

THE INFLUENCE OF THE TYPE OF ANTI-FOAMING ADMIXTURE AND SUPERPLASTICIZER ON THE PROPERTIES OF SELF-COMPACTING MORTAR AND CONCRETE

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Abstract. To prevent excessive air entrainment, superplasticizers (SPs) should not only be compatible with cement, but also should not create the air-entraining effect in mortar. In order to counteract excessive air entrainment, anti-foaming admixtures (AFAs) can be used to prevent the formation of air bubbles. This paper investigates the influence of the type, amount and time of introduction of AFAs on air-entrainment, rheological properties and workability loss of self-compacting mortar. The research results prove that AFAs decrease air-content in mortar. Mortar containing an AFA does not undergo segregation, as is the case with mortars of a similar degree of fluidity with no AFA and incorporating an SP only. Moreover, mortar with an AFA keeps initial consistency for longer in comparison to mortar with a SP only. The properties of hardened mortar mixes are also investigated. The research results show that AFAs do not have a significant influence on the compressive strength of mortar mixtures. The compressive strength of mortar mixtures incorporating an AFA is similar to mortar with a non-air-entraining SP. Moreover, AFAs increase significantly the flexural strength of mortar. In most cases, AFAs do not decrease the absorbability of mortar. Only two types of AFAs increase slightly the absorbability of mortar. In order to explain this phenomenon, a research was performed investigating the porosity structure according to the EN 480-11 (1999) and SEM analyses of two types of self-compacting concrete (SCC): one made of mortar with an AFA and the other without AFA. The freeze-proof resistance of SCC (made of the same mortar mixes) was also investigated according to PN-88/B-06250 (2003). The results show that the type of AFA and SP significantly influences porosity characteristics and frost-resistance of SCC.

Keywords: anti-foaming admixture, superplasticizer, self-compacting mortar, rheological properties, workability loss, compressive strength, flexural strength, porosity.

1. Introduction

One of the important properties of self-compacting concrete (SCC) is self liberation of air bubbles introduced during concrete mixing (Kamal *et al.* 2002; Khayat 2000; Szwabowski, Łaźniewska-Piekarczyk 2009). It is puzzling that when it comes to self-compactibility this phenomenon is not taken into consideration in commonly used self-compacting tests (European Project Group 2005). The examination of SCC (Szwabowski, Łaźniewska-Piekarczyk 2007, 2008a, b) has shown that there is frequently an issue with too high air content in mixtures even though their flow diameter and flow time comply with the guidelines (European Project Group 2005). It is probably due to adding a superplasticizer (SP) because other experiments also have shown that the new generation SPs induce the air-entraining effect. Further experimental data (Mosquet 2003) confirm that an SP based on polycarboxylate has high air-entraining effect on SCC. The air content is higher in case of an elevated w/c ratio and can even exceed 8%. It should be mentioned that a mixture with such high air content can also have a relatively good flow diameter equal to 660 mm, correspond-

ing to the SF2 class. Independently of these advantageous conditions, the liberation of a sufficient air quantity from the mixture did not take place. The air entrainment of the mixture was even higher.

The presence of functional groups in superplasticizer molecules – namely etheric, hydroxyl, and carboxyl – decreases the surface tension of water, produces a defloculation of conglomerates, and increases wettability not only of cement grains but also of the whole mineral framework. There are superplasticizers which only show a dispersion effect without decreasing the surface tension of water solutions. These are for example: hydrocarboxylic acid salts, sulphonated melamine-formaldehyde resins, formaldehyde polycondensates salts of a beta-naphthalene sulphonic acid.

As researchers demonstrate in their papers (Kucharska 2000; Kurdowski 2003; Mosquet 2003; Ramachandran 1995), air bubbles trapped in the presence of hydrophilic surfactants should not adhere to cement and aggregate particles, being uniformly dispersed in the concrete mix. Moreover, according to the researchers (Kurdowski 2003), these bubbles are slightly bigger than those trapped under the influence of an air-entraining

agent, but their stability is lower. Air bubbles trapped in the presence of an air-entraining agent, reach the size of $20\div 250\text{ }\mu\text{m}$. Moreover, they adhere to the surface of cement particles. The air bubbles can be unstable due to the floating of large air bubbles, fading of air bubbles that are less than 0.10 mm in diameter, and coalescence of air bubbles (Khayat *et al.* 2002; Khayat 2000; Kobayashi *et al.* 1981; Litvan 1983). During concrete hardening, these air voids are not filled with hydration products because a C-S-H gel is preferentially formed on cement particles or between them. As far as the freeze-resistance of concrete is concerned, it would be the best if air bubbles were of $0.05\div 0.10\text{ mm}$ in diameter and had the spacing factor of $0.15\div 0.20\text{ mm}$. Nonetheless, the issue of the critical value of air voids spacing in freeze-resistant concrete, depending on its type, is still a problem open to discussion (Szwabowski, Łaźniewska-Piekarczyk 2009).

According to Sakai *et al.* (2006), the SP type is crucial regarding the size and share of air voids trapped under its influence, although as the time passes and the concrete hardens, further changes of these proportions usually take place. With the addition of polycarboxylate superplasticizers, air voids have smaller diameters than air voids trapped by using lignosulphonate or naphthalene superplasticizers.

The results of other researchers (Łaźniewska-Piekarczyk 2008; Mosquet 2003) also show that new generations of SPs present air-entraining action, which was proved too by the experiments of one of the authors (Szwabowski, Łaźniewska-Piekarczyk 2009a, b).

Air-entrainment of a fresh mix may decrease its flow depending on the degree of initial fluidity. The decrease in flow is the result of internal compression of air bubbles and lower density of a fresh mix. The research results (Sakai *et al.* 2006; Fagerlund 1999; Neville 2000) showed that air-entrainment may also initially increase the flow when a fresh mix is originally of low fluidity. However, subsequent increasing of the air-entrained admixture decreases the diameter of flow of fresh mixes.

Taking these results into consideration makes it possible to conclude that the overall accepted criteria for evaluating the properties of concrete mixes are insufficient in this scope, and do not guarantee a self-release (liberation) of air. Research results (Neville 2000; Grodzicka 2005) proved that larger air voids, formed e.g. because of too low compacting, cause higher decrease of concrete strength than smaller air voids produced by an air-entraining agent. For this reason, it is important to eliminate the air-entraining effect of a SP, because under its influence large air voids are formed (Szwabowski, Łaźniewska-Piekarczyk 2008b; Fagerlund 1999). Based on the results of this experiment, it can be concluded that the commonly applied tests of SCC mixtures are insufficient in this respect, and the results obtained do not guarantee a proper self-escaping of air. This can be achieved by increasing the mixture flowability through adding more SP. Care must be taken, however, not to allow segregation.

Due to this fact, in order to prevent excessive air entrainment, SPs should not only be compatible with cement, but also do not create the air-entraining effect in the paste.

To eliminate the excess of air content in a fresh self-compacting mix, anti-foaming admixtures (AFAs) can be used (Łaźniewska-Piekarczyk 2009b). Unfortunately, AFAs are not commonly used in self-compacting concrete, even though the functioning mechanism of anti-foaming admixtures is known. It may be explained in the following way. Active components are distributed around gas bubbles, displacing surfactant molecules. As a result, the thickness of a lamella wall built from a surfactant causes its destabilization and results in the fracture or coalescence of the bubble.

Components and their proportions used in AFAs, as well as in SPs, are known only to their producer. They may be mineral oils, silicone oils, organic modified silicones, hydrophobic constant molecules (silica, waxes, higher fatty acid soaps, alcohols and fatty acids), emulsifiers, polyalcohol or alcohol derivatives of organic compounds. They may also be mixes of the above active components acting in a synergetic way. Unfavorably, high prices and unknown influence on the properties of fresh and hardened concrete mixes do not favor wider use of AFAs.

Unfortunately, the best type of AFA used for decreasing air volume in mortar of SCC is not known. So it is advisable to carry out proper tests aiming to verify the influence of different types of AFAs on the properties of fresh and hardened self-compacting mortar mixes. The results of such investigations are presented in the paper.

2. Methodology of the research

The effectiveness of AFAs, depending on their type and the most air-entrained SP (identified on the basis of earlier tests on SCC (Szwabowski, Łaźniewska-Piekarczyk 2008a), were checked by tests of flow and air entrainment of mortar in accordance with EN 1015-3:2000/A2:2007 (2007) and EN 1015-7:2000 (2000) respectively (Figs 2 and 3).

The process of mixing mortars started with dry ingredients (Table 1 and Fig. 1), then a SP was added, and next an AFA of a particular type (Tables 1 and 2). The mixing of mortar components was carried out according to the procedure described in EN 197-1:2002 (2002).

To compare the influence of the admixtures used on the investigated properties of fresh and hardened self-compacting mortar mixes, *M* mortar without admixtures was made. The non-self-compacting *M* mortar (Table 1), was subjected to densification through shaking. The mortar was put in three layers in a container, which formed a part of the apparatus for testing the volume of air content. Each layer was shaken before laying another one.

After 20 minutes air-entrainment, diameter and time flow measurements were carried out for all mortar mixes, because, according to the authors' many investigations, after this time the effectiveness of a SP is the highest. Time flow was measured at the flow diameter of 250 mm . The findings of various researchers prove that a concrete mix is self-compacting when the flow diameter of a SCC mortar is higher than 250 mm .

To assess the influence of a given AFA on the time induced loss of mortar workability, the mortar flow was

checked after 20 and 60 minutes, counting from the time of mixing the remaining components.

The properties of hardened mortar mixes were described by means of the following methods: density test in accordance with the EN 1015-10:2001 (2001) standard, compression strength and flexural strength in accordance with EN 1015-11:2001/A1:2007 (2001), and absorptivity (w_w) in accordance with PN-88/B-06250 (2003). All tests of hardened mortars were done on 4x4x16 cm mortar specimens 28 days after treating them in 20 °C water.

The porosity structure characteristics of two types of self-compacting concrete (made of *M1*, *M2* and *M2-f* mortar mixes) were examined in accordance with the EN 480-11 (1999) and SEM analysis procedures.

Table 1. Composition of mortar mixes (*M*); cement CEM II B-S 32.5 R: 541 [kg/m³], w/b = 0.40; sand (Fig. 1): 890 [kg/m³]

Series	w^1/b	SP [%m.C]	SP	AFA [%m.C]
M	0.40	0.00	—	0.00
$M1$		PCP		
$M2$		0.77	PCE*	2.21
$M2-a$				4.42
$M2-a1$				2.21
$M2-b$				
$M2-c$				
$M2-d$				4.42
$M2-d1$				1.11
$M2-d2$				2.21
$M2-e$				
$M2-f$				

¹water + admixtures liquid part

Table 2. Types of SPs and AFAs and their components

Symbol	AFA/SP based on:
SP	
PCP	based on polycarboxylate
PCE	based on polycarboxylic ether
AFA	
<i>a</i>	froth breaker based on PDMS/ silicone oil/ hydrophobic silica
<i>b</i>	froth breaker based on mineral oil or amidol wax
<i>c</i>	froth breaker based on alcohol derivative of saturated fatty alcohol, mineral oil and PE wax
<i>d</i>	fiakyl derivative of saturated fatty alcohol/mineral oil/PE and amidol wax
<i>e</i>	alkoxyl derivative of fatty alcohol, 100%
<i>f</i>	Polyalcohol

The freeze-proof resistance of SCC (made of *M1*, *M2* and *M2-f* mortar mixes) according to PN-88/B-06250 (2003) was also investigated. After 28 days concrete samples 100×100×100 mm were subjected to freezing and thawing in water in the temperature of ±20 °C (four cycles per day). According to this standard, concrete is frost-resistant when after *n* cycles its compressive strength decrease is lower than 20% or its decrease in mass is lower than 5%.

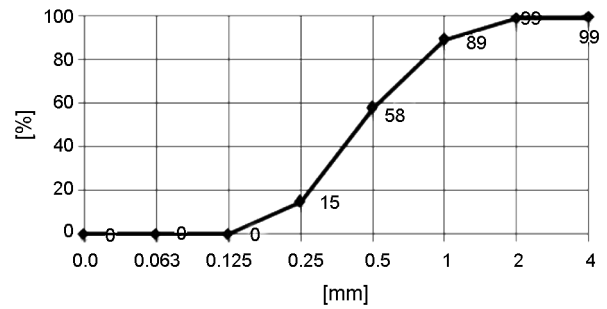


Fig. 1. The grain size distribution of sand

3. Mortar research results and their analysis

The research results of fresh mortars are presented in Table 3 and in Figs 3 and 4. The analysis of the results indicates that different types of SPs produce very different volumes of air in mortars.

Table 3. Research results of fresh self-compacting mortar mixes

Series	A _c [%]	D [mm]	T ₂₅ [sec]
<i>M</i>	4.0	110	4
<i>M1</i>	3.0	310	6
<i>M2</i>	12.0	260	2
<i>M2-a</i>	3.0	299	6
<i>M2-a1</i>	3.0	270	6
<i>M2-b</i>	5.5	363	5
<i>M2-c</i>	3.8	391	4
<i>M2-d</i>	3.4	370	6
<i>M2-d1</i>	3.4	340	6
<i>M2-d2</i>	4.0	320	6
<i>M2-e</i>	5.4	306	6
<i>M2-f</i>	2.8	360	5

PCP produced only 3% of air volume in mortar. In case of the *M* mortar mixes (mechanically compacted, without SP) 4% of air volume was observed. The *M2* mortar was characterized by as much as ca. 12% of air volume. This volume was the effect of the PCE action. The air entrainment from SP is an undesirable side effect. Nevertheless, other researchers' results proved that SPs based on a different type of polycarboxylic ether produce very different air volumes in SCC (2% and 10%). Thus, the influence of the type of SP base on air volume in SCC needs further examination. The *M*, *M1*, *M2* mortar mixes were considered a reference in the assessment of the effectiveness of AFAs.

The research results presented in Table 3 prove that polyalcohol-based AFA was highly effective in decreasing air content in mortar, increasing its flow diameter, and decreasing its flow time (Figs 2 and 3), without any segregation (Fig. 4). However, beyond the saturation point of AFA, there was no further reduction in air volume of mortar and SCC. Research results (Table 3) show that the type c AFA produces a higher flow diameter of mortar, but is characterized by 5.4% air content (Fig. 2).

The test results for mortar mixes presented in Figs 2 and 3 prove that due to the reduction of air entrainment in

mortar and its rheological properties, the most effective mixes contained an AFA based on polyalcohol.

In case of fresh mortar with AFA, the loss of its initial consistency was slower (Figs 5 and 6). Thus, due to the use of AFAs, it is possible to decrease the necessary dosage of a SP in order to achieve suitable flow of self-compacting mortar or a self-compacting concrete mix.

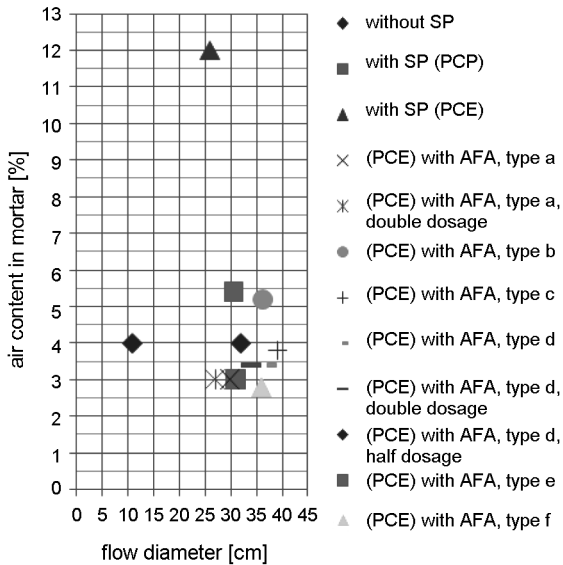


Fig. 2. The influence of the type of AFA and SP on the relationship between the flow diameter and air content in mortar

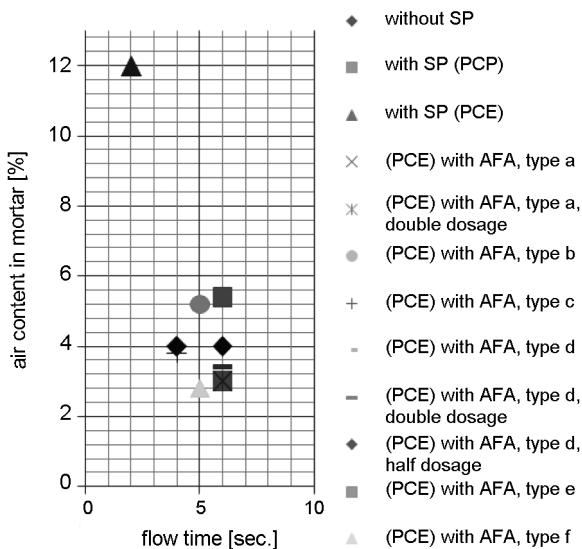


Fig. 3. The influence of the type of AFA and SP on the relationship between flow time and air content in mortar

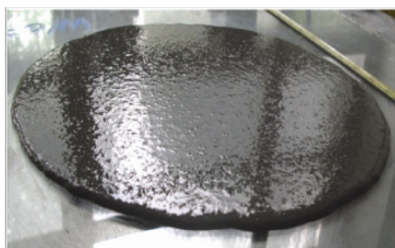


Fig. 4. The segregation-free highly fluid mortar incorporating an AFA

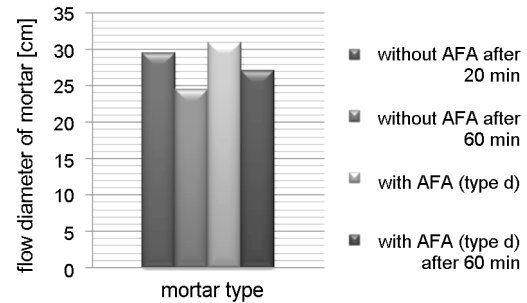


Fig. 5. The influence of the type of AFA on the flow diameter of mortar mixes (after 20 and 60 minutes)

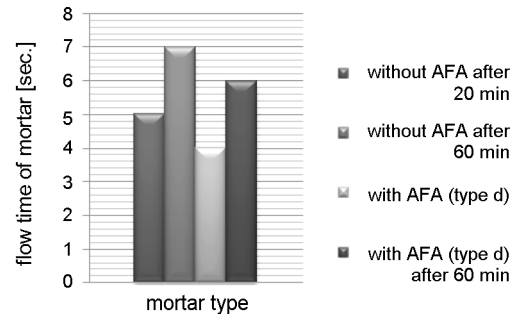


Fig. 6. The influence of the type of AFA on the flow time of mortar mixes (after 20 and 60 minutes)

In Table 4 the test results on the properties of hardened mortar are presented. The research results proved that different types of AFA (except type *e*) do not influence the compressive strength of mortar mixes. The compressive strength of mortar incorporating an AFA (except AFA type *e*) is similar to that of mortar without any AFA. However, all types of AFAs increase significantly the flexural strength of mortar.

In Figs 7 and 8 the relationship between air volume in fresh mortar mixes and compressive or flexural strength of hardened mortar is presented.

The research results suggest that the type of admixture has significant influence on the relationship between the parameters analyzed.

Table 4. Research results of hardened self-compacting mortar

Series	ρ [kg/m ³]	f_{cm} [MPa]	f_{ct} [MPa]	Absorptivity [%]
<i>M</i>	540.3	59.0	6.3	0.0
<i>M1</i>	549.2	58.1	7.0	0.0
<i>M2</i>	529.5	49.0	5.4	3.0
<i>M2-a</i>	542.0	58.0	12.0	0.7
<i>M2-a1</i>	570.1	59.5	12.0	0.0
<i>M2-b</i>	571.2	59.3	11.7	0.6
<i>M2-c</i>	536.6	53.4	11.3	1.8
<i>M2-d</i>	581.0	61.7	11.9	0.1
<i>M2-d1</i>	580.0	62.5	10.5	0.0
<i>M2-d2</i>	580.1	62.0	10.4	0.0
<i>M2-e</i>	540.2	51.6	9.9	1.4
<i>M2-f</i>	582.3	65.0	11.0	0.0

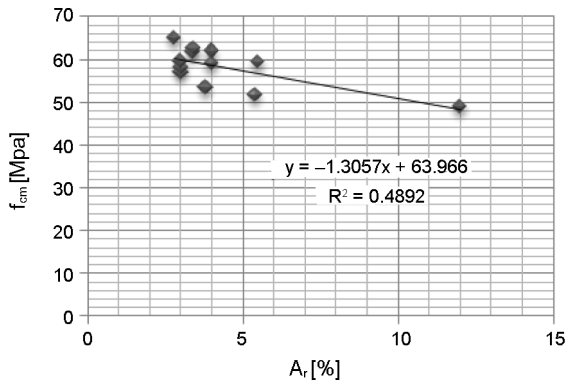


Fig. 7. The relationship between the air volume in a mix and the compressive strength of hardened mortar

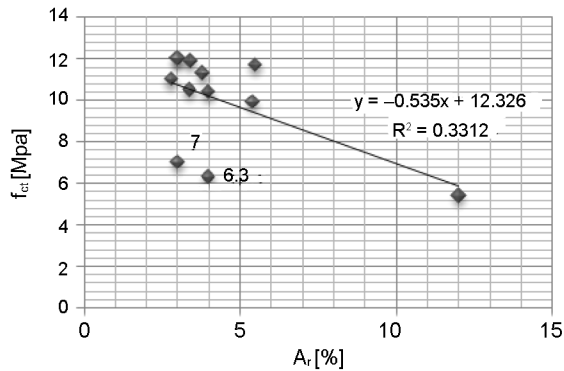


Fig. 8. The relationship between the air volume in a mix incorporating AFA and the flexural strength of hardened mortar

In Table 4 the absorptivity test results of the mortars are presented. The AFA types *c* and *e* increase the absorptivity of mortar significantly. The air volume in mortar does not correspond to the absorbability of mortar, only capillary air voids influence this property.

Fig. 9 presents the side views of hardened mortar mixes obtained using a SP, and a SP introduced with an AFA.

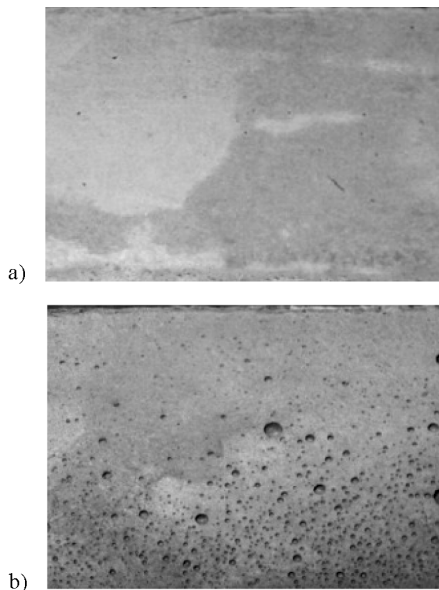


Fig. 9. The differences in side porosity of hardened mortar with a SP (a) and SP with the type *f* AFA (b)

It was noticed that different types of combinations of admixtures produce different air void characteristics of mortar (Fig. 9).

4. Concrete research results and their analysis

The reason why AFAs influence the mechanical properties of mortar mixes lies probably in the internal porosity characteristics of mortar, modified by the AFA action. In order to confirm this assumption, the porosity structure characteristics of two types of self-compacting concrete (SCC) made of *M1*, *M2* and *M2-f* were investigated (Tables 5–7). The porosity characteristics for SCC research results obtained in accordance with EN 480-11 (1999) are presented in Table 8 and in Figs 10–12. This research proved that AFAs generate negative changes in the values of the porosity structure parameters. The value of the air void space factor is bigger, when a SP and an AFA are incorporated. Moreover, the air void volume with a diameter lesser than 300 μm is smaller. This suggests that these changes are not positive in respect of frost-resistant SCC. Nonetheless, SCC with entrainment from SP (as an undesirable side effect SP), *SCC-M2* and SCC with an AFA, *SCC-M2-f*, were freeze-proof but SCC, *SCC-M1*, with a “non-entraining” SP was not freeze-proof (all concrete mixes were subjected to 300 cycles of freezing and thawing). When an air-entraining admixtures is used along with a “normal” SP (without entrainment from SP as an undesirable side effect SP), investigated SCC is frost-resistant.

Table 5. The mix composition of self-compacting mixes, $w^1/b = 0.40$; CEM II/B-S 32.5 = 541 kg/m^3

Series	Sand	Gravelly aggregates				SP	AFA
	[kg/m ³]						
	0–2	2–4	2–8	8–16	[% m.C.]		
	[mm]						
<i>SCC- M1</i>	890	200	228	256	0.77	0.00	
<i>SCC- M2</i>						0.00	
<i>SCC-M2-f</i>						2.21	

¹ water + liquid chase of admixture

Table 6. Chemical characteristics of CEM II/ B-S 32.5 R

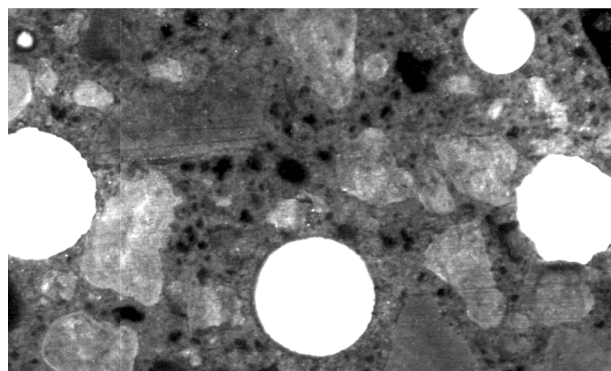
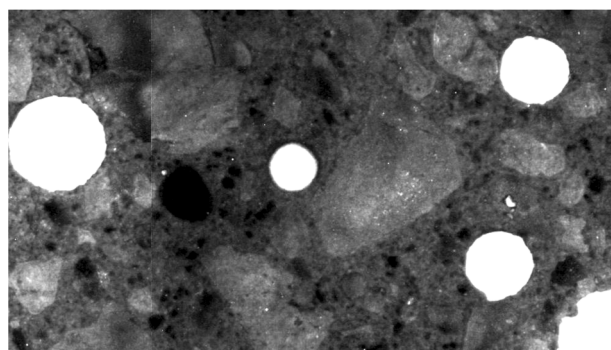
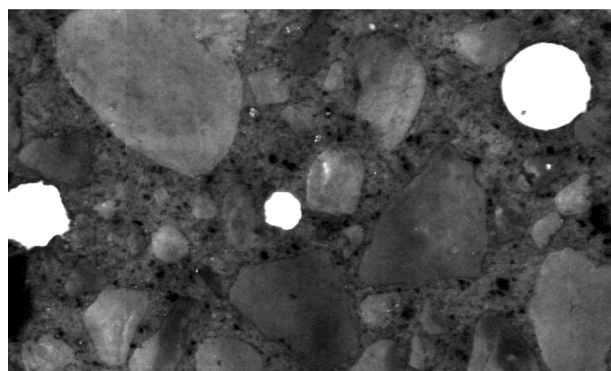
Cement components [%]							Specific surface [m^2/kg]
SiO_2	CaO	Al_2O_3	Fe_2O_3	MgO	Na_2Oe	SO_3	
24.7	56.7	6.3	2.3	2.9	0.70	3.2	325

Table 7. Characteristics of admixtures

Admixture type	Admixture base	Density [g/cm^3]	Concentration, [%]
Superplasticizer	Polycarboxylate (PCP)	1.09	34
Anti-foaming admixture	Polyalcohol	1.05	30

Table 8. The test results for the porosity characteristics of hardened concrete

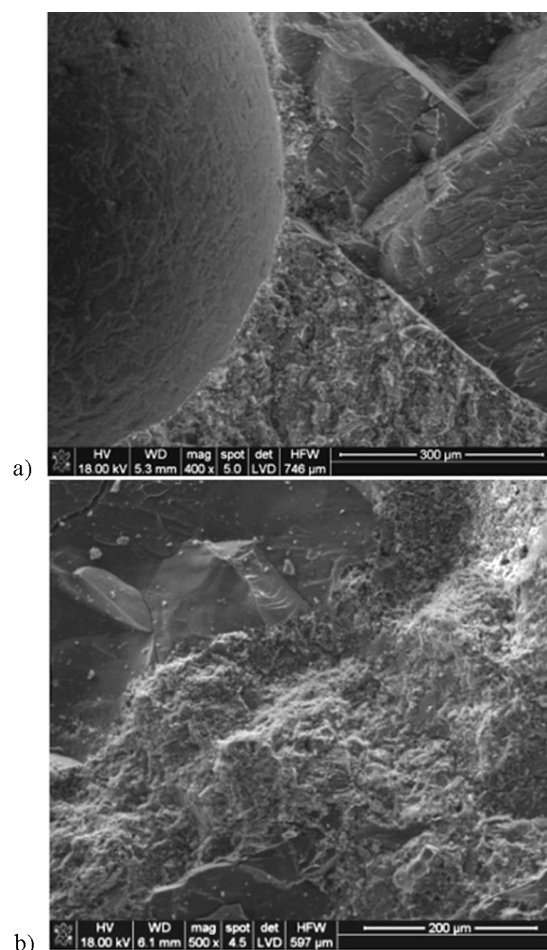
Symbol	A [%]	α [mm ⁻¹]	\bar{L} [mm]	A_{300} [%]
SCC- M1	1.76	10.75	0.85	0.21
SCC- M2	4.47	20.83	0.29	1.55
SCC-M2-f	2.10	15.04	0.58	0.25

**Fig. 10.** Porosity characteristics of SCC-M1**Fig. 11.** Porosity characteristics of SCC-M2**Fig. 12.** Porosity characteristics of SCC-M2-f

The problem of theoretical and practical values of porosity structure has been considered in previous publication (Szwabowski, Łaźniewska-Piekarczyk 2009) and needs further studies.

SEM (*Scanning Electron Microscopy*) research results carried out by other scientists investigated the influence of an air-entraining SP, non-air-entraining SP and

AFA on the structure of SCC. Figs 13–15 present these results. It is clearly visible that admixtures influence the SCC structure significantly. Admixtures generate different porosity, CSH structure and contact zone in SCC.

**Fig. 13.** SEM research results of SCC for non-air-entraining PCP: a) porosity; b) CSH phase

There are no visible large air voids in Fig. 13. This is the result of the application of non-air-entraining PCP. The CSH phase is homogeneous and compact. The cement paste encloses the aggregate tightly. No calcium hydroxide was observed.

In Fig. 14 the effect of the application of air-entraining PCE on the quality of SCC structure is presented. This structure is the most amorphous. There are a lot of large air voids in this SCC structure (with the diameter exceeding 560 μm).

The structure of SCC with an AFA and air-entraining PCE is less heterogeneous than the structure of SCC with air-entraining PCE (compare Figs 14 and 15).

To sum up, the research results presented in the paper prove that the type of AFA and SP has significant influence on the properties of self-compacting mortar mixes and the structure of SCC. The reasons of the influence of admixtures will be explained in the next publications by the authors.

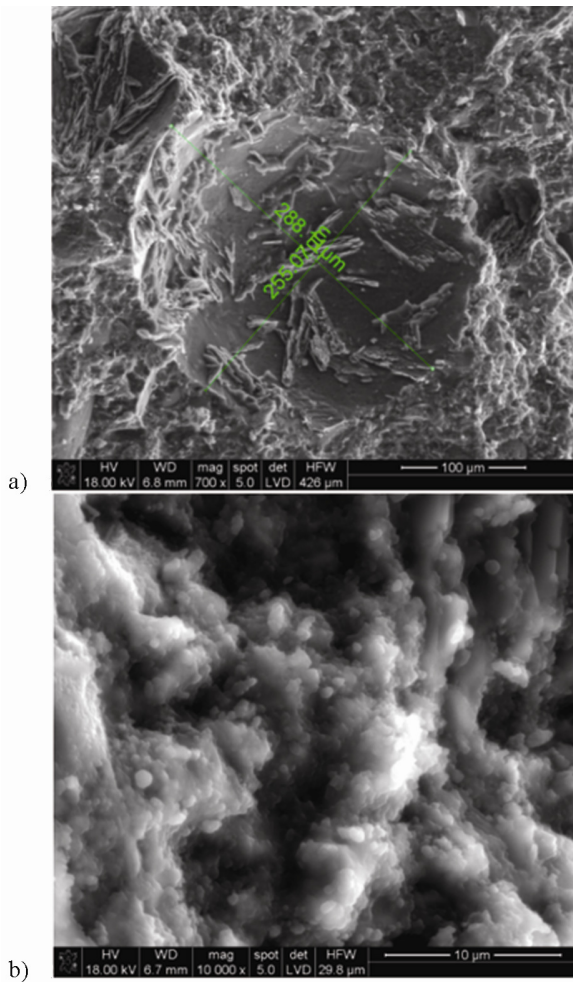


Fig. 14. SEM research results of SCC for air-entraining PCE: a) porosity; b) CSH phase

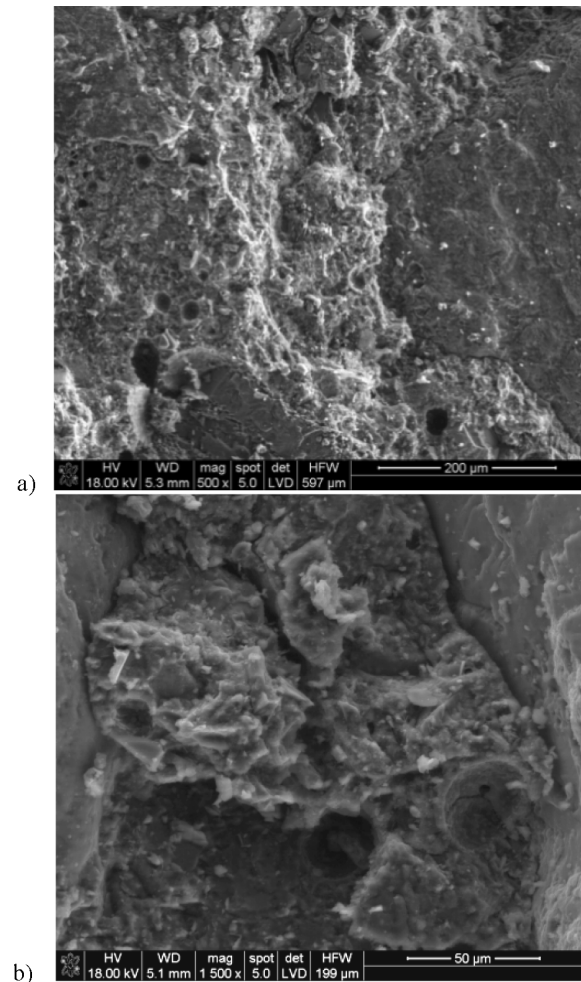


Fig. 15. SEM research results of SCC for air-entraining PCE and AFA: a) porosity; b) CSH phase

5. Conclusions

In the scope of the tests performed the following conclusions can be proposed:

- A polyalcohol-based AFA was highly effective in decreasing air content in mortar, increasing its flow diameter and decreasing its flow time without any segregation. However, beyond the saturation point of AFA, there was no further reduction in air volume of mortar and SCC. The research results showed that the type *c* AFA produces a higher flow diameter of mortar, but unfortunately it is characterized by 5.4% air content.
- Mortar containing an AFA does not undergo segregation, as is the case with mortar mixes with no AFA of a similar degree of fluidity (caused by higher amount of a SP). Thus, with the use of AFAs, it is possible to decrease the necessary dosage of a SP in order to achieve a suitable flow of self-compacting mortar or a self-compacting concrete mix. Moreover, test results prove that mortars with an AFA maintain initial consistency for a longer time in comparison to mortar with a SP only.

- Anti-foaming admixtures (except the type *e* AFA) did not produce significant difference in compressive strength of mortar mixes. However, the admixture type had a significant influence on the relationship between air volume in mortar mixes and their compressive strength.
- Compressive strength of mortar mixes incorporating an AFA is similar to that of mortar without an AFA. However, an AFA increases the flexural strength of mortar.
- In most cases, an AFA did not decrease the absorptivity of mortar. Only AFA types *c* and *e* slightly increased the absorptivity of mortar.
- An AFA generates a negative change of the values of the porosity structure parameters. The value of air void space factor is bigger when a SP and AFA are incorporated. Moreover, air void volume, with a diameter lesser than 300 μm is smaller. This suggests that the changes are not positive in respect of frost-resistant SCC. Nonetheless, SCC with an air-entraining SP and SCC with an AFA were freeze-proof. SCC with a not entraining SP was not freeze-proof. Thus, the problem of theoretical and practical values of porosity structure parameters needs further studies.

- The type of SP and AFA admixture has a significant influence on the SCC structure. Admixtures generate different porosity, CSH structure and contact zone in SCC.

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