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Influence of Polycarboxylic Ether Superplasticizer (PCE) on the Absorption Capacity of Sulfate- and Potassium-Based Superabsorbent Polymers (SAPs) for Use in Cement-Based Materials

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Abstract

Superabsorbent polymers (SAPs) have a highly hydrophilic network, absorbing and retaining large amounts of water or aqueous solutions, making them useful for internal curing in cement-based materials. Absorption is influenced by the SAPs characteristics, the medium, and the testing method. In many studies reporting on the use of SAPs in cementitious materials, their absorption capacity is usually tested via demineralized water or artificial cement filtrate solution (which does not always include the presence of superplasticizers). PCE-based superplasticizers are among the most used chemical admixtures for concrete, especially when fluid/self-compacting mixtures are produced, and their chemical composition can strongly influence the absorption capacity of different types of SAPs (due to the presence of polymer chains with sulfonic groups (-SO_3H)). This study focused on determining the absorption capacity of three commercial SAPs for use as internal curing agents. The absorption capacity was investigated via the tea-bag method, filtration test, and slump-flow test with cement pastes. The chemical composition significantly influenced absorption: SAP A (with higher K_2O content) presented lower absorption with the superplasticizer, whereas SAPs B and C (higher SO_3) were less impacted because the high polarity favored absorption. The results indicate that the addition of superplasticizers can negatively affect the performance of SAPs in specific applications in civil construction. This impact highlights the need for further investigations into the absorption and desorption mechanisms of SAPs in the presence of these additives, with the aim of optimizing dosing strategies and improving field application methodologies.

Keywords Superabsorbent polymers · Absorption · Tea-bag · Filtration · Slump-flow · Superplasticizer

1 Introduction

Superabsorbent polymers (SAPs) are materials capable of absorbing and retaining large quantities of liquid relative to their mass, due to their polymeric network with high hydrophilic potential [1, 2]. Their applications range from everyday uses, such as hygiene products, to agriculture and, more recently, the construction industry [3].

SAPs are enhanced hydrogels that can absorb up to a thousand times their weight in water, while typical hydrogels absorb about ten times their weight. Understanding the swelling process of SAPs is crucial for optimizing their use in various applications and adapting synthesis parameters to develop new SAPs for specific uses [4].

In Fig. 1, the left image shows the SAP in its dry state, whereas the right image shows the polymer in its swollen state. Swelling occurs due to the absorption of the solution and dissolution of ions into the polymer network structure. The green areas highlight the interconnections of the primary polymer chains, represented in black, illustrating how the polymer network expands in volume while remaining insoluble [5].

The chemical composition of SAPs significantly influences their absorption capacity [6]. Factors such as the

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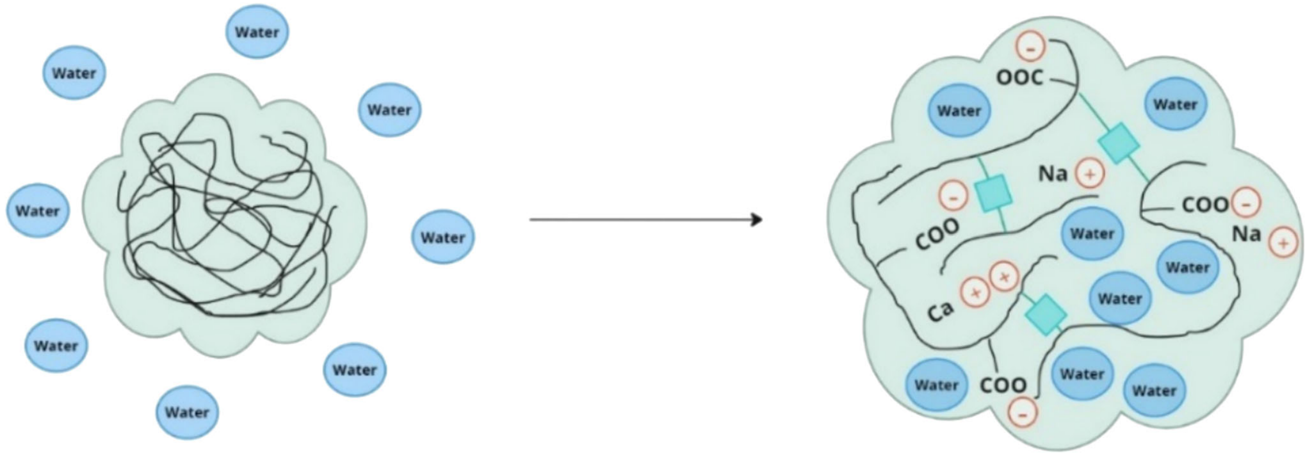


Fig. 1 Schematic of an SAP network in the collapsed (dry) state without water molecules taken up (left) and in the swollen state with absorbed water molecules and ions dissolved inside the network structure (right)

composition of hydrophilic functional groups, degree of crosslinking, ionic charge, and solubility are determinants [6, 7]. SAPs with different functional groups can actively influence the nucleation and crystallization of calcium hydroxide (CH) in cement pastes. This effect occurs by modifying the water-to-cement ratio (w/c) and the Ca2 + concentration in the pores formed by the SAPs, affecting crystallization pressure and CH orientation [8]. SAPs with carboxylate groups strongly interact with Ca2 +, releasing water slowly, delaying nucleation, and forming small, dispersed crystals [9]. Those with sulfonate groups exhibit moderate interaction, controlled water release, and may generate elongated or modified crystals [10]. Amide-functionalized SAPs have low interaction, gradual water release, and form homogeneous crystals [10]. Hydrophobic groups show minimal interaction, leading to limited and discontinuous water release with heterogeneously [11].

As for crosslinking agents, or reticulation agents, are chemical substances used to form covalent bonds between polymer chains. These bonds prevent the polymer from dissolving in the presence of water, providing the necessary structure for water retention. Commercial polymers, often by petroleum industry products, consist of a network based on acrylate, acrylic acid, and acrylamide. Common crosslinkers include copolymerized bifunctional monomers, such as N,N’-methylenebisacrylamide, which is widely used [12].

The grain shape is related to the production technique and the application of the SAP. Spherical particles are characteristic of the inverse suspension production technique, while granular particles are obtained by gel or bulk solution polymerization [13]. Granular particles are common in self-curing concretes, while spherical particles are used in lightweight concretes, reducing the material density without compromising strength [14].

A relevant factor that affects the absorption kinetics of SAPs is the ionic concentration of the solution [14]. The presence of ions in the solution can limit the electrostatic repulsion of functional groups, reducing the expansion capacity and flexibility of the polymer network. The reduction in osmotic potential due to high ionic concentrations also affects absorption. Additionally, ions in the solution can compete with the functional groups of SAPs for the water in the mixture [15]. Importantly, in cementitious mixtures, SAP comonomer, such as 2-acrylamido-2-methylpropane sulfonic acid (AMPS), is less sensitive to ions distributed in the cement paste, especially the formation of complexes with calcium, which can reduce the absorption capacity of SAPs [5, 16–18]. When SAP is in a cementitious environment, the ionic strength of the solution, resulting from the presence of dissolved ions (Ca2$^+$, Na$^+$, K$^+$, and SO$_4$2$^-$, among others), alters the osmotic potential of the pore solution, influencing its swelling capacity. This effect occurs due to the reduction of the osmotic gradient between the polymer interior and the pore solution [15, 18].

Additionally, the interaction with multivalent cations (Ca2$^+$, Mg2$^+$, and Al3$^+$) and anions such as SO$_4$2$^-$ and OH$^-$ can affect ionic crosslinking and the stability of polymer chains, reducing the SAP’s absorption capacity [19, 20]. The presence of additives, such as silica fumes and superplasticizers, which are widely used in high-performance cementitious mixtures, also influences absorption [21].

The particle size is also a factor associated with the absorption kinetics of SAPs, which are determined by their synthesis, emulsifiers, and sieving parameters. The particle size can be adjusted during post-production, as can the shape, chemical composition, and crosslinker. Particles smaller than 100 μm in the dry state and a few hundred micrometers in the swollen state are commonly used as internal curing agents in concrete [13, 22]. Finer particles provide smaller pore sizes with better distributions but in larger quantities. Ultrafine SAP powders are indicated to mitigate autogenous shrinkage, improve cement hydration, and increase the strength of high-performance concrete [23].

The addition of SAPs has also shown significant benefits for the durability of structures, acting on various degradation mechanisms over time. A study [24] demonstrated that with the presence of SAPs, there was a 10.7% increase in compressive strength, due to the storage and release of water by the SAP, as well as a 9.3% reduction in drying shrinkage. Additionally, SAPs contribute to freeze–thaw resistance, an important factor in cold regions, and reduce carbonation by up to 45%, thanks to improved hydration and reduced permeability. The presence of SAPs was also able to reduce acid rain penetration by up to 41% in concrete with SAP addition [25].

In large-scale concrete [26], the addition of SAPs reduced shrinkage deformation by 55% after 7 days and prevented cracking for 24 months, while the reference walls showed cracks as early as the 1 st week. Moreover, no signs of corrosion were observed in the treated walls, which were more resistant to carbonation and chloride penetration. Life cycle analyses revealed that concrete with SAPs showed a reduction of up to 14% in maintenance costs over 100 years, mainly due to crack mitigation and the promotion of autogenous curing [27].

Chloride ion penetration, which can cause reinforcement corrosion, is a critical factor for the durability of structures. The use of SAPs improves the resistance of concrete to chloride penetration by refining its microstructure and reducing permeability when used in adequate dosage. A study [25] has shown that 2‰ of SAP improves resistance in marine environments, but above 4‰ can generate excess voids, compromising this resistance. Furthermore, the presence of SAPs in autogenous curing of cracked mortar reduces chloride diffusion, especially in mixes with an effective W/C ratio $\leq$ 0.42, promoting crack sealing and improving impermeability, with a more significant effect on smaller cracks [28].

However, the absorption kinetics of SAPs in cement-based solutions can be affected by the active presence of silica and superplasticizer, influencing the absorption capacity [29]. To make the use of SAPs in concrete viable, accurate estimation of their absorption capacity is essential; otherwise, a reduction in concrete strength, durability, and workability may occur [30].

Therefore, this work aims to characterize three types of SAPs and investigate their absorption capacity through three distinct methods, relating the properties obtained with the absorption characteristics of the materials and exploring the influence of ionic action based on previously established discussions in the literature and presented throughout the text. Additionally, the methodology for analyzing SAPs in an aqueous medium with the addition of superplasticizers is highlighted, a topic that is still rarely addressed in the literature. Finally, a dosage of the three SAPs for future applications is proposed, along with suggested methodologies for their implementation.

2 Materials and Methods

Three types of SAPs, named A, B, and C, were analyzed in this study. These SAPs were produced for cementitious materials. The polymers used are copolymers of acrylamide-sodium acrylate. SAP A and B were chosen because they have been used by one of the authors in the previous studies [20], while SAP C was selected for being a commercial product, making it more accessible. A dosage of 2‰ of SAP was adopted in relation to the binder content, a volume that has already been used in the previous studies [21, 24, 31–33]. This quantity is effective in enhancing the concrete's properties without causing an excessive increase in porosity. The methodologies adopted for their characterization and absorption analysis are presented below.

The particle size distribution of dry SAP particles was studied using laser granulometry equipment, using the Fraunhofer method, with 14% obscuration, using a CILAS 1090 Laser Particle Size Analyzer. The reagent used as a dispersant was 99.8% alcohol, which was chosen because SAP does not absorb it [34]. To analyze the diameter of the particles, the software "The particle Expert" was used to obtain the granulometric curve and the parameters d_{10}, $d_{50}ed_{90}$.

Using the X-ray fluorescence (XRF) method, it was possible to identify the chemical composition based on the fluorescence radiation energy emitted by the SAP. The test was carried out using EDX-720 type equipment with a vacuum atmosphere.

X-ray diffraction (XRD) analysis was also carried out. Using this method, it was possible to analyze the presence of crystalline regions or amorphous halos in the material. Cu-ka tube radiation was used, with a 2Θ scanning range from 5 to 100º. The sweep speed was 2 degrees/minute, with a current of 30 mA and voltage of 40 kV.

Finally, to understand the morphology of the particles and the production technique of the SAPs, scanning electron microscopy (SEM) was employed using the Hitachi TM 3000. The dried powder samples were subjected to ultrasound for 60 s. Through the combined analysis of the characterization tests, the goal is to better understand the material's nature and the absorption process of each SAP.

Three methods were used to analyze the absorption of SAPs: tea-bag method, filtration method, and slump-flow test, which will be discussed in detail below.

2.1 Tea-Bag Method

The method followed the recommendations of TC 260-RSC from RILEM (International Union of Laboratories and Experts in Construction Materials, Systems, and Structures) [21]. Initially, ensuring that the tea-bag does not allow the SAP to escape during the test is essential. This study used bags measuring 8 cm wide and 10 cm high, with approximately 0.5 g of dry SAP. Care must be taken to ensure that the SAP amount is not too high; filling the bag could restrict the absorption of the polymer. First, the mass of liquid absorbed by the tea-bag must be determined so the dry tea-bag (m_a) was weighed.

The tea-bag was then briefly immersed in the study liquid and placed on dry paper to remove liquid for approximately 30 s, after which its mass was determined (m_b). This process must be done with at least ten tea-bags, thus obtaining the average absorption of the tea-bag (m_0), according to Eq. 1, which was used in the subsequent SAP absorption calculation.

$$m_0 = \frac{1}{n}.\sum_{i=1}^{n}(m_{bi} - m_{ai}) \tag{1}$$

Next, the dry tea-bag to be used in the procedure was weighed (m_1). The SAP particles were then placed into the tea-bag, and the mass was recorded (m_2). The tea-bag was then immersed in at least 200 ml of liquid. For this study, absorption analysis was conducted in water and a solution of water and the superplasticizer Glenium 51, with a mass ratio of 1:0.23. The container was covered with cling film to prevent carbonation and evaporation. The tea-bag plus the SAP set weighed 1, 5, 10, 30, 60 min, 3, and 24 h after the SAP came into contact with the liquid (m_3), permanently removing excess moisture. After the tea-bag was weighed, it was reinserted into the liquid until the subsequent measurement [4, 35]. To ensure the reliability of the study, at least three samples of each SAP were prepared. The absorption capacity (AC) is given by Equation 2.

$$AC = \frac{m_3 - m_2 - m_0}{m_2 - m_1} \tag{2}$$

2.2 Filtration Method

Recommendations for this assay were also determined by RILEM's TC 260-RSC [21]. Before the process begins, it is crucial to perform a preliminary test to ensure that after the SAP is added to the liquid, the excess test liquid allows the polymer to swell freely until maximum absorption. The mass of dry SAP used was approximately 0.1 g (m_1). The absorptions in water and water with superplasticizer (1:0.23) were studied. The dry SAP was placed in a beaker with 200 ml of solution (m_2). The container was covered with film paper, and the entire solution was filtered for 1, 5, 10, 30, 60 min, 3, and 24 h after SAP/liquid contact. The filter paper used was saturated with the test fluid to avoid interference with the results. The filter was folded to have less contact with the funnel, reducing the possibility of clogging [4]. Measurements were made in triplicates, ensuring the statistical significance of the test. Therefore, the amount of filtered fluid (m_3) was recorded. Equation 3 is used to obtain SC.

$$AC = \frac{m_2 - m_3}{m_1} \tag{3}$$

2.3 Slump-Flow Method

Initially, a reference UHPC was prepared without the addition of SAP. High initial strength Portland cement (Brazilian type CP V–ARI) and silica were used as the binders. The properties are shown in Table 1.

The sand was obtained from building materials stores in the region, dried, and then sieved through a 600 µm sieve. The average density under dry conditions was 2.47 g/cm3, and in saturated surface dry condition, it was 2.48 g/cm3. The water absorption was 0.42%.

Marble and Granite Waste (MGW) was obtained from a regional company. The residue was collected in a tailings pond and subjected to pre-drying in the sun. After drying, the material was piled and subjected to sieving on a 600 µm sieve. The particle size of the material ranges from 3.39 µm to 69 µm. X-ray fluorescence revealed that the material used was predominantly composed of SiO_2 (66.52%), Al_2O_3 (13.05%), Fe_2O_3 (8.01%), K_2O (7.06%), and CaO (4.06%).

The superplasticizer used was based on polycarboxylate ether, pH 4–6, with a density between 1.067 g/cm3 and 1.107 g/cm3 and 30% of solids. The mix design of the reference mixture is shown in Table 2. The mixing time since the materials' first contact with water was recorded and maintained for the remaining tests. The mixing method adopted is also presented in Table 2.

The slump-flow test was used to analyze the cement medium's absorption capacity based on NBR 13276: Mortar for laying and covering walls and ceilings—Determination of the consistency index [36]. The consistency test was carried out by measuring the diameter of the spread of the fresh material, with no blows being made on the consistency table.

The amount of SAP for all tests was set at 0.2% (2.21 g), of mass of the binders, and the SAP was inserted dry at the beginning of the process, together with the cement and silica. Water additions were made by assigning different absorption capacities (ACs) and, consequently, different values of total w/b [25], as shown in Table 3. The higher the

Table 1 Physical–chemical characterization of CPV ARI Portland cement and silica (data provided by the manufacturers)

Binders	Test reference		Method	Result	Requirement
Cement	Insoluble residue (%)		ABNT NM 15	1.36	≤ 3.5
	Loss of ignition (%)		ABNT NM 18	3.54	≤ 6.5
	Magnesium oxide (%)		ABNT NBR 14656	3.27	≤ 6.5
	Sulfur trioxide (%)		ABNT NBR 14656	4.09	≤ 4.5
	Sodium oxide (%)		ABNT NBR 14656	0.06	–
	Potassium oxide (%)		ABNT NBR 14656	1.22	–
	Specific area (Blaine) (cm^2/g)		ABNT NBR 16372	4359	–
	Specific mass (g/cm^3)		ABNT NBR 16605	3.05	–
	Fineness index—#75 μm (%)		ABNT NBR 11579	0.02	≤ 6.0
	Fineness index—45 μm (%)		ABNT NBR 9202/85	0.47	–
	Normal consistency water (%)		ABNT NBR 16606	32.6	–
	Setting start (min)		ABNT NBR 16607	151	≥ 60
	Setting end (min)		ABNT NBR 16607	211	≤ 600
	Expandability (mm)		ABNT NBR 11582	0.1	≤ 5
	Compressive strength (MPa)	1 day	ABNT NBR 7215	29	≥ 14
		3 days		39.5	≥ 24
		7 days		43.9	≥ 34
		28 days		50.9	–
Silica	Specific mass (g/cm^3)		ABNT NBR 16605	2.2	–
	Specific area (m^2/g)		ABNT NBR 16605	15,000—30,000	–
	Minimum silicon dioxide content (%)		ABNT NBR 14656	85	–

AC value adopted, the greater the amount of water added to check whether the SAP could absorb the additional water. The premise adopted is that, at the ideal dosage, the water absorbed by the SAP does not interfere with the spreading of the mortar; therefore, the slump-flow result that most closely matches the reference is sought [37].

The criteria for selecting the AC and w/b values for SAP A were based on the results of the tea-bag and filtration tests, considering the reductions reported in the literature for the effect of SAP on cementitious materials compared to water. After the initial test, a comparison was made with the reference UHPC to adjust the AC value. SAPs B and C, as will be discussed later, exhibited similar absorption behaviors that were superior to those of SAP A, guiding the selection of initial AC and w/b values that were subsequently adjusted based on the materials' behavior relative to the reference.

Once the AC of the SAPs was obtained based on the slump-flow test analysis of the UHPC, the reinterpretation of the Powers model [38] discussed by Jensen and Hansen [39, 40] was used. This approach allowed estimation of the extra water needed for the UHPC binders to achieve maximum hydration.

According to Eq. 4 [28], the extra water/cement factor (w/c)$_e$ is obtained. This equation is only used for water/cement factors (w/c) lower than 0.36. Equation 5 [28] was used to determine the amount of polymer (SAP/c). The (SAP/c) value is given as a percentage of the mass of the

Table 2 Reference mixture and mixing method

Reference mix					
571.79 C (kg/m^3)	S (kg/m^3)	Sa (kg/m^3)	MGW (kg/m^3)	sp (%)	w/b
	114.36	686.14	343.07	1.5	0.25
Mixing method					
Process	Action			Time (secs)	Speed (rpm)
Paste mixture (Drill with screw attachment for mortar)	Mixing dry binders (cement and silica) + dry SAP			30	2000
	Addition of 50% water + 50% superplasticizer + mixing of all the paste components			180	2000
Mortar (Mortar mixing machine)	Homogenization of aggregates (sand and MGW) + absorption water			60	60
	Paste mixture + aggregates			60	135
	Addition of 50% water + 50% superplasticizer			30	60
	Mixing of components			390	135

C cement, *S* silica, *Sa* sand, *MGW* marble and granite waste, *sp* (%) superplasticizer, and *w/b* water–binder ratio

Table 3 Absorption capacity variations studied

SAP = 0.2%	AC = 40 w/b = 0.33	AC = 45 w/b = 0.34	AC = 50 w/b = 0.35	AC = 60 w/b = 0.37	AC = 65 w/b = 0.38
SAP A	x	x	x	–	–
SAP B	x	–	x	–	x
SAP C	–	x	–	x	x

w/b water–binder ratio and *AC* absorption capacity

binders.

$$(\mathrm{w/c})_{\mathrm{e}} = 0.18*(\mathrm{w/c}) \tag{4}$$

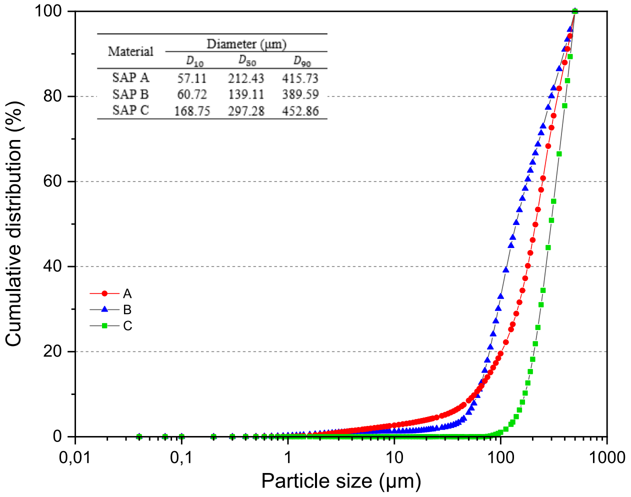
$$(\mathrm{SAP/c}) = \frac{(\mathrm{a/c})\mathrm{e}}{\mathrm{AC}} \tag{5}$$

3 Results and Discussion

3.1 Characterization Results

3.1.1 Granulometric Composition

With respect to the analysis of the granulometric composition, as shown in Fig. 2, SAPs B and C present more uniform curves than SAP A does. As for grain size, B has a curve that is more displaced to the left, as the material has smaller grains. Further to the right, curve C, which has larger diameter particles than the other curves, can be observed. It also presents the values of the effective diameter D_{10}, D_{50}, and D_{90}.The sizes of the SAPs studied can affect the maximum absorption time and the homogeneity of the SAP in concrete.



Material	Diameter (μm)		
	D_{10}	D_{50}	D_{90}
SAP A	57.11	212.43	415.73
SAP B	60.72	139.11	389.59
SAP C	168.75	297.28	452.86

Fig. 2 Particles size distribution of used superabsorbents polymers

3.1.2 X-Ray Fluorescence (XRF) Method

The results of the analysis of the chemical composition of the SAPs via the XRF method are shown in Table 4. Superabsorbent polymers formed by K_2O(potassium oxide) can interact with calcium ions ($Ca2^{+}$) in cementitious materials and other aqueous solutions. These interactions can have a significant effect on the properties of SAPs and the performance of cementitious materials. SO_3 SAPs are sulfonated polymers that contain sulfonate functional groups ($–SO_3^{-}$). These groups are highly polar and attract water molecules [41], giving the SAP a high absorption capacity.

3.1.3 X-Ray Diffraction (XRD)

According to the XRD results, **Error! Reference source not found.**3, the materials do not exhibit regions of crystallinity. Instead, an amorphous halo is present in SAPs A, B, and C, indicating that the polymers do not have an ordered mesh structure. Material A presents an intensity of the amorphous halo that differs from that of the other materials. B and C are very similar, corroborating the XRD results (Fig. 3).

3.1.4 Scanning Electron Microscopy (SEM)

SEM analysis was conducted, as shown in Fig. 4. For SAP A, at a magnification of 100 ×, it is observed in (a) that the grains that make up the material have a granular shape. This

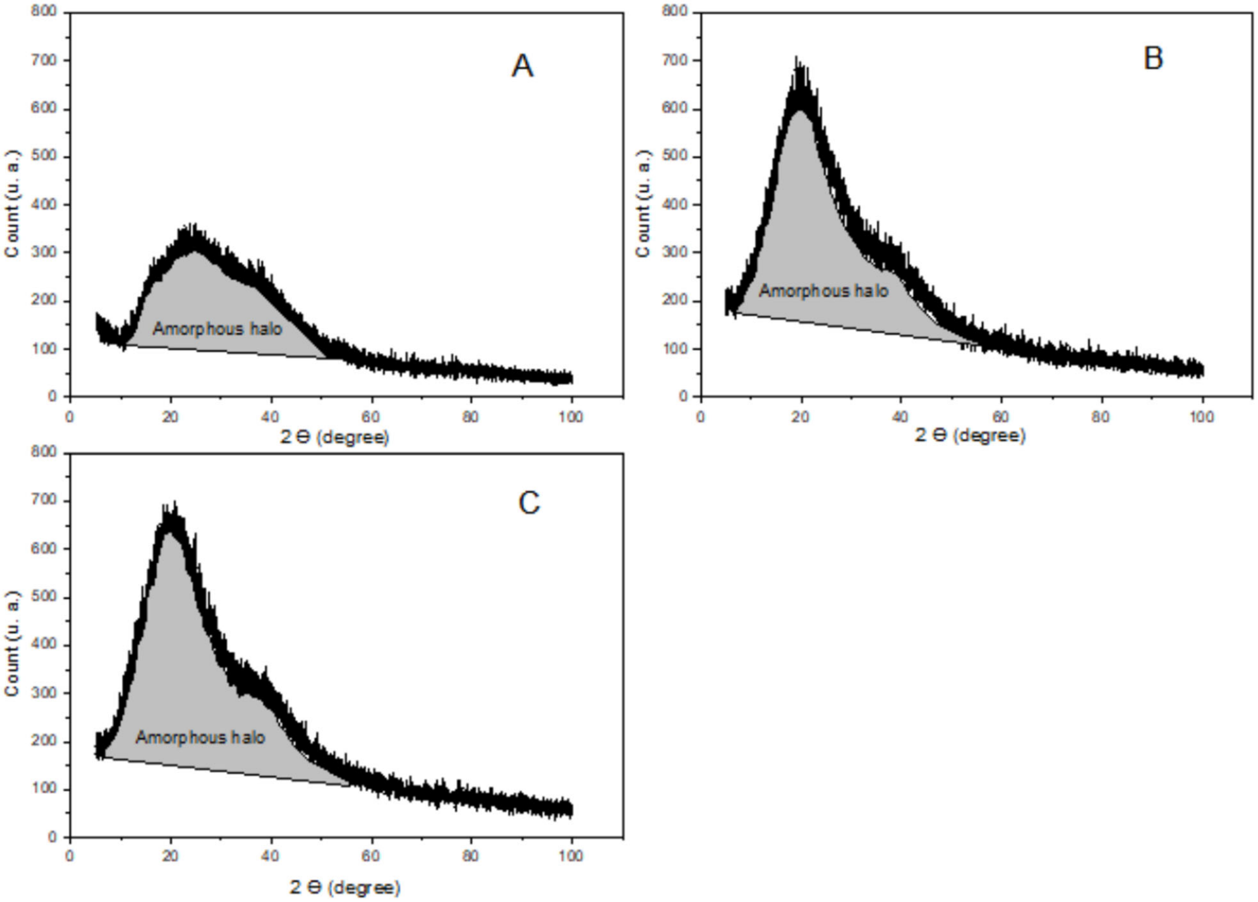


Fig. 3 X-ray diffraction of SAPs A, B, and C indicates amorphous halos

Table 4 SAPs chemical composition (%)

SAP	K_2O	SO_3	Na_2O	P_2O_5	SiO_2
SAP A	99.38	0.62	–	–	–
SAP B	–	88.18	6.82	3.60	1.4
SAP C	–	87.91	6.99	3.56	1.54

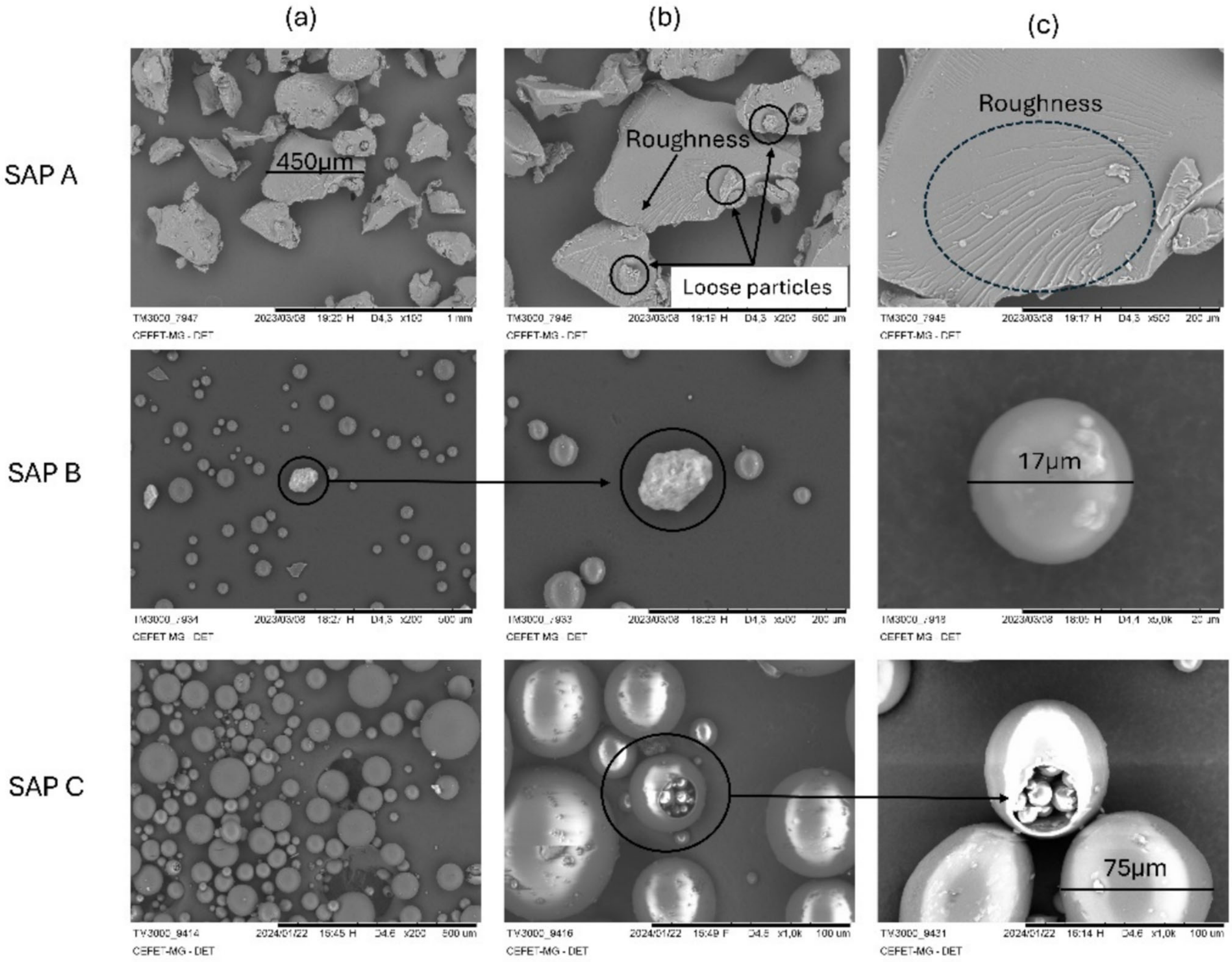


Fig. 4 Scanning electron microscopy SAP A, B, and C

finding indicates that the production method involved gel or bulk polymerization.

SAP particles synthesized through bulk polymerization typically have a smoother surface and are more irregular and varied, while gel polymerization produces rougher and more uniform surface particles. Therefore, it is assumed that the method used was bulk polymerization [42]. In (b), with a magnification factor of 200, it is possible to observe roughness on the surface and the presence of loose particles. By increasing the degree of approximation, in (c), a region with roughness can be observed that may be linked to the production process.

SAP B, as shown in Fig. 4, is mostly formed by spherical particles, as shown in (a). This factor indicates that the polymerization production technique used was reverse suspension. However, what stands out in (b) is the presence of some granular elements, which may come from the production process or some contamination. The spherical structure is further emphasized in image (c), where the surface appears visually uniform.

Finally, for SAP C, as shown in Fig. 4a, the particles are also spherical, which reflects the same production method used in B. This characteristic corroborates the similarity of the XRF and XRD results. In (b), a sphere can be seen in the center of the figure with several SAP particles inside, as highlighted in (c). This phenomenon was not observed in other SAPs and may be linked to some change in the production method used by the manufacturer.

Lastly, it is worth observing the variation in grain size. SAP A (a) has grains of 450 µm, and SAP C (c) has grains of 75 µm. For SAP B (c), grains of 17 µm are observed, which is consistent with the laser granulometry data. The shape and size of the SAP can change the distribution of the curing water in the matrix and the size and distribution of the macropores.

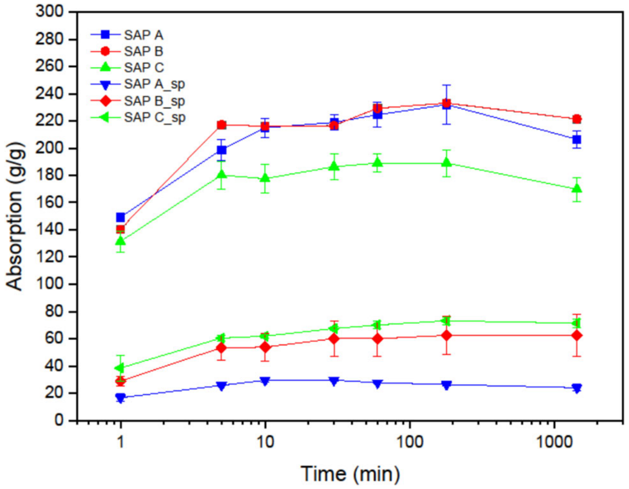


Fig. 5 Development of SAP absorption in tea-bag method

3.2 Absorption Results

3.2.1 Tea-Bag Method

Once the characterization of the SAPs was completed, a tea-bag test was carried out to analyze the absorption capacity of the materials, Fig. 5. The water absorption capacity of polyacrylate-based polymers can reach up to 1000 g/g [29], while other studies report values in the range of 300 g/g [43]. This variation is due to several factors, such as particle size, chemical composition, crosslinking degree, and environmental conditions. Therefore, it is crucial to determine the specific absorption capacity for each polymer under study. Furthermore, research on polymer absorption in aqueous solutions containing superplasticizers is still in its early stages, highlighting the need for further investigations in this area.

SAPs A and B, in the presence of only water, obtained closer absorption, with SAP C having a lower absorption value. This behavior is assumed to be associated with the larger size of the SAP C grains, which provide a smaller specific surface area and can affect the absorption capacity [44].

Notably, for all three materials, there was a reduction in absorption in the 24-h measurement in water, a factor that may be linked to the closure of the tea-bag pores due to the increase in the volume of SAPs [15, 29]. Observations reveal that the absorption values in water are higher than those in water with a superplasticizer (SAP A_sp, SAP B_sp, and SAP C_sp). Figure 6a and b visually exemplifies the reduction in absorption of materials when analyzed in water compared with a water and superplasticizer solution.

This is related to the ionic nature of the action of the superplasticizer in contact with water, which alters the swelling behavior of the SAP due to ionic changes in the mixture. The superplasticizer used has a polymer chain composed of sulfonic groups (-SO_3H), which are strongly hydrophilic and ionizable. An analysis of the solution under study revealed that the excess of ions can influence the absorption capacity of SAPs even at low concentrations [45]. This is because the swelling capacity of SAP depends on the balance between the repulsive forces between the ionized groups and the osmotic force that defines the direction of absorption or release of water.

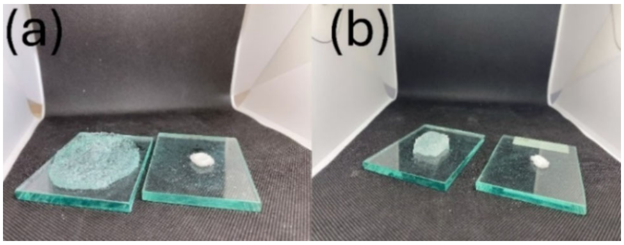


Fig. 6 SAP A (**a**) Tea-bag in water (**b**) Tea-bag in water with superplasticizer

The sulfonic group of the superplasticizer can dissociate in the presence of water, thus forming sulfate anions (SO_3-) and hydrogen cations (H^+). The presence of SO_3- can compete with water molecules for SAP absorption sites, reducing polymer absorption.

Interestingly, SAPs B and C exhibited similar behavior in this second analysis. This similarity may be attributed to the similar chemical compositions observed in the XRF analysis, which could lead to analogous behavior in the presence of ionic solutions [29, 46]. SAP A is mainly formed by K_2O, which contains ionic groups such as potassium (K^+) that can interact with sulfate anions, forming ionic complexes that affect the absorption of the SAP.

SAPs B and C, which are mainly formed by SO_3, are highly polar, thus attracting water molecules, which can favor the absorption of the SAP. Both the SAPs and the superplasticizer have sulfonic groups, which can generate ionic interactions between the SAP polymer chains and the superplasticizer molecules. In this situation, ionic interactions involve like charges, resulting in electrostatic repulsion, which can affect the dispersion and distribution of the components in the aqueous medium.

Therefore, all the SAPs studied had a reduced absorption capacity in the presence of the superplasticizer; however, the distinct ionic interactions due to the components that form SAPs promoted a distinction between absorptions. It can also be discussed how adding the superplasticizer makes the solution more acidic. Knowing that SAPs are composed of polymers with non-ionizable functional groups, pH can change the degree of ionization of these groups, altering the absorption capacity [47].

Table 5 presents the maximum values and deviations for the materials and methods under study, which are obtained after 24 h of testing. Only for the tea-bag method with water,

Table 5 Maximum absorptivity in the tea-bag method

Method	Absorption (g/g)		
	SAP A	SAP B	SAP C
Tea-bag _water	231,91 ± 14,38	233,06 ± 3,04	189,06 ± 9,83
Tea-bag_sp	23,97 ± 1,20	62,66 ± 15,51	73,38 ± 3,16

the maximum was observed in 3 h. It can be observed that absorption does not increase significantly after a few hours, which may be associated with the SAP having reached its maximum absorption capacity. The SAP absorption–desorption process is crucial in determining the effectiveness of internal concrete curing and varies depending on the a/agl ratio and hydration time [15]. The dispersion values observed, especially in SAP B_sp, may increase the risk of inadequate mixture compositions when the polymer is added to cementitious materials due to inaccuracies in the expected absorption values for SAPs [4]. To reduce the risk of deviations during the test, it is important to control laboratory conditions and standardize the operating procedure, avoiding measurement errors, variations in immersion time, incomplete drainage, temperature and humidity fluctuations, heterogeneity in SAP distribution, and evaporation during measurement, among others. In addition to the basic precautions mentioned, other relevant factors have already been studied to analyze discrepancies in absorption results using the tea-bag and filtration methods for cementitious media [48]. It has been observed that convection conditions, that is, the movement of liquid during the test, can influence the absorption rate and efficiency. Additionally, water-to-cement ratios affect the composition of the pore solution, directly impacting absorption capacity. The hydration times during the preparation of the cement paste filtrate also interfere with the total ionic concentration, influencing absorption dynamics. Another relevant factor is the presence of interstitial water, which is not effectively absorbed but remains between the particles, affecting the measurement of absorption capacity.

3.2.2 Filtration Method

The results of the filtration method are shown in Fig. 7. Importantly, the absorption rates obtained via the filtration method, both in water and with the superplasticizer solution, were higher than those obtained using the tea-bag method. This difference can be attributed to the direct contact between the SAP and the liquid in the filtration test, where the SAP samples remain immersed throughout the process. This continuous absorption during filtration increases the apparent absorption value of the material [29].

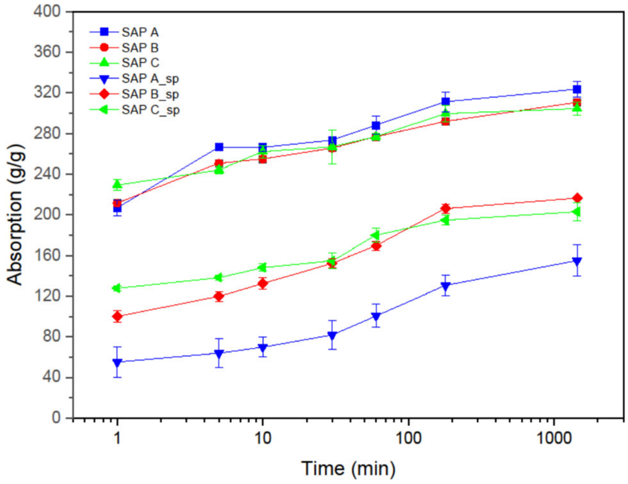


Fig. 7 Development of SAP absorption in filtration method

Table 6 Maximum absorptivity in filtration method

Method	Absorption (g/g)		
	SAP A	SAP B	SAP C
Filtration _water	323,70 ± 7,80	310,67 ± 6,15	304,55 ± 6,67
Filtration_sp	154,92 ± 15,45	216,53 ± 1,52	203,11 ± 9,00

Liquid retention between particles during the process is noteworthy. In the tea-bag method, this could potentially be removed by the surface drying process of the wet tea-bags before weighing [4, 21]. This retention process is more effective on larger diameter grains with greater interparticle space [4].

Analyzing Table 6, compared to Table 5, the difference in water absorption capacity between the filtration and tea-bag methods was approximately 92 g/g for SAP A, 77 g/g for SAP B, and 115 g/g for SAP C, which has larger diameter particles. The same behavior does not occur for the analysis of water with the superplasticizer, where the most significant difference is for SAP B, which may be influenced by the ionicity and viscosity of the solution.

The general comparison between SAP behavior in water and in solution with a superplasticizer mirrored the results of the tea-bag analysis (Fig. 8). There was no reduction in absorption over time, with maximum absorption achieved within 24 h. Notably, SAPs B and C exhibited similar absorption in the presence of the superplasticizer, while the absorption of SAP A was lower, as observed in the tea-bag test. It is important to highlight that, to reduce deviations in the test, one should ensure the uniform distribution of the polymer in the medium, standardize the filtration time, and carefully monitor any leaks or blockages in the filter, as well

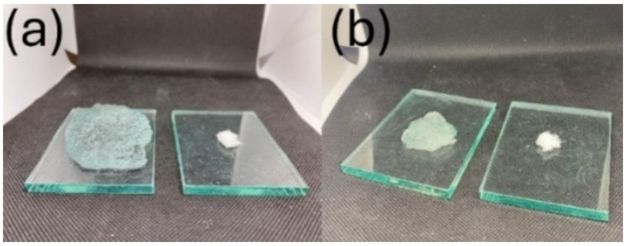


Fig. 8 SAP A (**a**) Water filtration (**b**) Water filtration with superplasticizer

as control significant temperature and humidity variations in the laboratory.

3.2.3 Slump-Flow Test

Finally, Fig. 9 shows the absorption relative to the reference UHPC dosage, without the SAP. In cementitious mixtures, the absorption values are expected to be lower than those obtained in water and water with a superplasticizer. This occurred because the SAP competes with aggregates, cement particles, and other solids for water, and the ionic composition of mixture changes over time. During cement hydration, ions, such as Ca^{2+}, Na^{+}, K^{+}, and OH^{-} [49], form and affect SAP absorption. Ca^{2+} ions can cross-link SAP polymer chains, increasing their rigidity and decreasing their absorption capacity.

SAP A, as with previous methods, showed lower absorption than the other methods did. Potassium (K^{+}) can interact with ions present in the cement paste, such as calcium (Ca^{2+}). This process of ion exchange and the formation of ionic complexes can affect the polymer structure and SAP absorption. Furthermore, this interaction can lead to a reduction in the strength of cementitious materials.

For SAPs B and C, sulfonate groups ($-SO_3-$) are also prone to forming ionic complexes with Ca^{2+}, reducing the SAP absorption capacity. The interaction of sulfonates and Ca^{2+} can lead to the formation of insoluble precipitates, such as calcium sulfate ($CaSO_4$), which can be deposited in the cement matrix. However, sulfonate groups are highly polar and attract water molecules, which may have led to greater absorption of SAPs B and C to SAP A, as observed below.

For SAP A, an initial test absorption of 50 g/g was adopted, and the fluidity evolution curve was far above the reference line, indicating that the SAP could not absorb all the additional water. Reducing to AC = 45 g/g brought the curve closer to the reference value. At AC = 40 g/g, the curve was below the reference line, indicating that the SAP absorbed more water than predicted. Thus, the absorption of SAP A in the reference UHPC was determined to be between 40 and 45 g/g, with AC = 42.5 g/g being adopted.

SAP B started with AC = 40 g/g, and the curve was well below the reference value. Since SAP B presented higher absorption than did SAP A in the other tests, was tried AC = 50 g/g, which was still insufficient. Finally, AC = 65 g/g produced values compatible with the reference curve, indicating its absorption capacity in the cement medium. SAP C started with AC = 45 g/g, based on the similar characteristics of SAP B. As this value was insufficient, AC = 65 g/g was tested, which was too high. Finally, AC = 60 g/g was tested and adjusted adequately to the reference curve. In a previous study with sodium polyacrylate SAP in a cementitious medium containing a polycarboxylate-based superplasticizer, the absorption rate ranged from 27 to 33 g/g[17]. In another study, the superplasticizer varied at 0.1%, 0.3%, and 0.5%. Maximum absorption capacities ranging from 22 to 30 g/g were observed [50]. It is observed that these values fall within the same range, indicating a consistent trend. Variations in absorption can be attributed to the composition and particle size distribution of the SAPs used, the formulation and dosage of the cementitious medium, the mixing method, and other factors that influence the absorption capacity of the polymers.

3.2.4 Dosage Proposal

The reference mixture has w/b = 0.25. Adopting Eq. 4, it is estimated that $(w/c)_e = 0.045$. According to Eq. 5, obtaining the percentage of polymer to be incorporated, in percentage, about the mass of the binders is possible. Adopting the AC values obtained in the slump-flow test, for SAP A, AC = 42.5 g/g, (SAP A/c) = 0.11%. For SAP B, AC = 65 g/g, (SAP B/c) = 0.07% and for SAP C, AC = 60 g/g, (SAP C/c) = 0.08%. It is, therefore, possible to define the SAP incorporations in the laboratory.

It is worth noting that in all three absorption analysis methods, the ambient temperature and relative humidity were controlled. The standard indicates that temperatures between 10 and 30 °C do not significantly affect absorption [21]. However, in real-world mixing project applications, the behavior of SAP may deviate from what was predicted in the laboratory. At lower temperatures, rapid absorption may be delayed, but long-term capacity remains stable. At higher temperatures, evaporation may reduce liquid retention and accelerate polymer degradation [51]. Regarding relative humidity, it is recommended to store SAP in sealed containers, protected from sunlight. In laboratory tests, a relative humidity of 60% is adopted to promote maximum absorption. However, on construction sites with RH < 40%, evaporation occurs more rapidly, potentially compromising SAP retention and the internal curing of concrete [52]. Therefore, it is important to analyze the expected field conditions to improve the assessment of dosage parameters.

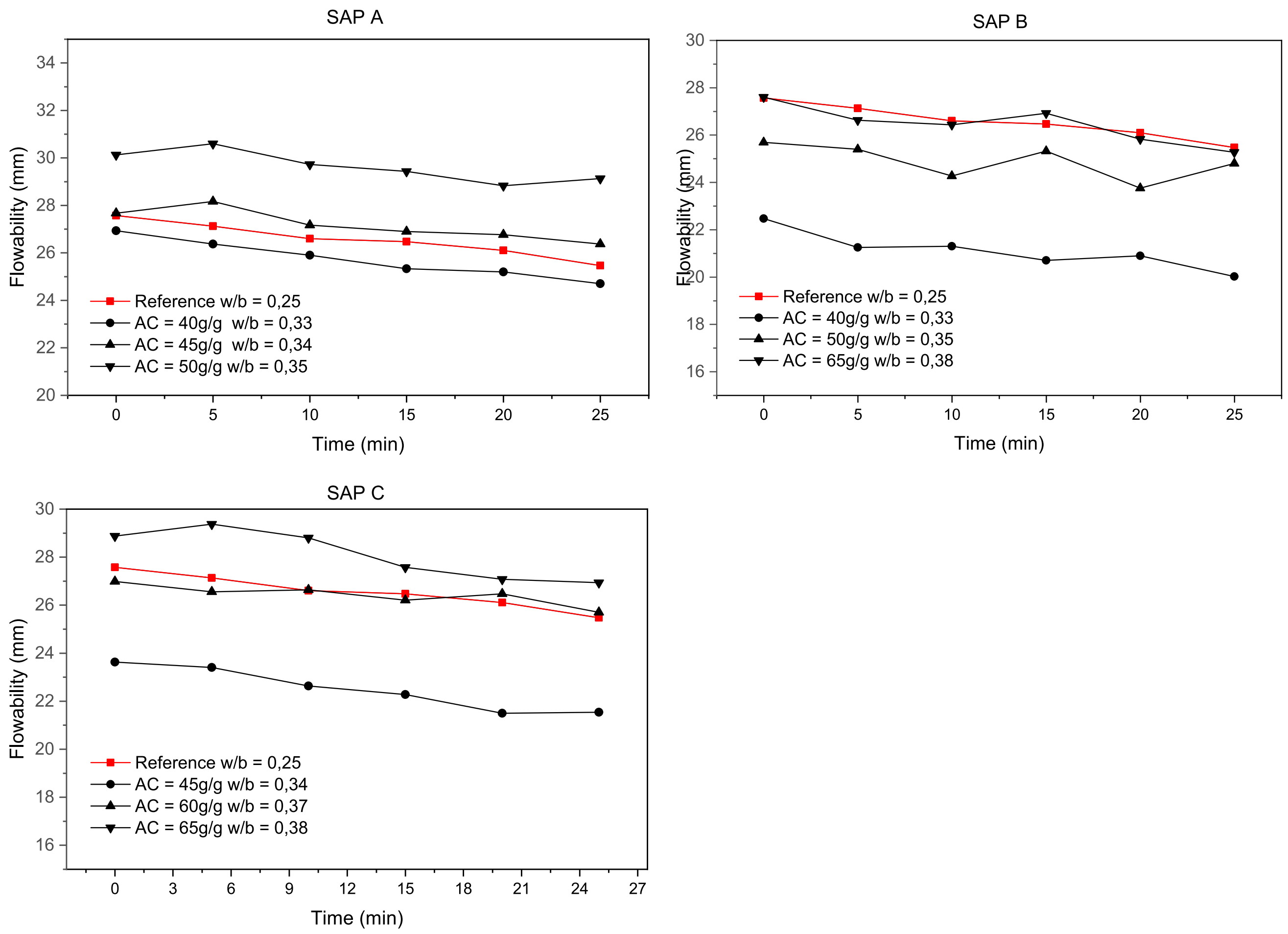


Fig. 9 Absorption behavior of SAPs A, B, and C with different w/b factors in comparison with the reference

The reduction in workability due to the addition of SAP should also be considered in the dosage and application process, so as not to affect field execution. Adjustments to the water/binder ratio and the dosage of superplasticizer should consider the desired minimum workability [39, 53], considering the SAP absorption and desorption predicted for field conditions [15].

In that regard, recent studies have investigated mixing protocols of incorporating SAPs into concrete at laboratory and plant scale and compared the results of absorption and shrinkage mitigation between laboratory conditions and real conditions. In [47, 49], the authors presented a method for adding the SAPs to the conveyor belt of a concrete plant, within sealed water soluble bags, being then delivered to the mixer together with all other dry materials. The authors found that the absorption capacity defined in laboratory combining absorption tests with slump-flow tests in fresh concrete mixtures was compatible with the one observed on the field. The same was valid for the effects of the SAPs on the strength and shrinkage of the real structure and the specimens produced and tested under laboratory conditions [54] [27].So adjustments to the mixing sequence and adaptations in placement and compaction methods can also be useful tools in the successful execution of concretes with SAP addition in large-scale applications [55–57].

4 Conclusions and Suggestions for Futures Studies

In the tea-bag analysis, higher absorption was observed in the medium with only water compared to the solution with superplasticizer for all the SAPs studied. This is because the superplasticizer is composed of a polymer chain with sulfonic groups (-SO$_3$H). In the presence of water, the sulfonic group of the superplasticizer can dissociate, forming sulfate anions (SO$_3$-) and hydrogen cations (H$^+$). The presence of SO$_3$- can compete with water molecules for SAP absorption sites, reducing polymer absorption. This behavior was also noted in the filtration test. However, in the filtration method, absorptions were higher due to the direct contact of the SAP with the liquid, and during the filtration process, the SAP samples remained immersed in the test fluid.

The chemical composition of the SAPs was a determining factor for their absorption capacity. In the presence of the superplasticizer, SAP A presented lower absorption results than other samples did. This is due to its composition, which is composed primarily of K_2O, which contains ionic groups such as potassium (K^+). These ions can interact with sulfate anions, forming ionic complexes that affect absorption. Conversely, SAPs B and C, composed of SO_3, are less impacted because they are highly polar and attract water molecules, which can favor SAP absorption. In the slump-flow test, this behavior was repeated, but it is worth noting that all absorptions were reduced due to the presence of Ca^{2+} ions. These ions can form cross-links between SAP polymer chains, increasing the rigidity of the polymer and decreasing absorption capacity. Thus, the absorption kinetics is closely linked to the ionic composition of the SAP and the absorbed solution. This underscores the need for further studies on absorption prediction, allowing a more precise definition of the optimal dosage for each SAP and ensuring a more efficient use of additional water in the system.

It is also important to highlight the need to understand the influence of the desorption process of SAPs on dosage processes. Study [15] addresses the processes that govern internal curing in concrete with SAPs. The desorption process occurs in two phases: osmotic stage and moisture stage. In the first, water is released quickly, driven by osmotic pressure, releasing the water freely into the cement paste. Then, in the moisture stage, desorption continues at a slower pace, with the remaining water being released under the influence of the moisture gradient. Since this water is transported more slowly, it is considered vapor, as the cement paste is already hardened. These processes affect the effectiveness of internal curing and, consequently, the properties of the concrete. Therefore, in the future studies, this analysis would be highly valuable to complement the investigations.

Other suggestions for future studies may also investigate optimized combinations of SAP-plasticizer dosages, as well as potential modifications in SAP formulations, to ensure that their absorption capacity is less affected by the presence of superplasticizers. Furthermore, it is crucial to analyze the long-term behavior of SAPs in different cementitious environments, particularly regarding durability in severe conditions, while also considering their economic feasibility on a large scale.

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Data Availability No data are associated with this article.

Declarations

Conflict of interest The authors declare that they have no conflict of interest.

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