

Reduction of shrinkage by Superabsorbent polymers (SAP) in fibre reinforced mortars

Rostami, Rohollah ; Klemm, Agnieszka; C.R. Almeida, Fernando

Published in:
Construction and Building Materials

DOI:
[10.1016/j.conbuildmat.2021.123109](https://doi.org/10.1016/j.conbuildmat.2021.123109)

Publication date:
2021

Document Version
Author accepted manuscript

[Link to publication in ResearchOnline](#)

Citation for published version (Harvard):

Rostami, R, Klemm, A & C.R. Almeida, F 2021, 'Reduction of shrinkage by Superabsorbent polymers (SAP) in fibre reinforced mortars', *Construction and Building Materials*, vol. 288, 123109.
<https://doi.org/10.1016/j.conbuildmat.2021.123109>

General rights

Copyright and moral rights for the publications made accessible in the public portal are retained by the authors and/or other copyright owners and it is a condition of accessing publications that users recognise and abide by the legal requirements associated with these rights.

Take down policy

If you believe that this document breaches copyright please view our takedown policy at <https://edshare.gcu.ac.uk/id/eprint/5179> for details of how to contact us.

Reduction of shrinkage by Superabsorbent polymers (SAP) in fibre reinforced mortars

Rohollah Rostami^{*1a}, Agnieszka J. Klemm^{1a} and Fernando C. R. Almeida^{2b}

¹ Glasgow Caledonian University, School of Computing, Engineering and Built Environment, 70 Cowcaddens Road, Glasgow, G4 0BA, UK

² Federal University of Minas Gerais, Department of Materials Engineering and Construction, Av. Presidente Antônio Carlos, 6627, Belo Horizonte, MG 31270-901, Brazil

ABSTRACT

Fibre reinforced mortars (FRM) containing fly ash (FA) and ground granulated blast-furnace slag (GGBS) may suffer from volumetric changes and despite fibre reinforcement, internal curing may be required. This paper evaluates the efficiency of Superabsorbent Polymers (SAP) in reduction of shrinkage. Autogenous (AS), plastic (PS) and drying (DS) shrinkage were analysed for FRM with three types of cement (CEM I, CEM II/B-V and CEM III/A) and three types of SAPs (different water absorption capacities and different particles sizes). The experimental results showed that SAP substantially contribute to the total shrinkage reduction in cementitious mortars and reduce susceptibility to crack formation. Reduction of plastic and autogenous shrinkage is highly dependent on particle sizes. Finer SAPs lead to better performances with reductions reaching 75% for PS and 124% for AS. Reduction of drying shrinkage, although substantially lower (up to 12%), can be achieved by application of SAPs with lower water absorption capacities. The effect of particle sizes is less important for drying shrinkage.

KEYWORDS: Superabsorbent polymers (SAPs), Polymeric fibres (PF), Plastic shrinkage, Autogenous shrinkage, Drying shrinkage

*Corresponding author, Ph.D. Student, E-mail: Rohollah.Rostami@gcu.ac.uk

^a Professor, E-mail: A.Klemm@gcu.ac.uk

^b Ph.D., E-mail: fernando@demc.ufmg.br

1. INTRODUCTION

For the last couple of decades, Supplementary Cementitious Materials (SCMs) have been successfully used to improve sustainability of Portland cement-based materials by reduction of clinker, responsible for high CO₂ emissions during its production [1, 2]. Fly ash (FA), from coal combustion (pozzolanic material), and ground granulated blast-furnace slag (GGBS), from pig iron production (latent-hydraulic material), are well-known SCMs and both are viable solutions to partially substitute Portland cement (PC) [3-5].

Volume changes, resulting from shrinkage in concrete, are still a major problem and they may compromise durability of structures [6, 7]. Many parameters may influence concrete shrinkage cracking, including water/cement ratio, type of cement, fibres, admixtures, structural element geometry, fines content, exposure conditions [7-9].

In a very early age (before hardening), concrete is susceptible to **plastic shrinkage (PS)**. In an initial period of hydration, a highly exothermic reaction of C₃A with calcium sulphate (CaSO₄) takes place, leading to formation of ettringite. During the next stage (dormant period), reactions slowdown and concrete maintains its plastic state (fluid or semi-fluid), highly susceptible to cracking. Plastic shrinkage occurs as a result of rapid evaporation of water from surfaces, which exceeds the amount of water available to replenish surface by bleeding. A direct consequence of this disproportion is a build-up of high hydraulic pressure (capillary pressure), which increases in an inverse proportion to the radii of water menisci between solid particles [10, 11]. As a result, an increase in contracting forces induces both settlement and shrinkage deformation along the surface of concrete [7, 11].

Autogenous shrinkage (AS) of cement-based materials is a bulk deformation of sealed concrete in isothermal conditions, without any external forces. It occurs during hardening, predominantly as a result of emptying of capillary pores due to insufficient supply of water in hydration process [12]. This, in turn, decreases internal relative humidity of cement paste and consequently leads to self-desiccation [13]. It creates empty pores due to chemical shrinkage, resulting in a decreased radius of menisci curvature and increased tensile stresses in pore water [13-15]. Thus, AS can also lead to formation of cracks and deterioration of cementitious materials which, in turn, impair strength, durability and aesthetics.

Concrete may also experience **drying shrinkage (DS)**, which is referred to a reduction in bulk volume due to a loss of water to the surrounding atmosphere [16, 17]. DS begins from setting and continues during hardening process, throughout the whole service life of concrete [18]. Internal relative humidity in concrete pores is reduced and affects capillary stresses, disjoining pressure, movement of interlayer water, and surface free energy [19, 20]. When water is lost from concrete pores, capillary pressure builds up and leads to formation of water-air menisci. This, in turn, induces stresses within the solid skeleton and consequently shrinkage takes place. Moreover, at low relative humidity (especially below 50%), the force from desorption of water on the hindered adsorption regions leads to a decrease in disjoining pressure and surface tension [21]. DS is also influenced by the surface free energy due to a loss of adsorption of water on surface [16, 21]. At the end, both visible and micro cracks can be formed.

In order to mitigate the negative shrinkage effects, polymeric fibres are commonly used [22, 23]. However, their efficiency is often limited, especially in large surface elements or in severe environments [24, 25].

Internal curing agents, such as Superabsorbent polymers (SAP), can be used to further reduce these phenomena. This additional reduction of shrinkage is the subject of the presented study. As it has been previously reported SAP ability to provide water during hydration process [26, 27] can lead to notable reductions of autogenous shrinkage [28-30], plastic shrinkage [28, 31-32] and drying shrinkage [28, 29] in plain cementitious materials (without SMCs and fibre reinforcement).

Therefore, the **main objective** of this paper was to evaluate the efficiency of Superabsorbent Polymers (SAP) in reduction of autogenous, plastic and drying shrinkage in fibres reinforced mortars containing SCMs. Since the effect of SAPs in cementitious matrices depends on a number of parameters (polymer shape, size, crosslinking density, chemical structure and ionic concentration of surrounding pore solution) [26-34], three types of SAPs with different particles sizes and different water absorption capacities (WAC) were analysed.

2. MATERIALS AND METHODS

Three types of commercial cements have been used in this study, in accordance with BS EN 197-1:2011 [35]: CEM I 52.5N (Portland cement - PC), CEM II/B-V 32.5R (PC-FA), and CEM III/A 42.5N (PC-GGBS). Table 1 presents chemical characteristics of cements and SCM contents as

provided by the manufacturers. Particles size distributions (PSD) of cements, shown on Fig. 1, were determined by a Mastersizer laser diffractometer (air as dispersant). PSD curves for different cements are comparable and no significant variations were observed.

Table 1 Chemical compositions of CEM I, CEM II/B-V and CEM III/A

	SCM (%)	SiO ₂ (%)	Al ₂ O ₃ (%)	Fe ₂ O ₃ (%)	CaO (%)	MgO (%)	SO ₃ (%)	K ₂ O (%)	TiO ₂ (%)	ZnO (%)	LOI (%)
CEM I	0	20.1	4.9	2.57	64.3	2.2	3.2	0.27	0.0	0.0	2.39
CEM II/B-V	30	32.69	13.13	3.29	43.48	1.33	0.4	1.26	0.56	0.02	0.16
CEM III/A	50	24.50	8.99	1.76	57.13	5.33	0.0	0.0	0.58	0.0	1.19

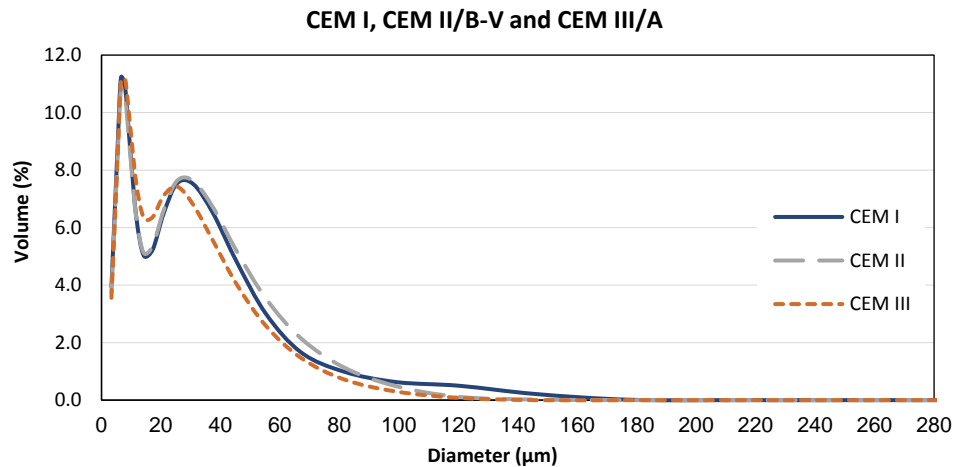


Fig. 1 Particle size distributions of CEM I, CEM II/B-V and CEM III/A.

Mortars were produced with fine sand, with 90% of particles were smaller than 0.425 mm. Micro polypropylene fibres (PF) used in this study had length of 6 mm, diameter of 18 μm and density of 0.91 kg/m³. Three types of cross-linked SAPs were investigated (called here SAP A, C and E). Table 2 summarises properties of SAPs, including their basic chemical composition, particles size range, and sorption characteristics. SAP A and C had similar particle sizes and SAP C and E similar compositions (modified polyacrylamide). Water absorption capacities (WAC) of SAPs in DI water and in three cement paste solutions have been determined gravimetrically, by the Tea-bag Method [36, 37]. All analyses of SAP samples were carried out in standard laboratory conditions at a temperature of 21 ± 2 °C and a relative humidity of 40 ± 5%. SAP E had the

highest absorption capacity in all cement solutions, while the lowest water absorption was recorded for SAP A.

Table 2 Characterisation of SAPs

SAP	Type	Particles size range (µm)	Modes values (µm)	WAC ¹ Deionized water	WAC CEM I Solutions	WAC CEM II/B-V Solutions	WAC CEM III/A Solutions
SAP A	copolymer of acrylamide and acrylic acid	30-140	102.51± 0.43	340 g/g	34 g/g	33 g/g	25 g/g
SAP C	modified polyacrylamide	30-140	95.19± 0.44	290 g/g	36 g/g	37 g/g	33 g/g
SAP E	modified polyacrylamide	20-130	76.74 ± 0.22	340 g/g	40 g/g	46 g/g	40 g/g

¹ WAC: water absorption capacity

Shapes and sizes of SAPs particles (dry condition) and PF were additionally characterized by the SEM image analysis and presented in Fig. 2. Although all samples show irregular shapes, SAP A and C have similar particles sizes (Fig 2a and b) and SAP E, smaller particles (Fig 2c).

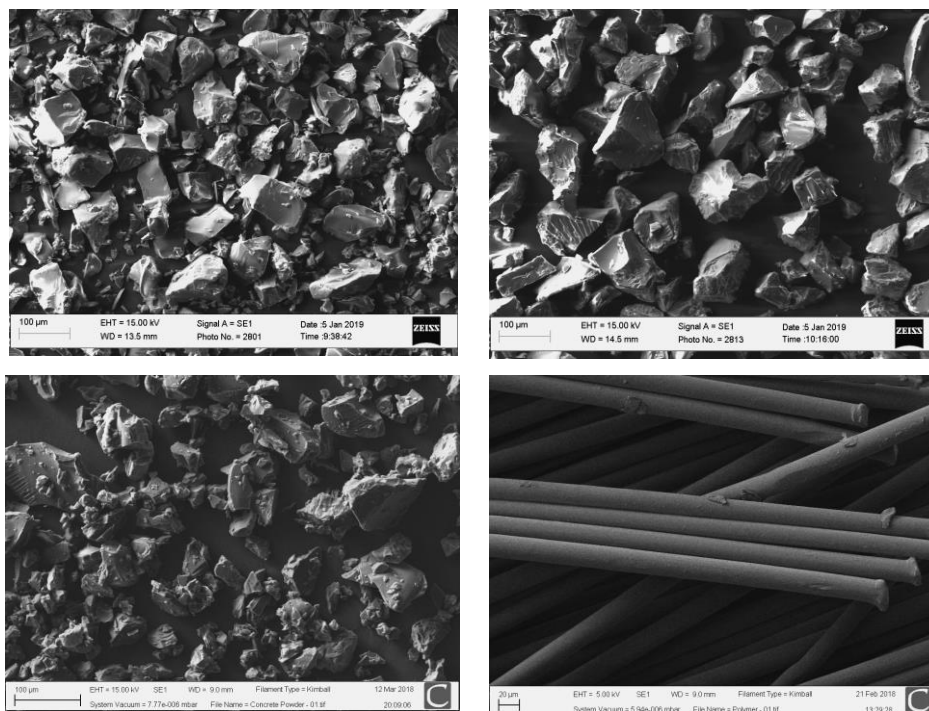


Fig. 2 SEM micrographs of SAP A (a), SAP C (b), SAP E (c) and PF (d).

Table 3 shows fifteen different compositions, which were designed to carry out this study.

Table 3 Mix proportions of materials

Cement	Sample Name	SAP type	SAP Content	PF Content	(w/b)effective ratio	(w/b)total ratio
CEM I	I	-	-	-	0.47	0.47
	IF	-	-	0.50%	0.47	0.52
	IF-A	A	0.25%	0.50%	0.47	0.58
	IF-C	C	0.25%	0.50%	0.47	0.58
	IF-E	E	0.25%	0.50%	0.47	0.58
CEM II/B-V	II	-	-	-	0.47	0.45
	IIF	-	-	0.50%	0.47	0.50
	IIF-A	A	0.25%	0.50%	0.47	0.56
	IIF-C	C	0.25%	0.50%	0.47	0.56
	IIF-E	E	0.25%	0.50%	0.47	0.56
CEM III/A	III	-	-	-	0.47	0.47
	IIIF	-	-	0.50%	0.47	0.52
	IIIF-A	A	0.25%	0.50%	0.47	0.58
	IIIF-C	C	0.25%	0.50%	0.47	0.58
	IIIF-E	E	0.25%	0.50%	0.47	0.58

All mortars were prepared with the binder: sand proportion of 1:2 (by weight) and with the effective water/binder ratio $(w/b)_{\text{eff}}$ of 0.47 [38]. The total w/b ratio is the sum of the effective water/binder ratio and the additional w/b (i.e., amount of water absorbed by SAP). This additional water is required in order to keep the same consistency of mortars (140 ± 2 mm in flow table test) [39]. Both SAPs and PF were added to the mixtures in amounts of 0.25 % and 0.50 % by mass of cement respectively.

Plastic shrinkage (PS) cracking of mortars was analysed by the optical microscope based on ASTM C1579-13 [40]. The specimens were prepared in $160 \times 140 \times 40$ mm³ moulds and, for the restraint conditions, three $140 \times 20 \times 0.2$ mm aluminium plates were placed equally and symmetrically on two steel threaded bars (8 mm diameter). The temperature during the experiment was maintained at 36 ± 3 °C, and the relative humidity at 30 ± 10 %. After 24h, a minimum of 50 measurements of crack widths were determined for each sample, by using the optical microscopy technique (Fig. 3a) with a resolution of 0.05 mm.

Autogenous shrinkage (AS) tests were carried out using the corrugated tubes method according to ASTM C1698 (ASTM 2009b) [41]. The final setting time (BS EN 480-2:2006 [42]) was used as a starting point of AS measurements. Bulk deformations of sealed mortars specimens were

analysed under constant environmental conditions ($T = 21 \pm 2^\circ\text{C}$ and $\text{RH} = 40 \pm 5\%$), not subjected to external forces (Fig. 3b). The change of length was recorded every 30 minutes, during the first day, then at least twice a day until 28 days, following by daily measurements until 180 days. At least three specimens were tested for each mortar.

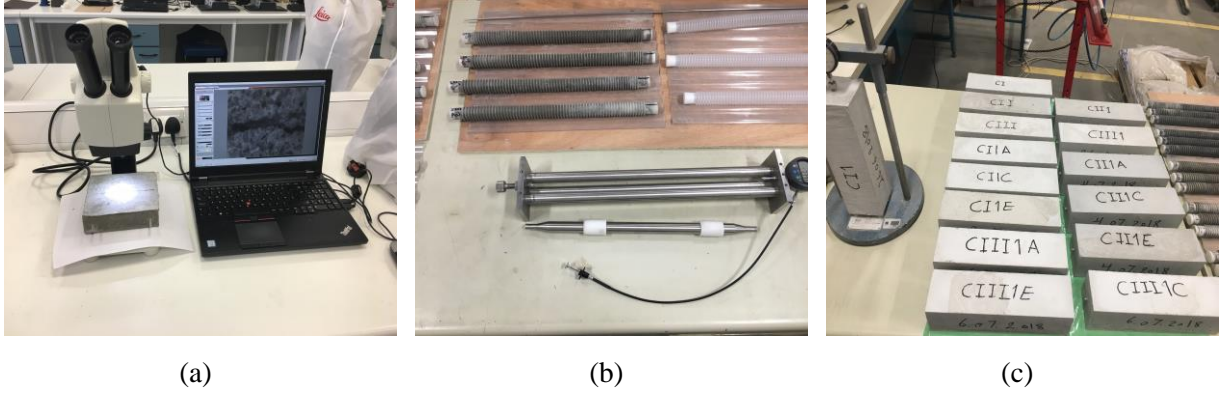


Fig 3. Shrinkage measurements: (a) Cracks analyses by optical microscope (b) Corrugated tubes and digital bench dilatometer for AS (c) samples of DS tests.

Drying shrinkage (DS) of mortars was measured on prismatic specimens ($75 \times 75 \times 280 \text{ mm}^3$) according to BS ISO 1920-8:2009 [43]. Samples were demoulded after 24h and the unsealed specimens were left drying in laboratory environment ($T = 21 \pm 2^\circ\text{C}$ and $\text{RH} = 40 \pm 5\%$) for 180 days. Length and mass of specimens were measured daily in the first 28 days and weekly between 28-180 days (Fig. 1c).

Total and additional shrinkage reductions (SAP induced) were subsequently calculated using Eq. (1) and (2).

$$Rt (\%) = \frac{s_{SAP} - s_{CEM}}{s_{CEM}} \times 100\% \quad (1)$$

$$Ra (\%) = \frac{s_{SAP} - s_{PF}}{s_{PF}} \times 100\% \quad (2)$$

Where $Rt (\%)$ and $Ra (\%)$ are respectively total and additional percentages of shrinkage reduction at the end of testing, s_{SAP} is the strain measured in SAP mortars, s_{CEM} is the strain measured in reference mortars (no SAP, no fibres), and s_{PF} is the strain measured in mortars with polymeric fibres (without SAP).

3. RESULTS AND DISCUSSION

3.1 Plastic shrinkage

Fig. 4 shows the maximum average cracks widths identified on samples surfaces after 24 hours of severe environmental exposure.

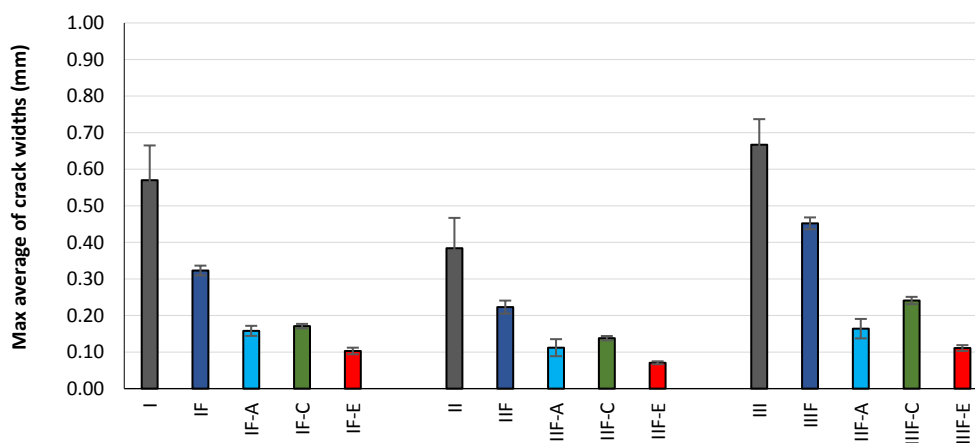


Fig. 4. Maximum averages of crack widths (mm).

The reference samples had the crack widths greater than 0.2 mm. Usually, cracks around 0.1 mm width can be visible by the naked eye and cracks below 0.05 mm width are very fine and difficult to be identified (even with an optical microscope) [44].

Overall, CEM II/B-V mortars had the narrowest cracks and CEM III/A mortars the widest cracks. This can be attributed to a decrease of total bleeding water and bleeding rate of fly ash cement [45]. Furthermore, it is related to the lower w/b of CEM II/B-V samples, and lower susceptibility to plastic shrinkage cracking. On the contrary, in CEM III/A samples, GGBS leads to prolonged setting times [46], resulting in higher bleeding rate. This, in turn, results in advanced tensile capillary pressure and the higher probability of plastic shrinkage cracking. Moreover, the addition of polymeric fibres reduces PS by providing bridging forces across the cracks. The results presented here are in a good agreement with the previously reported studies [22, 23, 47, 48]. Further substantial drop in plastic shrinkage was observed in all samples containing SAPs (Figs. 4 and 5).

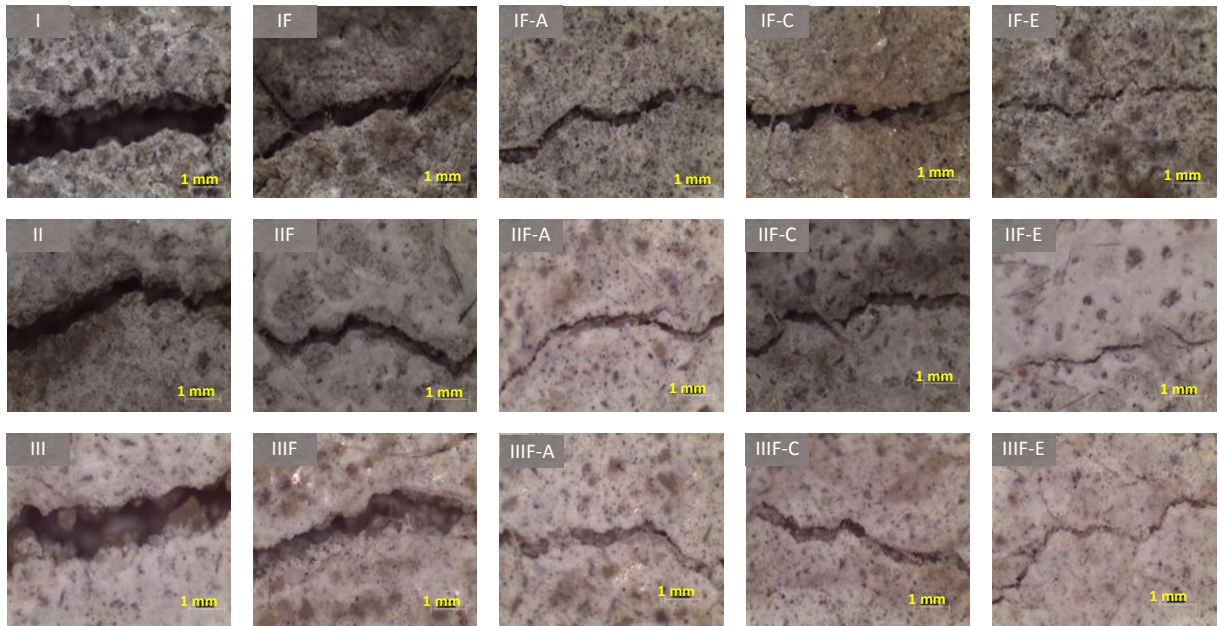


Fig. 5. Maximum averages of crack widths (16x magnification).

Depending on the types of cement and SAP, additional reduction level varied from 33% to 76% when compared to FRM reference samples. The role of SAPs is to slow down capillary pressure by providing stored water. This, in turn, controls restrained cracking and leads to a significant decrease of plastic shrinkage. Regardless the type of cement, SAP E mortars had the finest crack widths, while SAP C samples had the largest ones, confirming the prominent effect of particle sizes of polymers (similar chemical compositions). SAP with finer particles and higher WAC (Table 2) resulted in a longer water retention in plastic state and avoidance of PS cracking.

3.2 Autogenous shrinkage

Fig. 6 shows development of autogenous shrinkage (AS) in the reference mortars (no SAPs) during the first 180 days. The curves represent the trends in AS development.

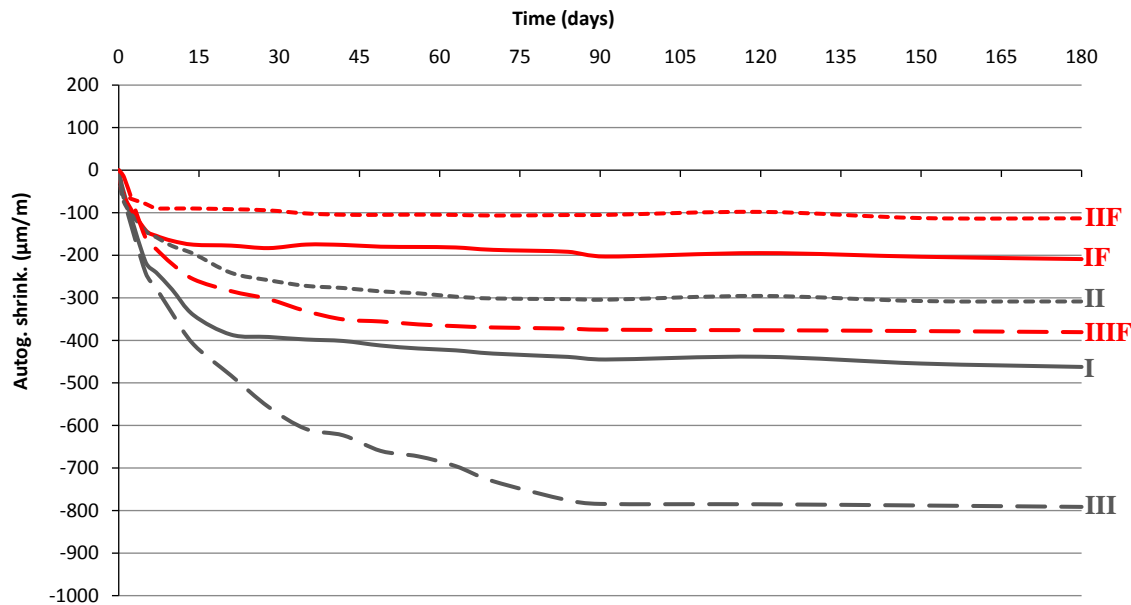


Fig. 6. Autogenous shrinkage development of reference mortars (without SAPs) during 180 days.

After an initial sharp increase (15 days) the progress of autogenous shrinkage slows down in all reference samples (CEM I, CEM II and CEM III). At 180 days, AS for plain mortars with CEM I, CEM II/B-V and CEM III/A reached approximate values of -450 , -300 and -780 $\mu\text{m}/\text{m}$ respectively, due to a different degree and rate of reactions [5, 49, 50]. Overall, delayed initial reactivity rate and lower water consumption during PC-FA pozzolanic reactions lead to reduced chemical shrinkage in samples with CEM II/B-V. On the other hand, the latent-hydraulic GGBS (CEM III/A) increases hydration degree by speeding up clinker reactions in early ages (dilution effect) and a denser microstructure with smaller pores is formed. This, in turn, leads to the higher tensile stresses triggered by water menisci between pores' walls, resulting in higher AS. Such a behaviour was also reported by [51, 52].

The same general pattern of AS, although reduced, was also observed for FRM. At 180 days, absolute values of AS were -210 , -115 and -380 $\mu\text{m}/\text{m}$, respectively for IF, IIF and IIF samples. This reduction is more explicit in matrices with CEM III/A cement (50% of reduction compared to sample III, without PF). For CEM I and CEM II/B-V samples, this decrement was around 30% and 25%, respectively. Polymeric fibres (PF) can retain some water during early ages and influence hydration processes in different types of cement [53]. These changes in water availability affect the rate and degree of hydration, especially in the presence of SCMs [30, 54].

For example, the highest difference in AS can be attributed to the physical presence of GGBS [55, 56]. It is well known that (up to 30 h), the filler effects dominate leading to an increased, and sometimes also faster, reaction of PC phases. This is due to the presence of more space relative to the amount of clinker (dilution effect) and increased nucleation rates [54-57]. Thus, the higher early reaction rate in GGBS mixes results in higher chemical shrinkage triggered by self-desiccation process [54, 55, 58-61]. GGBS can also refine capillary pores in blended paste, and thus contribute to the greater shrinkage [58, 61].

When PF is added to the mix, the initial retention of water acts as an internal curing agent. This alters dilution effect of PC and GGBS hydrations during early ages, significantly reducing AS [30]. Moreover, PFs act as nucleation sites for the secondary hydration products from SCM reactions. This enhances bonding between fibres and matrix, resulting in improved mechanical properties and reduced autogenous shrinkage [53, 62].

Fig. 7 shows the effect of SAPs addition on AS development of mortars produced with CEM I. As illustrated, all SAPs can further limit AS in FRM and the extent of mitigation (between 76 - 122%) depends very much on the type of polymer.

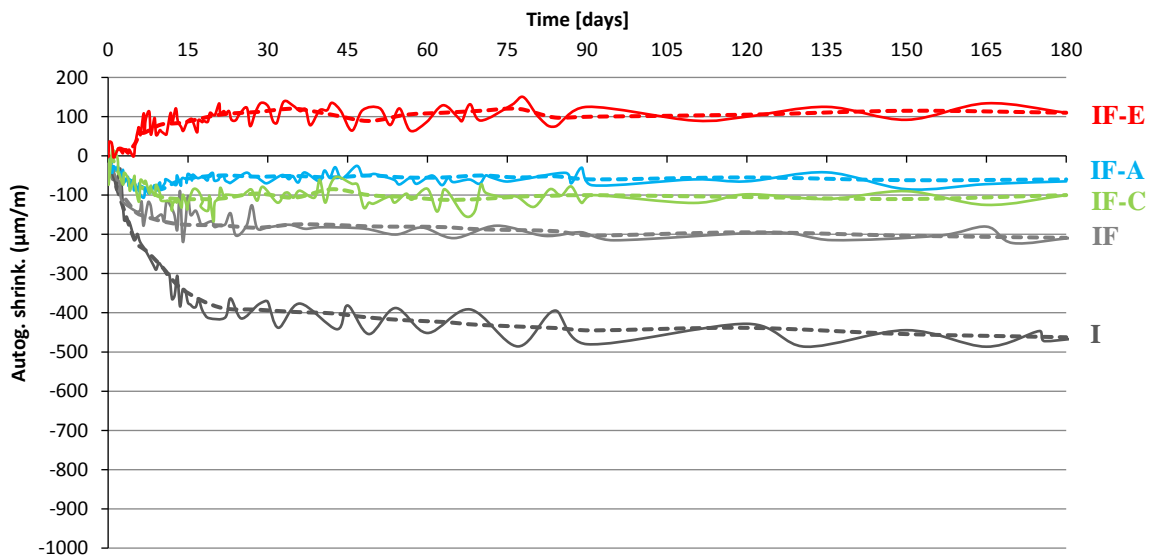


Fig. 7. Autogenous shrinkage development in CEM I (PC) during 180 days. Dotted lines represent the trends in AS development.

In general, SAP C performed similarly to SAP A, although SAP A showed a slight swelling between the first and second week. This pattern of increasing volume was also found in SAP E mortars triggered by crystallization of portlandite or re-absorption of bleeding water from the fresh mix just after setting [12]. After the second week, no significant changes have been observed in SAP mortars. At the end of testing (180 days), AS values were approximately: -60, -110 and 100 $\mu\text{m}/\text{m}$ respectively for samples with SAP A, SAP C and SAP E. This means an additional reduction in the range of 46 - 92%.

Fig. 8 presents autogenous shrinkage results of CEM II/B-V mortars with fibre and different SAPs. Similarly to CEM I samples (shown in Fig. 7) the highest reduction of shrinkage was recorded for samples with SAP E, followed by SAP A and SAP C. However, this reduction is more distinct in matrices with CEM II/B-V, where AS was almost eliminated.

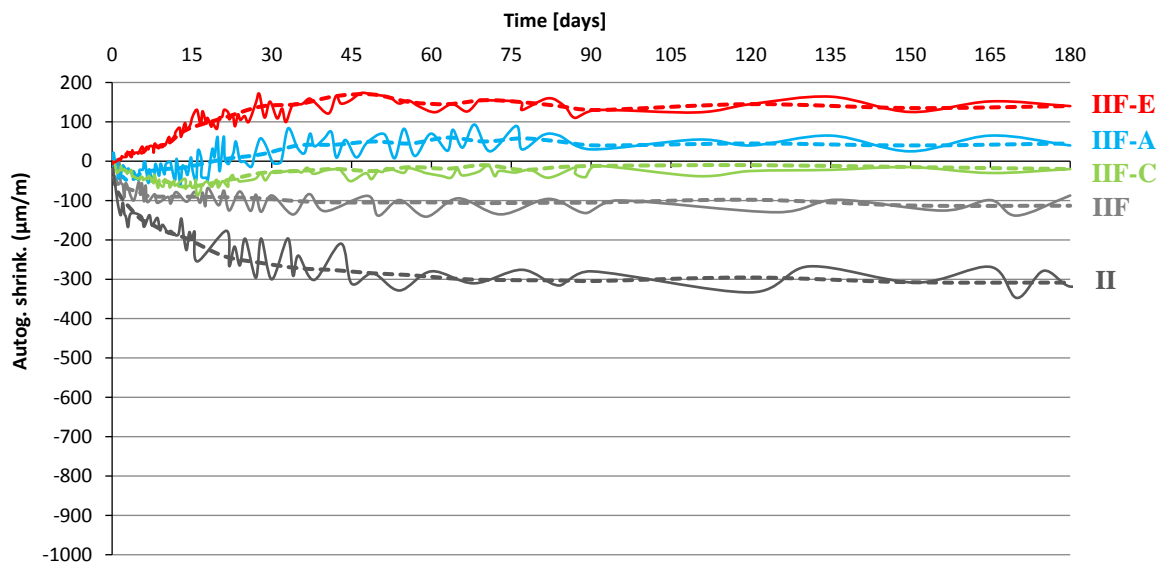


Fig. 8. Autogenous shrinkage development in CEM II/B-V (PC-FA) during 180 days.

Dotted lines represent the trends in AS development.

All SAP samples experienced a noticeable volume expansion during the first 45 days, in particular mortars with SAP C and E. SAP C samples swelled from near -90 to -20 $\mu\text{m}/\text{m}$, while SAPs A and E mortars, from almost 0 to 30 and 140 $\mu\text{m}/\text{m}$. Samples with SAP E experienced higher expansion due to SAP fineness and faster absorption kinetics. At 180 days, FRM mortars with SAP A, C and E reached AS values of 40, -22 and 150 $\mu\text{m}/\text{m}$, respectively. This represents an additional reduction in the range of 68 -124%.

Autogenous shrinkage development for CEM III/A mortars is shown in Fig. 9. Again, when compared to the reference fibre reinforced mortar (IIIF), all SAP samples showed a significantly reduced AS (above 80%). Analogously to the CEM I pattern (Fig. 7), mortars containing SAP A performed similarly to those with SAP C, while SAP E samples showed the greatest AS reduction. At 180 days deformations reached approximately: -85, -140 and 40 $\mu\text{m}/\text{m}$ respectively for the mixes with SAP A, C and E. Addition of SAPs clearly leads to mitigation of autogenous shrinkage in all fibre reinforced mortars. However, the extent of mitigation depends very much on type of SAP and cement and the range of this additional AS reduction varies between 30-55%.

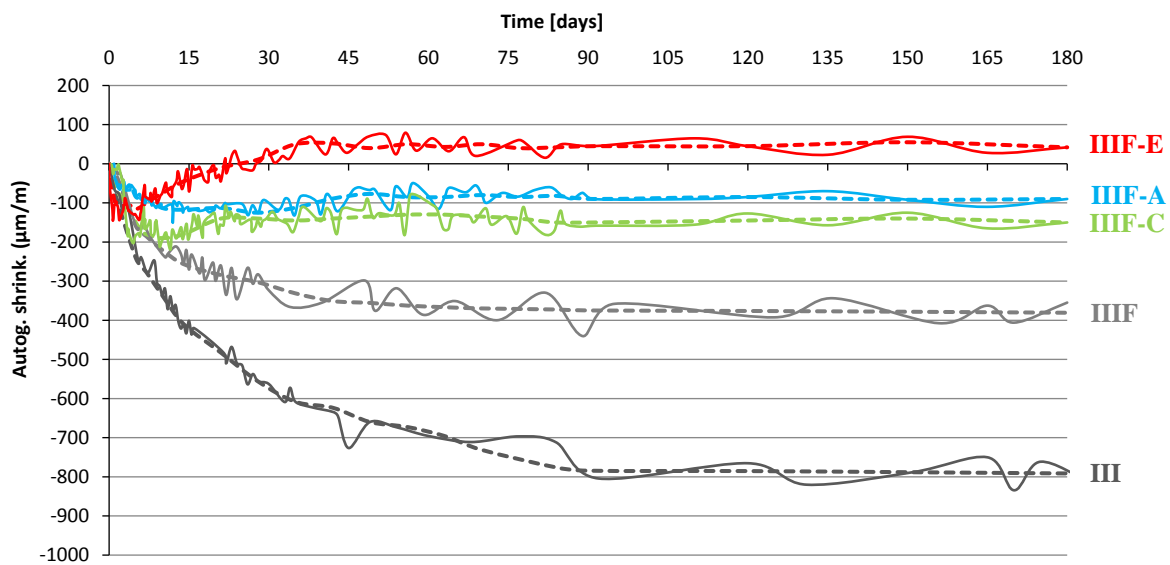


Fig. 9. Autogenous shrinkage development in CEM III/A (PC-GGBS) during 180 days. Dotted lines represent the trends in AS development.

This is evidenced by the prolonged swelling pattern in CEM II/B-V and CEM III/A mortars during the first 45 days (not observed in CEM I mortars). SAPs promote precipitation of further C-S-H phases (later reactions of FA and GGBS) leading to an overall bulk expansion of hardened matrix. This effect is more prominent for SAP E due to its very fine particles and greater water absorption capacity for all cementitious solutions (Table 2). Thus, the contribution of SAP in SCM-FRM is twofold: internal curing for additional AS reduction (in the first two weeks), as well as prolonged hydration for pozzolanic and latent-hydraulic materials. Further hydration products can be formed by later water release from SAP, as also reported by [28, 30, 50, 63].

3.3 Drying shrinkage

Fig. 10 shows drying shrinkage (DS) development in all samples up to 180 days.

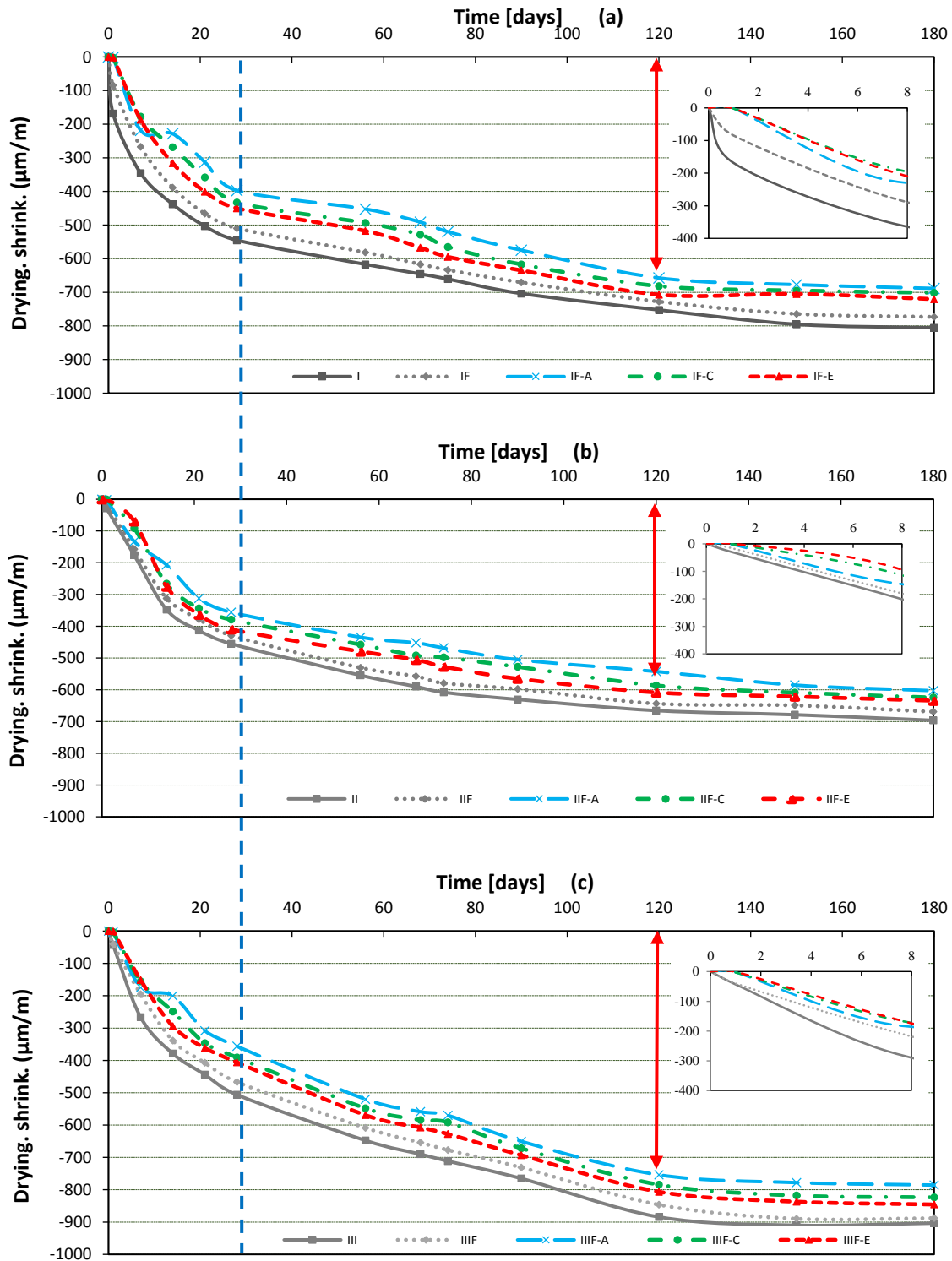


Fig. 10. Drying shrinkage development of all samples (a) CEM I (PC), (b) CEM II/B-V (PC-FA) and (c) CEM III/A (PC-GGBS).

During the period of 120 days drying shrinkage curves for all reference mortars have accentuated slopes, particularly during the first 28 days (blue dash line). This is due to the increased cement hydration rate [64] and early water evaporation processes. After this period, when hydration process was completed and the equilibrium of humidity was reached, no further significant changes were recorded (red arrows). Drying shrinkage was considerably more prominent in CEM III/A mortars due to the reaction of calcium sulphate (from PC) with alumina (from GGBS) [65], as well as the higher AS (Fig. 6).

Contrarily, CEM II/B-V with low water/binder ratio (Table 3) showed the lowest drying shrinkage due to the lower amount of water lost to the surrounding atmosphere and denser microstructure with minimized susceptibility to crack formation. Relatively high DS values can be attributed to high w/b ratios, which are in line with the previously reported studies [52, 60]. Addition of polymeric fibres can reduce DS by approximately 5% at the age of 180 days. The improvement of bond strength between fibres and cementitious matrix results in a physical restraining of shrinkage, leading to the limited micro-cracking inside mortars.

As shown in Fig. 10, drying shrinkage in FRM can be further reduced by application of SAP, regardless the cement type. Drying shrinkage in mortars with SAP is directly proportional to the water absorption capacity (WAC) of the polymer (Table 2); the lower WAC, the lesser water retained in the mix and the lower DS. Thus, the most effective in reduction of shrinkage was SAP A, owing to its lowest WAC. In contrast to autogenous and plastic shrinkage, the effect of particle sizes is less important for drying shrinkage.

However, during the first 7 days, a notably lower DS was recorded in samples with SAP E due to its contribution to the reduction of AS and PS. Moreover, water in small pores, created by SAP E, is less likely to evaporate [67]. Thus, the differences in recorded values of drying shrinkage are linked to water availability, porosity and sorption behaviour of SAP.

Mass loss and shrinkage curves of mixtures with SAP are shown in Fig. 11.

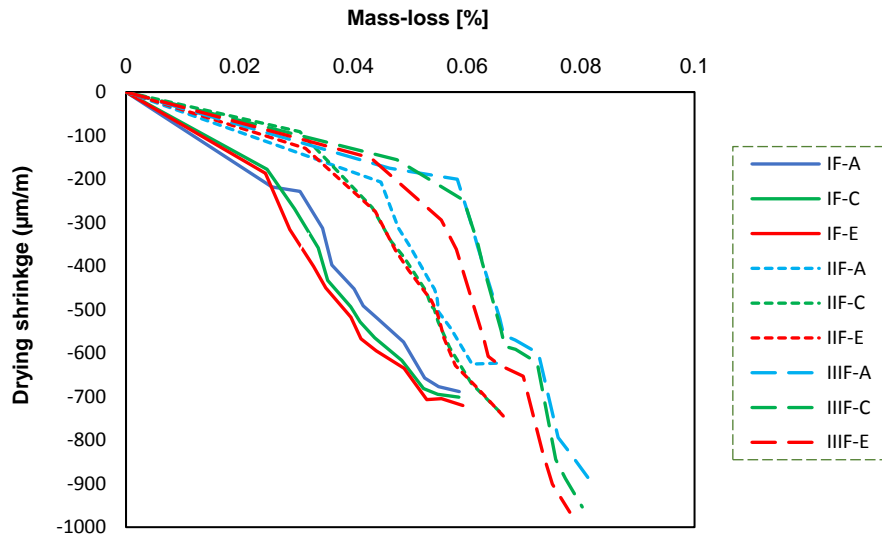


Fig. 11. Mass loss-shrinkage relationship for FRM with SAPs.

For the same level of mass loss, mortars containing SAP with lower WAC experienced lower drying shrinkages. In the initial stages ($DS \leq -300 \mu\text{m/m}$), CEM I curves had the higher inclination (in relation to mass loss axis) compared to CEM II and CEM III curves. This higher inclination is due to the initial higher availability of free water. Concurrently, the magnitude of drying shrinkage is influenced by SAP absorption capacities. With the prolonged time, shrinkage progressively increases in mortars containing SAP with higher WAC due to formation of additional coarse pores.

3.4 Shrinkage reduction levels by SAP

Table 4 shows the quantitative analysis of additional reduction of different types of shrinkage facilitated by SAPs (Eqs. (1) and (2)). The calculation of reduction levels was based on the final measurements for each shrinkage test, i.e., at 24 h for plastic shrinkage (measure of cracks widths) and at 180 days for autogenous and drying shrinkages.

Based on these analyses, it is clear that the greatest shrinkage reductions were recorded in reinforced mortars containing SAP E. As SAP was added, plastic shrinkage of FRMs decreased by 30–75% when compared to the corresponding reference samples with PF. Also, autogenous shrinkage of SAP mortars was 30–124% lower than in samples containing only PF. This “additional” reduction of PS and AS increases as particle sizes of SAP decreases. Drying

shrinkage is strongly correlated to the type of binder. Nevertheless, the maximum additional reduction of drying shrinkage was recorded for samples with SAP A, with the lowest WAC.

Table 4. Plastic, autogenous and drying shrinkage reduction levels for SAP modified mortars compared to both reference samples

Sample Name	Description	Total reduction			Additional reduction		
		PS	AS	DS	PS	AS	DS
I	CEM I (PC)	-	-	-	-	-	-
IF	CEM I, PF	50%	30%	5%	-	-	-
IF-A	CEM I, PF, SAP A	73%	87%	14%	46%	57%	10%
IF-C	CEM I, PF, SAP C	69%	76%	12%	39%	46%	8%
IF-E	CEM I, PF, SAP E	82%	122%	11%	64%	92%	7%
II	CEM II/B-V (PC-FA)	-	-	-	-	-	-
IIF	CEM II, PF	40%	25%	3%	-	-	-
IIF-A	CEM II, PF, SAP A	65%	113%	15%	42%	88%	12%
IIF-C	CEM II, PF, SAP C	60%	93%	13%	33%	68%	10%
IIF-E	CEM II, PF, SAP E	80%	149%	12%	67%	124%	9%
III	CEM III/A (PC-GGBS)	-	-	-	-	-	-
IIIF	CEM III, PF	30%	50%	2%	-	-	-
IIIF-A	CEM III, PF, SAP A	75%	87%	13%	65%	37%	12%
IIIF-C	CEM III, PF, SAP C	65%	80%	10%	50%	30%	9%
IIIF-E	CEM III, PF, SAP E	84%	105%	8%	76%	55%	8%

Table 4 illustrates better effectiveness of SAP in reducing autogenous shrinkage than drying shrinkage. This is attributed to the main role of SAP as internal curing agent. Its high capacity to absorb water from fresh mix and release over time can control shrinkage, during initial stages of cement hydration [28, 30, 67-72]. Hence, this effect is more pronounced for autogenous and plastic shrinkages.

On the other hand, lower reduction of drying shrinkage by SAP is related to the lower water availability for longer period of time (here measured up to 180 days). When SAP collapses, no (or little) extra water is free to be either consumed (by cement hydration) or to evaporate in drying processes. Therefore, autogenous shrinkage (and also plastic shrinkage) is significantly more reduced by SAP than drying shrinkage.

The results of volumetric changes of fibre reinforced mortars demonstrated the outstanding efficiency of superabsorbent polymers in promoting additional reduction of shrinkage for any type of cement.

4. CONCLUSION

Superabsorbent polymers (SAP) facilitate substantial additional reduction in plastic, autogenous and drying shrinkage in fibre reinforced mortars (FRM) with supplementary cementitious materials. However, the level of efficiency depends on either particle sizes and/or water absorption capacity (WAC), as well as cement type used:

- The biggest effect on crack propagation in plastic shrinkage, up to 75% can be achieved by application of fine SAPs (SAP E). The effect of different water absorption capacities (SAP A and C) is of a lesser importance;
- Although different cements show distinct autogenous shrinkage performances, the overall behaviour for SAP mortars with different binders is almost identical. The effect of water absorption capacity on AS is very limited in FRM with blended cements. However, SAP effectiveness depends on the particle sizes. The significant reduction of autogenous shrinkage up to 124% is observed for mortars with SAP E, with finer particle size ($76.74 \pm 0.22 \mu\text{m}$);
- Drying shrinkage in FRMs is the least sensitive to SAPs, where the type of cement plays a critical role. Reduction of DS is more pronounced for mortars with CEM II/B-V (PC-FA). SAP effectiveness depends mainly on water absorption capacity. The highest reduction of drying shrinkage takes place in mortars with the lowest WAC (SAP A) up to 12%.

Acknowledgements

The authors acknowledge Hanson Cements (UK), Lafarge (UK) and Ecocem (Ireland) for the supply of cements, and BASF Construction Chemicals GmbH (Trostberg, Germany) for the supply of SAPs.

References

- [1] Scrivener, K.L., John, V.M. and Gartner, E.M., 2018. Eco-efficient cements: Potential economically viable solutions for a low-CO₂ cement-based materials industry. *Cement and Concrete Research*, 114, pp.2-26.

- [2] John, Vanderley M., Marco Quattrone, Pedro CRA Abrão, and Fabio A. Cardoso. "Rethinking cement standards: Opportunities for a better future." *Cement and Concrete Research* 124 (2019): 105832.
- [3] Juenger, M.C., Snellings, R. and Bernal, S.A., 2019. Supplementary cementitious materials: New sources, characterization, and performance insights. *Cement and Concrete Research*, 122, pp.257-273.
- [4] Suraneni, P., Hajibabae, A., Ramanathan, S., Wang, Y. and Weiss, J., 2019. New insights from reactivity testing of supplementary cementitious materials. *Cement and Concrete Composites*, 103, pp.331-338.
- [5] Skibsted, J. and Snellings, R., 2019. Reactivity of supplementary cementitious materials (SCMs) in cement blends. *Cement and Concrete Research*, 124, p.105799.
- [6] Ghafari, E., Ghahari, S.A., Costa, H., Júlio, E., Portugal, A. and Durães, L., 2016. Effect of supplementary cementitious materials on autogenous shrinkage of ultra-high performance concrete. *Construction and Building Materials*, 127, pp.43-48.
- [7] Combrinck, R., Steyl, L. and Boshoff, W.P., 2018. Interaction between settlement and shrinkage cracking in plastic concrete. *Construction and Building Materials*, 185, pp.1-11.
- [8] Ghourchian, S., Wyrzykowski, M., Baquerizo, L. and Lura, P., 2018. Susceptibility of Portland cement and blended cement concretes to plastic shrinkage cracking. *Cement and Concrete Composites*, 85, pp.44-55.
- [9] Sant, G., Lura, P. and Weiss, J., 2009. The influence of temperature on unrestrained volume changes in cementitious materials. In *International RILEM conference on concrete durability and service life planning 'ConcreteLife '09'*, Haifa, Israel.
- [10] Lura, P., Pease, B., Mazzotta, G.B., Rajabipour, F. and Weiss, J., 2007. Influence of shrinkage-reducing admixtures on development of plastic shrinkage cracks. *ACI materials journal*, 104(2), p.187.
- [11] Kayondo, M., Combrinck, R. and Boshoff, W.P., 2019. State-of-the-art review on plastic cracking of concrete. *Construction and Building Materials*, 225, pp.886-899.
- [12] Snoeck, D., Jensen, O.M. and De Belie, N., 2015. "The influence of superabsorbent polymers on the autogenous shrinkage properties of cement pastes with supplementary cementitious materials". *Cement and Concrete Research*, 74, pp.59-67. <https://doi.org/10.1016/j.cemconres.2015.03.020>.
- [13] Wyrzykowski, M. and Lura, P., (2016). Effect of relative humidity decrease due to self-desiccation on the hydration kinetics of cement. *Cement and Concrete Research*, 85, pp.75-81.
- [14] Jensen, O.M. and Hansen, P.F., 2001. "Water-entrained cement-based materials: I. Principles and theoretical background". *Cement and concrete research*, 31(4), pp.647-654. [https://doi.org/10.1016/S0008-8846\(01\)00463-X](https://doi.org/10.1016/S0008-8846(01)00463-X).
- [15] Lura, P., Jensen, O.M. and Van Breugel, K., 2003. Autogenous shrinkage in high-performance cement paste: An evaluation of basic mechanisms. *Cement and concrete research*, 33(2), pp.223-232.
- [16] Di Bella, C., Wyrzykowski, M. and Lura, P., 2017. Evaluation of the ultimate drying shrinkage of cement-based mortars with poroelastic models. *Materials and Structures*, 50(1), p.52.
- [17] Hu, X., Shi, Z., Shi, C., Wu, Z., Tong, B., Ou, Z. and De Schutter, G., 2017. Drying shrinkage and cracking resistance of concrete made with ternary cementitious components. *Construction and Building Materials*, 149, pp.406-415.
- [18] Gilbert, R.I., Castel, A., Khan, I., South, W. and Mohammadi, J., 2018. An Experimental Study of Autogenous and Drying Shrinkage. In *High Tech Concrete: Where Technology and Engineering Meet* (pp. 33-41). Springer, Cham.

- [19] Nežerka, V., Havlásek, P. and Trejbal, J., 2020. Mitigating inclusion-induced shrinkage cracking in cementitious composites by incorporating recycled concrete fines. *Construction and Building Materials*, 248, p.118673.
- [20] Cheng, S., Shui, Z., Yu, R., Sun, T. and Zhang, X., 2017. Multiple influences of internal curing and supplementary cementitious materials on the shrinkage and microstructure development of reefs aggregate concrete. *Construction and Building Materials*, 155, pp.522-530.
- [21] Duckheim, C. and Setzer, M.J., 2008. Drying shrinkage mechanisms of hardened cement paste. *Proc Creep Shrinkage Durab Mech Concr Concr Struct*, 2008, pp.49-55.
- [22] Bertelsen, I.M.G., Ottosen, L.M. and Fischer, G., 2020. Influence of fibre characteristics on plastic shrinkage cracking in cement-based materials: A review. *Construction and Building Materials*, 230, p.116769.
- [23] Mazzoli, A., Monosi, S. and Plescia, E.S., 2015. Evaluation of the early-age-shrinkage of Fiber Reinforced Concrete (FRC) using image analysis methods. *Construction and Building Materials*, 101, pp.596-601.
- [24] Yousefieh, N., Joshaghani, A., Hajibandeh, E. and Shekarchi, M., 2017. Influence of fibers on drying shrinkage in restrained concrete. *Construction and Building Materials*, 148, pp.833-845.
- [25] Aly, T., Sanjayan, J.G. and Collins, F., 2008. Effect of polypropylene fibers on shrinkage and cracking of concretes. *Materials and Structures*, 41(10), p.1741.
- [26] Wyrzykowski, M. and Lura, P., 2013. Controlling the coefficient of thermal expansion of cementitious materials—A new application for superabsorbent polymers. *Cement and Concrete Composites*, 35(1), pp.49-58.
- [27] Rostami, R. and Klemm, A.J., 2020. Influence of superabsorbent polymers on properties of fibre reinforced mortars containing fly ashes. *Roads and Bridges*, 19, pp.149-163.
- [28] Montanari, L., Suraneni, P. and Weiss, W.J., 2017. Accounting for water stored in superabsorbent polymers in increasing the degree of hydration and reducing the shrinkage of internally cured cementitious mixtures. *Advances in Civil Engineering Materials*, 6(1), pp.583-599.
- [29] Almeida, F.C., Rostami, R. and Klemm, A.J., (2019). The Effect of SAP on Volumetric Changes and Microstructural Alterations in PC-GGBS Matrices. In *International Conference on Application of Superabsorbent Polymers & Other New Admixtures Towards Smart Concrete* (pp. 97-105). Springer, Cham.
- [30] Almeida, F.C. and Klemm, A.J., 2018. Efficiency of internal curing by superabsorbent polymers (SAP) in PC-GGBS mortars. *Cement and Concrete Composites*, 88, pp.41-51.
- [31] Ghourchian, S., Wyrzykowski, M., Plamondon, M. and Lura, P., 2019. On the mechanism of plastic shrinkage cracking in fresh cementitious materials. *Cement and Concrete Research*, 115, pp.251-263.
- [32] Meyer, D.M., Boshoff, W.P. and Combrinck, R., 2020. Utilising super absorbent polymers as alternative method to test plastic shrinkage cracks in concrete. *Construction and Building Materials*, 248, p.118666.
- [33] Almeida, F.C. and Klemm, A.J., 2018. Effect of GGBS on water absorption capacity and stability of superabsorbent polymers partially crosslinked with alkalis. *Journal of Materials in Civil Engineering*, 30(12), p.04018315.
- [34] Klemm, A.J. and Sikora, K.S., 2013. The effect of Superabsorbent Polymers (SAP) on microstructure and mechanical properties of fly ash cementitious mortars. *Construction and Building Materials*, 49, pp.134-143.
- [35] BS (British Standard). 2011. Cement. Part 1: Composition, specifications and conformity criteria for common cements. EN 197-1. Brussels, Belgium: BS.

- [36] Almeida, F.C., Rostami, R. and Klemm, A.J., (2018). Characterization of Polyacrylamide based Superabsorbent Polymers for potential use in PC Matrices with Supplementary Cementitious Materials. In MATEC Web of Conferences (Vol. 199, p. 02023). EDP Sciences.
- [37] Mechtcherine, V., Snoeck, D., Schröfl, C., De Belie, N., Klemm, A.J., Ichimiya, K., Moon, J., Wyrzykowski, M., Lura, P., Toropovs, N. and Assmann, A., (2018). Testing superabsorbent polymer (SAP) sorption properties prior to implementation in concrete: results of a RILEM Round-Robin Test. *Materials and Structures*, 51(1), p.28.
- [38] Lura, P., Winnefeld, F. and Fang, X., 2017. A simple method for determining the total amount of physically and chemically bound water of different cements. *Journal of Thermal Analysis and Calorimetry*, 130(2), pp.653-660.
- [39] BSI 2006b. BS EN 1015-3. Methods of Test for Mortar for Masonry - Part 3: Determination of Consistence of Fresh Mortar (by Flow Table). British Standards Institution.
- [40] ASTM 2013. C1579-13. Standard Test Method for Evaluating Plastic Shrinkage Cracking of Restrained Fiber Reinforced Concrete (Using a Steel Form Insert). bsi International - American Society for Testing and Materials, pp.1-7.
- [41] ASTM C1698-09. Standard Test Method for Autogenous Strain of Cement Paste and Mortars. ASTM International, 2009, American Society for Testing and Materials.
- [42] BS EN 480-2. Admixtures for Concrete, Mortar and Grout - Test Methods - Part 2: Determination of Setting Time. 2006, British Standards Institution.
- [43] BS EN 1920-8. (2009). "Part 8: Determination of drying shrinkage of concrete for samples prepared in the field or in the laboratory." British Standard, 50.
- [44] Serpukhov, I. and Mechtcherine, V. 2015. Early-age shrinkage of ordinary concrete and a strain-hardening cement-based composite (SHCC) in the conditions of hot weather curing. IN: C. Hellmich, B. Pichler, J. Kollegger (Eds.), *Mechanics and physics of creep, shrinkage and durability of concrete and concrete structures (Proceedings of CONCREEP 10)*. pp. 1504-1513.
- [45] Ghourchian, S., Wyrzykowski, M. and Lura, P., 2016. The bleeding test: a simple method for obtaining the permeability and bulk modulus of fresh concrete. *Cement and Concrete Research*, 89, pp.249-256.
- [46] Rostami, R. and Klemm, A.J., 2019, March. Effect of superabsorbent polymers on plastic shrinkage cracking and properties of fresh state mortars reinforced by polymeric fibres. In *Proceedings of the International Conference on Sustainable Materials, Systems and Structures (SMSS2019): New Generation of Construction Materials* (pp. 614-621). RILEM.
- [47] Kim, J.H.J., Park, C.G., Lee, S.W., Lee, S.W. and Won, J.P., 2008. Effects of the geometry of recycled PET fiber reinforcement on shrinkage cracking of cement-based composites. *Composites Part B: Engineering*, 39(3), pp.442-450.
- [48] Bertelsen, I.M.G., Ottosen, L.M. and Fischer, G., 2019. Quantitative analysis of the influence of synthetic fibres on plastic shrinkage cracking using digital image correlation. *Construction and Building Materials*, 199, pp.124-137.
- [49] Scrivener, K.L., Lothenbach, B., De Belie, N., Gruyaert, E., Skibsted, J., Snellings, R. and Vollpracht, A., 2015. TC 238-SCM: hydration and microstructure of concrete with SCMs. *Materials and Structures*, 48(4), pp.835-862.
- [50] De la Varga, I., Castro, J., Bentz, D. and Weiss, J., 2012. Application of internal curing for mixtures containing high volumes of fly ash. *Cement and Concrete Composites*, 34(9), pp.1001-1008.
- [51] Schröfl, C., Mechtcherine, V. and Gorges, M., 2012. Relation between the molecular structure and the efficiency of superabsorbent polymers (SAP) as concrete admixture to mitigate autogenous shrinkage. *Cement and concrete research*, 42(6), pp.865-873.

- [52] Liu, J., Ou, Z., Mo, J., Wang, Y. and Wu, H., 2017. The effect of SCMs and SAP on the autogenous shrinkage and hydration process of RPC. *Construction and Building Materials*, 155, pp.239-249.
- [53] Chinchillas-Chinchillas, M.J., Orozco-Carmona, V.M., Gaxiola, A., Alvarado-Beltrán, C.G., Pellegrini-Cervantes, M.J., Baldenebro-López, F.J. and Castro-Beltrán, A., 2019. Evaluation of the mechanical properties, durability and drying shrinkage of the mortar reinforced with polyacrylonitrile microfibers. *Construction and Building Materials*, 210, pp.32-39.
- [54] Lothenbach, B., Scrivener, K. and Hooton, R.D., 2011. Supplementary cementitious materials. *Cement and concrete research*, 41(12), pp.1244-1256.
- [55] Scrivener, K.L., Juilland, P. and Monteiro, P.J., 2015. Advances in understanding hydration of Portland cement. *Cement and Concrete Research*, 78, pp.38-56.
- [56] Scrivener, K.L., Lothenbach, B., De Belie, N., Gruyaert, E., Skibsted, J., Snellings, R. and Vollpracht, A., 2015. TC 238-SCM: hydration and microstructure of concrete with SCMs. *Materials and Structures*, 48(4), pp.835-862.
- [57] Berodier, E. and Scrivener, K., 2015. Evolution of pore structure in blended systems. *Cement and Concrete Research*, 73, pp.25-35.
- [58] Valcuende, M., Benito, F., Parra, C. and Miñano, I., 2015. Shrinkage of self-compacting concrete made with blast furnace slag as fine aggregate. *Construction and Building Materials*, 76, pp.1-9.
- [59] Zhang, W., Hama, Y. and Na, S.H., 2015. Drying shrinkage and microstructure characteristics of mortar incorporating ground granulated blast furnace slag and shrinkage reducing admixture. *Construction and building materials*, 93, pp.267-277.
- [60] Jiang, C., Yang, Y., Wang, Y., Zhou, Y. and Ma, C., 2014. Autogenous shrinkage of high performance concrete containing mineral admixtures under different curing temperatures. *Construction and Building materials*, 61, pp.260-269.
- [61] Lee, K.M., Lee, H.K., Lee, S.H. and Kim, G.Y., 2006. Autogenous shrinkage of concrete containing granulated blast-furnace slag. *Cement and Concrete Research*, 36(7), pp.1279-1285.
- [62] Ghafari, E., Ghafari, S.A., Costa, H., Júlio, E., Portugal, A. and Durães, L., 2016. Effect of supplementary cementitious materials on autogenous shrinkage of ultra-high performance concrete. *Construction and Building Materials*, 127, pp.43-48.
- [63] Trtik, P., Muench, B., Weiss, W.J., Herth, G., Kaestner, A., Lehmann, E. and Lura, P., 2010, September. Neutron tomography measurements of water release from superabsorbent polymers in cement paste. In *Proceedings of the international conference on material science and 64th RILEM annual week*, Aachen, Germany (pp. 6-10).
- [64] Zhang, W., Hama, Y. and Na, S.H., 2015. Drying shrinkage and microstructure characteristics of mortar incorporating ground granulated blast furnace slag and shrinkage reducing admixture. *Construction and building materials*, 93, pp.267-277.
- [65] Hooton, R.D., Stanish, K., Angel, J.P. and Prusinski, J., 2009. The effect of ground granulated blast furnace slag (slag cement) on the drying shrinkage of concrete—a critical review of the literature. *Slag Cement Concrete*, pp.79-94.
- [66] De Belie, N., Soutsos, M. and Gruyaert, E., 2018. *Properties of fresh and hardened concrete containing supplementary cementitious materials*. Cham, Switzerland: Springer.
- [67] Ye, H., Cartwright, C., Rajabipour, F. and Radlińska, A., 2017. Understanding the drying shrinkage performance of alkali-activated slag mortars. *Cement and Concrete Composites*, 76, pp.13-24.
- [68] Mechtcherine, V. and Reinhardt, H.W. eds., 2012. *Application of super absorbent polymers (SAP) in concrete construction: state-of-the-art report prepared by Technical Committee 225-SAP (Vol. 2)*. Springer Science & Business Media.

- [69] Craeye, B., Geirnaert, M. and De Schutter, G., 2011. Super absorbing polymers as an internal curing agent for mitigation of early-age cracking of high-performance concrete bridge decks. *Construction and building materials*, 25(1), pp.1-13.
- [70] Hasholt, M.T., Jensen, O.M., Kovler, K. and Zhutovsky, S., 2012. Can superabsorbent polymers mitigate autogenous shrinkage of internally cured concrete without compromising the strength?. *Construction and Building Materials*, 31, pp.226-230.
- [71] Schröfl, C., Mechtcherine, V. and Gorges, M., 2012. Relation between the molecular structure and the efficiency of superabsorbent polymers (SAP) as concrete admixture to mitigate autogenous shrinkage. *Cement and concrete research*, 42(6), pp.865-873.
- [72] Shen, D., Wang, X., Cheng, D., Zhang, J. and Jiang, G., 2016. Effect of internal curing with super absorbent polymers on autogenous shrinkage of concrete at early age. *Construction and Building Materials*, 106, pp.512-522.