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ARTICLE



The influence of granite powder waste and the addition of fly ash on concrete bleeding

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Abstract

Bleeding is a common problem in concrete slabs, and may lead to serious damage. The goal of this article is to understand the impact of alternative binders and their properties on the bleeding of concrete. Therefore, the impact of the type of binder on the bleeding process is investigated. The results show that the addition of granite powder or fly ash allows for the increased control of the bleeding process. It was found that a finer particle size distribution (PSD), an increased specific surface area (SSA), and a higher bulk density may reduce the amount of dispensed water in the concrete mix. Furthermore, the use of additives with an increased SSA leads to even a 30% reduction of the bleeding rate of mixes. The utilization of additives with a finer PSD than cement enables a 37% reduction of the bleeding rate of mixes. The influence of bleeding on compressive strength was assessed using destructive and non-destructive tests: Replacing 30% of cement with granite powder leads to a 30% reduction of concrete strength after 28 days of curing; on the other hand, replacing 30% of cement with siliceous fly ash leads to an 18% reduction of strength. Importantly, bleeding was also found to lead to the heterogeneity of the physical and mechanical properties across the concrete section. Consequently, the proper control of the bleeding process leads to more homogeneous properties of concrete across its cross-section.

KEYWORDS

bleeding, cementitious composites, fly ash, granite powder

1 | INTRODUCTION

Bleeding in concrete manifests through the accumulation of a thin water layer on its top surface, and may have a significant impact on its properties. Excessive bleeding

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may cause crucial changes in the long-term properties of concrete (e.g., decreased freeze-thaw resistance, cracking, or delamination). In general, two types of bleeding can be distinguished: normal bleeding, or channeled bleeding. Normal bleeding is connected with the fundamental physical laws that describe the process of sedimentation on heterogenous mixes. It is also correlated with chemical processes in cementitious systems. On the other hand, channeled bleeding occurs when cementitious mixes have a large amount of pores that coalesce

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over time, thereby creating channels. Those channels significantly accelerate the process of water extraction from the mix. Air channels are mainly associated with the properties of the subsurface layer of concrete. Scientists have found that water dispersed from a mix increases the water-to-cement ratio (w/c) of the mix.^{4,5} This is the main reason for the reduced mechanical and physical properties of concrete close to its surface. Powers⁷ described the physical laws related to concrete bleeding, and divided this process into two stages: constant and diminishing bleeding. Morris and Dux⁸ stated that bleeding in cementitious pastes is the result of the selfweight consolidation of cement grains. Perrot et al.9 stated that plastic shrinkage may significantly influence bleeding in cementitious systems. They also indicated that a higher amount of dispensed water influences the bleeding process and increases the plastic shrinkage-related changes of the material (initial cracking, initiating the creation of water extraction channels). Josserand et al. developed a mathematical model of the bleeding process based on the compressibility of the granular skeleton. Giaccio and Giovambattista¹⁰ observed that in concrete slabs, lower and upper levels are characterized by different concrete strengths (differing by even up to 30%). They¹⁰ attributed this to bleeding, which in turn leads to the increased porosity and deformability of concrete. Olorunsogo¹¹ investigated the impact of ground granulated blast furnace slag (GGBS), and the different particle size distribution (PSD) of GGBS, on bleeding in cementitious mortars. He observed that the addition of GGBS to the mix leads to a decrease in the bleeding rate, and also that the PSD does not have a vital impact on bleeding. Josserand et al. described bleeding as an aging consolidation process. They stated that the bleeding effect of concrete, which results from the consolidation of the self-weight of the granular skeleton, leads to synchronized physical, chemical, and mechanical effects. This allowed others to create models of the bleeding process. 12,13 Yim et al. 12 investigated the impact of different compressed air pressure on the bleeding process in cementitious mixes. They observed that cementitious materials demonstrate various phenomena under high pressure, and they proposed the application of a pressure vessel test for the evaluation of the bleeding process. Massoussi et al. 14 observed that bleeding can not only be considered as the consolidation of soft porous materials, but it actually has a heterogeneous nature. This in turn leads to the development of water extraction channels in the cement paste. As a result of this observation, there is less confidence that the bleeding process can only be associated with aging consolidation. Further research

focused on the understanding of the impact of the rheological properties of the mix on the bleeding process.

The bleeding process also leads to damage in concrete structures. 15,16 Uncontrolled bleeding is almost always associated with concrete damage. 17,18 Bleeding influences the homogeneity of a material structure across the cross-section of concrete, and also causes variations of the physical and mechanical properties of concrete. This is particularly relevant in concrete slabs, where excessive bleeding leads to cracking, delamination, and damage of the structure of subsurface concrete.5,19 It is known that bleeding depends on the cement type, ¹⁸ admixtures, ^{20,21} w/c ratio, ^{20,22} aggregate size and type, ²³ and the scale of the used cementitious mixes (i.e., concrete, mortar, or paste). 17,24 Concrete subjected to uncontrolled bleeding may need expensive repair, or even replacement of the entire concrete element.25,26

The significant impact of the bleeding process on the durability of concrete requires the control of this process. To control the bleeding process, the following question is crucial: How do the physical properties of binder grains influence the bleeding process of concrete, and consequently its mechanical properties and durability?

2 RESEARCH SIGNIFICANCE

The study investigated the influence of the physical properties and the type of granite powder (GP) grains or fly ash (FA; which were used in the cement mixes) on bleeding. The article focuses on formulating, observing, and describing the relation between the bleeding process and different types and amounts of additives. A division of the bleeding process into phases was proposed, with each phase being different with regards to the volume of water dispensed from the mixes over time. To understand the impact of bleeding on the heterogeneity of the physical and mechanical properties of cementitious materials, ultrasonic pulse velocity (UPV) tests were performed. The tests showed a strong correlation between the bleeding process and the durability of concrete. Furthermore, for the first time, the effect of using GP waste on the bleeding process in concrete is studied. The findings highlight the importance of the functionalization of wastes that are used in concrete. This work will be helpful for researchers studying concrete durability, especially the concrete used in concrete slabs or floors. The focus was on the impact of bleeding on concrete properties, and also its practical relevance. It should be noted, however, that the development of (new) physical and/or mathematical models related to the process is beyond the scope of the this work.

	CEM I 42.5R	Granite powder	Fly ash	Water	Superplasticizer Sikament 400/30	00 0	Aggregate 2/8 mm	Aggregate 8/16 mm
Specific surface area (cm ² /g)	3650	4150	3490	_				
Bulk density (g/cm ³)	2.454	2.415	2.435	1.0	1.150	1.867	1.712	1.625

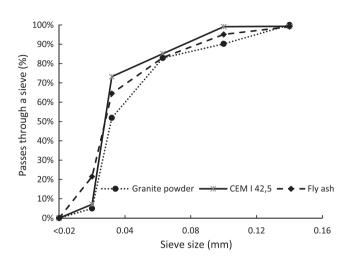


FIGURE 1 Particle size distribution of the binders

3 | MATERIALS AND METHODS

3.1 | Materials used in the research

Portland cement CEM I 42,5 R (Odra, Opole, Poland), siliceous FA (PGE Opole), GP waste (Strzegom, Poland), coarse and fine aggregate mixes (KSM Żelazna Kopice, Poland), and water (tap water) were used in the research. The physical properties of the ingredients are presented in Table 1. The PSD of the used materials is given in Figure 1. The aggregate was dried for 7 days and mixed every 24 h in order to achieve the same level of drying. Drying took place in a laboratory with standard temperature conditions (21 \pm 3°C) before the test in order to reduce humidity and to obtain a stable humidity of the aggregate.

The chemical compositions of the binders used in the research are described in Table 2 (all data from the Manufacturers).

Seven concrete mixes were investigated: a reference mixture with only Portland cement (CEM I 42,5 R), and mixtures containing 10%, 20%, and 30% of FA and GP (of the total mass of the cement) as a cement replacement. To improve the workability of the mixes, superplasticizer Sikament 400/30 (pH value: 5.5 ± 1.0 ; chloride content: $\leq 0.1\%$; sodium oxide equivalent: 0.1%) was used. The mix proportions used are presented in Table 3.

The concrete was mixed in three stages according to the EN 206 standard.²⁷ First, the aggregate, starting with

the coarsest and ending with the finest, was placed in the mixer. The dry aggregate was mixed for about 60 s to homogenize the mix. Then, the aggregates were mixed with 1/3 of the target amount of water for 2 min. After adding the binder, mixing was continued for 4 min, with the plasticizer slowly being added during the mixing.

3.2 | Fresh concrete properties

3.2.1 | Bulk density

The bulk density (BD) of the mixes was measured according to ASTM C642-13 Standard.²⁸ A mixture was placed and compacted into a prepared vessel with a known volume. After compacting, the vessel (with the mix) was weighed. On this basis, the BD of the mixes was calculated.

3.2.2 | Consistency of the mix

Consistency was tested using an Abrams cone according to EN 12350–2.²⁹ At the beginning, all measurement equipment was prepared according to standard.²⁷ Concrete was placed in the cylinder in three layers, each compacted with 25 bar strokes. Within 5–10 s, the mixture was demolded by lifting the cone. The entire test lasted no more than 2.5 min. The drop in the cone was measured by checking the difference in the height of the mold and the highest point of the demolded material.

3.2.3 | Bleeding

Immediately after mixing, the mixture was carefully placed in the measurement container. The process of laying and compacting individual layers was carried out according to EN 480-4 standard³⁰ (Figure 3). The concrete mixture was laid in three layers, and each layer of the same volume was compacted 25 with a steel rod (with a rounded end) with a diameter of 16 mm. After compaction, the concrete surface was leveled, with the least number of smoothing movements possible, until the effect of a smooth sheet was obtained. The container was placed on a vibration-free surface and covered with a lid. The temperature in the room

TABLE 2 Chemical composition of the binders used in the research (%)

	CEM I 42.R	CEM I 42.R		wder	Fly ash	
Chemical compound	Result	SE ± (%)	Result	SE ± (%)	Result	SE ± (%)
Fe_2O_3	<u>—</u>	_	2.54	1.85	_	_
MgO	1.54	2.14	6.42	2.45	2.45	2.15
Al_2O_3	5.73	3.47	20.17	3.12	30.65	4.56
SiO_2	18.86	2.98	54.11	3.65	54.33	1.96
SO_3	5.89	2.14	_	_	_	_
K ₂ O	1.30	3.58	3.23	2.85	2.51	2.57
CaO	63.94	2.93	8.18	2.65	3.34	0.99
FeO	2.75	5.01	4.02	1.89	4.78	1.25
NaO	_	_	3.09	2.47		_
Na ₂ O	_	_	_	_	1.95	3.54

TABLE 3 Mix proportions of the designed concrete mix (kg/m³)

Series of mixes	Cement CEM I 42,5 R (kg/m³)	Water	FA	GP	Aggregate 0/2 mm	Aggregate 2/8 mm	Aggregate 8/16 mm	SP Sikament 400/30	w/c Ratio
REF	310	175	_	_	715	445	717	3.41	0.57
FA10	281.4	175	27.9	_	715	445	717	3.41	0.62
FA20	257.7	175	51.9	_	715	445	717	3.41	0.68
FA30	238.6	175	71.5	_	715	445	717	3.41	0.73
GP10	281.4	175	_	27.9	715	445	717	3.41	0.62
GP20	257.7	175	_	51.9	715	445	717	3.41	0.68
GP30	238.6	175	_	71.5	715	445	717	3.41	0.73

Abbreviation: FA, fly ash; GP, granite powder; w/c ratio, water-to-cement ratio.

during the test was $20 \pm 2^{\circ}$ C, while the relative humidity was $65\% \pm 5\%$. Water from the concrete surface was collected at 10-min intervals in the first 40 min, and then every 30 min until the volume of extracted water stopped increasing. To facilitate the liquid collection, a 2-cm wide spirit level was placed under the edge of the container. The PN-EN 480-4 standard allows the use of washers with a dimension of up to 5 cm. The cylinder remained covered with a lid throughout the test. The filtered liquid was collected in plastic cups weighing 2.00 g and then weighed on scales with an accuracy of 0.01 g. Water was collected with the use of a pipette. The test scheme is presented in Figure 2. For each research series, the test was performed at least five times (for statistical evaluation of the results). Based on the measured values of the dispensed water in the bleeding process, the bleeding rate was calculated, according to standard EN 480-4,²⁸ as:

$$B_{\mathbf{r}} = \frac{V_1}{V_2} * 100\%(\%) \tag{1}$$

where V_1 is the total volume of the dispensed water in the bleeding process (ml) and V_2 is the volume of the concrete mix used in the test (ml).

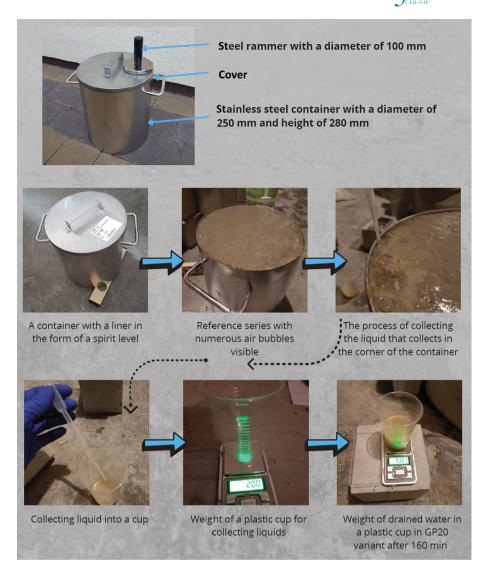
3.3 | Hardened concrete properties

3.3.1 | Compressive strength

After determining the consistency of the mixture, samples for the compressive strength tests were cast in molds with dimensions of $15 \times 15 \times 15$ cm. Their inner surface was covered with a release agent. Concrete was placed in two layers and compacted using a vibration table. The cubes were demolded after 24 h and placed in water $(20 \pm 2^{\circ}\text{C})$ to ensure optimal curing. The compressive strength was tested in accordance with EN 12390-3. The samples were tested after 14 and 28 days, with a minimum of six samples being tested in each condition.

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FIGURE 2 Apparatus for the concrete bleeding testing according to EN 480-4, ³⁰ and the activities performed during the water drainage test.



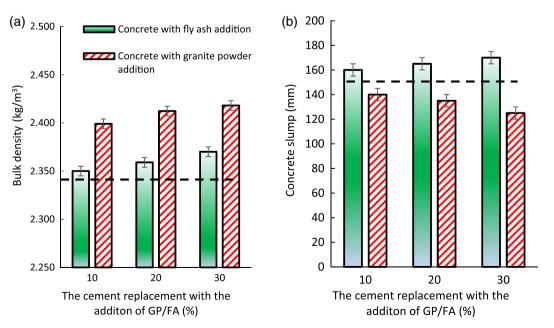


FIGURE 3 Properties of the fresh concrete with a partial replacement of cement with the addition of granite powder (GP)/fly ash (FA): (a) bulk density and (b) slump

3.3.2 | UPV measurements

Stored 14 or 28 days samples were measured and prepared for investigation. The tests were carried out according to the methodology described by Stawiski and Kania⁵ using an ultrasonic pulse analyzer (Procequ, Switzerland HQ) equipped with conical heads. An UPV test was conducted for the concrete samples prepared for compressive strength testing (Section 3.3.1). Before the destructive tests, UPV measurements were performed to investigate the UPV across the height of the concrete. Testing locations were marked at every 5 mm in a vertical direction, where the velocity of the ultrasonic wave was then measured. The spatially resolved UPV measurements allowed the compressive strength in different parts of the specimen to be estimated, thereby enabling the effects of local defects or pores on the local measurements to be considered.

4 | RESULTS AND DISCUSSION

4.1 | Properties of the fresh concrete

Figure 4 shows the properties of the fresh concrete with a partial replacement of cement with GP or FA. The

concrete modified with GP/FA was characterized by an increased BD (Figure 3a), with modification with GP leading to a significant increase in the BD of fresh concrete. 32,33 However, the addition of GP or FA led to a different effect on concrete workability (Figure 3b). The GP allowed for more dense mixes when compared with the reference concrete. On the other hand, the addition of FA in concrete mixes changed the consistency to be more liquid, which is related to the spherical morphology of FA grains. 34

Figure 4 presents the results of the bleeding measurement in the different mixes.

The mass of water dispensed in the bleeding process in the concrete depended on the material used as the partial cement replacement (Figure 4a,b). The reference concrete (100% Portland cement) dispensed the highest amount of water in the hardening process. The partial replacement of cement with GP or FA resulted in a reduction of the mass of dispensed water. The GP provided a higher reduction of dispensed water when compared with the FA. The error bars show that the FA30 and GP10 series have nonsignificant changes of values (within the range of error bars). It is possible that these series have statistically similar results. Moreover, the results of the reference series and FA10 series are similar.

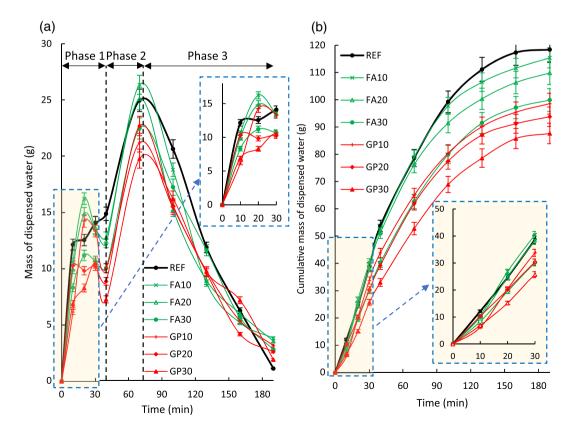


FIGURE 4 The bleeding process in the concrete with the partial cement replacement with granite powder (GP)/fly ash (FA). (a) Mass of water dispensed over time. (b) Cumulative mass of water dispensed over time

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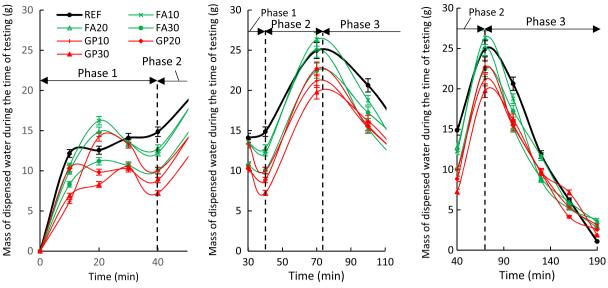


FIGURE 5 Proposed division of the bleeding process in three phases for the concrete modified with the addition of granite powder (GP)/fly ash (FA): (a) Phase 1, (b) Phase 2, and (c) Phase 3

When analyzing Figure 4a, it can be stated that the bleeding process is characterized by three phases. All the observed phases are presented in Figure 5.

In Phase 1 (first 30–40 min of the process, Figure 5a), an increased variability of the mass of dispensed water was observed. Moreover, in Phase 1, numerous changes in the dynamics of the process for all the mixes were noted. Phase 1 may be related with the normal type of bleeding. Phase 2 (Figure 5b) started after 30/40 min, and continued until 70/80 min after mixing. In this short phase, the highest amount of dispensed water was measured. This phase also allows for the possible creation of air channels. Phase 3 (Figure 5c) started after 70/80 min, and ended when the concrete was completely hardened. In this phase, there was a decrease in the amount of dispensed water at each testing time. Based on these measurements, Figure 6 presents a hypothetical description of bleeding in concrete.

Each of the bleeding phases proposed above can be further divided into two stages. In fresh concrete (liquid phase), bleeding leads to more heterogeneous properties (local density and w/c ratio) across the section's height. In hardened concrete, the bleeding process leads to changes in hardening and porosity across the section height of the specimens. The description of the bleeding process in the phases is similar to that proposed by Massoussi et al. ¹⁴ However, they stated that the bleeding process is similar to a function of yield stress in cement pastes. Herein, the process is divided based on the peaks of the amount of water dispensed.

In Stage 1 of Phase 1, a clear impact of the mixing process was observed. In this stage, the mix was homogenous

(i.e., it had the same density and w/c ratio across the section). In Stage 2 of Phase 1, a lower impact of the mixing process was observed. Heavier grains start to settle, while finer grains move up due to gravity (sedimentation process). This led to an increased density and a decreased w/c ratio at the bottom of the specimen (normal bleeding). In Stage 1 of Phase 2, the initiation of the hydration process and an increase in nucleation were observed. In this stage, the intermolecular forces between the particles in the mixture increased and gravity dominated the sedimentation process. Moreover, the effect of the intermolecular and chemical forces of the hydration process was decreased. The water channels that transfer water in the bleeding process across the section's height were created (the differences in local density and the w/c ratio observed in Stage 2 of Phase 1 were greater). The creation of water channels accelerated the bleeding and led to an increased amount of extracted water (channeled bleeding). Stage 2 of Phase 2 is connected with the start of the hardening process. At the top of the mix, a thin water film was created and grew over time. The material was at the transition between the liquid and the solid phase. The hydration and nucleation processes were dominant in this stage. Chemical reactions connected with nucleation led to a significant decrease in the amount of extracted water, which is in turn reduced the influence of channeled bleeding on the bleeding process. Stage 1 of Phase 3 describes the hardened composite. In this stage, the bleeding process affected the porosity and hardening of the composite—at the top of the composite, porosity was increased, and hydration was significantly slower than at the bottom, which may be connected with an increased w/c ratio. This led to a lower hardness

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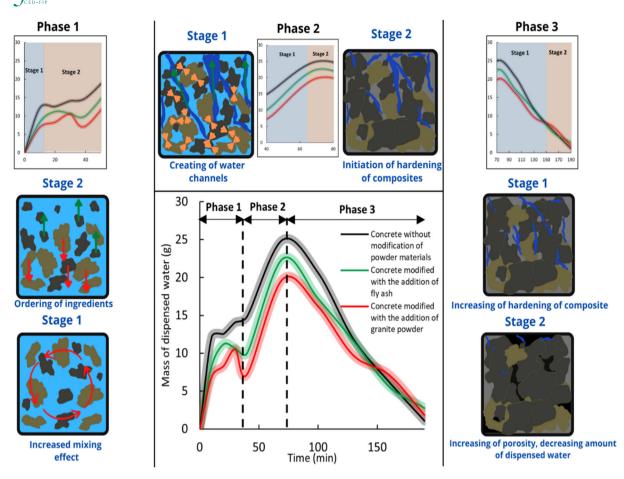


FIGURE 6 Hypothetical description of the bleeding process in concrete with a partial cement replacement with the addition of granite powder or fly ash (a detailed description is provided in the text below)

and strength of the top section of the composite. The occurrence of water channels leads to a variable effective w/c ratio across the cross-section of the concrete, which in turn affects its properties. Stage 2 of Phase 3 describes an almost fully hardened concrete. The amount of water dispensed in this stage was not great; however, the effect of the bleeding process was significant. In concrete characterized with high bleeding, more heterogeneous properties across a section of the composite's height are expected. Figure 7 shows the bleeding process in cementitious composites, and helps to understand its complex nature. Problematic processing of the bleeding process is associated with the phase change in cement-based materials, the consolidation process, the chemical reactions of cement hydration, and the physical influence of intermolecular forces.

4.2 | Properties of hardened concrete

Figure 8 presents the compressive strength of the concretes (Figure 8a) and the correlations between their

compressive strength and the bleeding process (Figure 8b).

The compressive strength of the reference concrete (with 100% cement) was the highest (Figure 8a). The partial replacement of cement with GP or FA led to a decrease in compressive strength at 14 and 28 days. The use of GP as a partial cement replacement resulted in a higher reduction in compressive strength when compared with the concretes modified with the addition of FA. The increased bleeding led to an increase in the compressive strength of the composites (Figure 8b). The increased bleeding rate of the concrete led to a lower w/c ratio in the composites, and therefore a higher compressive strength. However, the bleeding process primarily affects the microstructure of the material close to the surface. It should be noted that compressive strength is only affected by surface changes to a small degree. A limitation of using GP as a material to decrease bleeding in concrete may be the fact that it has a negative effect on the mechanical properties of concrete. GP may be an appropriate material to control the bleeding process, but it should be functionalized in order to improve the

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FIGURE 7 Scheme of the bleeding phenomena process in cementitious composites

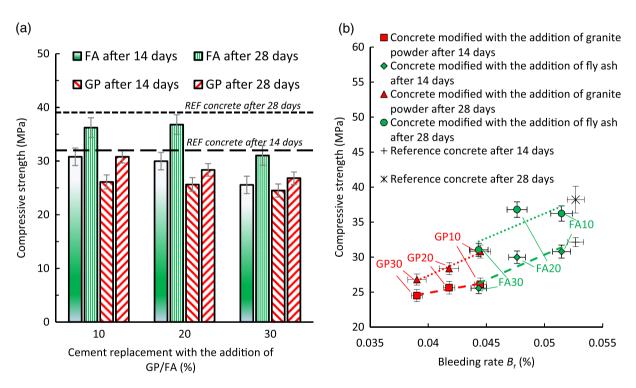


FIGURE 8 (a) Compressive strength. (b) Correlation between compressive strength and the bleeding rate B_r of the concrete modified with the addition of granite powder (GP) or fly ash (FA), which was assessed after 14 and 28 days of curing

properties of grains (which will lead to an increase in the concrete's strength). To obtain more precise results, more samples should be tested in the future. Therefore, the compressive strength test is not expected to be sensitive to the effect of the bleeding process in cementitious composites. To describe the effect of bleeding on compressive strength, an investigation of samples from the top and the bottom of the section of concrete should be performed. In order to investigate other dependencies related to the influence of the bleeding process on the properties of concrete, Figure 9 was used.

Figure 9a presents a correlation between the w/c ratio and the bleeding rate of the concrete mixes. Increasing the w/c ratio led to a reduction in the bleeding rate in the mix. Moreover, the additives caused a lower amount of water to be dispersed from the mix. The higher specific surface area (SSA) of the GP or FA modified binders used as a cement replacement may have a significant influence on this. Our observation allows us to state that the PSD of cement replacement materials has an impact on the bleeding process. The prismatic shape of GP grains led to a higher water demand and a lower bleeding ratio of the

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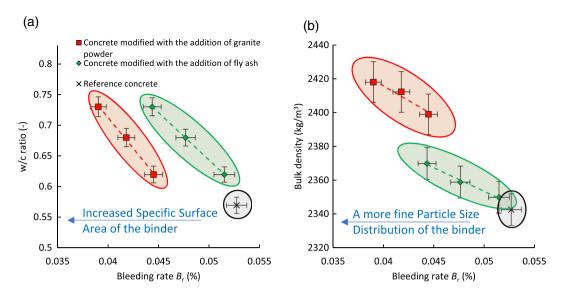
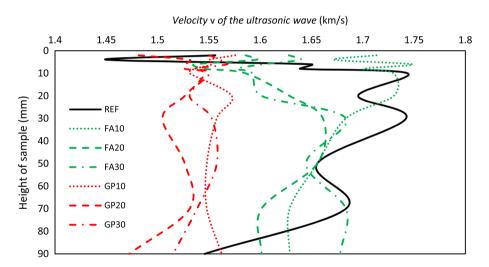


FIGURE 9 Correlations between (a) water-to-cement (w/c) ratio and (b) bulk density and bleeding rate B_r in the concrete modified with the addition of GP/FA



Velocity of the FIGURE 10 ultrasonic wave in the concrete samples modified with the addition of granite powder (GP)/fly ash (FA)

mix. Moreover, a higher cement replacement with GP or siliceous FA led to a larger reduction in the bleeding rate of the mixture. Similar observations were made for the correlations between the BD and bleeding rate of the mix (Figure 9b). However, Figure 9b shows that the addition of GP to concrete enables a higher BD of the concrete to be achieved. These observations may be related to the finer PSD of GP when compared with cement or FA.

To analyze the impact of the bleeding process on the mechanical properties of concrete, the methods of analyzing the properties of composites across the section's height should be used. Figure 10 presents the results of the ultrasonic wave velocity analysis.

The results of UPV investigations are commonly characterized by variable curves. These curves describe the characterization of the material in the investigated places (usually each 5 mm), which may result in irregular curves. The investigated material may have some local cracks, pores, or defects, which could in turn decrease the velocity of the ultrasonic waves. This allows the heterogeneity of the tested materials to be investigated. The addition of GP and FA led to more homogeneous concrete across the section's height (Figure 10). The modification of concrete with GP or FA enabled improved properties of the composite at the top of the samples to be achieved, which has a direct impact on the durability of the composites. The addition of GP, when compared with FA, led to more homogenous properties of the composite. However, the modification of concrete with the addition of GP led to a lower strength of the composite when compared with the FA or the reference concrete. It should be noted that relatively low values of the mean velocity of ultrasonic wave are observed. The authors believe that these results are lower when compared with

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the typical mean velocity of the ultrasonic wave in concrete (generally 2.8-3.4 km/s), which is due to the significant impact of bleeding. This observation is based on the differences in the velocity of the ultrasonic wave value for the reference series (100% cement) and the samples modified with the addition of FA or GP. FA led to insignificant changes in the UPV test when compared with the reference series. The samples modified with GP are characterized by lower UPV values, and also lower bleeding. The observed impact of GP may be potentially significant in the future when developing more homogeneous concrete. However, the reactivity of GP should be improved in order to achieve better mechanical properties of concrete.

5 CONCLUSIONS

On the basis of this research, a significant gap related to the impact of GP or FA on the bleeding of concrete has been filled. A new bleeding mechanism, which is associated with the physical properties of binders, was proposed, and its positive effect on the hardening process and the properties of hardened concrete were described. The following major conclusions can be drawn based on the presented study:

- The use of novel binders may result in a reduction of concrete bleeding, which facilitates the controlling of this process-modification of a concrete mix with GP (30%) leads to a reduction of bleeding by up to 25%, whereas the addition of FA (30%) leads to a reduction of extracted water by up to 15%,
- The decrease in the amount of water dispensed in the bleeding process in concrete modified with the addition of GP and FA is connected with the increased SSA, finer PSD, and increased BD of those binders. The UPV test showed a variability of concrete properties across the section's height. Replacing cement with the addition of GP or FA leads to a significant improvement of the concrete's fresh properties, resulting in a more homogenous material. On the other hand, GP-modified concrete is characterized with a 6% lower UPV when compared with FA modified concrete.

Future work needs to focus on understanding and numerically modeling the bleeding process, while also considering the impact of the physical properties of alternative binders on concrete bleeding. Moreover, more research on the impact of the bleeding process on structural elements is highly desired, and will allow to better relate the scientific results with practical applications. Ultimately, a positive effect of GP waste on some important properties of concrete was observed. In order to enhance this impact, attempts should be made to functionalize GP (e.g., mechanically-by grinding, or chemically—by prespraying or pretreatment).

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DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available on request from the corresponding author. The data are not publicly available due to privacy or ethical restrictions.

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