



HAL
open science

Effect of Excessive Bleeding on the Properties of Cement Mortar

Mimoune Abadassi, Youssef El Bitouri, Nathalie Azéma, Eric Garcia-Diaz

► **To cite this version:**

Mimoune Abadassi, Youssef El Bitouri, Nathalie Azéma, Eric Garcia-Diaz. Effect of Excessive Bleeding on the Properties of Cement Mortar. *Construction Materials*, 2023, 3 (2), pp.164-179. 10.3390/constrmater3020011 . hal-04081168

HAL Id: hal-04081168

<https://imt-mines-ales.hal.science/hal-04081168>

Submitted on 25 Apr 2023

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.

Article

Effect of Excessive Bleeding on the Properties of Cement Mortar

Mimoune Abadassi, Youssef El Bitouri *, Nathalie Azéma and Eric Garcia-DiazLMGC, IMT Mines Alès, University of Montpellier, CNRS, F-30100 Alès, France;
mimoune.abadassi@mines-ales.fr (M.A.)

* Correspondence: youssef.elbitouri@mines-ales.fr

Abstract: The bleeding of cementitious materials corresponds to the settlement of the granular skeleton accompanied by the accumulation of water at the surface (bleed water). Part of this water (internal bleeding) remains trapped under the aggregates (sand or gravel) or the reinforcements. The excess of this trapped water can weaken the bond between the cementitious matrix and the aggregates (or the reinforcements), which affects the mechanical performance and durability of the material. This study aims to investigate the effect of excessive bleeding induced by superplasticizer on the properties of mortars. For this, a study of cement paste bleeding in the presence of superplasticizer was carried out. The effects of the water-to-cement ratio (w/c) and the superplasticizer (SP) dosage on this bleeding have been characterized. Then, the influence of the proportion of sand on the bleeding was examined by varying the sand/cement (s/c) ratio. The water trapped by sand (internal bleeding) was determined by the difference between the external bleeding on the cement paste and the external bleeding on the corresponding mortar. The results show that the internal bleeding increases with the s/c ratio and the SP dosage, until it reaches a plateau. The effect of the internal bleeding on the mechanical properties and the porosity of the mortar were then examined. Microscopic observations were made to assess the quality of the paste/sand bond. The results showed that the internal bleeding causes a degradation of the paste/sand bond (a more porous bond), resulting in a decrease in the mechanical strength (by 30% for compressive strength and 25% for flexural strength) of the hardened mortar.

Keywords: bleeding; cement paste; mortar; superplasticizer

Citation: Abadassi, M.; El Bitouri, Y.; Azéma, N.; Garcia-Diaz, E. Effect of Excessive Bleeding on the Properties of Cement Mortar. *Constr. Mater.* **2023**, *3*, 164–179. <https://doi.org/10.3390/constrmater3020011>

Received: 2 March 2023

Revised: 17 April 2023

Accepted: 18 April 2023

Published: 21 April 2023



Copyright: © 2023 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (<https://creativecommons.org/licenses/by/4.0/>).

1. Introduction

Bleeding of cementitious materials corresponds to the upward displacement of a water film and the settlement of solid skeleton under gravitational forces, resulting in water accumulating at the top [1–3]. At the cement paste scale, bleeding occurs due to the difference in density between the solid skeleton and water. It is affected mainly by the particles' shape, the size, and the specific surface [3,4].

Several studies focused on the description of bleeding in cementitious materials. Thus, the first works of Powers described bleeding as a particular case of sedimentation [3,4]. Jossierand et al. [2] described bleeding as an ageing consolidation process. Recently, Mas-soussi et al. [5] showed that bleeding is a process that cannot always be described by homogeneous consolidation theory and is rather of a heterogeneous nature. The authors showed the existence of an induction period. This induction period is followed by an acceleration period during which extraction channels can form, leading to a local increase in permeability and, therefore, to an acceleration in bleeding [5]. These extraction channels can be the consequence of the presence of defaults (e.g., air bubbles) and/or admixtures, such as superplasticizer.

Furthermore, the development of modern concretes requires the use of specific admixtures, such as superplasticizers. Since superplasticizers make it possible to limit the agglomeration of cement particles [6,7], they are widely used in modern concretes in order

to improve workability without adding additional water [8,9]. However, this type of admixture could induce some problems. In fact, the addition of superplasticizer induces a de-flocculation effect, leading to the release of water trapped among agglomerated cement particles [10]. The release of this trapped water increases the bleeding capacity and reduces the yield stress [10]. It is worth noting that the high dosage of superplasticizer can induce an excessive bleeding that can affect the properties of the cementitious material. When bleeding is not excessive, it limits the effects of drying shrinkage. In addition, excessive bleeding is usually accompanied by segregation and, therefore, alters the homogeneity of the cementitious material.

Furthermore, part of the bleed water can be trapped among flocculated cement particles or under the aggregates or reinforcement (in the case of reinforced concrete), often referred to as internal bleeding [11]. This trapped water can weaken the adhesion (matrix/inclusions) and increase the porosity, which affects the mechanical properties and durability of the cementitious material [1,12,13].

Although many works examined bleeding of cementitious materials [1,2,5,10,14–17], only a few focused on the effect of bleeding on the properties of hardened cementitious materials [18–21]. According to Han and Wang [21], the excessive bleeding makes the concrete more porous, weakens the bond between the cement matrix and the subsurface of aggregates, and induces a non-uniformity of strength. Han and Wang [21] also report that bleeding leads to a high w/c ratio in the upper region of a fresh cement paste, which increases the porosity of the hardened cement paste in this region. The authors specify, by carrying out tensile tests by splitting, that bleeding alters the mechanical properties of cement paste. This alteration would be linked to the hydration products and the change of the microstructure after bleeding.

In the literature, there is a lack of data on the characterization of internal bleeding, the influence of SP on bleeding (excessive bleeding) [10,14,17], and the consequences of excessive bleeding on the properties of the hardened cementitious material. Thus, this study aims to examine the influence of excessive bleeding induced by the addition of polycarboxylate-based superplasticizer (PCE) on the properties of cement mortar. The effect of the proportion of sand on the bleeding is examined by varying the sand/cement ratio (s/c). The effects of trapped water (internal bleeding) on the porosity, on the mechanical strengths of mortars, and on the quality of the paste/sand bond are examined.

2. Materials and Methods

In this work, an ordinary Portland cement CEM I 52.5 R provided by LafargeHolcim (Port-la-Nouvelle) was used. This cement has a low sulphide content (<0.2%) and is compliant with the European standard (NF-EN-197-1) [22]. Its density measured using a pycnometer was 3.12 g/cm³. Its Blaine specific surface was 0.44 (m²/g). The particle size distribution, determined using a laser granulometer (Beckman Coulter LS 13 320, Pasadena, CA, USA), ranged from 0.04 to 84 µm, with an average diameter of 12 µm (Figure 1). The chemical composition of the cement is presented in Table A1 (Appendix A).

The superplasticizer (SP) used was a commercial polycarboxylate (PCE) supplied by Masters Builders company with a dry extract of 19.5% and a density of 1.05 g/cm³.

Standardized sand (0/4 mm) with a density of 2.65 g/cm³ was used. Its absorption coefficient was 1.85%, determined in accordance with NF EN 1097-6 standard [23].

The mortars were prepared with different sand/cement (s/c) ratios (from 0.3 to 2.4) and water/cement ratios (w/c) (0.45 and 0.6) in a standardized mortar mixer according to the following sequence: 30 s (s) of mixing the cement (560 g) and water (deionized) at low speed (first mix); then, adding sand, and stopping mixing for 90 s; then, scraping and homogenization for 30 s; and finally, 60 s of mixing at high speed (final mixing). For mortars containing superplasticizer, the same mixing procedure was applied, except that 90% of total water was added during the first mixing, and the rest of the water mixed with the superplasticizer (SP) was added during the last mixing phase (delayed addition).

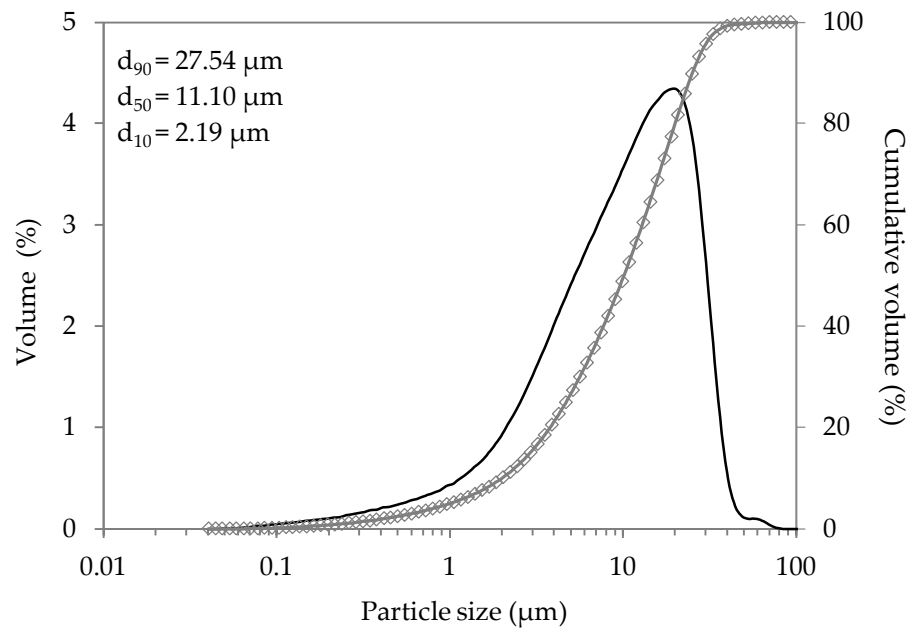


Figure 1. Particle size distribution of cement (Line: Volume (%); Line with diamonds: Cumulative volume (%)).

There are several methods for quantifying the external bleeding of cementitious materials [5,10,14,24,25]. In this study, the external bleeding was evaluated by a conventional method adapted from ASTM standards [24–27] and by turbidimetry measurements using TurbiLab from Formulation (Figure 2). The bleeding apparatus (conventional method) consisted of a cylindrical and transparent column (10 × 35 cm) fitted with a cover to limit evaporation (Figure 2). These columns were wide enough to limit the wall effects. The column was placed vertically on a horizontal plane in a room regulated at 20 ± 2 °C and away from any vibrations. This conventional method is suitable for cement paste and mortar. The turbidimetry method was performed according to the method described by El Bitouri and Azéma [10]. This method is suitable only for cement paste and allows turbid bleeding to be accurately determined. The bleeding capacity was determined as follows:

$$\text{Bleeding capacity}(\%) = \left(H_e / H_i \right) \times 100 \tag{1}$$

where H_i is the initial height of the sample, and H_e is the thickness of bleed water. This bleeding capacity was determined after a sufficiently long time to have a stabilization of the bleeding. This stabilization of external bleeding depends on several factors, in particular, the w/c ratio and the SP dosage. The stabilization occurs after a few minutes or even a few hours for cement paste (or mortar) with low w/c ratio and/or low SP dosage, whereas for cement paste (or mortar) with high SP dosage, the stabilization of bleeding occurs after several hours due to the retarding effect.

A preliminary study on the effect of vibration before turbiscan measurements was carried out. It appears that the vibration increases the bleeding capacity.

Figure 3 presents a comparison between bleeding capacity determined by conventional and turbidimetry (without vibration) methods. It can be noted that the obtained results are almost comparable, except for high dosage of SP. In fact, as reported by El Bitouri and Azéma [10], in the presence of superplasticizer, the bleed water becomes turbid due to the finest particles dispersion, and conventional methods underestimate the bleeding thickness.

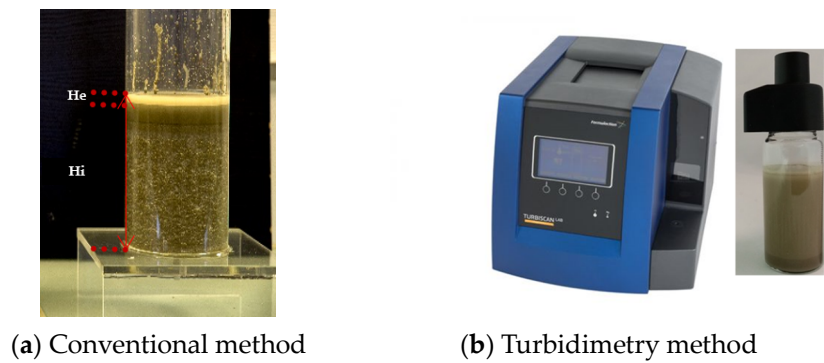


Figure 2. The apparatus used for the bleeding measurements.

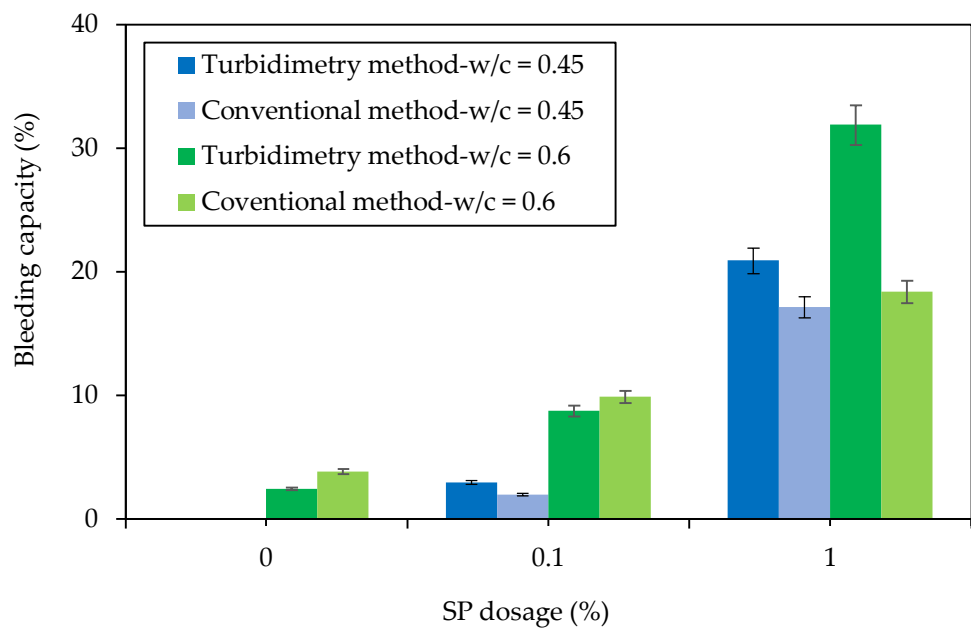


Figure 3. Comparison between bleeding capacity determined by conventional and turbidimetry (without vibration) methods.

In order to assess the effect of bleeding on the quality of the cement paste/sand bond in hardened mortars, polished sections were prepared for observation under an optical microscope (Leica Laborlux 12 POL S equipped with Sony digital camera single CDD 1600 × 1200 pixels).

Yield stress measurements were carried out using a rotational rheometer AR2000Ex from TA Instruments equipped with Vane geometry. The internal diameter was 28 mm, and the outer cup diameter was 30 mm. The resulting gap was 1 mm. Calibration of shear stress and shear rate constants were carried out using the methodology based on the Couette analogy described by Ait-Kadi et al. [28]. The static yield stress was measured according to the stress growth procedure [29,30]. This procedure consisted of the application of a very low shear rate (0.005 s^{-1}) maintained constant during 180 s. A pre-shear phase, consisting of the application of a shear rate of 100 s^{-1} during 30 s followed by a resting time of 2 min, was performed before the stress growth procedure. The static yield stress corresponds to the peak in the shear stress–time (or strain) curve (Figure 4).

The amount of consumed SP was determined by the depletion method [31] using total organic carbon measurements. Measurements were performed using Vario TOC cube from Elementar.

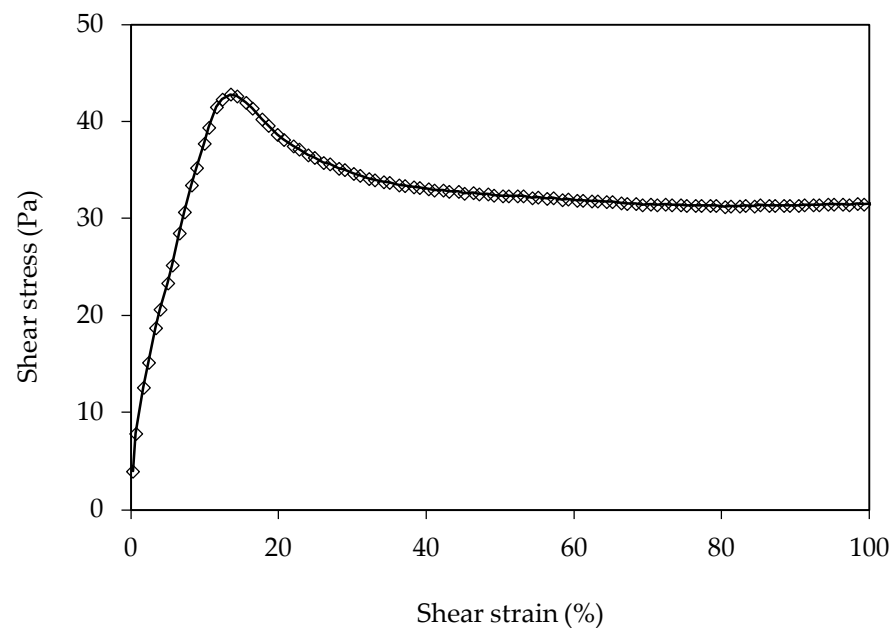


Figure 4. Example of the yield stress measurement (cement paste with $w/c = 0.45$ and 0% SP).

Mercury intrusion porosimetry was used to determine porosity of the cement mortars. The measurements were carried out on samples of $1 \times 1 \times 1 \text{ cm}^3$ obtained by coring the test specimens. It should be noted that the porosity determined by mercury intrusion made it possible to characterize only part of the microstructure of the capillary pores with diameters of approximately $0.5 \mu\text{m}$ and of the pores of the hydrates with entry diameters of approximately 10 nm .

Compressive and flexural strength were determined after 28 days in accordance with NF EN 196-1 standard [32]. Indeed, for each mortar, three specimens were made and placed in prismatic polystyrene molds of dimensions $14 \times 14 \times 16 \text{ cm}^3$. This type of mold allowed the limiting of the loss of water. These specimens were demolded after 24 h and kept in water in a temperature-controlled room ($20 \pm 2 \text{ }^\circ\text{C}$).

All the tests were performed in triplicate.

3. Results and Discussion

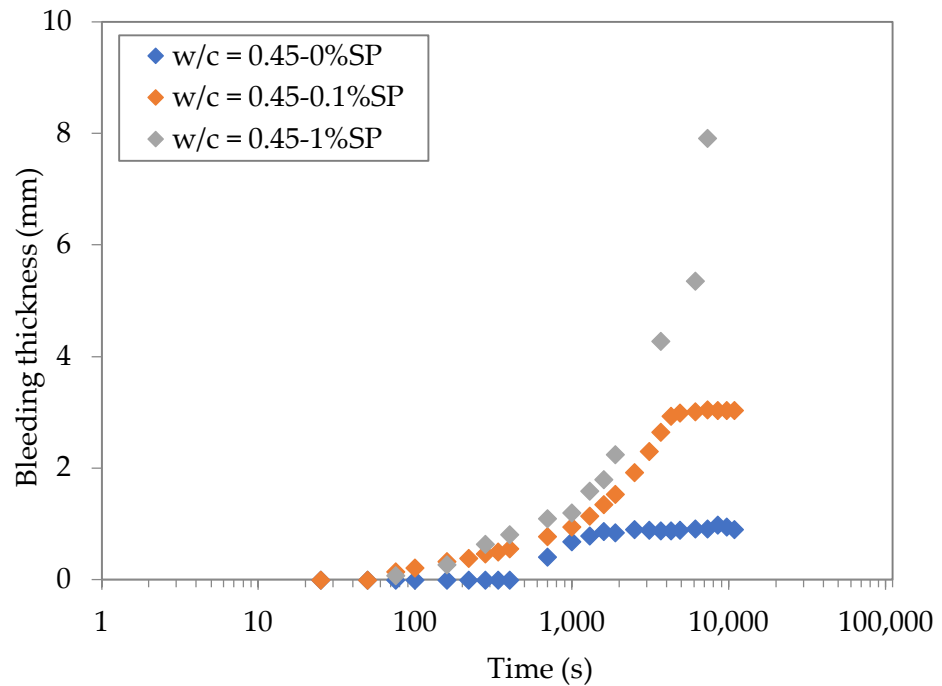
3.1. Effect of SP on Cement Paste Bleeding

The evolution of cement paste bleeding as a function of time is shown in Figure 5. It can be observed that bleeding of cement paste takes place in at least three stages. After an “induction” period, bleeding thickness increases quickly and reaches a plateau. It can be noted that bleeding stabilizes after 30 min for cement pastes without SP and in 2 to 3 h for pastes with 0.1% SP, while it continues to evolve for cement paste with 1% SP beyond 24 h and sometimes even up to 48 h. The evolution of bleeding of non-admixed cement paste is similar to that obtained by Massoussi et al. [5].

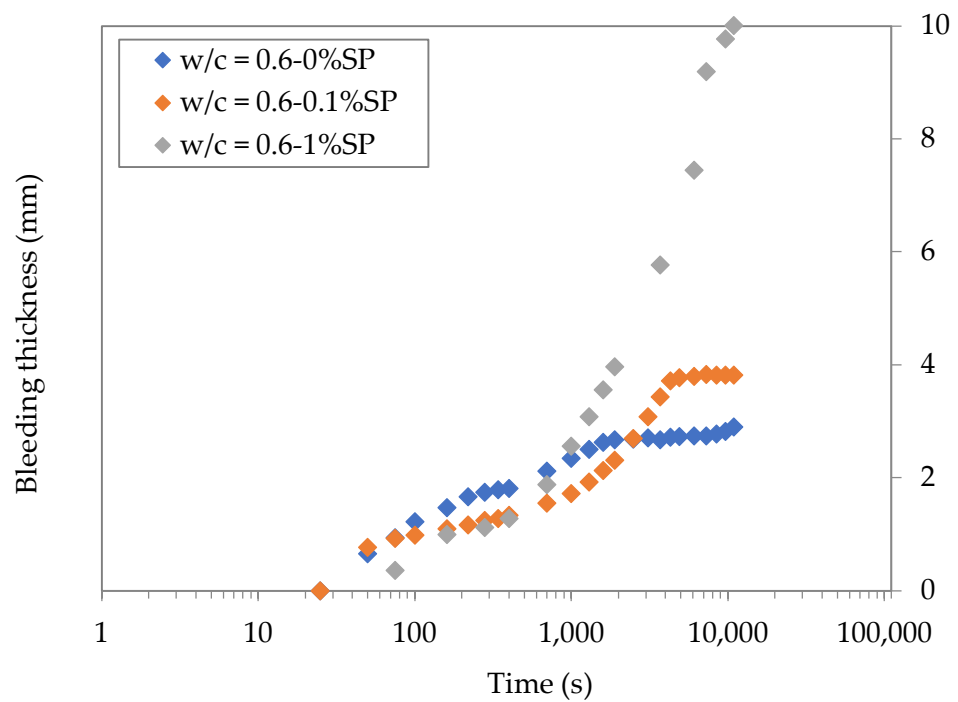
Bleeding capacity corresponds to the bleed water at the plateau (measured when the bleeding is stabilized). As shown in Figure 5, the time required for the stabilization of bleeding depends on the SP dosage.

Figure 6 shows the effect of w/c ratio and SP dosage on the bleeding capacity of cement paste. It can be noted that the bleeding capacity increases with the w/c ratio. This increase is related to the modification of the solid volume fraction, which affects the granular skeleton (permeability) and the drainage of water towards the upper surface. In addition, for all cement pastes (with w/c ratios of 0.45 and 0.6), the bleeding capacity also increases with the SP dosage. This increase can be explained by the modification of the granular skeleton by de-flocculation of the cement particles and the release of the water trapped among the agglomerates [10]. In fact, the addition of SP reduces the magnitude

of attractive colloidal forces [15,17,33]. Moreover, if the increase in the w/c ratio does not induce a significant retarding effect on the hydration of the cement, the increase in SP dosage induces a retarding effect that decreases the structural build-up during the dormant period [34,35]. Thus, cement paste with SP has more time to bleed than cement paste without SP due to this retarding effect.



(a)



(b)

Figure 5. Example of the evolution of cement paste bleeding as a function of time determined by turbidimetry measurements: (a) cement paste with w/c ratio of 0.45 and (b) cement paste with w/c ratio of 0.6 (with vibration).

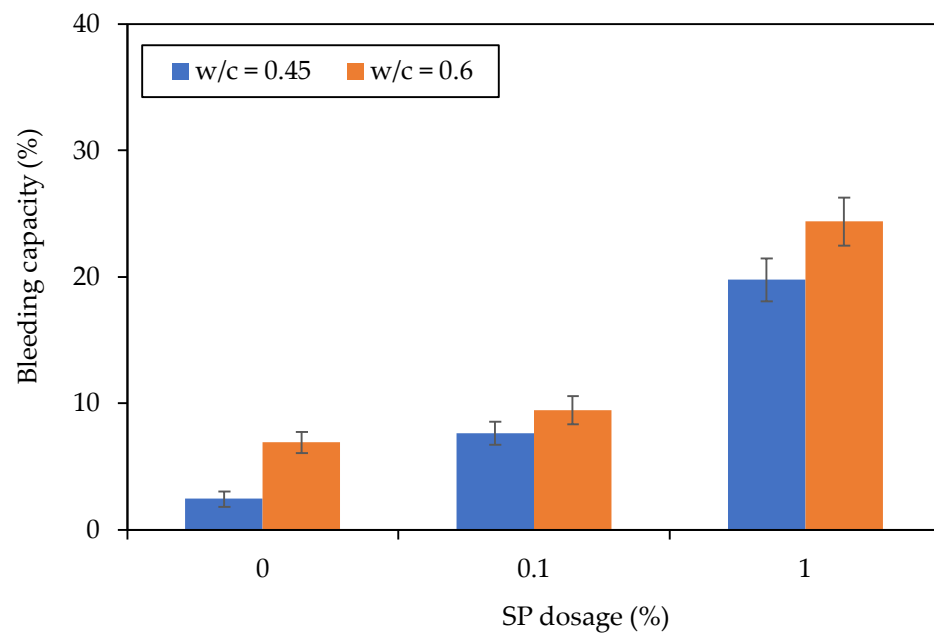


Figure 6. Effect of SP dosage and w/c ratio on the bleeding capacity determined by Turbiscan after vibration.

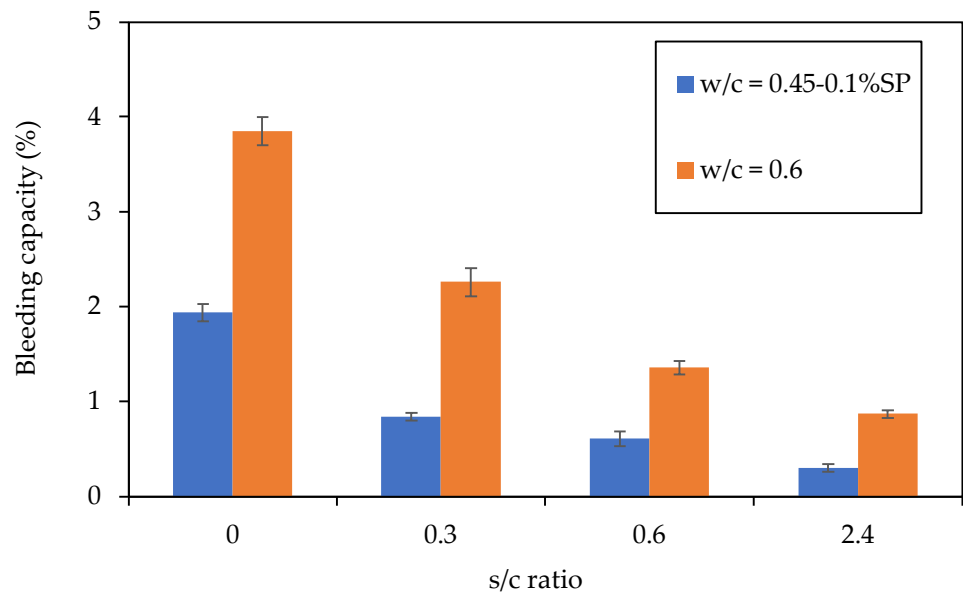
3.2. Effect of sand on Bleeding Capacity

In order to investigate the effect of sand inclusions on bleeding, different mortars were formulated by varying the sand-to-cement ratio (s/c) from 0.3 to 2.4 with the same volume of cement paste. The evolution of the bleeding capacity as a function of sand content and SP dosage is presented in Figure 7 and the data are available in Tables A2–A4 in the Appendix A. The results show that the presence of inclusions leads to a decrease in the bleeding capacity. This decrease could be explained by the fact that the inclusions constitute obstacles for the upward displacement of water. Consequently, part of the bleeding water would be retained inside the mortar.

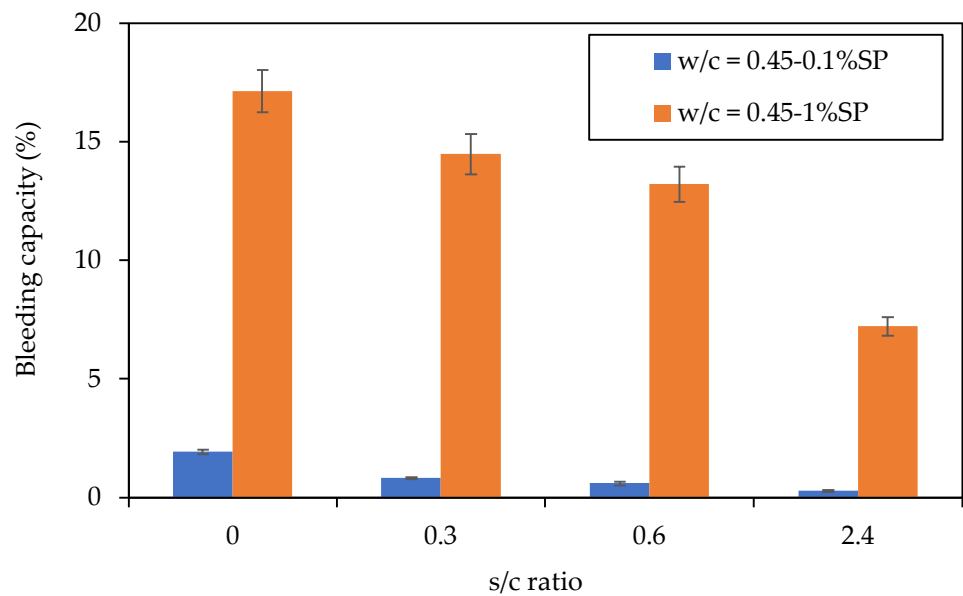
Figure 7b shows the variation of the bleeding capacity as a function of the s/c ratio and SP dosage. It can be noted that the external bleeding capacity increases with SP dosage. This increase would be related to the enhanced dispersion of the cement particles. It is important to emphasize that from a certain SP dosage, the mortar can display significant segregation, since the suspending phase, which is the cement paste, is no longer capable of maintaining sand in suspension. This is due to the decrease in viscosity and yield stress of the cement paste due to the presence of SP.

3.3. Evaluation of Internal Bleeding

The evaluation of the trapped water, called internal bleeding, is complex and is rarely reported in the scientific literature. An indirect approach based on modeling has been proposed by Yim et al. [16]. The authors used a consolidation model associated with experimental measurements of external bleeding. Internal bleeding was determined indirectly by validating the modeling approach on external bleeding results. However, this approach has several limitations and requires assumptions about the model parameters. Moreover, the effect of SP has not been examined. In this study, a simplified approach to estimate the internal water volume is used. It is assumed that the total water trapped in mortar is the sum of the water trapped among flocculated cement particles and the water trapped by sand.



(a)



(b)

Figure 7. Effect of s/c ratio on the bleeding capacity (a) w/c = 0.45–0.1%SP and w/c = 0.6 (b) effect of SP dosage.

At the cement paste scale, the trapped water among flocculated cement particles can be assessed by the difference between the bleed water in cement paste without SP (flocculated state) and the bleed water in cement paste with 1% SP (deflocculated state).

At the cement mortar scale, the water trapped by sand is the difference between the bleed water in the corresponding cement paste and the bleed water in the mortar.

The effect of sand/cement (s/c) ratio on the trapped water is examined. Figure 8 presents the obtained results. It can be noted that the water trapped by sand increases with s/c ratio until reaching a plateau when the proportion of sand is greater than 60% of cement (wt%). In addition, the water trapped by sand increases with w/c ratio and SP dosage, suggesting that segregation of sand may play a role in trapping the water. In fact, from a certain SP dosage or a certain w/c ratio, cement paste is not able to maintain sand in suspension. For example, cement paste with a w/c ratio of 0.6 and 0.1% SP does not

display any yield stress. This cement paste is, therefore, more sensitive to the segregation of sand. This segregation could participate to trap more water that is expected to bleed.

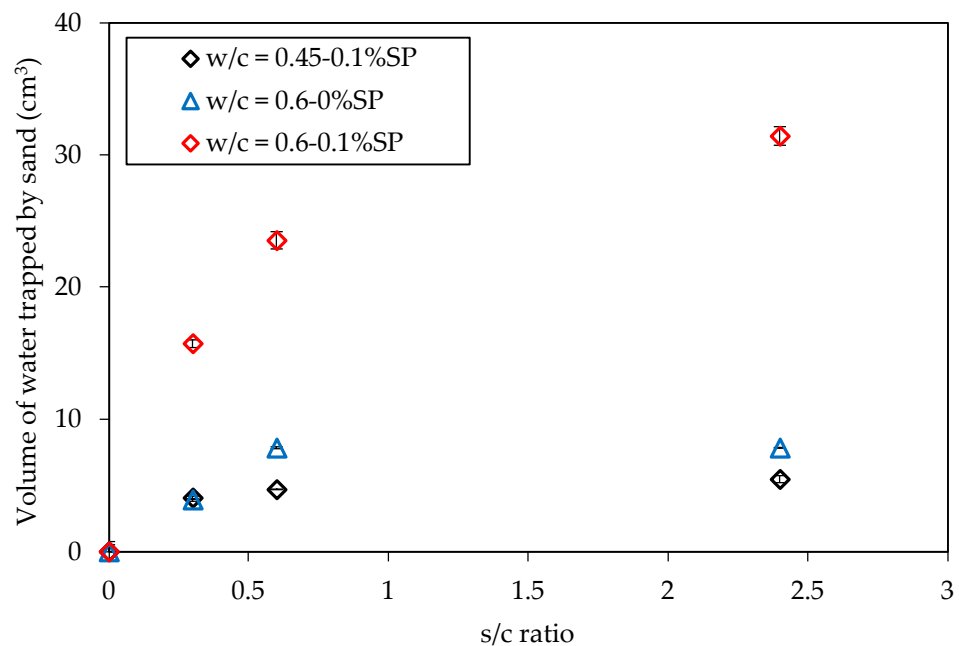
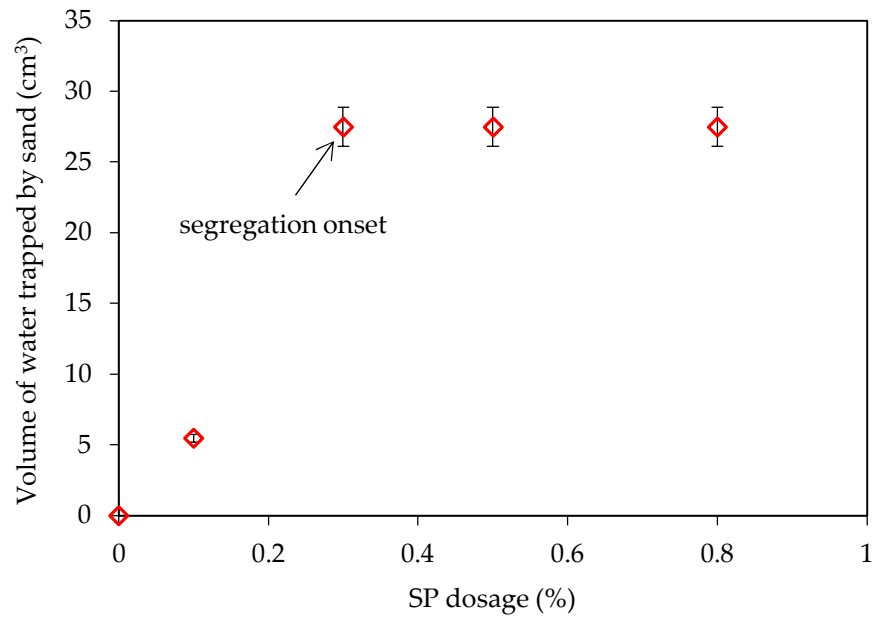


Figure 8. Effect of sand/cement ratio (s/c) on the water trapped by sand.

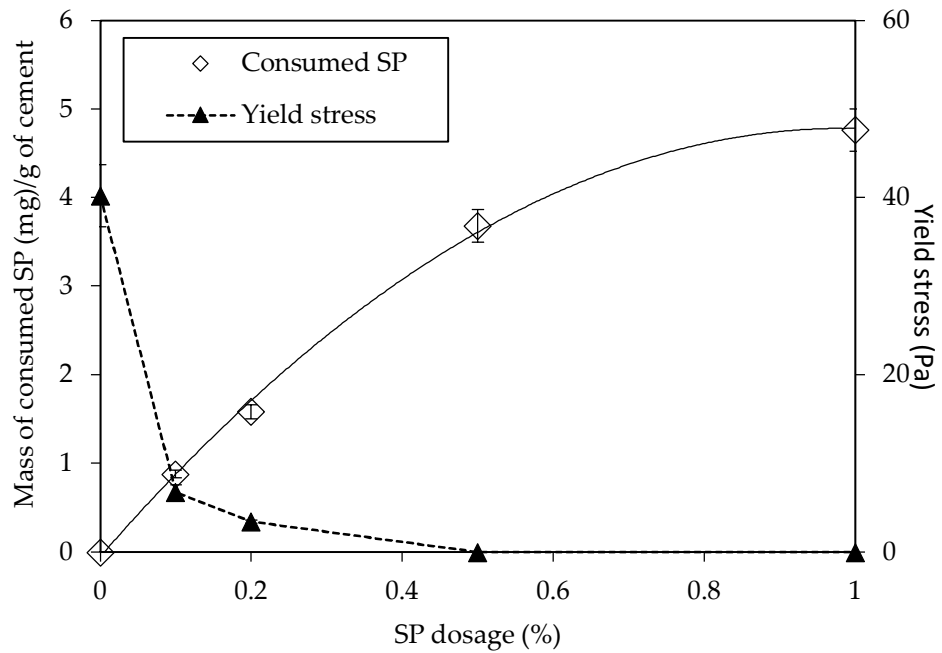
In order to more deeply illustrate the effect of segregation, Figure 9a shows the water trapped by sand as a function of SP dosage. As shown in this figure, the water trapped by sand increases with SP dosage. It is worth noting that the water trapped by sand remains almost constant from a certain SP dosage (0.3%). At this dosage, the segregation onset is observed. In fact, the segregation onset means that the static yield stress of cement paste is not high enough to maintain sand in suspension. As shown in Figure 9b (The yield stress and TOC data are available in Table A4 in the Appendix A), the dosage of 1% SP is a high dosage at the saturation plateau (determined via measurements of total organic carbon. At this dosage, cement paste does not display any yield stress. The static yield stress of cement paste with 0.3% SP is very low, indicating that the cement paste is not able to hold sand grains in suspension. Thus, when segregation occurs, the grains of sand will trap the same volume of water.

3.4. Effect of Internal Bleeding

The characterization of the mechanical properties allowed the analysis of the effect of water trapped by sand on the compressive and flexural strengths of the mortar at 28 days. The mortars were made with s/c of 2.4 and w/c of 0.45 and 0.6, for which the volumes of water trapped by sand were determined. Figure 10 shows the compressive and flexural strengths as a function of the proportion of the volume of water trapped by sand in relation to the total volume of water (internal bleeding) and the related data are summarized in Table A5 in the Appendix A. The results show a decrease in compressive and flexural strength with the internal bleeding. Compressive strength decreases by 30%, while the flexural strength decreases by 25%. This decrease would be related to the increase of the porosity of the cement paste/sand bond. This porosity degrades the bond and weakens the mechanical resistance of the mortar.



(a)



(b)

Figure 9. (a) Effect of SP dosage on the water trapped by sand for mortar with $w/c = 0.45$ and $s/c = 2.4$. (b) Amount of consumed SP and yield stress of cement paste as a function of SP dosage.

The porosity determined by mercury intrusion makes it possible to characterize only part of the microstructure of capillary pores with diameters of approximately $0.5 \mu\text{m}$ and of hydrate pores with inlet diameters of approximately 10 nm [36]. The pore size distribution of mortars after 28 days is presented in Figure 11. Two categories of pores are identified: capillary porosity and hydrate pores. The influence of the w/c ratio on capillary porosity is well documented in the scientific literature [36]. However, regarding the porosity of hydrates, Powers and Brownyard [37] assumed it to be independent of w/c ratio. However, this hypothesis has been discussed by other authors [38]. The results obtained do not show

a significant effect of the w/c ratio or of the SP dosage on the porosity of the hydrates (<10 nm). On the other hand, the capillary porosity increases with the w/c ratio. The results obtained do not allow a conclusion, for the moment, on the effect of SP on this porosity. It seems that SP decreases capillary porosity [39,40], but further analyzes are underway to confirm these findings. It should also be noted that the porosity of the cement paste alone is necessary to be able to draw conclusions on the effect of sand inclusions and, in particular, the trapped internal water.

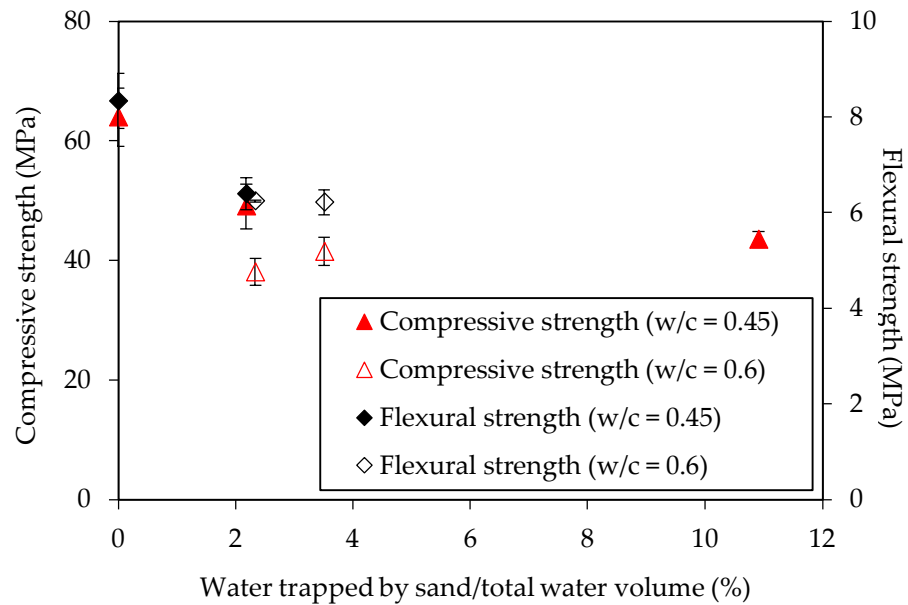


Figure 10. Effect of water trapped by sand on mechanical strength.

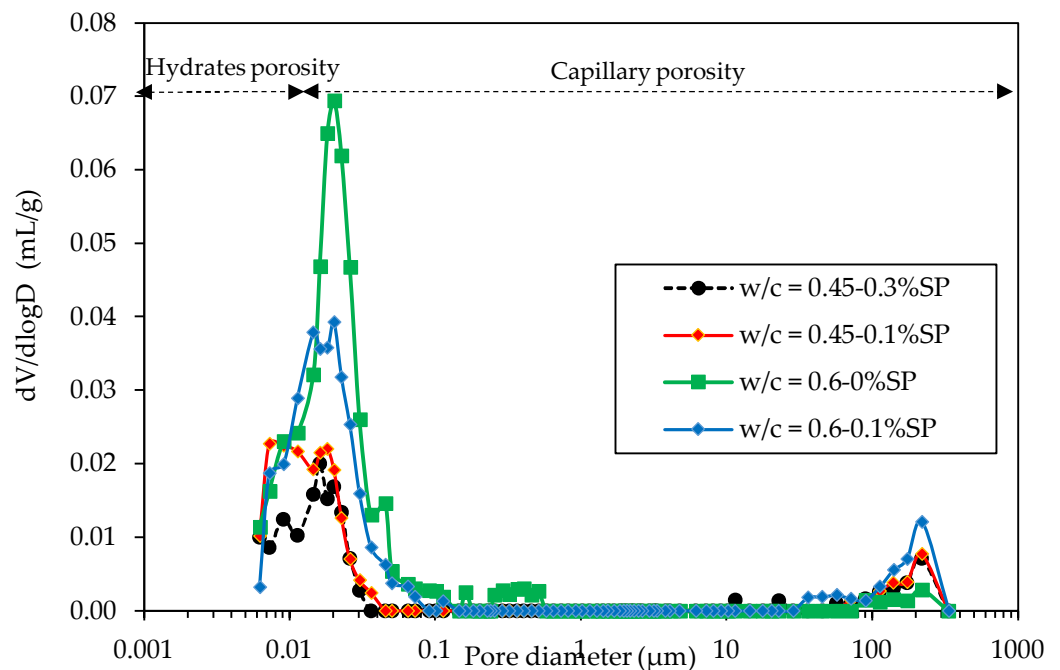


Figure 11. Porosimetry of mortars.

Figure 12 shows some observations of the mortar samples under an optical microscope. Visible defects can be noted, in particular, at the cement paste/sand interfaces. These defects are due to the wall effects [41,42] as well as to internal bleeding, that is to say, the bleed water remained trapped by sand. The observations suggest more defects for sample (b)

than for samples (a) and (c). Note that the trapped water for sample (a) is approximately 5.5 cm^3 , 27.5 cm^3 for sample (b), and 11.8 cm^3 for sample (c). There seems to be a correlation between the water trapped by the sand and the presence of defects at the interfaces. Other analyses are in progress to complete the obtained results.

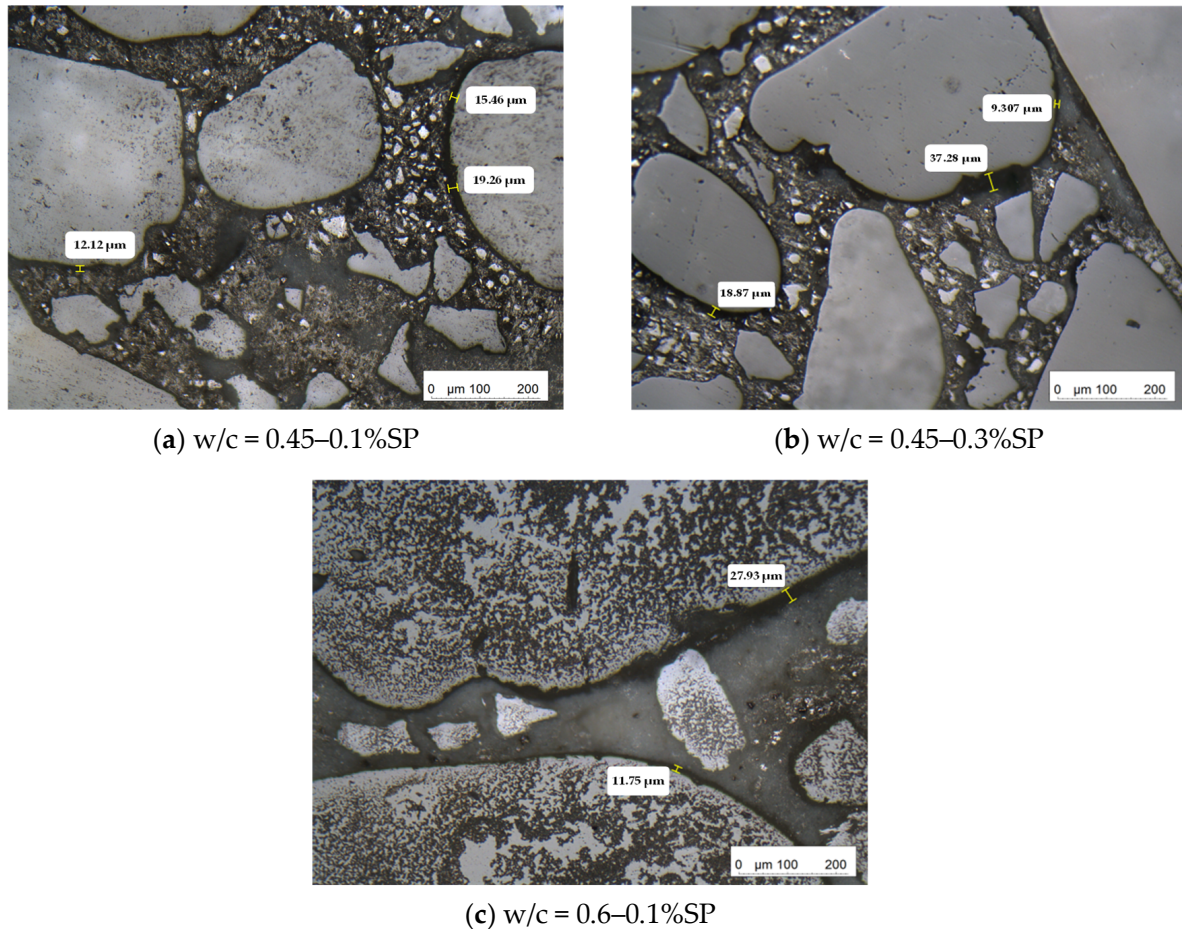


Figure 12. Observations of the microstructure of hardened mortars under the optical microscope: (a) $w/c = 0.4-0.1\%$ SP, (b) $w/c = 0.45-0.3\%$ SP, and (c) $w/c = 0.6-0.1\%$ SP.

4. Conclusions

This study investigated the effect of excessive bleeding on the properties of mortar.

The results obtained showed that the bleeding of the cement paste increases with w/c ratio and SP dosage. Moreover, the presence of sand inclusions leads to a decrease in the external manifestation of bleeding since the grains of sand constitute obstacles for the upward displacement of water, leading to the trapping of part of the bleeding water.

A simple and quick approach has been proposed to quantify the water trapped by sand. It can be retained that the water trapped by sand increases with sand/cement (s/c) ratio and SP dosage. When the proportion of sand exceeds 60% (wt% of cement), the water trapped no longer increases. Moreover, beyond a threshold of SP dosage, the static yield stress of the cement paste is not high enough to maintain sand in suspension, which leads to segregation, and the amount of water trapped (internal bleeding) no longer increases.

The water trapped by sand (internal bleeding) leads to an increase in porosity and a degradation of the cement paste/sand bond. This increase in porosity leads to a decrease in the compressive and flexural strength of the hardened mortar.

Author Contributions: Conceptualization, methodology, validation, investigation, data curation, and writing—original draft preparation, M.A. and Y.E.B.; supervision, Y.E.B., N.A. and E.G.-D. All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Data Availability Statement: Data presented in this study are available in the Appendix A.

Conflicts of Interest: The authors declare no conflict of interest.

Appendix A

Table A1. Chemical composition of cement.

Chemical Analysis ^a	%wt
SiO ₂	19.5
Al ₂ O ₃	5.37
Fe ₂ O ₃	2.24
CaO	62.7
MgO	1.27
SO ₃	3.79
Na ₂ O	0.05
K ₂ O	0.85
TiO ₂	0.19
SO ₃ (%)	3
Cl ⁻ (%)	0.03
Ignition loss ^b (950 °C)	2.4

^a XRF data, ^b TGA.

Table A2. Bleeding capacity determined by conventional method (column).

w/c	SP Dosage (%)	s/c	Bleeding Capacity (%)	
			Mean Value (%)	Standard Deviation
0.45	0	0	0.00	0.00
		0	1.94	0.09
		0.3	0.84	0.04
	0.1	0.6	0.61	0.08
		2.4	0.30	0.04
		0	15.90	1.40
	0.3	2.4	1.77	0.16
		0	18.70	1.60
	0.5	2.4	3.54	0.10
		0	21.30	1.80
	0.8	2.4	4.80	0.27
		0	17.14	0.90
1	0.3	14.49	0.85	
	0.6	13.22	0.74	
	2.4	7.22	0.40	
	0	3.85	0.15	
0.6	0	0.3	2.26	0.15
		0.6	1.36	0.07
		2.4	0.87	0.04
	0.1	0	9.87	0.47
		0.3	6.02	0.40
		0.6	3.99	0.25
	0.3	2.4	1.49	0.21
		0	13.33	1.42
	1	2.4	5.79	0.85
		0	18.40	1.90

Table A3. Determination of the volume of water trapped by sand.

w/c	SP Dosage (%)	s/c	Bleed Water (cm ³)	Mean Value (cm ³)	Standard Deviation	Volume of Water Trapped by Sand (cm ³)
0.45	0.1	0	7.9	7.85	0.79	0.0
			8.6			
			7.1			
		0.3	3.9	3.77	0.13	4.1
	3.8					
	3.7					
	0.6	3.2	3.14	0.03	4.7	
		3.2				
		3.1				
	2.4	2.3	2.36	0.28	5.5	
		2.1				
		2.7				
0.6	0	0	15.5	15.71	0.22	0.0
			15.9			
			15.8			
		0.3	11.8	11.78	0.09	3.9
	11.9					
	11.7					
	0.6	7.9	7.85	0.07	7.9	
		7.9				
		7.8				
	2.4	7.8	7.85	0.02	7.9	
		7.9				
		7.9				
0.1	0	46.5	47.12	0.54	0.0	
		47.5				
		47.4				
	0.3	31.5	31.38	0.29	15.7	
31.1						
31.6						
0.6	23.2	23.56	0.67	23.6		
	24.3					
	23.2					
2.4	15.9	15.68	0.70	31.4		
	16.3					
	14.9					

Table A4. TOC and yield stress measurement data.

SP Dosage (%)	Mass of Consumed SP (mg/g of Cement)		Yield Stress (Pa)	
	Mean Value	Standard Deviation	Mean Value	Standard Deviation
0	0.00	0.00	40.2	3.5
0.1	0.88	0.04	6.8	0.8
0.2	1.59	0.08	3.5	0.2
0.5	3.68	0.18	0.0	0
1	4.76	0.24	0.0	0

Table A5. Determination of the volume of water trapped by sand and mechanical data.

w/c	SP Dosage (%)	Volume of Water Trapped by Sand (cm ³)		Compressive Strength (MPa)		Flexural Strength (MPa)	
		Mean Value	Standard Deviation	Mean Value	Standard Deviation	Mean Value	Standard Deviation
0.45	0	0.0	0.00	64.00	4.9	8.30	0.1
	0.1	5.5	0.3	49.10	3.7	6.40	0.3
	0.3	27.5	1.4	43.50	1.4	6.10	0.3
	0.5	27.5	1.4				
	0.8	27.5	1.4				
0.6	0	7.9	0.4	38.10	2.2	6.30	0.10
	0.1	11.8	0.6	41.60	2.4	6.20	0.30
	0.3	19.6	1.0				

References

- Loh, C.-K.; Tan, T.-S.; Yong, K.-Y.; Wee, T.-H. An experimental study on bleeding and channelling of cement paste and mortar. *Adv. Cem. Res.* **1998**, *10*, 1–16. [CrossRef]
- Josserand, L.; Coussy, O.; de Larrard, F. Bleeding of concrete as an ageing consolidation process. *Cem. Concr. Res.* **2006**, *36*, 1603–1608. [CrossRef]
- Powers, T.C. *The Bleeding of Portland Cement Paste, Mortar, and Concrete: Treated as a Special Case of Sedimentation*; Portland Cement Association Bulletin. No. 2: Chicago, IL, USA, 1939. Available online: <http://www.concrete.org/publications/internationalconcreteabstractsportal.aspx?m=details&i=8504> (accessed on 3 March 2022).
- Powers, T.C. *The Properties of Fresh Concrete*; John Wiley & Sons: Hoboken, NJ, USA, 1968; pp. 533–652.
- Massoussi, N.; Keita, E.; Roussel, N. The heterogeneous nature of bleeding in cement pastes. *Cem. Concr. Res.* **2017**, *95*, 108–116. [CrossRef]
- Zhang, Y.-R.; Cai, X.-P.; Kong, X.-M.; Gao, L. Effects of comb-shaped superplasticizers with different charge characteristics on the microstructure and properties of fresh cement pastes. *Constr. Build. Mater.* **2017**, *155*, 441–450. [CrossRef]
- Zingg, A.; Winnefeld, F.; Holzer, L.; Pakusch, J.; Becker, S.; Gauckler, L. Adsorption of polyelectrolytes and its influence on the rheology, zeta potential, and microstructure of various cement and hydrate phases. *J. Colloid Interface Sci.* **2008**, *323*, 301–312. [CrossRef] [PubMed]
- Plank, J.; Sakai, E.; Miao, C.W.; Yu, C.; Hong, J.X. Chemical admixtures—Chemistry, applications and their impact on concrete microstructure and durability. *Cem. Concr. Res.* **2015**, *78*, 81–99. [CrossRef]
- Scrivener, K.L.; Juilland, P.; Monteiro, P.J.M. Advances in understanding hydration of Portland cement. *Cem. Concr. Res.* **2015**, *78*, 38–56. [CrossRef]
- El Bitouri, Y.; Azéma, N. Contribution of turbidimetry on the characterization of cement pastes bleeding. *Adv. Cem. Res.* **2022**, *35*, 1–30. [CrossRef]
- Tan, T.S.; Wee, T.H.; Tan, S.A.; Tam, C.T.; Lee, S.L. A consolidation model for bleeding of cement paste. *Adv. Cem. Res.* **1987**, *1*, 18–26. [CrossRef]
- Hoshino, M. Relationship between Bleeding, Coarse Aggregate, and Specimen Height of Concrete. *ACI Mater. J.* **1989**, *86*, 185–190. Available online: <https://www.scopus.com/inward/record.uri?eid=2-s2.0-0024621829&partnerID=40&md5=c842f70873d72c4c9ff7bd7bf1235ff5> (accessed on 1 April 2022).
- Khayat, K.H.; Guizani, Z. Use of viscosity-modifying admixture to enhance stability of fluid concrete. *ACI Mater. J.* **1997**, *94*, 332–340. [CrossRef]
- Peng, Y.; Lauten, R.A.; Reknes, K.; Jacobsen, S. Bleeding and sedimentation of cement paste measured by hydrostatic pressure and Turbiscan. *Cem. Concr. Compos.* **2017**, *76*, 25–38. [CrossRef]
- Perrot, A.; Lecompte, T.; Khelifi, H.; Brumaud, C.; Hot, J.; Roussel, N. Yield stress and bleeding of fresh cement pastes. *Cem. Concr. Res.* **2012**, *42*, 937–944. [CrossRef]
- Yim, H.J.; Kim, J.H.; Kwak, H.G.; Kim, J.K. Evaluation of internal bleeding in concrete using a self-weight bleeding test. *Cem. Concr. Res.* **2013**, *53*, 18–24. [CrossRef]
- Perrot, A.; Rangeard, D.; Picandet, V.; Mélinge, Y. Hydro-mechanical properties of fresh cement pastes containing polycarboxylate superplasticizer. *Cem. Concr. Res.* **2013**, *53*, 221–228. [CrossRef]
- Ishigaki, K.; Kurumisawa, K.; Nawa, T. The effects of bleeding on the microstructure of hardened cement paste. *Cem. Sci. Concr. Technol.* **2012**, *66*, 111–118. [CrossRef]
- Boel, V.; Helincks, P.; Desnerck, P.; De Schutter, G. Bond behaviour and shear capacity of self-compacting concrete. In Proceedings of the Design, Production and Placement of Self-Consolidating Concrete: Proceedings of SCC2010, Montreal, QC, Canada, 26–29 September 2010; Springer: Berlin, Germany, 2010; pp. 343–353.

20. Zhu, W.; Gibbs, J.C.; Bartos, P.J.M. Uniformity of in situ properties of self-compacting concrete in full-scale structural elements. *Cem. Concr. Compos.* **2001**, *23*, 57–64. [[CrossRef](#)]
21. Han, J.; Wang, K. Influence of bleeding on properties and microstructure of fresh and hydrated Portland cement paste. *Constr. Build. Mater.* **2016**, *115*, 240–246. [[CrossRef](#)]
22. *NF-EN 197-Part 1*; Composition, Specifications and Conformity Criteria for Common Cements. The European-French Standard: European Committee for Standardization: Brussels, Belgium, 2012.
23. *NF-EN 1097-6*; Tests for Mechanical and Physical Properties of Aggregates—Part 6: Determination of Particle Density and Water Absorption. The European-French Standard: European Committee for Standardization: Brussels, Belgium, 2022.
24. *ASTM C940-10a*; Standard Test Method for Expansion and Bleeding of Freshly Mixed Grouts for Preplaced-Aggregate Concrete in the Laboratory. ASTM International: West Conshohocken, PA, USA, 2010.
25. Josserand, L.; De Larrard, F. A method for concrete bleeding measurement. *Mater. Struct. Constr.* **2004**, *37*, 666–670. [[CrossRef](#)]
26. *ASTM C243-95*; Standard Test Method for Bleeding of Cement Pastes and Mortars. ASTM International: West Conshohocken, PA, USA, 2017.
27. *ASTM C232/C232M-09*; Standard Test Methods for Bleeding of Concrete. ASTM International: West Conshohocken, PA, USA, 2021.
28. Ait-Kadi, A.; Marchal, P.; Choplin, L.; Chrissemant, A.S.; Bousmina, M. Quantitative analysis of mixer-type rheometers using the couette analogy. *Can. J. Chem. Eng.* **2002**, *80*, 1166–1174. [[CrossRef](#)]
29. Mahaut, F.; Mokéddem, S.; Chateau, X.; Roussel, N.; Ovarlez, G. Effect of coarse particle volume fraction on the yield stress and thixotropy of cementitious materials. *Cem. Concr. Res.* **2008**, *38*, 1276–1285. [[CrossRef](#)]
30. El Bitouri, Y.; Azéma, N. Potential Correlation between Yield Stress and Bleeding. In Proceedings of the SP-349: 11th ACI/RILEM International Conference on Cementitious Materials and Alternative Binders for Sustainable Concrete, Online, 7–10 June 2021; American Concrete Institute: Farmington Hills, MI, USA, 2022; Volume 349, pp. 479–494. [[CrossRef](#)]
31. Déjardin, P. Volume effect of the adsorbed layer on the determination of adsorption isotherms of macromolecules by the depletion method. *J. Phys. Chem.* **1982**, *86*, 2800–2801. [[CrossRef](#)]
32. *NF-EN 196-1*; Methods of Testing Cement—Part 1: Determination of Strength. The European-French Standard: European Committee for Standardization: Brussels, Belgium, 2016.
33. Neubauer, C.M.; Yang, M.; Jennings, H.M. Interparticle Potential and Sedimentation Behavior of Cement Suspensions: Effects of Admixtures. *Adv. Cem. Based Mater.* **1998**, *8*, 17–27. [[CrossRef](#)]
34. El Bitouri, Y.; Azéma, N. On the “Thixotropic” Behavior of Fresh Cement Pastes. *Eng* **2022**, *3*, 677–692. [[CrossRef](#)]
35. Zhang, K.; Mezhev, A.; Schmidt, W. Chemical and thixotropic contribution to the structural build-up of cementitious materials. *Constr. Build. Mater.* **2022**, *345*, 128307. [[CrossRef](#)]
36. Ollivier, J.P.; Torrenti, J.M. (Eds.) La structure poreuse des bétons et les propriétés de transfert. In *La Durabilité des Bétons Chapter: 3*; Presses de l'école Nationale des Ponts et Chaussées: Paris, France, 2008; pp. 51–133.
37. Powers, T.C.; Brownyard, T.L. Studies of the physical properties of hardened Portland cement paste. *J. Proc.* **1946**, *43*, 101–132.
38. Jennings, H.M.; Bullard, J.W.; Thomas, J.J.; Andrade, J.E.; Chen, J.J.; Scherer, G.W. Characterization and modeling of pores and surfaces in cement paste: Correlations to processing and properties. *J. Adv. Concr. Technol.* **2008**, *6*, 5–29. [[CrossRef](#)]
39. Aghaee, K.; Sposito, R.; Thienel, K.-C.; Khayat, K.H. Effect of additional water or superplasticizer on key characteristics of cement paste made with superabsorbent polymer and other shrinkage mitigating materials. *Cem. Concr. Compos.* **2023**, *136*, 104893. [[CrossRef](#)]
40. Mor, A.; Mehta, P.K. Effect of superplasticizing admixtures on cement hydration. *Cem. Concr. Res.* **1984**, *14*, 754–756. [[CrossRef](#)]
41. Ollivier, J.P.; Maso, J.C.; Bourdette, B. Interfacial transition zone in concrete. *Adv. Cem. Based Mater.* **1995**, *2*, 30–38. [[CrossRef](#)]
42. Scrivener, K.L.; Crumbie, A.K.; Pratt, P.L. A Study of the Interfacial Region Between Cement Paste and Aggregate in Concrete. *MRS Online Proc. Libr.* **1987**, *114*, 87. [[CrossRef](#)]

Disclaimer/Publisher’s Note: The statements, opinions and data contained in all publications are solely those of the individual author(s) and contributor(s) and not of MDPI and/or the editor(s). MDPI and/or the editor(s) disclaim responsibility for any injury to people or property resulting from any ideas, methods, instructions or products referred to in the content.