A comprehensive review of cementitious grouts: composition, properties, requirements and advanced performance

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ABSTRACT

Although grouting is a widespread process mainly used for soil treatment and for filling cracks/voids in concrete structures, grout injection is still a challenging step. Due to the different performance required for the numerous fields of application, encompassing several injection methods and different design approaches, it is essential to understand how the components of the grout (cement, aggregates, supplementary cementitious materials, chemical admixtures) affect the workability, stability, injectability, consistency, rheology and, as a result both the composition and the aforesaid properties, also the mechanical strength of the material and the effectiveness and long term performance of the overall grouting application. As a matter of fact, all cementitious materials can suffer deterioration processes that affect the serviceability and durability of structures and jeopardizing their safety, requiring maintenance/recovery works whose cost can, overall the structure life cycle, result even higher than the construction one. This may be especially true in the case of grouting applications, e.g. in prestressed concrete structures, where the state of deterioration is not visible and its non-inspectable progress might lead to catastrophic structural failures. To address all these issues, researchers have developed self-healing cementitious materials which have proved to be an interesting option, as they are able to prolong the lifetime of structures, reducing the environmental impact all along its life cycle. The literature points out that many self-healing mechanisms are effective in concrete and mortars. However, this technology has been barely applied in grouts. In this context, this work presents a comprehensive overview of cementitious grouts with focus on their composition, properties, application technologies and conditions that can affect the overall material and application performance. In addition, this review also provides an overview of self-healing technologies applied to grouts as well as the research gaps in the field of self-healing grouts that should be desirably filled to exploit their benefits in structural and infrastructural applications.

Keywords: cement grout; constituent materials; applications; self-healing

1. INTRODUCTION

The first records of grouting date to the early 19th century, when it was used as a corrective measure in soils [1,2]. Over time, the injection technique was improved and grouting started being used in ports, canals, tunnels, mining wells, bridges, dams [1]. The improvement of injection technologies and new grout formulations were fundamental to consolidate the grouting no longer just a remedial measure but as a stage of its own in the construction process of structures and infrastructures [1]. Due to this growth, technical standards were developed in order to establish a uniform approach to materials, processes and methods. However, some of these standards are quite vague about composition and do not limit the scope of a particular property [3,4]. Johnson [5] points out that even providing a definition of “grout” is a complex task. The most acceptable one (although it is more a characteristic than a definition) is that “grout” describes many types of injectable fluid materials that can be designed and handled in countless ways to achieve a desired result [6]. Therefore, a cementitious grout is considered a fluid mixture consisting of cement, and in case supplementary cementitious materials, fine aggregates, water and chemical admixtures, which is injected for filling cracks and voids, bonding precast concrete elements, stabilizing soils, sealing joints, fillings ducts of posttensioning tendons in prestressed elements, among others [3]. Clearly the composition and properties of grouts change according to the field of application. For example, low viscosity grouts are generally not recommended for lifting structures works because there could be a water flow in the rock. For this application, grouts with fast setting time are the most indicated [7].

Grouts for filling post-tensioning ducts have to present high stability and fluidity to cover the largest area of the ducts, as in case of movement of the structure it can cause losses. Additionally, unstable grouts can exhibit sedimentation and bleeding. The free water can freeze/evaporate (depending on the weather conditions) and lead to some expansion and/or corrosion problems [8]. In precast concrete the connection between the elements is always a critical point, and the grout used has to present good bond strength and the lowest shrinkage [9]. There are numerous applications available in the literature on possible grout applications. Among others, grouts for soil nailing [10], steel reinforcement [11], structural repair [12], soil erosion treatments [13], mansory[14], pavement [15]and tunneling [16] should be mentioned in this review. Regarding the properties, fluidity is always a critical point for grouts. On the one hand, a good fluidity is required to completely and effectively fill all free regions in the intended grouting “domain”, whereas, on the other, an excessively high fluidity might result into likewise high bleeding rate, affecting the performance of the application. Excess free water can even lead to some expansion and/or reinforcement corrosion problems. Many publications have investigated the effects of adding supplementary cementitious materials (SCM) [17–23] and chemical admixtures [24–26] not only to govern the fluidity but also in targeting to other properties, including permeability, durability and strength.

The grouting industry also tried to address issues related to the negative impact caused by the construction sector proposing innovations which range from new eco-friendly compositions [27–29] to new grout formulations for well-known applications [11].

Due to the variety of materials and application fields, this study will focus on mapping the most relevant publications of cementitious grouts providing useful information to understand how different components affect the properties in the fresh- and hardened-state. The state of the art is organized into sections that cover from composition to properties, highlighting some critical parameters that must be evaluated. In addition, the review discusses the design of functionalized grouts using self-repair technology, focusing on current advances in the implementation of this technology in cementitious grouts.

2. GROUTING METHODS

As mentioned before, the first grouting methods emerged in the field of soil improvement and remediation. In that time, the main purpose was to improve the soil strength and reduce its permeability, in order to increase the bearing capacity and stability [30,31]. Nowadays, grouts can be employed in several different applications and the grouting methods (permeation, compaction, jet, compensation, backfill, injection grouting) vary according to the grout type, its mechanisms and field of application [30,32].The selection of grouting method will depend not only on the grout composition but also on several factors, including geological characteristics of the site, climate conditions, the objective of grouting, types of cracks, budget and time for project execution [6]. For example, masonry grouting requires attention to parameters such as distance between the injection holes, the injection pressure, the water absorption capacity and physical/chemical characteristics of the masonry [33]. In the case of filling the soil cracks, the selection of the method will depend on the rheology, filtration trend and permeability into the tortuosity of the fissure, among other characteristics. Table 1 presents some characteristics of the most commonly used grouting methods.

Table 1- Main grouting methods used in geological applications and their respective characteristics

Permeation grouting	***Compaction grouting***	***Jet grouting***	***Compensation grouting***
It is the most common and conventional method. It is used in soft-ground works, that is, joints, fractures or voids are filled at low injection pressure without disturbing, displacing or creating any change in volume in the soil formation or structure. Permeation grouting is also known as cement grouting, chemical grouting and pressure grouting. This method makes soils and rocks less permeable. Thin fluid grouts (low viscosity, non-particulate grouts) are essential to obtain adequate penetration, although they cannot permeate into very thin voids [31,34].	This method is also known as low mobility grouting and it was developed to improve soil stability and fix settlement problems during tunneling operations. The grout does not penetrate nor permeate through the soil voids. It displaces the soil, creating lenses that control the lifting and re-levelling of structures. High pressure is required and very thick grouts are used to prevent or limit hydrofracturing. The grout must be workable enough to be pumped with low mobility, as it must remain as a growing mass (without expanding) [31,34,35].	In this method, a jet of pressurized fluid is used to erode the soil (creating a cavity) which is then filled with grout. It can be applied for reinforcing foundations, building retaining structures and impermeable barriers, preventing soil movements and in tunnelling projects, in addition to stabilizing soft ground and sealing vertical joints. As this application requires ultra-high pressure, it is important that the grout resists structural breakage (yield stress must be achieved without destabilizing the suspension). The rheological properties of the grout must be carefully designed and measured, as the grout has to tolerate high shear rates during the entire pumping process [36].	Thick grout is used to compact soil particles (increasing the stiffness and strength) and to stabilize and mitigate settlements. Compensation grouting is also known as *fracture grouting*. The objective is to minimize the movement of the soil that would affect existing structures, e.g., it is used to adjust ground levels as tunnels are excavated in order to balance the excavation-induced settlement [34].

3. MAIN COMPONENTS OF CEMENTITIOUS GROUTS

Grouts can be based on solutions or on binders. Cementitious grouts are considered as particulate grouts, that is, they are mainly composed by particulates derived from the clinker suspended in water. The final properties are influenced by several parameters including water/cement (w/c) ratio, cement composition, ambient temperature, mixing time and speed [37].

It is important that cementitious grouts completely fill voids and joints, have good adhesion to surfaces (e.g., concrete, rocks, mortar), are chemically and mechanically resistant and minimally shrink to prevent the appearance of micro-cracks. Depending on the application or formulation, cementitious grouts may present limited ability to penetrate fine soils or voids [38]. In this respect, several studies seek to optimize the grout mixture [39–41]. The effectiveness of the optimization is normally evaluated by assessing workability, volume stability, porosity, strength, injectability and durability performance. Fig. 1 provides a comprehensive overview of parameters that must be considered for a successful grouting.

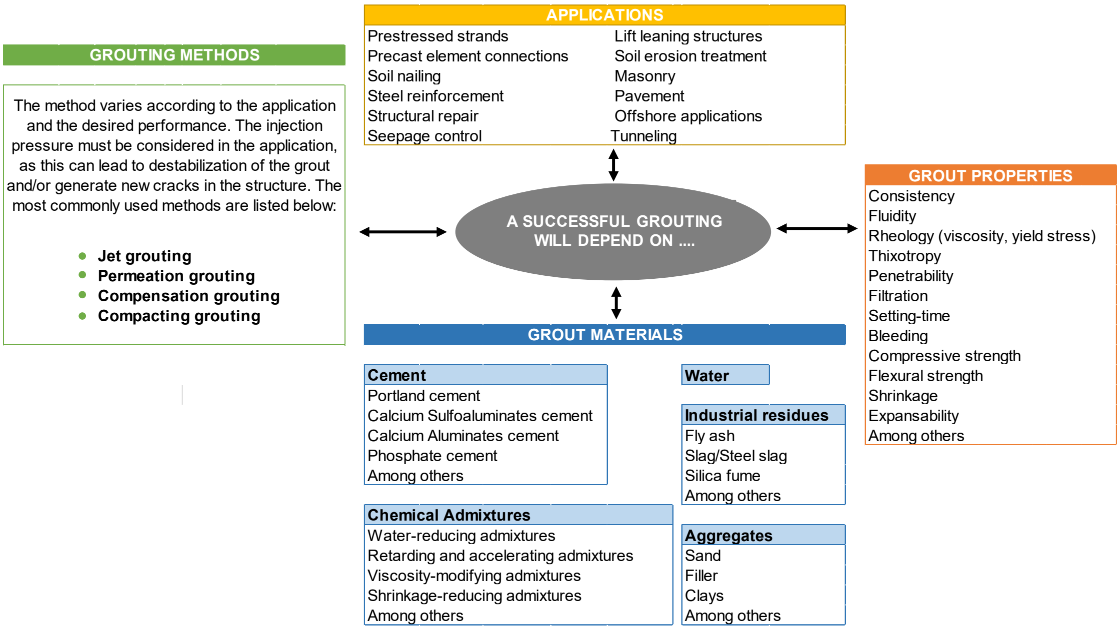

Fig. 1. Overview of some parameters required for a successful grouting.

As mentioned earlier, cement-based grouts are composed by cement, aggregates, supplementary cementitious materials and chemical admixtures. The main binder of most cementitious grouts is Ordinary Portland Cement (OPC) due to its engineering characteristics, low costs, predictable durability and high compatibility with concrete structures [30,38,40,42,43]. Supplementary cementitious materials (SCM) are discarded coproducts or industrial by-products and are also known as mineral admixtures. Normally, SCM replace part of the OPC content [19,44–46].The most conventional SCMs are fly ash, slags, silica fume and calcined clays. Supply reductions of these by-products are already noticed and new SCMs are being studied as alternatives, such as rice husk ash, calcined dredging sediments, steel slag and natural pozzolans (although this is not a new field of application, but rather the recovery of an old technology) [47–49].

Fly ash (FA) is a by-product from coal combustion with pozzolanic properties. It is categorized in low-calcium FA (Class F) and high-calcium FA (Class C), being class C the most used type due to its better pozzolanic property [50]. Although FA improves the workability of the fresh concrete due to a lubrication effect [49], its use in non-shrinkage grouts did not produce the same result. Kim et al. [20] reported that additions of FA with different particle size (ground fly ash (GFA) and raw fly ash (RFA)) did not improve the workability because, as the authors claimed, less cement was available for the hydration reaction as the fly ash content increased. The flow time of mixes with GFA were slightly reduced compared to 100% OPC grout whereas additions of RFA increased the flow time up to 27 seconds (compared to reference time: 44 s). In addition, both studied FA types improved bleeding, reduced the setting time and decreased the compressive strength in early ages. Adding Class F FA (20%, 35%, 50% and 65% by volume) decreased the yield stress [51] and additions of microfine fly ash (MFA) (from 0 to 40% by weight) reduced the apparent viscosity [18].

Slags are impurities separated from the metal during the smelting processes. They can be classified as ferrous (from iron and steel), nonferrous (from copper, nickel, lead and zinc) and non-metallurgical (e.g., phosphorus slag). The most used types in civil construction are slags produced during the ironmaking process. When iron is smelted and cooled from a blast furnace, a granular vitreous product is formed. This material is known as blast furnace slag (BFS). Different types of BFSs (such as granulated, expanded, pelletized) are produced depending on cooling method [52]. Adding high amounts of BFS in concrete increases the setting time because its hydration is relatively slow [49]. Effects on fresh and hardened properties of steel fiber-reinforced grout (SFRG) by additions (0, 20 and 40% of cement by weight) of ground granulated blast furnace slag (GGBS) in two types of cement grouts (Type 1 cement is ordinary Portland cement while Type 3 cement is high early strength (HES) cement) were studied by Kim et al. [8]. The authors observed, for both cements, an increase in the flow table measurements with the increase of GGBS amount; the increase in HES cement was more evident than in OPC: 40% of GGBS produced an increase in setting time of 10.5 % for OPC and 35% for HES cement. Regarding the flow time, an increase in flowability, i.e. a flow time reduction, was observed for both types of cement. For type 1, the reduction was 17.3% and for type 3, it was 23.8%. These results are resumed in Fig. 2, in which relative values with respect to reference case are shown for each parameter represented: flow (time and table), bleeding and initial setting time.

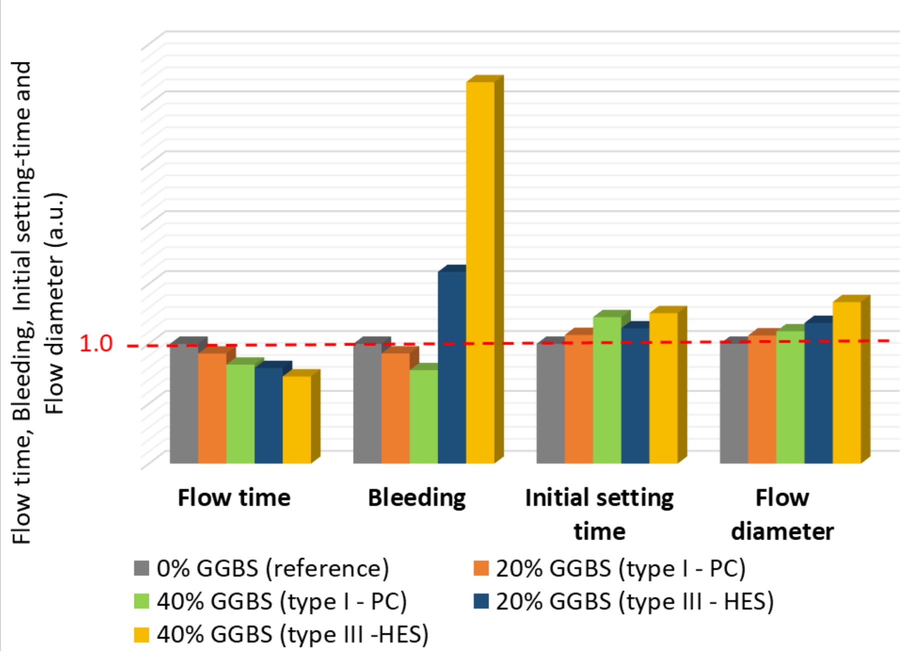

Fig. 2. Effect of cement replacement by weight (Type I Portland Cement and Type III High Early Strength cement) by GGBS on flow (time and diameter), bleeding and initial setting-time. Adapted from [8].

Silica fume (SF), also reported as microsilica or condensed SF, is one of the most popular choices for increasing microstructure compactness and, as a results, strength of cementitious composites due to its high silica content and fineness. When added to cement, an increase of the yield stress and plastic viscosity was reported [48,53]. Small amounts of SF (5, 10 and 15% by weight of cement) in high-performance cementitious grouts (water/binder (w/b) equal to 0.33) improved the performance when compared to the grout without SF. With 5% and 10% of SF, the flow time was reduced to 25 and 27 seconds, respectively (reference starting from 39 s). While additions of 15% decreased fluidity, increasing flow time to 55 seconds. In Fig. 3 the effect of SF in the compressive strength is shown. Lower additions of SF (5% and 10%) decrease the long-term strength in comparison to the reference grout, even after 56 days curing. However, with 15% SF, after 28 days, the strength reaches higher values than in the grout without SF as addition [12] .

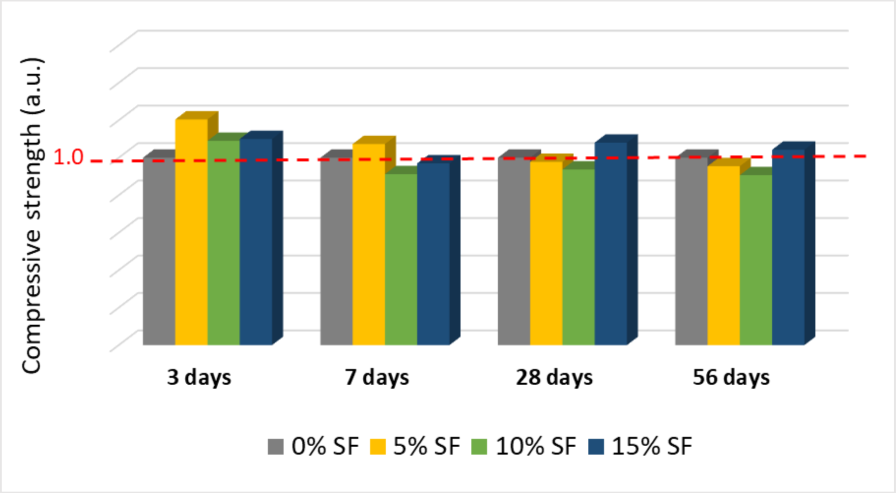

Fig. 3. Effect of the use of silica fume on compressive strength. Adapted from [12].

Fine aggregates can also affect the properties of grouts depending on the mineralogical composition, particle size distribution, geometrical parameters, apparent weight, surface texture. For grouting is important to use fine aggregates in order to avoid the formation of plugs that impair the penetrability [54]. Normally, the maximum particle size used is up to 2 mm. Lim et al. [55] studied the influence of sand grading on strength and flow. Grouts with three different sand gradings (≤ 1.18 mm, ≤ 0.90 mm and ≤ 0.60 mm) were prepared. The w/c ratio varied from 0.61 to 0.67 and the cement/sand (c/s) ratio was equal to 1. The authors reported that, for any sand gradation, the increase of w/c decreased the flow time and the compressive strength. In addition, by setting the w/c at 0.61 for any sand gradation, the efflux time was always the same. Also, the finer sand (≤ 0.6 mm) in grout with w/c = 0.67, yielded the highest results of compressive and flexural strength.

Chemical admixtures also change the fresh and the hardened state properties of grouts. They are classified according to their function: water-reducers, retarders, viscosity-modifiers, air-entrainers, shrinkage-reducers. Water-reducing admixtures (WRA), also known as superplasticizers (SP) reduce the water content while maintaining fresh performance. Examples of WRA are lignosulphonates, casein, polynaphthalene sulfonates (PNS), polymelamine sulfonates (PMS), vinyl copolymers, polycarboxylates and acrylic copolymers [56,57]. They also help to minimize flocculation in microfine cement-based grouts [58]. Cement-based grouts (w/b from 0.4 to 0.8) for radioactive waste isolation were prepared with 30% of cement replaced by FA (by weight), polypropylene (PP) fibers and PNS-based SP [59]. SP additions enhanced the flowability and viscosity but worsened the bleeding. The use of SP combined with PP fiber enhanced the compressive strength, increased the efflux time, setting time and bleeding for any w/b ratio. The authors explain that worsening of bleeding is due to the bleeding paths created by the hydrophobic fibers and by the retarding effect caused by SP on cement reactions. The authors also observed a decrease in compressive strength in grouts with only PP fiber. They justified that reduction was caused by the "redistribution of the void structure and the presence of weak interfacial bonds between the fiber and the fly ash grains" [59].

The admixtures that modify the cement hydration rate will mainly change the setting time and strength development depending on the type of admixture. The most common accelerators are inorganic salts (calcium and sodium hydroxide, potassium carbonate, sodium fluoride and sodium aluminates), water glass (sodium silicate solution) and ethanolamine [60–62]. Lignosulphonate-based admixture is on its hand a well-known retarding agent that also has water-reducing effect. In addition, hydroxycarboxylic acids, inorganic compounds (those with zinc, tin, borate, or phosphate) and sugar derivatives also retard cement hydration [56,63].

Viscosity-modifying admixtures (VMA), also known as water-retaining (WR) or anti-washout admixture (AWA), are used to enhance stability and cohesion. The most commonly used VMAs are natural polymers (welan gum, xanthan gum, alginates), semi-synthetic polymers (cellulose-ether derivatives, alginates derivatives) and synthetic polymers (polyethylene oxide, polyvinyl

alcohol) [56]. Grouts intended for underwater sealing of cracks, offshore structures and pre-stressed ducts usually contain this type of admixture [64,65]. Combined use of SP with VMA in high-fluid grouts is instrumental in increasing the stability of the mixtures avoiding the segregation and bleeding promoted by the SP. Saric-Coric et al [66] studied the interaction between a cellulose-based VMA with two different types of high-range water reducers (HRWR) based on sulfonates, PNS and a PMS. Grouts (w/c = 0.4) with PNS presented better dispersant efficiency than grouts with PMS. After 1 hour of preparation, the grout with PNS showed a reduction of 9% in the mini slump flow diameter, while the reduction obtained with PMS was around 36%. The authors also reported that addition of a cellulose-based VMA increased the HRWR demands between 10 and 40% (to achieve the same fluidity of the grouts with PNS/PMS and without VMA). The VMA did not change the consistency but reduced the bleeding (for both HRWR) and increased the yield value and plastic viscosity [66].
Fig. 4 [67] shows the effects on consistency by changing the VMA type and w/c ratio. From the figure, it is observed that increasing hydroxyethyl cellulose (HEC), bleeding decreased and yield stress and plastic viscosity increased. Even with increasing w/c, HEC behaved the same way. Similar response was observed when increasing the amount of welan gum (WG). However, a higher sensitivity to increase of w/c is observed.

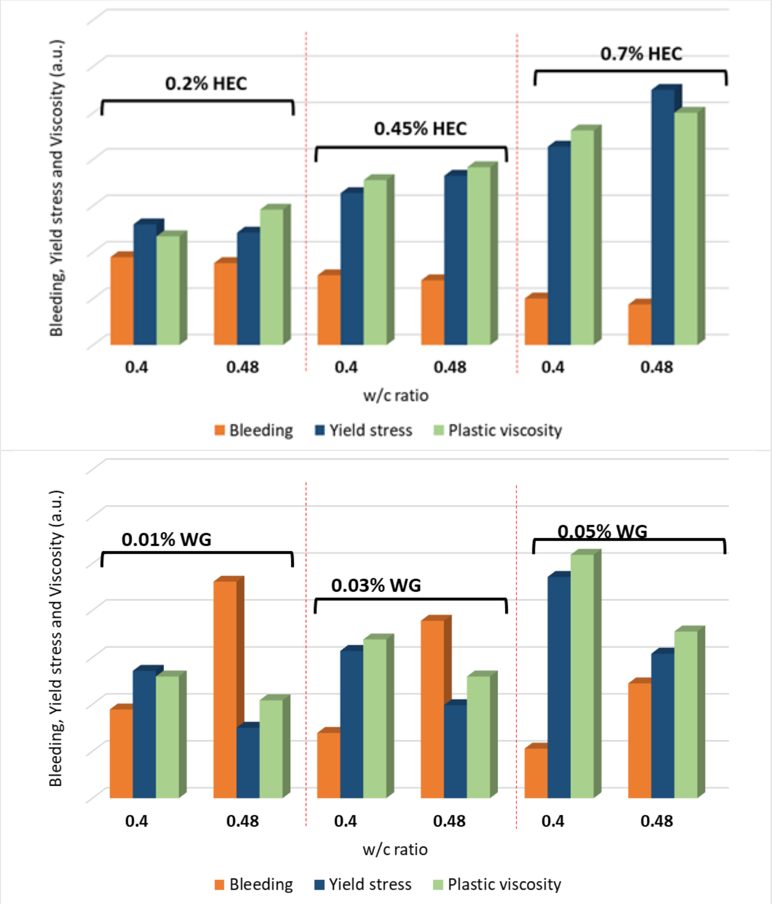

Fig. 4. Effect of VMAs (liquid hydroxyethyl cellulose (HEC) and welan gum (WG)) on bleeding, yield stress and plastic viscosity. Adapted from [67].

Shrinkage-reducing admixtures (SRA) are considered an important chemical additive in the design and production of highly fluid grouts as they delay water absorption [24]. SRA is a surfactant that adsorbs on the water-air interface of pore solution of cementitious materials and on the liquid-vapor interface of clinker, reducing the interfacial energy and the surface tension. Hence, it increases the dispersion of cement particles [24,56]. A shrinkage mitigation study was carried out by adding 1% and 2% (by weight of cement) of glycol-based SRA in a high-performance grout (HG) to be used in post-tensioned concrete structures [26]. The HG (w/b =

0.3) consisted of a binder composed by 80% OPC, 10% zirconium SF and 10% FA The SRA slightly increased the flowability compared to the grout without SRA: the flow table measurements of grouts containing 0%, 1% and 2% of SRA were 130 mm, 135 mm and 140 mm respectively. The compressive strength was also higher than for the reference grout. At 7 days, no significant differences were observed for specimens with and without SRA (strength around 30 MPa). At 28 days, the specimens with 2% of SRA achieved the highest strength of 60 MPa, while the strength of specimens with 1% and 0% of SRA was nearly 45 MPa. Regarding "free shrinkage" (considered by the authors as the sum of autogenous and drying shrinkage), the specimens with SRA exhibited lower shrinkage strains values than the specimen without SRA. At all ages (total of 40 days), there was a decrease in shrinkage with the increase in SRA: the specimen with 2% of SRA showed -636 µƐ which was respectively 56% and 21% lower than those of 0% and 1% SRA.

4. PROPERTIES OF CEMENTITIOUS GROUTS

The properties of the grouts which have to be assessed depend on the grouting process and application. For example, for soil stabilization, the grout has to reduce voids in the soil and to increase the load capacity. As the injection is usually done under high pressure, it is important to evaluate its consistency and rheology. In tunnelling works, grout should set early, thus, it is equally important to assess the setting time as well as resistance to chemical attack or erosion by water seepage [68].

As known, porosity is an inherent characteristic of cementitious materials that directly influences permeability [69]. Water permeation causes damage to the structure due to the interaction of dissolved ionic species (chloride, sulphates and carbonates) with the matrix. The reduction of the w/c ratio leads to a reduction in the total porosity but it also interferes in fluidity. As durability is a result of the interaction of the grout with the environment, a high permeability will negatively affect the durability [70]. For better durability, the w/c ratio should be as low as possible. However, low w/c ratio may decrease the fluidity affecting the grout injectability. If a specific application requires higher w/c ratios, the permeability can be reduced by adding permeability reducing admixtures [71–75].

Usually, workability, consistency and stability are the main properties studied in the fresh state, while the hardened state is often characterized by compressive strength, shrinkage and injectability [30]. Environmental conditions of the site should always be considered as they may change the performance of the grout. For example, the temperature (not only the environmental but also the grout temperature) changes setting time, rheology, injectability and stability [41,76].

Table 2 shows some properties that change according to the grout composition and application.

Table 2 - Summary of the main fresh and hardened state properties of grouts

Mix composition	**Application**	**w/b ratio**	**Flow time (s)**	**Mini-slump (cm)**	**Setting time (initial)**	**Setting time (final)**	**Bleeding rate (%)**	**Plastic viscosity (Pa.s)**	**Yield stress (Pa)**	**Compressive strength (MPa)**	**Flexural strength (MPa)**	**REF**
OPC 53 Grade, SF, Class F FA, fine sand, polycarboxylate ether-based SP	Grouting for precast construction and ground stabilization	0.25-0.8	0.8-45	15	4-10 h	18-25.5 h	0-3	0-0.2	4 - 15	20-35 (7d) 35-55 (28d/56d)	4-10 (28d); 5-12 (56d)	[17]-
microfine OPC, colloidal nanosilica, microfine FA, polycarboxylate-based SP	not described	1.0-2.0	31.73-38.21	33.4-37.5	4.2-8.3 h	8.6-13.5	1.3-6.2	0.0141-0.0379	1.07-2.73	5.0-14.0 (28d)	2.0-4.0 (28d)	[18]-
OPC CEM I 42.5R, ladle furnace slag and blast furnace slag	not described	1.0	8.5-9.0	-	-	-	-	-	-	1.02-6.29 (7d) 2.05-11.41 (28d) 1.98-16.90 (90d)	0.51-2.45 (7d) 1.16-3.5 (28d) 1.29-4.63 (90d)	[27]
OPC (ASTM Type I), SF, VMA, polycarboxylate ether-based high-range water reducer	Not described	0.35-0.48	19-22.5	22-24	-	-	0.017-0.92	4.6-23.3	6.7-28.7	28.3-62.3 (28d)	-	[67]
OPC CEM I 42.5 N, nanosilica, Type F polycarboxylic acid-based SP	Not described	-	42-68	13.4-19.6	-	-	-	0.029-0.419	0.061-0.103	34.0-61.2 (1d) 53.4-99.8 (3d) 92.3-113.0 (7d) 124.0-142.7 (28d)	-	[77]-
OPC CEM I 32.5 N, polynaphthalene-based SP, polycarboxylate ether-based SP	Not described	0.33-0.5	-	-	-	6.5-22	1.2-5.6	-	0-58	11.5-26 (3d); 16.4-45 (7d); 22.5-58 (28d) 31-41 (28d)	-	[78]-
cement, ground granulated blast furnace slag, steel fiber, naphthalene-based and polycarboxylate-based SP	Grouting for prestressed concrete structures	0.45-0.75	22.5-50.22	-	5.50-8.15	-	0.6-4.69	-	-	-	5.19-11 (28d)	[8]

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Mix Composition	**Application**	**w/b or ratio**	**Flow time (s)**	**Mini-slump (cm)**	**Setting time (initial)**	**Setting time (final)**	**Bleeding rate (%)**	**Plastic viscosisty (Pa.s)**	**Yield stress (Pa)**	**Compressive strength (MPa)**	**Flexural strength (MPa)**	**REF**
OPC CEM I 42.5R, polycarboxylate-based SP	Grouting for sealing of concrete cracks	0.5	-	-	-	-	-	-	-	51.5-63.1 (28d)	7.4-10.4 (28d)	[75]
OPC (ASTM Type I), granulated blast-furnace slag, Class C FA, polysaccharide-based anti-washout agent, polynaphthalene-based SP	Grouting for water rich and broken rock stratum	0.6-1.2	30-60	17.5-36.0	14.8-17.5h	24.6-29.7h	-	-	-	7-16 (28d) 7-20 (91d)	0.5-2.25 (28d) 1.0-2.4 (91d)	[79]
OPC Type 10, Class F and Class C FA, melamine formaldehyde condensate-based SP, polysaccharide-based anti washout agent	Not described	0.4-1.3	30-140	-	5-20	7.5-25	0-40%	-	-	7.5-37.5 (28d) 12.5-42.5 (91d) 6-16 (28d) 53.4-99.8 (3d) 92.3-113.0 (7d) 124.0-142.7 (28d)	-	[80]
OPC CEM I-42.5R, natural zeolite, polycarboxylic ether-based SP, VMA (welan gum)	Not described	0.6	13.25-66.35	6.8-16.8	-	7.9-13.7	-	0.0299-0.2693	3.03-19.43	-	-	[81]
Type 10 CSA-CAN A5 cement, cellulose-based VMA, polynaphtalene sulfonate and polymelamine sulfonate high-range water reducers	Not described	0.4	39-225	7.7-14.5	7.1-12.15	8.5-14.0	0.06-0.3	0.06-0.16	0.7-24.9	26-32 (7d) 31-41 (28d)	-	[66]
OPC Type II, class F FA, ground granulated blast furnace slag	Mansory grouts	0.668-0.972	-	20.3-28.0	-	-	-	-	-	4.8-13.6 (7d) 4.5-24.1 (14d) 7.1-26.9 (28d) 9.2-35.6 (42d) 8.9-36.0 (56d) 20.8-41.9 (90d)	-	[82]

4.1 WORKABILITY

Workability is a term used to indicate the fresh state performance of cementitious materials in which the mixture must be cohesive and no segregation between different particle sizes should be observed [83,84]. It encompasses two main aspects, namely fluidity and cohesion, and is affected by water content, aggregate type, aggregate/cement ratio, chemical-physical characteristics of cement, presence of admixtures, temperature (environmental conditions), among others [84].

As the requirements for the grout workability change according to the field of application, it is not easy to establish a value or a range for the different parameters employed to measure and quantify workability. Flowability, compactability and stability qualitatively describe this property, although it can also be described quantitatively by measurements of viscosity, flow time and flow table spread [83,85].

Chemical admixtures (especially SP) and SCM are used to improve workability [39,79,86,87]. Erdem et al. [88] studied how workability of cementitious grouts (w/b ratio of 0.4 and 0.5) is affected by two types of SP (polycarboxylic-ether type and naphthalene formaldehyde sulfonic acid type), limestone and FA. They concluded that workability is closely related to consistency (flow measurements) and stability (bleeding results). Moreover, the type of SP was the variable that most affected the robustness of the workability performance, followed by the influence of the w/b ratio. Kim et al. [20] studied the changes on workability replacing OPC (amounts of 10, 20 and 30% by weight) by two types of FA (ground fly ash (GFA) and raw fly ash (RFA)). They found that the flow of the grout increased with the decrease of RFA, being not affected by GFA. In addition, all samples were quite stable because no bleeding occurred in all the mix conditions.

Setting time is also related to the grout workability. The initial setting time is the time when the workability and plasticity of the grout begins to decrease. A fast increase in viscosity and yield stress rapidly decreases the penetration capacity of the grout. In that situation, it is necessary to increase the injection pressure, making filtration more likely to occur. For that reason, it is preferable that grouts have longer initial setting time, ensuring better penetrability. Cementitious grouts that require fast setting are modified with chemical admixtures. These grouts are typically more viscous (i.e., exhibit low workability and fluidity) and require high pressure pumping systems [89]. Similar to other early age/fresh state properties, setting time is influenced by cement type, w/c and s/c ratios, by SCM additions and additives. Increasing w/c, setting time increase [79]. Shannag [12] demonstrated that SF in grouts (with Type-I OPC) decreased the initial setting time, while FA (grouts with Type-II OPC) increased it [90]. For microfine cement-based grouts, the initial and final setting time increased up to 5 hours, (starting from 8h) when FA dosage was up to 40% [18].

4.2 BLEEDING

According to He et al. [70] bleeding shows the stability of the grout indicating whether the material has sufficient cohesion and water retention capacity to prevent stratification and segregation. As a matter of fact, when the mixing water flows to the top, solid particles settle causing sedimentation. The bleeding capacity is expressed as the relation between the volume of water released and the initial volume of the grout [86,91]. Lombardi [92] affirms that a stable grout should not present more than 5% sedimentation.

Bleeding is also related to the durability and permeability. Excessive bleeding increases the porosity of the grout resulting in loss of strength [93,94]. Additionally, it can affect the grout performance in ground treatment, as the grouted site can present partial filling due to uneven settlements [95]. In tunneling operations, bleeding can cause structural failures because the partial filling can favor the appearance of preferential paths for water flow [5].

Different variables can influence the bleeding rate, the w/c being the main one [22,67,93]. Tests varying the w/c ratio, between 0.6 and 1.2, demonstrated that the increase of bleeding is directly proportional to the increment of w/c [42]. The same behavior has been reported by [80]. Some practices can be applied to stabilize the grout mixture and minimize the bleeding effect, including addition of SCMs, chemical admixtures and changes in solids fractions [75]. Fig. 5 presents the effects of replacing cement by slags studied by Perez-Garcia et al.[27]. They reported that additions of 30, 40 and 50% (% in weight) of different types of slags (unprocessed ladle furnace slag (LFS) and GGBS) in cementitious grout (w/c =1) decreased bleeding (for any slag type and percentage tested) without affecting significantly the flow time. Sha et al. [79] verified that additions of 30% (in weight) of GGBE combined with Class C FA (40% in weight) in a cement-based grout (water/solid (w/s) ratio between 0.6 and 1.2) reduced the bleeding ratio. Gopinathan et al. [93] investigated ultra-fine slags (UFS) additions in a grout (w/c = 0.3, 0.35 and 0.4) with two types of SP (sulfonated naphthalene formaldehyde (SNF) type and poly carboxylate ether (PCE) type). The results showed that the bleeding of the mixture with w/c = 0.4 and 10% (by weight of cement) of UFS was reduced to zero for any SNF dosages (from 0.4 to 1.2% by weight of cement). The same behavior was seen for the mixtures containing w/c = 0.35, 15% (by weight of cement) of UFS and PCE dosages of 0.6 and 0.85% respectively.

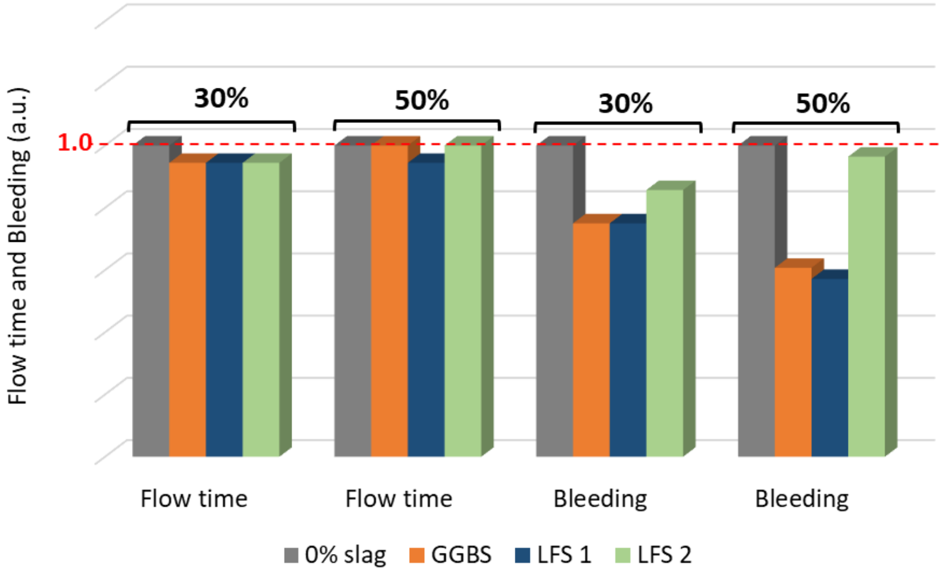

Fig. 5. Effect of replacing OPC with 30% and 50% (% in weight) of slags (LFS and GGBS) on flow time and bleeding. Adapted from [27]

4.3 CONSISTENCY

Consistency reflects the grout plasticity which is important for the injection process [70]. According to technical standards, the consistency can be reported as fluid, plastic or flowable and it is often measured by flow tests.

Fluid consistency is defined as " (1) the consistency at which a grout will form a nearly level surface without vibration or rodding and (2) the consistency of a grout that has an efflux time of less than 30 seconds through an ASTM C939 [96] flow cone" [91]. Additionally, flowable consistency is defined as " (1) the consistency at which a grout will form a nearly level surface when lightly rodded and (2) the flowable consistency has a flow of 125 to 145 percent after five drops of the flow table described in ASTM C230 [97]" [91,98]. Lastly, plastic consistency is “(1) the consistency at which a mixture subjected to a constant shearing stress undergoes increasing deformation without rupture” [91]and (2) the plastic consistency has a flow of 100 to 125 percent after five drops of flow table [98].

Similar to other cementitious materials, the grout consistency is affected by the particle size distribution of solids and the w/c ratio [42,79]. Mineral additions and chemical admixtures are widely used to control the consistency. Krishnamoorthy et al. [87] investigated how the required SP dosage (sulphonated naphthalene formaldehyde condensate SP) changes depending on the replacement of OPC by SCM (Class C FA (20, 30 and 40%), GGBS (20, 30 and 40%) and SF (5 and 10%)). All % by weight of cementitious material. The time of efflux of the grout (w/c ratio between 0.25 and 0.40) was measured through Marsh cone apparatus. They reported that SP dosage varied according to the type of SCM and the dosage increased with the amount of cement replaced by SCM. For the mixtures with FA or GGBS, only increasing the amount of water was enough to reduce de SP dosage up to 75%, to achieve the desired fluidity.

Zhang et al. [18] studied the fluidity and spreading ability of microfine-cement-based grout (w/s = 1.2) containing additions of microfine fly ash (MFA). Increasing MFA contents (0, 10, 20, 30 and 40% by weight of cement), the flow time decreased and the mini-slump diameter increased. This behavior is similar to other studies that also observed better flow due to the addition of FA [27,99]. High-volume FA grouts (w/b = 0.4-1.3) did not show significant changes in fluidity. For w/c ratio between 0.4 and 0.65, additions of FA (cement replacement by weight) between 50% and 75% reduced the flow time up to 80%. For w/c ratio of 0.8, 1.0 and 1.3, the flow was constant, regardless of any change in water and FA contents [80].

Grout containing natural zeolite as VMA (w/c = 0.6) exhibited less fluidity when compared to a grout with WG-type VMA (additions of 0.05, 0.10 and 0.15% by weight of cement) [81]. In this work, measurements of flow diameter and Marsh cone flow time revealed that an increase of zeolite additions (20, 30 and 40% by weight of cement) reduces the grout flowability due to the higher water retention capacity of the zeolite. For a mixture containing 0.25% of SP, the increase of zeolite addition from 20% to 40%, decreased the flow diameter from 99 mm to 68 mm and the flow time varied from 20.76 seconds to 66.35 seconds, respectively.

4.4 INJECTABILITY

Injectability is also a parameter required to ensure adequate grout performance. According to Miltiadou-Fezans and Tassos [100] injectability is associated with penetrability, fluidity and stability. A grout with great workability (that is to say good flowability, compactability and stability) does not guarantee adequate injection as this process requires a great understanding of fluid mechanics, grouting methods and physical/chemical characteristics of the local to be grouted. Many studies in the literature test different methods to predict penetrability, simulating the diffusion flow in various porous media, aiming to establish a relation between the grout composition (w/c ratio, rheology, granulometric distribution, fluidity, stability), site characteristics (granulometric grading, ambient characteristics, voids volume, among others), and injection pressure [101–103]. Knowing the penetrability, it is possible to indicate if the granulometric distribution of the grout is suitable for the smaller volume/widths of voids/cracks.

Jorne et al [104] demonstrated how grout injection varies according to porous media, void volume, water content and granulometric distribution. The authors reproduced the injection of a lime-based grout (w/b = 0.5) in different porous media (dry and pre-wet) varying grain size ranges of limestone sands and crushed brick. They concluded that soils formed mainly by fine particles absorb more water, which decreases injectability. The water absorption was also elevated in dry porous media which can stimulate grout segregation.

As discussed previously fluidity and stability also contribute to the success of injectability. Fluidity is not only related to flow time but also should be expressed by rheological measurements which is strongly influenced by the w/c ratio and by as powder fineness and particle size distributions [17,95,105,106]. Rheology can be described by different analytical models and the cementitious grout is generally characterized as a non-newtonian fluid. The rheological behavior is described by two parameters: yield stress (τ_0) and plastic viscosity (μ). Both parameters define the Bingham constitutive equation, employed to characterize the behavior of the grout (Eq. 1), where τ is the shear stress (Pa) and $\dot{y}$ is the shear rate (s^{-1}) [107].

$$\tau = \tau_0 + \mu\dot{\gamma} \quad (1)$$

Dhir et al [3] explains that the stability of the mixture is directly proportional to its viscosity and inversely proportional to its fluidity. The suspension is stable when its plastic viscosity is high. However, the fluidity (which is essential for grout) will only be high when the yield value and plastic viscosity are both low. An option to increase the penetrability of the grout is to increase the w/c ratio. However, mixtures tend to segregate with increasing the amount of water. It is also important to know that as the w/c ratio increases, changes in viscosity (after reaching its critical value) are not easily measured. Therefore, to produce a low viscosity grout is much more appropriate to maintain low w/c ratio and select better types of cement and chemical additives, rather than just increasing the amount of water [108].

Correlations between yield stresses, unconfined compressive strength (UCS) and w/c ratio were studied in cement-zeolite blended specimens. The authors revealed that zeolite additions of more than 30% and an increase in w/c ratio led to a decrease in yield-stress. The increase in pressure (from 100 kPa to 500 kPa) increases the yield stress which is justified by the volume change during grout consolidation [28]. Güllü et al. [109] found that FA additions (0-100%) in cement grouts (w/c = 0.75 to 1.5) decreases the yield stress and, the apparent and plastic viscosities.

Liu et al. [106] studied the influence of clay, sand and setting-time modifier on shear stress, shear rate and viscosity of cementitious grouts. The results indicate that the yield stress increased with the increase of clay dosage for w/s = 0.6 and 1. Viscosity slightly varied with low clay dosages (≤ 10% by weight of cement) and rapidly increased with dosage of 30% and 50% (by weight of cement), remaining stable after 40 min. The mixtures containing clay, cement, sand and modifier showed that a suitable content of sand can improve cohesion. With the same modifier dosage, the yield stress increased when the sand/cement (s/c) ratio was increased up to 1.5. However, it decreased for s/c =2.

Sonebi et al. [22] concluded that additions of GGBS and polycarboxylic acid-based SP decreased the yield stress and plastic viscosity, while adding nanosilica in cement-based grouts increased both [77]. The addition of polynaphthalene sulfonic-based SP (from 0.2% to 1.2%) in grout used for prestressing works, decreased the yield stress and increased the sedimentation with increasing SP dosages [110].

The yield stress and plastic viscosity measured at different temperatures and resting times show how the initial shear stress, equilibrium viscosity and even the flocculation rate can vary under these conditions. It is important to understand the Brownian motion of the particles, as the interactions between them can weaken/strengthen and, in this way, favor (or not) agglomeration, flocculation and loss of workability [111].

Penetrability of grouts is also affected by the extent of the filtration. Filtration phenomenon can occur during grouting as the particles of water and cement/fine aggregates gradually separate from the grout flow (only water penetrates in spaces/cracks) and block the flow path, increasing the penetration resistance of the grout[112]. Adequate water retentivity is essential for grout materials, as otherwise it can decrease fluidity changing the yield stress and viscosity [67,113]. The water retentivity can be measured by several instruments, such as sand column, pressure chamber, filter pump, PenetraCone, NES method, among others [114–116].

The factors that influence filtration stability are w/c ratio, grout pressure, maximum grain size and grain size distribution. Grouts with high w/c ratio tend to have less problems with filtration. However, a high w/c increase porosity affecting the durability. Regarding the grain size, for a good penetration result, it is recommended that the maximum particle size of the suspension should be at maximum one third of the aperture through which the mixture has to be grouted [105]. If the grout had only a single grain size/shape, it would easily penetrate in any fracture/soils. However, this is an illusory scenario, as grouts are not monodisperse systems. Thus, it is essential to assess the best grain size and the particle distribution. Bohloli et al. [117] showed that filtration stability depends on the grain size. They evaluated (through filter press) grouts composed by water and cement (three cement type were tested; D_{95} of the cements ranged from 17 to 25 µm). The cement with D_{95} = 17 µm had the best filtration stability, while cement with D_{95} = 18 µm exhibited the lowest. Despite the D_{95} values of both cements are close, the grains of cement (with D_{95} = 18 µm) agglomerated, forming particles ≥ 75 µm (clogging the filter).

The success of grouting also depends on the magnitude of the pressure applied for injection [118–120]. To fill all spaces, a minimum pressure is required to overcome the shear resistance between the grout flow and the walls of the space to be filled. By increasing the injection pressure, the grout rheology can change and filtration can decrease, improving injectability [121]. However, higher pressures are recommended up to a certain limit. Although with high pressure the cracks expand (facilitating the grout flow), high pressure can replicate the cracks, deform them and hinder penetrability [122]. Moreover, during the injection, high grouting pressure might cause segregation or even favor the agglomeration of finer particles due to filtration tendency.

4.5 MECHANICAL PROPERTIES

Similar to other cementitious materials, the mechanical properties of the grout are characterized by compressive and flexural strengths. The use of OPC is advantageous, as the grout develops higher strength in early ages. The effect of different SP on strength was studied by [78]. Additions (from 0.5 up to 3.5% by cement weight) of polycarboxylate-and polynaphthalene-based SP on cement-based grout (w/c = 0.33, 0.4 and 0.5) showed that the strength increased over time for both SPs. The increase caused by PCE was slightly higher compared to the polynaphthalene type, especially for grouts with a w/c ratio of 0.4 and 0.5. Regarding the increase in the amount of SP (for the same w/c), in general, there was no increase in compressive strength with the increase of SP, for all ages tested, the strengths were very similar.

Saric-Coric et al. studied grouts (w/c = 0.4) containing cellulose-based VMA and two types of HRWR (PNS and PMS). The results indicated that grouts containing VMA exhibited lower compressive strength (at 7 and 28 days) than those without VMA. Furthermore, PMS additions increased the compressive strength more than those with PNS additions [66].

Early strength improvement in microfine-cement-based grout containing colloidal nanosilica (NS) was verified by Zhang et al. [18]. The grout with 2% (by cement weight) of NS required 5.8 h to reach a strength value of 0.45 MPa whereas the one with 1% (by cement weight) of NS took 6.5h. Another study [123] evaluated that the addition of 16% (cement replacement by weight) of SF improved early (1d and 3d) and long-term (90d) strength, while the same FA dosage improved the compressive strength over longer periods (90d). Fonseca et al. [82] observed that the replacement of OPC in grouts (w/c = 0.795) by large amounts of FA (40, 50 and 60% by weight of cement) reduced the compressive strength. Fig. 6 shows that, over time, the strength increased for any percentage of FA; however, all mixtures with FA presented strength values below the reference (grout without FA). The low strength values are explained by the authors due to the different aggregates used (since the grouts are slightly sensitive to aggregate), non-parallel caping and misaligned endplates (as they used an alternative casting method) and/or FA flocculation.

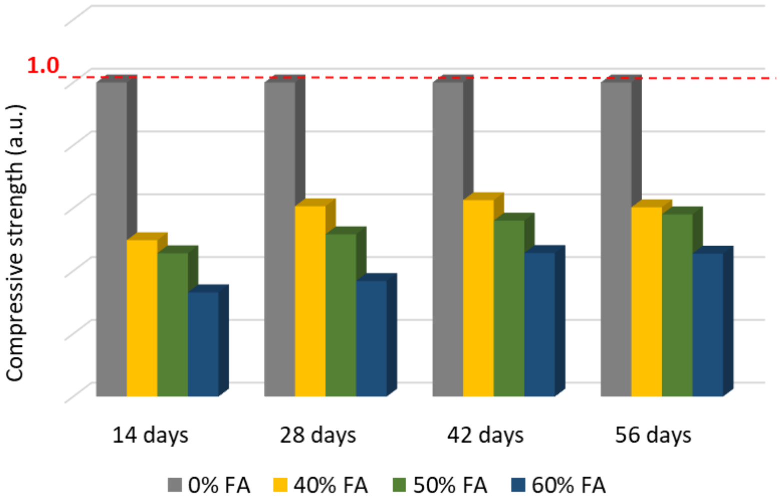

Fig. 6. Effect of replacing OPC with FA on compressive strength. Adapted from [82]

4.6 SHRINKAGE

All cementitious materials undergo physical and chemical changes that lead to a volume reduction process known as shrinkage. It starts with volume reduction during the cement hydration and goes on all along hardening and drying processes, resulting in the formation of cracks [124,125] if the corresponding deformation is restrained and the restraint generates stresses higher than the material tensile strength. Shrinkage is influenced by curing conditions, type and content of cement, w/c ratio, type and size of aggregate, admixture additions.

An excessive shrinkage in grouts will facilitate the entry of harmful substances (e.g., chloride and carbonate ions) impairing its durability [24]. Excessive bleeding in very fluid grout (with high w/c ratio) promotes plastic shrinkage due to the gradual evaporation of the bleed water layer [126]. Cementitious grout designed for connections (with low w/s ratio) can develop internal tensile stresses due to restrained early-age autogenous shrinkage. The grout can crack or lose bond between the grout/concrete interface [127]. The strategies to avoid or to reduce the shrinkage include reduction of cement content, use of mineral additions and fibers, use of shrinkage reducing admixtures (SRA), aggregate grinding, control of time and curing conditions. De La

Varga et al. [128] evaluated the use of lightweight aggregates (LWA) as an internal curing agent in cementitious grouts and conclude that LWA minimizes autogenous and drying shrinkage, thanks to its ability to supply pre-absorbed water to compensate its consumption. Shamsuddoha et al. [99] studied how SCM additions (microsilica, metakaolin and FA) can cause both linear and volume shrinkage in grouts designed for structural repair. In this study, linear shrinkage was determined conforming EN 12617 standard and volume shrinkage was determined by a cone test method. The authors identified that the volume shrinkage increased with a higher content of FA and microsilica, while additions of metakaolin decreased the shrinkage. Linear shrinkage increased with the increasing of FA content. Additionally, it was not affected by microsilica additions, but decreased with increasing the metakaolin content. Drying shrinkage increases with increasing w/c ratio and additions of FA proved to be advantageous in decreasing drying shrinkage [80]. Although the main functionality of permeability-reducing admixtures (PRA) is to make concrete less permeable, they have been applied to mitigate shrinkage in concrete. Commercially known also as crystalline admixtures, PRA can modify the early-age properties of a cementitious matrix as they easily react with moisture forming crystals that block pores and cracks [129].

5. CEMENTITITOUS GROUTS WITH SMART FUNCTIONALITIES

Over the years, the construction sector has focused on increasing durability to surpass the inherent deterioration of structures. In this context, the self-healing ability has inspired the design of smart cementitious systems, as they can repair a damage/defect by themselves, prolong the service life of structural applications and reduce maintenance costs. In the construction sector, and focusing on cement-based construction materials, self-healing mechanisms are divided in two categories: autogeneous, in which the healing process occurs due to carbonation and continuous hydration of unreacted cement grains, mainly in low w/c ratio composites [130] and automonous that "depends on the incorporation of unconventional engineered additions into the matrix to provide self-healing function" [131].

Numerous reviews on the subject were published [131–135], focusing on healing agents, self-healing mechanisms and methods to evaluate the healing efficiency. Table 3 presents some self-healing approaches explored in cementitious materials.

Table 3 – Summary of self-healing approaches: healing materials, crack width and comparison between autogenous and autonomous self-healing technologies

	Autogenous self-healing	**Autonomous self-healing**				
Healing Technology	Incorporating mineral admixtures, fibers, nanofillers, curing agent	Based on mineral admixtures	Microbial technology	Capsule technology	Vascular technology	Based on polymers
Materials/Healing Agent	SCM, Polyethylene fiber, Polypropylene fiber, carbon nanotube	crystalline admixtures and expansive agents (e.g., calcium sulfoaluminate, sodium aluminum silicate hydroxide, montmorillonite clay)	bacteria	Inorganic and organic compounds (sodium silicate solutions, sulfonates, benzoates, magnesium oxides, bentonite), and crosslinking polymers	crosslinking polymers (polyurethane, epoxy, polymethylmethacrylate, cyanoacrylate)	superabsorbent polymers (SAPs) oil sorbent shape memory materials
Self-healing crack width	Up to 150 μm	Up to 300 μm	up to 800 μm	up to 300 μm	up to 500 μm	up to 200 μm
Advantages	- good healing capability - good compatibility with the matrix	fast self-healing of cracks	- environmentally friendly - natural healing mechanism	- on-demand healing agent release - good efficacy in healing cycles	-on-demand curing agent release - good efficacy in healing cycles	- macro cracks can be treated - high recovery rate of mechanical properties – shape memory materials - Good efficacy in repeated healing cycles
Disadvantages	- Low effectiveness in healing cycles - Uncontrolled expansion may occur	- Mineral admixtures are consumed before cracking (If added directly into the matrix they will react with water) - Lack of control of expansion by expansive additive (uncontrolled expansion may cause damage) - Constant availability of water in the cracks	- Bacteria cannot be added directly to the matrix (need to be protected to prolong their lifetime) - Change of mechanical properties - Concerns about effectiveness in healing cycles	- Difficulty in preparing the capsules and limited amount of healing agent (only for microcapsules) - Concern on bonding between capsules and matrix - With a bicomponent resin, the healing efficiency may be low because the availability/release of both components cannot be controlled not occurring the polymerization reaction - Change in mechanical properties - Resistance of capsules (may break during mixing)	- Difficulty injecting the healing agent - Concern about bonding between capsules and matrix - Change in mechanical properties - Fragile material (may break during application)	- Low effectiveness in dry places - SAP does not form the barrier because it does not swell - High cost - Sensitive to increased temperature (early stimulation of the healing process) – shape memory materials
REF	[134]	[72,136–140]	[141–143]	[144,145]	[146,147]	[148]

A search in the Scopus database of the keyword “self-healing” in combination with “concrete/mortar/grout” reveals that the interest in this technology has been increasing (Fig. 7) over the last decade. From 2013 onwards, there has been an exponential growth in self-healing concrete. The same interest in self-repairing mortars is also evident, although the increase in the number of publications is not as impressive as concrete’s. This growth is a result of the cement industries concerns toward sustainability.

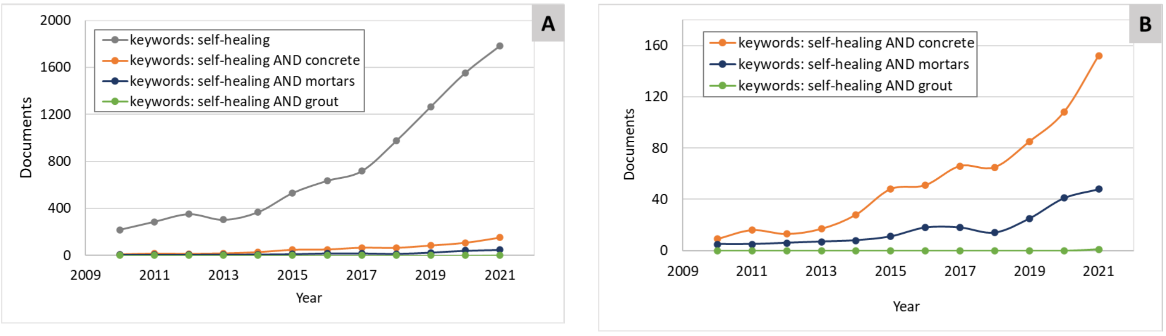

Fig. 7. A: Number of publications related to self-healing: B: Zoom-in on the documents published on self-healing cementitious materials. Source: Scopus® database.

Despite this great advance in both materials, the research on self-healing grouts is low. Table 4 presents the number of published documents related to self-healing of cement-based materials. In the last 10 years, the number of documents related to concrete and mortars is up to 800% greater than those about grout, which indicates a research gap in grouting field. Indeed, the number of papers on self-healing grout is practically zero (only one paper in 2021 was found with these keywords). Despite the low number of publications, this review will discuss self-repair methods that have already been applied to grouts. In view of the few studies found on the subject, in the absence of application of any method in grouts, articles that applied it to concrete and mortar will be discussed.

Table 4 - Number of academic publications in Scopus® database with keywords “self-healing”, “concrete”, “mortars” and “grouts” from 2010 to 2021

	keywords			
Year	**self-healing**	**self-healing AND concrete**	**self-healing AND mortars**	**self-healing AND grout**
2010	219	9	5	0
2011	287	16	5	0
2012	351	13	6	0
2013	303	17	7	0
2014	368	28	8	0
2015	531	48	11	0
2016	635	51	18	0
2017	717	66	18	0
2018	974	65	14	0
2019	1265	85	25	0
2020	1554	108	41	0
2021	1783	152	48	1
Total	**8987**	**658**	**206**	**1**

5.1 AUTOGENOUS AND AUTONOMOUS HEALING IN CEMENTITIOUS GROUTS.

As mentioned, autogenous crack healing is an inherent phenomenon of cement materials, and its main healing mechanisms include (a) hydration of unreacted cement, (b) precipitation of portlandite and (c) formation of calcite [131,136]. All these reactions can occur simultaneously but each of them has different reaction rates. At early ages, the hydration of anhydrous cement grains results in the filling of crack by calcium silicate hydrate (C-S-H) and Portlandite (CH). At later ages, the main mechanism is the formation of calcite [131].

The composition of the matrix and the crack width will influence the healing performance. The presence of water is essential for autogenous healing mechanism. It is a focus point when this technology is used in places with low water saturation [136]. Other strategies are used to overcome the unfeasibility for autogenous healing, e.g., additions of mineral admixtures, polymers, fibers, nanofillers, curing agents and coatings.

Unlike autogenous healing, the autonomous healing has several triggering mechanisms, which means that each of them will require a different condition to promote the healing. The main methods that have been studied are shape memory materials, capsules, vascular networks and bacteria additions.

Crystalline admixtures (CA) are products known mainly as permeability reducer admixtures that can be employed as a stimulator of the autogenous healing capacity. When reacting with water, water-insoluble deposits are formed blocking the cracks [72,149–151]. In mortars, CA was able to close cracks (width of 250–400 μm) and also reduce the water permeability rate[130]. The crystallization process is affected by wet/dry cycles and repeated crack-healing cycles can improve the healing efficiency [152,153]. In concrete, it was reported that additions of CA reduced compressive strength by 7,9% [154] and chloride permeability [73,155].

In grouts, Wang et al. [156] showed that the CA did not change the slump but increased both long-term (90 days) compressive strength and modulus of elasticity. Zeng et al. [16] investigated the performance of a commercial grout (for sealing tunnel leakage) by adding different amounts of CA between 0% - 1.6% (by weight of cement). The authors reported that there was no change in setting time and viscosity for any addition. With 0.8% of CA, the compressive strength was slightly increased. Between 0.8% and 1.2%, the increase in strength was better noticed. Starting at 1.2%, there were no significant changes.

SCM also has self-healing capability [138], but when combined with CA, they improve the self-healing properties even more. Li et al.[157] studied the healing effect of mortars containing CA and GGBS. The self-healing capability was determined by compressive strength recovery, crack closure ability and water absorption. The authors concluded that 1.2% (by cement weight) of CA increased the recovery of mechanical strength. In addition, self-healing was improved with the addition of up to 10% (by cement weight) GGBS. In this case, the strength of the specimens with cracks performed after 28 days (and cured for 56 days after cracking) was higher than those cured for 28 days. Above 10% of GGBS, the self-healing capability decreased. The benefits of GGBS on early age cracks were not very noticeable and the recovery rate was practically the same of the specimens with only CA.

In order to understand how the consumption of portlandite by FA might impact the crystallization process of CA, Wang et al. investigated the combination of FA with CA [158]. For this, they prepared mortar specimens (replacing OPC with 10%, 20% and 30% of FA by weight of cement) with 1.2% of CA, by weight of cement. The addition of CA not only contributed to the increase of compressive strength but even improved the recovery rate of compressive strength. The results also showed that the recovery strength was not improved with high amounts of FA. The authors concluded that FA compete with CA for calcium ions, weakening the crystallization capability of CA.

To improve healing of macro-cracks (width >0.5 mm), using superabsorbent polymers (SAP) with CA is considered a promising option. It has already been shown that materials with only SAP cannot completely heal the cracks [159]. The use of SAP is advantageous because it absorbs water, expands and seal large cracks. So, it is interesting to combine SAP with CA because large cracks can be healed by SAP while small cracks can be repaired by CA. This synergy was demonstrated by Li et al. [160] that studied for mortars the best CA type to obtain a total crack closure. They studied SAP combined with 5 types of CA (citric acid, silicon dioxide, sodium silicate, sodium carbonate and a commercial product) and concluded that citric acid was the most suitable CA to completely close the cracks.

Cao et al. [148] investigated the self-healing performance of a cementitious grout with oil sorbent (contents of 0%, 5% and 10% by grout weight). This absorbent polymer can swell and block cracks. The results of plastic viscosity and yield stress were 24, 28 and 42 mPa·s and 9.2, 9.9 and 10.6 Pa, respectively for the different addition ratios indicated above Thus, the grout flowability decreased with the increase of oil sorbent. Consequently, higher energy for the grout to be pumped is required as the yield stress increased. The authors also found that oil sorbents additions decreased the unconfined compressive strength at any dosage and age tested (28, 56 and 90 days).

As mentioned in Table 3, incorporating microcapsules and vascular networks are options for healing larger cracks. Encapsulation allows the healing agent to be released into the damaged area without suffering from environmental conditions. The release of core material may be time-dependent or triggered by external effects as diffusion, rupture and dissolution.

For a successful repairing effect, is important to know which chemical reactions and interactions are responsible for the healing process. As important as the chemical crosslinkers, is fundamental to understand from triggering process to structural factors, including diameter, wall-thickness, shape, dispersion of the capsules and vascular network pattern.

A capsule-based self-healing method was investigated by Liu et al. [161] who added 0, 1%, 3%, 5% and 8% (by grout weight) of urea/formaldehyde microcapsules (epoxy resin as core material) in a commercial cementitious grout (w/c = 0.13). The results showed that the flow decreased regardless of the number of capsules. The decrease was by 8% for grouts with less than 3% of capsule content. Above 3%, the reduction was up to 45%. Similar reduction was observed in compressive strength measurements at 1, 3 and 28 days for grouts containing more than 3% of microcapsules.

Bacteria have been explored to improve the durability and to remedy cracks of cement-based materials [143]. The microorganisms can be added by several methodologies, such as encapsulation, aggregates impregnation and mixed with water [141,142]. The crack sealing is a result of the precipitation of calcium carbonate ($CaCO_3$) [143]. Joshi et al. [162] evaluated the microbially induced carbonate precipitation (MICP) biobased approach on cementitious grouts to repair cracks in existing concrete structures. In the study, a mixture composed by cement, FA (cement replacement from 10% to 50%) and two bacterial suspension-binder ratios (0.45 and 0.5) was evaluated to repair artificial cracks. The cracks were made with a steel plate of 0.8 mm width and 20 mm depth - in horizontal and vertical positions. The water ingress was measured by sorptivity tests that showed that the bacterial grout treatment was very efficient. The sorptivity coefficient of untreated concrete was 0.03, while the coefficient of specimens with vertical and horizontal cracks were, respectively 0.005 and 0.002.

6. CONCLUSIONS

This work has compiled the relevant topics on the development of cementitious grouts highlighting the main constituent materials, properties and applications. "Grout" describes many types of injectable fluid cementitious materials and their properties vary significantly according to numerous possibilities of mixing design. Therefore, this work has first of all pointed out that there is no rigid pattern of grout behavior. As seen throughout the sections, small differences in formulation (from the granulometry of the aggregate to the excessive amount of SP) result into infinite possibilities of results. From the information gathered the following statements hold about the relationships between grout composition and application-oriented performance, in whose fields efforts have to be done for a better comprehension of the correlation and a likely prescription-to-performance based treatment/funneling of the existing data:

- Cementitious grout is mostly composed by cement, water, sand and additive. Grout mixture is mainly optimized by adjusting its water/cement and cement/solid ratios to achieve the desired performance. It is essential that the grout is fluid enough without losing cohesion and stability. The consistency is mainly affected by the amount of water. Very fluid grouts (with high water/cement ratio) can easily segregate.

- High amounts of water also promote bleeding. High bleeding rate favors sedimentation and increases porosity. Very porous grouts are more susceptible to the entry of aggressive substances and have low compressive strength. Normally, grout stability is ensured by chemical admixtures. However, large amounts can cause a reverse effect, which means that the excessive use not only increases bleeding, but also reduces the mechanical strength and impairs the penetrability.
- Adequate water retentivity is essential, otherwise flowability can decrease, which can promote filtration. The control of rheological behavior is essential for the injectability as the grout must withstand high rates of shear stress without destabilizing. As a grout normally requires high fluidity, the use of viscosity modifiers to enhance stability and cohesion is recurrent.
- Water-reducing admixtures provide workability and can increase the strength. Shrinkage-reducing admixtures are an important chemical additive in very fluid grout as they delay water absorption. Fly ash increases the workability, extends the setting-time and increases its impermeability. Slag additions decrease porosity and increase long-term mechanical properties. Silica fume increases fluidity, early and long-term strength, reduces the viscosity, decreases bleeding and porosity. In high quantities, silica fume can excessively increase the fluidity and impair the workability of the grout.
- Self-healing cementitious materials are designed to heal damage caused by, for example, mechanical stress or aging of the structure, in order to restore the original functionality, extend the life and safety of structures. Several publications reported the healing effect promoted by microencapsulation, mineral admixtures, bacteria, absorbent polymers, among others. Self-healing approach has been further explored in concrete and mortar, but it is not much explored in grouts. In the last 10 years, the number of documents related to cement and mortars is up to 800% greater than those about grout, which indicates a research gap in grouting field. Thus, a lot of research has to be done in this area, mainly focusing on the mechanisms/interactions of the grout matrix and healing agents.

Acknowledgement

The financial support from the ITN-MSCA SMARTINCS project is acknowledged by all the authors. This project has received funding from the European Union's Horizon 2020 research and innovation programme under the Marie Skłodowska-Curie grant agreement No 860006.

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