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Additives and Admixtures in Cement-based Grouts

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Abstract

Additives and admixtures are used in cement and non-cement-based grouts to modify their fluid and set characteristics. The ability to modify all fluid and set characteristics increases the durability, strength and penetrability of grout. Well designed formulations create balanced stable suspension grouts that reduce the cost of any grouting operation through increased grouting effectiveness by minimizing the cohesion and maximizing the penetrability of a grout via proper application of additives and admixtures.

Introduction

The use of a variety of additives and admixtures in microfine and regular cement-based grout formulations has become routine in advanced professional grouting practice. They play an important role in producing more durable grouts with improved rheological and set characteristics than could be obtained using only neat cement suspension grouts.

Neat cement grouts contain two products that are not very compatible: water and cement. Stokes' law governs the sedimentation of the particles; the finer they are, the slower the process. Segregation causes bleed paths to form unless a low water/ cement ratio is used. The resistance against pressure filtration of these grouts is poor, compared to balanced, stable suspension grouts. In rock and structural grouting, this phenomenon causes bleed water migration through the gelling grout. This creates pathways and bleed-pockets in the upper part of the cracks and crevices in which erosion and chemical attack can take place. Especially when grouting in water saturated media, laitance is formed which has no strength and is erodible. In soil grouting, the use of unstable grouts results in poor penetration and an unreliable end product.

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To obtain durable suspension grouts, the calcium hydroxide formed between the chemically resistant, hard ettringite in the cured cement paste, needs to be tied up and also turned into ettringite. The ability to modify viscosity, cohesion, bleed, geltime, pressure filtration, and strength of cement-based grouts is essential to ensure a cost effective and high quality end product. These modifications facilitate multiple grouting passes in soil grouting applications.

The "DNA" defining a modern day balanced suspension grout should be the particle size distribution, resistance against pressure filtration, bleed, initial and final gel and set times, evolution of cohesion and strength, not merely the water/powder ratio.

The properties of cement-based suspension grouts

The properties of cement-based grouts can be classified into two categories:

- Fluid characteristics: cohesion and its evolution with time, thixotropy, viscosity, pressure filtration coefficient, bleed, and initial and final gelation.
- Set characteristics: initial and final set-time, unconfined compressive strength, durability, resistance against chemical attack, and permeability coefficient.

Properties and characteristics of suspension grouts can be significantly altered by means of additives and admixtures. Often compromises need to be made between conflicting characteristics in the search for the ideal grout for a particular project.

The fluid characteristics of the grout can be evaluated to assess their effectiveness for the formation to be grouted by applying the .amenability theory (defined below) to assess the suitability of the selected formulation for the size of apertures and pore channels.

The grouting engineer must have a good understanding of the characteristics and the function of each component in the grout to formulate a balanced, stable, suspension grout with the desired rheology and set characteristics. It is obvious that the filling of a karst requires a different formulation than the filling of fine fissures in a rock formation or structure. Grouts placed in flowing water must have higher cohesion than grouts placed in stagnant water or in dry circumstances.

Fluid characteristics of cement-based suspension grouts

Viscosity, Cohesion and Thixotropy: For excellent background information on these matters, refer to publications by Khayat and Yahia (1997), Skaggs, Rakitsy, and Whitaker (1994), Naudts (1994) and Lombardi (1985).

The cohesion, apparent viscosity and thixotropy of a grout are related but impact differently on the rheology of a fluid. *Cohesion*, is a measure indicating how far and how easily the Binghamian fluid will flow through pore channels of a given size under the given pressure. *Thixotropy* relates to the time in which a certain cohesion value is obtained and the impact of the shear rate in obtaining this increase in cohesion.

Viscosity is strictly a measure of flowability, and can be measured with a coaxial cylindrical viscosity meter or even with a Marsh cone. The latter two properties are closely related: the higher the cohesion, the higher the apparent viscosity. Ideally, admixtures only marginally impact on the cohesion or viscosity at moderate to high shear rates, but cause a major increase once the grout moves slowly or is at rest. These grouts are often referred to as .stable suspension grouts with shear thinning rheology.

It has been recognized that the behaviour of suspension grouts can be characterized as a Binghamian fluid, as opposed to a Newtonian fluid. In a Binghamian model, interparticle forces between the solids result in a yield stress that must be exceeded in order to initiate flow. The shear stress "T" represents the transition between fluid-like and solid-like behaviour. The cohesion represents the resistance to flow until a minimum force is applied. The cohesion "C" - more so than the viscosity - is the property that prevents suspended particles from settling, or a grout from sagging in wide apertures.

It is clear from equation (1) that cohesion, plastic viscosity, and shear rate influence penetrability of a suspension grout. Therefore, the higher the cohesion and viscosity the higher the shear stress and hence the (grouting) pressure that will need to be applied to move the grout through the pore spaces.

(1)
$$\tau = C + \eta_{B} (dv/dx) \text{ or } \eta (dv/dx)$$

Where: τ = shear stress; C = yield value or cohesion; $\eta_{\rm B}$ = plastic viscosity; dV/dX = shear rate; η = apparent viscosity

The ease of placement and susceptibility to wash-out of a grout are directly related to its thixotropic properties. Thixotropic agents impart a unique property to the cement-based suspension grout as the shear rate applied to the grout falls there is a rapid increase in cohesion and viscosity (Figures 1 and 2).

As the grout is being placed, the velocity, and hence the shear rate, drops at the fringes of the grout cylinder, and the cohesion and viscosity rise substantially. This in turn increases the resistance to washout but reduces the ability to flow. Even at very low concentrations (0.05% by weight of cement or less) pre-hydrated biopolymers used in the base mix design provide a significant thixotropic effect as shown in Figure 1. When these admixtures are properly prehydrated and neutralized, they are very potent to produce suspension grouts with shear thinning rheology.

The penetrability of a grout into a formation is determined by the pressure applied, its particle size distribution, and the cohesion of the grout (Håkansson. et al., 1992). For a cylindrical flow channel of radius (r) it has been demonstrated that the distance "L" that a grout will penetrate through a flow channel with diameter 2r, is governed by the applied pressure (p) and cohesion of the grout (C) (Lombardi, 1985).

$$L = p x r$$

2C

Where:

L = length of the cylindrical flow channel;

p = applied pressure;

C = cohesion of the grout;

r = radius of cylindrical flow channel (or half the thickness of a seam or fissure).



Figure 1. Evolution of Viscosity



Figure 2. Thixotropy

The penetrability of grout is directly related to its cohesion. In general, the higher the cohesion of a grout the higher is the resistance to pressure filtration as described below and shown in Figure 3.



Figure 3. Evolution of Cohesion

Resistance against pressure filtration: If circumstances permit grouting with high pressures the grout's penetration can be enhanced if the grout has good resistance to pressure filtration. If an unstable grout is used, the grout will pressure filtrate (dry pack) causing a considerable reduction in penetration.

The pressure filtration coefficient is defined as the volume of water lost in the pressure filtration test divided by the initial volume of grout in the cylinder of the apparatus, divided by the square root of the of the filtration time in minutes.

Kpf= volume of water ejected

Initial volume of grout x (filtration time (min))^{1/2}

Neat cement grout mixes and unstable mixes have very poor resistance to pressure filtration. During grouting, unstable mixes will be subjected to substantial and almost immediate water loss where flow channels are narrow. The remaining grout forms a filtrate in the pores or fissures close to the injection point and refusal is reached quickly.

A balanced suspension grout refers to a grout that has a good resistance (low Kpf) to pressure filtration and a low to moderate cohesion. The relationship between pressure filtration and cohesion, based on recent in-house testing, is illustrated in Figure 4. Regular stable cement-based grouts have a low Kpf but high cohesion will limit their grout spread. By introducing high range water reducers the cohesion of the grout can be reduced without adding water, thus maintaining high resistance to pressure filtration.

The amenability coefficient (Ac) (Naudts, 1995)

The amenability coefficient (Ac) is a determined by dividing the apparent Lugeon value of the formation (Lu_{gr}) determined by using grout as the test fluid; by the initial Lugeon value of the formation determined during water testing (Lu_{wa}). A correction factor is applied to the Lugeon value determined using the grout to take into consideration the higher viscosity of the grout over water. For field applications in rock grouting, the following equation is used to calculate the apparent Lugeon value:

with $V_{Marsh gr}$: efflux time for 1 litre of grout, via the Marsh Cone.

 $Lu_{gr} = flow (litres/min) \times 1m \times 143 psi \times V_{Marsh gr} (sec)$

1 litre/min x L_{zone} (m) x P_{effective} (psi) x 28 sec

With $V_{Marsh gr}$: efflux time for 1 litre of grout, via the Marsh Cone.

Amenability is a measure of the suitability for a given suspension grout to permeate fissures and apertures, accessible to water, in the grout-zone. Since Luwa is directly related to the aperture size of the fissures and open pores intersected by a borehole, it is clear that not all of these apertures are accessible to a selected grout. Only the fissures or pore channels that are wide enough will accept the grout. Hence the permeability coefficient established with grout is directly related to the aperture width of the fissures intersected by the borehole that can accept grout. By dividing Lugrout by Luwa the percentage of open apertures intersected by a borehole accessible to grout is given.



Figure 4. Resistance to pressure filtration vs. cohesion

The amenability coefficient (determined during the first few minutes of grouting) will immediately signal if the selected formulation is suitable, somewhat suitable or not suitable at all. The amenability coefficient indicates where, or if, regular cement-based grout is no longer suitable and one has to resort to a formulation with finer particles. Expensive and useless grouting operations can be avoided if the amenability of the formation is recognized to be too low.

The amenability coefficient is calculated early in the operation. The apparent Lugeon value, however, can be calculated on a regular basis as the grouting operation progresses. The apparent Lugeon value combines flow and pressure: the main grouting data. In a properly executed grouting operation, the apparent Lugeon value will gradually decrease with time. If the apparent Lugeon value remains constant or increases, it means that gouge or drill cuttings are being displaced or that the grout is sagging or is being washed out. If on the other hand, the apparent Lugeon value is decreasing quickly, it means that "particle pick-up" is taking place or that the solids content is too high or that a grout with a lower pressure filtration coefficient is needed. The role of additives and admixtures is to optimize the effectiveness of formulations in relation to the hydrogeological conditions encountered in the formation.

Set characteristics of cement-based suspension grouts

Gel-time and set-time: The gel and set time of grout are affected by numerous factors including: the use of fillers, particle size distribution of the grout, ambient grout temperature, the use of retarders and accelerators, the solids content of the formulation and the medium being injected. The use of some retarders and accelerators also affects the durability and strength of the grout.

Bleed: The residual permeability of a rock formation injected with grout can be estimated by using the amenability coefficient as previously described, but also the bleed of the grout has to be taken into account since bleed pockets will be formed. In order for a grout to be considered .stable. by the classic school of grouters, the bleed should be less than 5%. Ideally, bleed should be less than 2%. The use of superplasticizers masks the ultimate bleed in a suspension grout by slowing down the settlement of the cement particles. For this reason, bleed should also be determined from samples (kept covered to prevent evaporation) that have reached final set.

Unconfined compressive strength: The strength of grout is affected by numerous factors including: solids content, type and percentage of fillers, cement content, grain size distribution, water content, as well as the presence of certain retarders, accelerators, and viscosity modifiers.

The resistance against pressure filtration and the execution of the grouting operation have a major impact on the ultimate strength of the grouted medium. During an extensive laboratory testing program using the In-situ Soil Injection System (ISIS) in 2000 (Landry, et al., 2000) using slag and non-slag based microfine cements, the authors discovered that sands grouted with only one pass had considerably lower strength than the same sands grouted in several passes allowing pressure filtration to take place. The difference could amount to a factor six!

Durability and resistance to chemical attack: Although related to strength properties the durability of a grout can be considerably enhanced through the use of various admixtures that will react with excess calcium hydroxide to form secondary ettringite. Through the use of accelerated aging tests, whereby samples of cured grout are exposed to an environment that is harsher (at a higher temperature, exposed to dynamic attack by placing the samples in a shaker bath) than the expected field conditions. Furthermore, the ability of the grout to withstand chemical attack over time can be evaluated.

Matrix Porosity and Permeability: The matrix porosity and permeability of a set grout can be an important factor. A more pervious grout is subject to faster deterioration that could, in turn, be accelerated if the environment has a low pH. Calcium hydroxide can be more readily leached out from the grout, if the permeability coefficient is high. Alternately, if a pervious grout is required in situations where strength is required but drainage is necessary, specially formulated cement foams can be used that have very high permeability and good strength characteristics.

Common additives and admixtures used in suspension grouts:

The characteristics that are desirable in a suspension grout have been outlined above.

How are additives and admixtures best used to produce a stable, balanced suspension grout? There is a myriad of additives and admixtures available on the market. Many products are sold under brand names. Manufacturers and distributors do not like to reveal details of the actual chemical composition. The application of a few additives and admixtures are detailed below as a guide for the reader.

Additives

Slag: Slag is a non-metallic by-product created during the smelting process of iron ore. In the smelter, layers of dolomite are placed between the ore. Slag is generally composed of silicates, aluminosilicates, calcium oxides and other base components. Slag is commonly used as an active component in the grout. It takes part in the hydraulic reaction between water and cement. It also reacts with other additives to create ettrengite. On its own, slag will cure very slowly and therefore must be used in conjunction with cement.

The low hydraulic reactivity of slag is ideal to delay initial set. This is particularly useful to control the set time of ultrafine cements. Most brands contain large amounts of slag and are blended with cement and other additives and admixtures before they are ground down to an ultra fine powder. Because of the large specific surface (Blaine fineness in excess of 8000 cm₂/g) and low matrix permeability and better chemical stability of ultrafine cement particles, the reactivity of the pure Portland cements is too high. Slag is commonly added to extend the set time of microfine cement-based grouts and to create a reaction with the calcium hydroxide. This reduces the matrix porosity and enhances the chemical resistance.

Fly-ash: Fly-ash (Type F) is a rather inexpensive filler with pozzolanic characteristics. This product reacts with calcium hydroxide (free lime) that is generated by the hydraulic reaction between the cement and water to form secondary ettringite. This creates a more durable grout, especially in environments where the free lime could be easily leached out. Fly-ash also slightly reduces segregation and enhances the water repellant characteristics of the grout, as well as, its resistance against pressure filtration.

Fly-ash slows down the hydraulic reaction and strength development. Fly-ash also reacts with lime to form a low strength grout. Grouts containing high concentrations of type F

fly ash (70.90% of the total solids content in the mix) cure very slowly and can be used for backfill grouting operations, in compensation grouting applications and flowable fill.

Fly-ash is a valuable component used to make ultrafine cement by either wet milling for immediate use in microfine cement-based grouts (Naudts and Yates, 2000).

It is blended with Portland cement and plays the same role as slag but reaches a lower unconfined compressive strength (at the same concentrations and mixing ratios).

There are two common types of fly-ash available: type C and type F. The difference lies in their chemical composition due to the various types of coal used in the combustion chamber. If the concentration of type C fly ash exceeds 15% by weight of cement, it could lead to rapid deterioration of the grout.

Natural Pozzolan: Natural pozzolan can be found in a natural state in different rock formations or they can be made from clay or schists burnt into cinder. Commonly known natural pozzolan such as pumice and trass have been effectively used to chemically react with the calcium hydroxide created during the hydration of cement to form secondary ettringite. By tying up the free calcium hydroxide, a more durable and competent end-product is achieved. The strength gain in the curing grout is slower and the exothermic reaction is less pronounced, which is beneficial when grout is placed in large quantities.

Trass: clay-phyllosilicate (1-1 structures) This natural pozzolan has been used for over 2,000 years. Trass-enhanced mortars have stood up for 20 centuries in marine environments. Trass is a powderised tuff stone mined in Andernach (Am Rhein) in Germany. Trass is extensively used in grouts for grouting sewers because it enhances the chemical resistance of the end product. Because of its ability to react with the free lime, the bond to concrete of grouts containing natural pozzolans is considerably better compared to those that lack it.

Silica Fume: Silica fume is a by-product of the production of silicon or alloys containing at least 75% silicon. The silica fume particles collected are spherical in shape with an average diameter of approximately 0.1 micron. The small particle size makes it act as small ball bearings, keeping the larger cement particles into position in the mix, enhancing the penetrability of the grout.

Silica fume is also useful to reduce the permeability of the cured grout and as a result enhance its durability. The stability and resistance against pressure filtration characteristics of a grout can be enhanced with the introduction of silica fume. Silica fume is also known to provide water-repellant characteristics to the grout.

Hooton (1990) researched and documented the qualities of silica fume enhanced grouts. During the last 15 years the authors have tested and used silica fume in numerous grouting applications. Especially for the grouting of structures, silica fume enhanced grouts are very suitable. Typical concentrations of silica fume in cement-based grouts vary between 4 and 10% b.w.o.c. Because of its fineness, it is a most valuable additive to ultrafine cement-based grouts. It also enhances the wet milling of cement-based suspension grouts.

Bentonite: Bentonite is one of the most common additives used in cement-based suspension grouts. It enhances resistance to pressure filtration, reduces bleed, enhances stability and penetrability, and increases the cohesion and viscosity of the grout. The barrel yield value of the bentonite is indicative of the stabilizing effect of the bentonite. A barrel yield of 110 or more is recommended for grouting applications.

Bentonite is a clay phylosilicate (2-1 structure) characterized by its great affinity for water. For grouting applications, a high yield sodium montmorillonite is recommended. They are composed of two layers of silica and one of aluminum. The weak Van der Waal link between the layers of silica makes it possible for bentonite particles to electrostatically attract other molecules of the opposite polarity. This phenomenon is particularly useful in grouting applications to curtail the segregation (i.e. minimize the bleed and reduce the pressure filtration coefficient) in cement-based suspension grouts.

Bentonite should be used as a pre-hydrated slurry. The authors found out that if the bentonite is not pre-hydrated first, it will lead to the cracking of the cured grout. Even if bentonite is introduced as a slurry, it should always be entered first in the mix. The mixing order has an impact on the rheology and even the strength of the cured grout. When bentonite is introduced as the last component, its stabilizing effects are vastly reduced.

Locally available fillers: The formulator should be familiar with the chemical composition of the locally available fillers such as waste products from industries, 'soils' and minerals, and consider incorporating them into the mix to produce inexpensive yet durable grouts. Initially, only the fluid characteristics of the mix can be assessed. The set characteristics and durability of the end product are typically not known and must be carefully considered, unless the grouting operation is only of a temporary nature.

Admixtures

Dispersants, high range water reducers and superplasticisers: These admixtures are used to reduce the viscosity of the grout and to reduce the speed of segregation of a given grout and consequentially, enhance its stability.

Deflocculators enrobe the cement particles with a film of the same negative charge. Consequentially, the particles reject each other and the formation of macroflocs is prevented. Typically defloculators used in grouts are naphthalene sulphonate or melamine based.

One important reason water reducers, dispersants, and superplasticisers are used is to improve the mixing of all ingredients. Consequentially, these admixtures should be introduced at the beginning of the mixing cycle and not at the end. In the case of mixes characterized by low viscosity, it is appropriate to introduce dispersants at the end of the mixing cycle. The latter saves money as the late introduction of dispersants requires smaller amounts.

Viscosity modifiers: Viscosity modifiers can be used to provide a wide range of flow properties. Viscosity modifiers with high molecular weight which are highly cross linked, when used in a fully neutralized solution or colloidal suspension, will provide a very .short flow. rheology. Short flow rheology can be characterized by gelled, highly water repellant consistency similar to mayonnaise. On the other hand, viscosity modifiers with a low molecular weight, more lightly cross-linked, will produce suspension grouts with .long flow. rheology, more resembling a .syrup.. One of the key attributes of viscosity modifiers is that they impart .shear thinning.. This means that when pressure is applied to the grout, it will flow. When the driving pressure is removed, the grout will quickly "thicken up".

Minute quantities of bio-polymers such as natural gums and starches, cellulose ester derivatives and hydrophobically modified polyarcylate polymers, polyethylene oxide or polyvinyl alcohol, make it possible to change the rheology of a suspension grout dramatically. In order to maximize the qualities of these products, they should be properly pre-hydrated and chemically neutralized.

Based on our research, viscosity modifiers can increase the resistance against pressure filtration by a factor 2 to 10, depending on the grout formulation.

Retarders, grout stabilizers: In the past, lignosulphonate - based superplasticisers were used to slow down the curing of cement-based suspension grouts. This was done to facilitate multiple grouting passes for soil grouting or to prevent the loss of access to a given grout zone. The effect of a commercially prepared retarder on the gel and set times of microfine cement-based grout is shown in Figure 5.

Citric acid has been used for the same purpose. A major loss in strength was noticed, in spite of citric acid's minimal impact on initial and final gelation times.

Schwarz (2000) performed very valuable work on the use of superplasticizers in conjunction with isopropyl alcohol to delay the evolution of the cohesion and extend the initial gel time. Isopropyl alcohol is an effective retarder that, when used in small quantities, will not affect the final strength of the grout. The isopropyl alcohol acts as a "super" superplasticizer, which prevents the initial setting stages of the reaction from occurring. During or following the final stage of grouting, sodium silicate can be injected which reverses the retardation process and causes the grout to set quickly.



Figure 5. Gel and set time modification using a retarder

Conclusions

Admixtures and additives are essential to create stable regular cement-based suspension grouts. When properly formulated, grouts will have the desired flow and set characteristics which results in a lower residual permeability of the medium to be grouted. There is a trade-off between stability and viscosity of a grout without sacrificing injection rate, penetrability and spread. The capability to modify all characteristics of grout results in a superior overall product. Consequentially, the effectiveness of a grouting operation is optimized, which results in a better end product and a reduction of the cost of the grouting program.

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