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Rheological Properties of Low pH Cement-Palygorskite Injection Grout

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ABSTRACT

The rheological properties of cementitious materials containing fine particles, such as mineral admixture were investigated using a Viso Star-L rheometer with cylindrical spindles. Selected features of the behavior of these materials are evaluated from a rheological perspective through literatures results for the past 30 years. One of the important factors that governs the ability of grout to penetrate fractures, channels and porous material is the rheology. The mineral admixture was clay palygorskite which can be used for early gelation because of its thixotropic properties as well as the powder quartz used as aggregate. The cementitious materials were designed and three recipes (MPG1, MPG2 and MPG3) were examined in this research by changing the mix proportions of the mineral admixture palygorskite and water content. For ensuring the accuracy of rheological measurement for the candidate's grouts, necessary verification was been evaluated such as, influence of mixing time, stability and "time effect" to the accuracy of viscosity measurement, and yield stress as well as the physical properties at 3, 7 and 28 days. The results showed that grouts recipes with quartzite aggregate of different granulometry, are chemically compatible and fulfill the requirement of containing no organic matter. Candidate's grouts found to satisfy the stable sedimentation criteria with sedimentation rates less than 5% after 2 hours. Furthermore, it was found that the grout recipe 3 followed by 1 showed lower yield stress and plastic viscosity which are (0.1 Pa and 0.269 Pas) and (0.288 Pa and 0.531 Pas) respectively.

Key words: **Low-pH cement, Grout, Rheology, palygorskite, physical properties, plastic viscosity**

1. Introduction

In general, grouting is used as a tool to improve or change the physical properties of material such as, deformability, permeability or strength. Grout design involves the study of the behavior of the grout in suspension in the fresh and hardened states. The required performances of a grout at the fresh state are: injectability, rate of maturation long term stability of the suspension, erosion resistance and limits or no bleeding (Toumbakari et al., 1999). In order to achieve high penetrability and chemical integrity, the use of finely ground materials (e.g. quartz powder) is necessary. Penetrability performance does not depend only on the maximum diameter of the particles contained in the grout. It is known that fine materials in suspension coagulate very easily due to inter-particle interactions. Thus, for achieving effective injectability the grout must be of low viscous but erosion-resistant and the idea is to have the clay mineral palygorskite as one component because of its strong thixotropic properties. However, the water content of the grout must increase, with detrimental effects on the stability of the suspension and the mechanical properties of the hardened grout. The type of cement selected for the preparation of grouts recipes should be compatible with groundwater chemistry and have a low pH. This paper aims to review selected developments of the rheology of cement based grout and deals with the case study of effect of the mixing procedure on the injectability performance of cement-palygorskite grouts composed of low-pH cement, palygorskite, quartz powder and distilled water. The effect of each mixing procedure time and "time effect" at different time intervals after preparation has been studied by means of the coaxial viscometer.

2. Review of literature

2.1 **Cement based grout**

Grouting for under-ground construction is a process for filling the fractures, voids or cavities existing in rocks to give the grouted materials improvement of water-tightness or mechanical characteristics. In Sweden, the primary projects dealing with grouts have concentrated on the grout itself. Most common type of grout used in Sweden is based on cement (Funehag, 2007). A great deal of effort on material producers were spent by construction companies and scientists to improve the cement so that it becomes a very adaptable grouting material. The cement-based grout has sometimes shown good results even when grouting is executed on small fractures.

Grout based on cement is extensively used for grouting, because it has advantages compared with other grouts, because it has a relatively low material cost and limited environmental impact (Eriksson, 2002). Cement based grout is a suspension consist of particles, these suspended particles when used for permeation grouting, have a significant influence on the penetrability and flow behavior of the grout. Through inter-particle forces resulting in non-Newtonian behavior, particles affect the grout rheology and yield stress. Improving bearing capacity under a sluice had been done in Dieppe, France, 1802, by using of the concept of injecting self-hardening cementitious slurry (Sio-Keong, 2005). In 1845 in the United State, the foundations of a flume was grouted by Worthen, and had been graduated to sealing a masonry pier on the New Haven Road at Westford, nine years later (Sio-Keong, 2005). Cement-based grouts in the history commonly used in permeation grouting of fractures in rock masses and soil pores deposits has been described in detailed by Houlsby (1990) and Littlejohn (2003). Most important features of cement based grouts in literature can be found by Hakansson (1993), Weaver (1991) and Schwarz (1997). Cement–based materials are able to stand unsupported without flowing under their own gravity and during setting they develop strength and stiffness (Banfill, 2003). Cement grout has a well-defined shear stress explained by Cambefort (1954), and immediately after mixing will be developed and characterized by its viscosity (Sio-Keong, 2005).

2.2 **Rheology**

The science focusing on the deformation and flow of matter, and the emphasis on flow means that it is concerned with the relationships between time, strain, stress and rate strain is referred to Rheology (Banfill, 2003 and Westerholm, 2006). A little work is reported concerning the rheology of cement-based grouts, while dense grout which is the paste of cement had been considered in the last decades. Due to an interaction of various physical and chemical natures of any suspension, arising from both the solid and the fluid phase, the rheological behavior of any suspension is considered as very complex (Hakansson, 1993). The first technical paper considering grouting of rock foundation under a dam was published by Rands, 1915 (Sio-Keong, 2005). Since 1954, various researchers had been studying the rheological properties such as viscosity and yield stress of grout including other influence factors which includes mixing time, stability, additives and degree of saturation. These studies, however, focused on solution grout properties itself such as microfine of cement and other used additive like bentonite clay because of poor permeation of pure cement grout due to its short setting time and high viscosity (Sio-Keong, 2005). In these studies also, the mixes found not to cover all the practical range of important properties such as water cement ratio for effective application of grouting like very fine of fractured rock and using high pH cement like Portland cement. The stages of cement grout after mixing interpreted schematically by (Klein and Polivka, 1958) as dormant, and a function of increasing of grout strength with curing time as power or exponential. In 1959, cement grout classified by Caron as Bingham's grouts, as it has rigidity and viscosity and both of them increase with time, and the movement cannot be achieved without some sort of yield stress (Sio-Keong, 2005). Injection of neat cement grout is controlled by viscosity and shear strength in the early and later stages, respectively as indicated by Raffle and Greenwood (1961). However they developed a graphical relation between the rheological characteristics of grout and its capacity to permeate soil. Cement pastes exhibit a complex rheological behavior affected by several physical and chemical factors such as: water-cement ratio, experimental and mixing procedure (intensity and time), temperature, vibration, time dependency, additives, admixtures and cement characteristics (Vom Berg 1979; Tattersall and Banfill, 1983; Lapasin et al., 1983; Hakansson, 1993).

a. Studies during the period (1975-1985)

In 1975, Littlejohn, highlighted that the range between 0.4-0.45 of water-cement ratio makes the grout as much as fluid so that it will be easily handled in both pumping and placing in a small diameter borehole and to keep sufficient continuity and strength after injection to act as a strengthening medium. He also stated that the viscosity and shear strength increased rapidly when the cement-grout has water-cement ratio less than 0.9, while Burgin (1979) reported that the water cement ratio is 0.6. Cement grout had been classified as a stable (Deere, 1982), when the bleeding result after 2 hours after completion of mixing less than or equal 5%. He stated also that the sedimentation and bleeding of grout will be reduced by a small amount of bentonite and appears to be preferable on one hand, on the other hand it will be not useful way to give improvement of grout specially in both pumpability and penetrability. Regarding the mixing time, Tattersall and Banfill (1983), highlighted that in order to get constant properties of grout for both the yield value and the plastic viscosity, 5 minutes time of grout mixing is sufficient. While there was nothing mentioned concerning the volume of mixed grout used. Rheological behavior influenced significantly by the particles characteristics and concentrations as well as the suspending medium (Nguyes, 1983). Two years later, analysis of grout flow conditions through a smooth rock fissure was done in (1985) by Lombardi. He came out with some conclusions that the maximum distance of grout can be reached and the flow rate and therefore the time necessary to complete the injection is determined by the yield stress and the viscosity respectively (Hakansson, 1993).

b. Studies during the period (1986-1995)

Rheological behavior of cement based grout or (paste) is influenced during the hydration of the cement by the progress of chemical reactions. Time dependency will be caused inevitably by the hydration process and changing the cement suspension from a fluid to a solid state. Thus, it is difficult to obtain the rheology of cement paste and to reproduce the data often difficult (Shaugnessy III and Clark, 1988), (Hakansson, 1993). There were many studies in (1992). The first one focused on grouted sand by cement based grout with additives which was presented by Vipulanandan and Shenoy, 1992. This study concluded that in order to penetrate a formation at reasonable pressure and rate, the maximum particle size must not exceed 1/3 to 1/10 of the size of void. The second study indicated that pore structure injected with a cement grout is a function of water-cement ratio and sedimentation behavior of the suspended particles which were stated by Helal & Krizek (1992). The third study concluded that plastic viscosity and yield stress increase for grout having bentonite as an additive and there will be an increase in the specific surface of microfine cement, in contrast there will be reduction with increasing plasticizing admixtures (Krizek and Helal, 1992).

Discussion took place regarding the permeation of cement based grout in (1992) also by Paoli et al., where they showed that the size of the particles governs the permeation of grout more than its viscosity and yield stress. Thus, reducing the size of cement grains could give improvements for both grout rheological properties and penetrabilityThis will give the grout more stability under infiltration pressure and hence reducing yield stress value (Sio-Keong, 2005). Håkansson et al. (1992) described and evaluated the flow properties of cement grouts depending on a measuring technique, based on rotational viscometry. They discussed also estimation of the flow properties by field equipment. Results showed that the flow properties of grouts influenced by different admixtures and additives. Conclusions drawn were that both yield stress and plastic viscosity will increase with increasing the specific surface and addition of bentonite, while they reduce by plasticizing admixtures. Flow properties can be improved by combining bentonite and plasticizers in the grout mixture.

c. Studies during the period (1996-2005)

Four years later, in 1996, rheological properties were studied by Shroff et al. They focused on microfine cement dust (MCD) based grout. This study concluded that the permeability of grout with (MCD) into medium sand around 7.89E-3 cm/sec. They commented that this grout grants higher adherent strength to the grouted mass. The effect of two different mixing procedures, one with a mechanical and one with an ultrasonic mixer, had been studied by (Toumbakari et al., 1999), the conclusion drawn that the grout dispersion improved by using ultrasonic mixing method especially when silica fume is added, also a lower water content than that of high turbulence mixing used to give the same penetrability of the grout. Hanehara and Yamada (1999), discussed the interaction between cement and the chemical admixture types from the cement hydration point of view. The combination of cement and chemical admixture, method of adding admixture, or the water-cement ratio have significant influence on the rheological properties of fresh concrete. They showed that the polycarboxylate type superplasticizer compatibility is affected by the amount of alkaline sulfates in cement.

The propagation of grout through porous media is influenced by the particle size distribution of cement and soil, rheological characteristics of the grout, water permeability of the soil and also by the degree of saturation of sand , (Perret et al., 2000). Nachbaur et al. (2001) studied the evolution of the structure of cement and pure tricalcium silicate pastes during the very first minutes following the end of mixing using of a special mixer type. Conclusions drawn indicated that during the very first minutes following the end of mixing showed the main evolution of the structure of the pastes occurred, while at setting time and few hours showed no change in interparticular forces. In 2001, Yahia and Khayat evaluated the yield stress of cement grouts depending on analytical model. Evaluated grouts made with 0-0.075 % of welan gum RMA, by mass of binder, and various concentrations of high-range water-reducer (HRWR) in addition to the different replacement values of silica fume and blast furnace slag. Results highlighted that the deducted yield stress can be quite different depending on the adopted analytical model. Lower yield stress value resulted by proposed model to estimate yield stress of high performance, pseudoplastic grout than the other models, while the values found to be close to those estimated using the De Kee model for mixtures made with 100 % cement.

In 2003, a study was carried out based on statistical model investigated the parameters of cement grout affecting fluidity, rheological behaviour induced bleeding and compressive strength. Factorial experimental design was adopted by Svermova et al. (2003), to assess the combined effects of the several factors such as water/binder ratio (W/B), dosage of both superplasticiser (SP) and viscosity agent (VA), and proportion of limestone powder as replacement of cement (LSP). Rheological properties were evaluated depending on Marsh cone, Mini-slump test, Lombardi plate cohesion meter, induced bleeding test and coaxial rotating cylinder viscometer. Various rheological characteristics of cement grout resulted from the statistical approach which showed that influence of the limestone powder and the dosage of SP and VA. Eriksson et al. (2004) conducted experiments where by number of measurements of grout properties relating to the rheology and penetrability of fresh cement-based grout were carried out. It was concluded that the rheology-related properties of grout vary more than the penetrability-related parameters; however, other parameters could significantly influence the grout properties such as cement condition, water–cement ratio and the mixing equipment.

One year later, Park et al. (2005) investigated the rheological properties of cementitious materials containing mineral admixtures (MA) such as finely ground blast furnace slag, fly ash and silica fume. This investigation was based on a Rotovisco RT 20 rheometer with a cylindrical spindle and grouped as three systems, one, two and three components by replacement of ordinary Portland cement with these mineral admixtures. Results of one-component, (OPC) and with increasing the dosage of PNS-based superplasticizer showed improvement in the rheological properties. The yield stress and plastic viscosity showed decrease with replacing OPC with fly ash (FA) and blast furnace slag (BFS) in two components system. However, both yield stress and plastic viscosity steeply increased with increasing SF, in the case of OPC-silica fume (SF) system. The last group three components systems, showed improvement in the rheological properties compared with sample based SF.

d. Studies during the period (2006-2010)

Development of a method to measure the shear strength of cement based grout in the field in simple and robust way have been presented by Axelsson and Gustafson in (2006). This method consists of a stick sinking in the grout so that direct measurement of the shear strength can be made. Results showed good agreement compared with measurements of the shear strength made in the lab., with a rotational rheometer. Effects of recycled metakaolin on the rheology and conduction calorimetry of cement pastes have been studied and compared to the effects of commercial metakaolin by Banfill and Frias in (2007). Results indicated similar effects on the rheology of cement paste of both calcined paper sludge and metakaolin. Vikan et al. (2007) studied the “flow resistance” of paste based on the influence of cement characteristics such as cement fineness and clinker composition and three different of plasticizers types. This investigation conducted depending on the area under the shear stress–shear rate flow curve. Results of the combined characteristic of cement showed either a linear or exponential function depending on the type and dosage of plasticizer. It was found that the correlation valid for a mix of pure cement and cement with fly ash, (4%) limestone filler in addition to the pastes depending on constant dosage of silica fume. The rheological behavior of cementitious pastes was also studied by Vikan and Justnes (2007), where the cement was replaced by raw silica fume (SF), densified (SF) and limestone. The area under the flow curve was to represent the effect of SF on the flow resistance. It was dependent on the dispersing ability of the plasticizer. Test results showed that the gel strengths increased with increasing of SF replacement of cement independently of plasticizer type. However, it was depending on the type of SF since pastes with raw SF developed higher gel strengths than with densified SF. For increasing limestone replacement both flow resistance and gel strength were decreased.

Eklund and Stille (2008), showed that the size and distribution of grains are significantly important for the ability of a grout to prevent passing obstructions in the flow path without the cement grains clogging and preventing further penetration which called "filtration tendency". They showed also that the grain-sizes used should be at least 2–3 times smaller than the aperture to be penetrated by the mixture. In (2008) also, Sahmaran evaluated the rheological properties of cement-based grouts, setting times and compressive strength experimentally through the effect of the replacement rate and fineness of natural zeolite. Results shown that the yield stress and apparent and plastic viscosities significantly increased by replacement rate of cement by natural zeolite keeping of SP content constant, while the fluidity and deformability of the cement-based grouts had reduced. In contrary, both yield stress and plastic viscosity decreased significantly with increasing the dosage of SP. Further decreasing in the rheological and workability properties of grouts showed with incorporation of a fine natural zeolite. In general, natural zeolite used with grouts showed pseudoplastic behavior, and with increasing the amount of zeolite and fineness in role increased the shear thinning grout behavior.

e. Studies during the period (2011- onward)

Takahashi et al. (2011) studied the effects of mixing energy on cement based grouts characteristics such as fluidity and hardening. The results showed that the deterioration in fluidity and setting properties caused by long mixing processes. This is due to different mixing durations which led to the acceleration in hydration kinetics and changes in microstructures and subsequent changes in dispersion states. In the same year, Sonebi and Malinov (2011) predicted the rheological behavior of grouts and evaluated the sensitivity of such parameters to the variation in mixture ingredients by using the development of artificial neural network (ANN) models. Results showed that accurate prediction of mini-slump, apparent viscosity at low shear, yield stress, and plastic viscosity values of the Bingham and modified Bingham models of the pseudo-plastic grouts by ANN which are within the practical range of the input variables as well. Yahia (2011) evaluated the effect of w/c, high-range water-reducer (HRWR) cement combinations, and supplementary cementitious materials (SCM) on non–linear rheological behavior of various grout mixtures experimentally. The yield stress of mixtures had lowered due to the incorporation of high-range water-reducer and thus enhancing deformability, while mechanical and durability performances has improved by silica fume. Results showed that, low w/c ratio gave shear-thickening behavior of high-performance structural grouts, while the shear-thinning behavior appeared at relatively higher w/c ratio.

Bouras et al. (2012), studied the influence of dosage rate of viscosity-modifying admixtures (VMA) on the steady state rheological properties of cementitious materials experimentally. These properties includes the yield stress, fluid consistency index and flow behavior index.

Results indicated that the rheological behavior of the material is in general dependent upon shear-rate interval considered, so the materials exhibit shear-thinning at sufficiently low shear-rates, while the pastes becomes shear-thickening at relatively high shear-rates due to repulsive interactions among the solid particles.

Optimization of the rheological parameters, hardened properties, and setting times of cement based grouts containing metakaolin (MTK), viscosity-modifying agent (VMA) and superplasticiser (SP) had been presented in (2013) by (Sonebi et al.). It showed that increased (SP) led to an increase in the fluidity and the setting times, decreasing in the rheological parameters and reduction in flow time and plate cohesion. While an increase in the (VMA) led to decrease in the fluidity and increase in the rest of parameters such as plate cohesion, Marsh cone time yield stress, and plastic viscosity. The results showed also an increase in cohesion plate, flow time, yield stress, plastic viscosity and improve compressive strength. On the contrary, it showed a decrease in mini-slump and setting times by using of (MTK). Zapata et al. (2013) studied the rheological properties of grouts in the fresh state effects of micro and nano-SiO_2 under various dosages of carboxylated-polyether-copolymer-type superplasticizer. Results based on marsh cone test showed a nonlinear behavior in grouts with micro and nano SiO_2 particles and chemical additions in saturation dosage, flow time and loss of fluidity compared to the control samples.

Rheological parameters of cement suspensions which are modified with different doses of two types of superplasticizers (SPs) were evaluated by Paravanova et al. (2013), using an alternative method of centrifugation. They showed that the additive dose, the additive type and the magnitude of centrifugal forces have changed the water/solid ratio, the critical normal and shear stresses ”yield values”. Results in general, indicated that dispersing effectiveness of SPs and rheological parameters of suspensions might be determined by the method of centrifugation. Feys et al. (2013), developed the Reiner–Riwlin transformation equation for the modified Bingham model. This equation can be valid for coaxial cylinders rheometers. Modified Bingham model is a second order term in the shear rate of Bingham model. Established equation presented to be compatible with the Reiner–Riwlin equation. This compatibility showed agreement with Bingham and Herschel–Bulkley models. They applied the same experimental data “yield stress values” in the three rheological models and then were compared with the yield values depending on slump flow. Results based on modified Bingham model showed independent of the non-linear behavior and most stable yield stress values.

2.3 **Conclusive remarks and scope**

Rheology plays an important role because of its scope for characterizing grout, fresh cement paste, mortar and concrete, and for understanding how they perform in practical applications. Ongoing research has included testing of three recipes of candidate grouts with respect to their physical and rheological properties. Depending on the presented literature studies, the following points have been considered in this study:

- The type of cement selected in this study for the preparation of grout should be compatible with groundwater chemistry and have a low pH which is Merit 5000 cement.
- The need for chemical integrity led to the choice of quartzite as aggregate used in this study.
- The particle size distribution of the solid mixture criterion required effective grinding of the aggregate material (powder quartz used) which governed the permeation of the grout. Reducing the inner friction of the grout can be achieved by small particles, which played as a lubricant for the larger particles. This role will also lead to the lowering of the water-cement ratio and increases the strength of the grout.
- The grout must be low viscous for achieving effective injectability but erosion-resistant and the idea was to have the clay mineral palygorskite as one component because of its strong thixotropic properties.
- Rheology is now seriously considered by users, rather than being seen as an inconvenient and rather specialized branch of cement science (Banfill , 2003).
- High water-cement ratios used in this study are (10, 11.4 and 15.7).
- Influence of mixing time was considered, the stirring times used in this study were (4, 8 and 12 minute), while the influence of time dependency studied after (20, 40 and 60 minute).

3. Case study

3.1 **Experimental program**

3.1.1 Materials

- Cement

Merit 5000 cement,was used through this work. The cement, is of low-pH, was delivered by the SSAB Merox AB, Oxelösund. The physical and chemical characteristics of the cement are shown in Table 1 and Fig. 1.

Table 1: Chemical and Physical characteristics of Merit 5000 cement according to SS-EN 196-1, 2 and 3

Analysis	Merit 5000 cement
MgO	16.6
LOI	-1.23
LOI compensated for S^{-2} oxidation	1.43
SO_3	0.085
Sulfide	1.33
cl^-	<0.01
Glass content %	99
Density kg/m^3	2912

Specific surface area (m^2/kg)	470
Moisture content	0.09
Initial setting	
Water content (%)	27.0
Setting time (min)	210
Compressive strength	
7 d (MPa)	23.3
28 d (MPa)	50.4

Fig.1. Merit 5000 cement

- Aggregate and fine material

The need for chemical integrity led to the choice of quartz powder for the major aggregate component. This powder which is so-called Norquartz 45 was delivered by the Sibelco Nordic, Lillesand, Norway. The particle size criterion selected required effective small size of the aggregate material to find ways of grouting of relatively finely fractured rock. The chemical compositions of the used materials and the particle size distribution are given in Table 2. Mineral admixtures (Palygorskite) had been used in order to increase early gelation of cementitious material because of its thixotropic properties. Once forced into rock fractures or channels in soil it stiffens and serves as a filter that prevents fine particles to migrate through it and further out. Due to its hydrophilic potential is too high, the grout will not get a high strength and density. The chemical formula is (Mg,Al) $2Si_4O_{10}(OH)_4(H_2O)$ and the material was delivered by the Greek enterprise Geohellas Co, Athens.

Table 2: Characteristics of Norquartz 45

Component	Average (%)

SiO_2	99.6
Al_2O_3	0.25
Fe_2O_3	0.02
LOI	0.15
Density (g/cm^3)	2.65
pH	6.5
Particle size distribution	
Size (µm)	Percent finer
<10	38
<20	68
45	99.2

3.1.2 Preparation of grout

The procedure was to mix Merit 5000 cement, palygorskite and quartze powders and adding the water for reaching a fluid state. The grouts were prepared by pouring the dry components in a glass beaker and adding distilled water under intense stirring with a rod until reaching the fluid condition.

3.1.3 Composition of Cement Grout Mix

For the study of permeation grouting three grouts termed MPG1, MPG2 and MPG3 were prepared with Merit 5000 cement and the compositions are shown in Table 3. The content of palygorskite varied to get the same fluidity which was required with the water content. The unit weight of various grout mixes was determined from the laboratory tests were1396, 1388 and 1305 kg/m^3, respectively.

Table 3: Grout components in weight percentages of solid total weight and mix proportions ratio with Merit 5000 cement (MC), palygorskite (P), quartzite aggregate (Q), and distilled water (W).

Grout		MPG1	MPG2	MPG3
Solid mixture components (%)	Palygorskite	13.5	19	24
	M cement	12	11	10.5
	Quartzite	74.5	70	65.5
Water/solid materials ratio		1.186	1.270	1.640
Density, kg/m^3		1396	1388	1305
Component ratios	W/MC	10	11.4	15.7
	Q/MC	6.28	6.28	6.28
	P/MC	1.14	1.71	2.28
	W/P	8.75	6.67	6.875

3.2 **Characterization of grout**

3.2.1 Compressive strength

The compressive strength was measured using cylinders sized 50 mm diameter x 67.5 mm length at the age of 3, 7 and 28 days (Fig. 2). Two measurements were performed for each sample and the results were given as an average. The samples were casted in the plastic tubes and left in a mould for 24 hours, after that they were stored in the aquarium covered by a plastic plate at room temperature until testing. At the age test required, the samples were de-moulded and left for a while before testing (Sievänen, 2006).

Fig. 2. Grout samples prepared for compressive strength test.

3.2.2 Rheological Properties of Grout

The yield stress and the viscosity are the prime rheological properties for cement grout, which need to be properly measured in view of the possible influence from the handling and testing process.

- **Measurement program**

For ensuring the accuracy of rheological measurement for candidate's grouts, so that the determined grout parameters are representative for practical use, these are possible influence of mixing time, stability and set time to the accuracy of viscosity measurement, and yield stress. All these were considered in the present experimental program.

- **Measuring Device for Viscosity**

Fig. 3 shows the equipment which is commonly used (Visco Star-L) and manufactured by JP Selecta S.A., Spain, i.e. rotational viscometer with immersion cylinder. This device consists of a coaxial-cylinder which is submerged in the test fluid and rotated at different rotational velocities (0.3-200 rpm) during the test. The resistance of the fluid against movement as measured in the test gives the values of viscosity.

Fig. 3. Rotary Viscometer with Coaxial-cylinder spindle

- **Stability of Cement-palygorskite Grout (Bleeding)**

Bleeding or stability of cement-based grouting suspensions is determined by putting the cement grout in a standard glass cylinder with a volume 1000 c.c. (Sio-Keong, 2005, Sievänen, 2006). The volume of clear water (dv) segregated on top of the suspension divided by the original grout volume (V) at about two hours after mixing (Fig. 4) to evaluate the stability of a suspension.

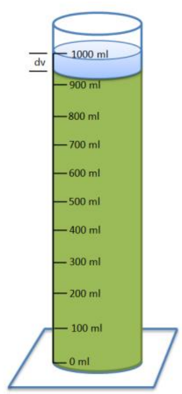


Fig. 4. Bleeding test for cement-palygorskit grout

- **Influence of mixing time**

Few information from literature review regarding the influence of mixing time, given by Tattersall and Banfill (1983); Paoli et al. (1992); Schwarz and Krizek (1992);and Sio-Keong (2005) were adopted for 5, 6-12, 1-10 and 5, 10-15 minutes as time for mixing the grout. For the purpose of quantifying the effect of mixing procedure on the viscosity of cement-palygorskite grout, three specimens stirred for duration of 4, 8 and 12 minutes were prepared for each grout mix for the viscosity measurement by using a viscometer mentioned above.

4. Results and Discussion

4.1 **Stability of grouts**

The results for the three cement grout mixes in the represent study are listed in Table 4. As shown in this table, all the grout mixes were found with sedimentation ratio less than 1%. All of the grouts mixes can are satisfactory for stable sedimentation criteria (i.e. sedimentation rate <5% after 2 hours) specified by Deere and Lombardi (1985) and Kutzner 1(996). This implies that using palygorskite mineral and quartz powder is good for minimizing segregation effect in permeation grouting work.

Table 4. Bleeding test results of candidate grouts.

Grout	Bleeding, %
MPG1	0.984
MPG2	0.275
MPG3	0.398

4.2 **Compressive strength**

Results of the uniaxial compression tests (shown in Fig. 5), after 3, 7 and 28 days are given in Table 5 and are plotted in Fig. 6. Generally, the grout samples showed fine fractures at failure for 3 days and a bit big fractures for 7 and 28 days. Also some samples exhibited plastic behavior with increasing diameter in the course of the compression. The strength rose when the content of palygorskite was increased from 13.5 to 19 % of the total solid mass and dropped when the contents of palygorskite and water content were increased as well. This indicates that thixotropic stiffening was more important than the formation of cementation bonds for the compressive strength (Pourbakhtiar, 2012).

Fig. 5. Compressive strength machine used in this study

Table 5. Compressive strength results

	Compressive strength, kPa		
Age, day	MPG1	MPG2	MPG3
3	11.6	23.7	20.9
7	27.1	31.5	28.3
28	54.23	79.3	72.8

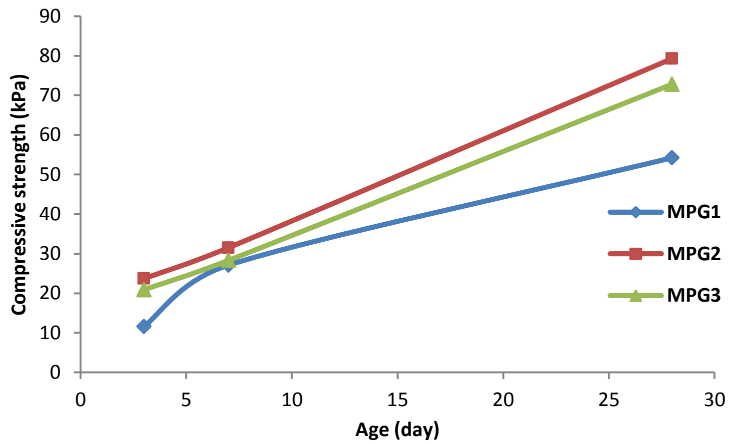


Fig. 6. Evolution of cement-playgorskite grout compressive strength

4.3 **Rheological properties**

4.3.1 Influence of mixing time

Minor differences in temperature (i.e. 0.4 to 0.6°C) was found between the same mix of grout stirred for 4 and 12 min in the present study while (1.2°C) for 8 min stirring time. The temperature of grout at the end of stirring was found generally decreasing slightly with increasing stirring time except for grout titled MPG1 of lower content of palygorskite ratio which showed an increase with increasing stirring time from 4 min to 8 min and then decreased at 12 min. This could be attributed to the completed hydration process in the light cement-palygorskite grout mixes based (water/solid materials ratio = 1.186 to 1.640). Figs. 7-9 show the plots of viscosity values as a function of time of three grout recipes measured during the Viscometer Tests. The temperature of grout as measured during the test for all stirring time (4-12 min) was found generally between 20.7 and 22.7°C in all three grouts.

Fluctuation of viscosity value measured during the test was found to be not sensitive to the effect of stirring time for grout with low palygorskite content and high W/P ratio (MPG1) under all stirring time (cf. Figs. 7-9). One fluctuation in the measured viscosity was observed during the test for the grout mix (MPG2) under the stirring time (4 and 12 min) and at (4 min) for grout mix (MPG3). However, it tends to stabilize after a measuring time of 100 seconds.

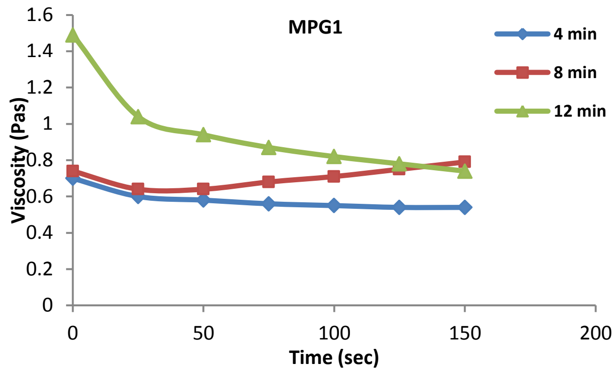


Fig. 7. Plot of viscosity of cement-palygorskite grout with time, MPG1

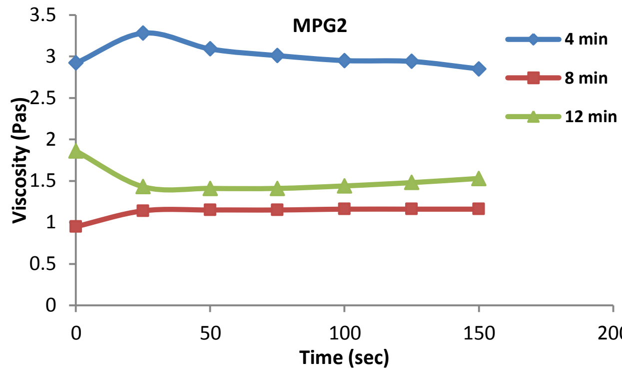


Fig. 8. Plot of viscosity of cement-palygorskite grout with time, MPG2

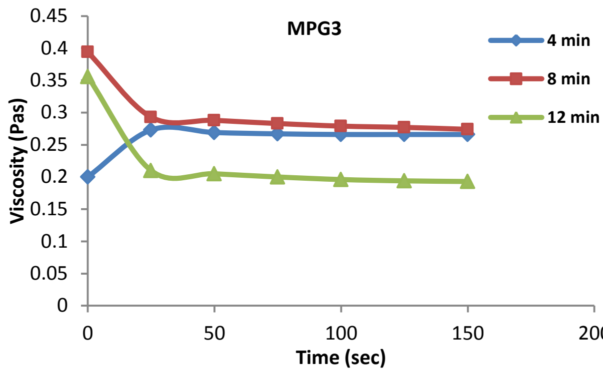
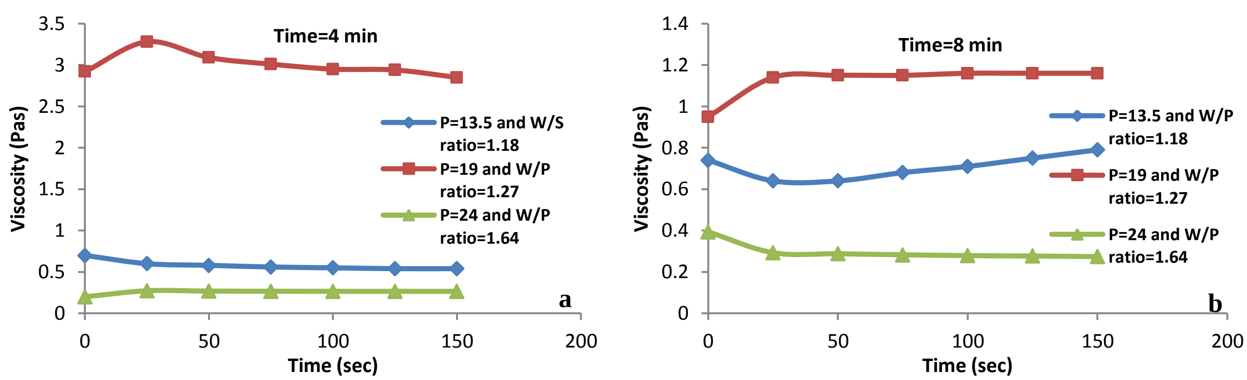


Fig. 9. Plot of viscosity of cement-palygorskite grout with time, MPG3

Fig. 10 shows the variation of the viscosity values measured for the cement-palygorskite grout prepared under stirring time for 4, 8 and 12 minutes, respectively. The results of these measurements show significantly high value of viscosity for grout mix (MPG2) of 19% palygorskite content ratio and minimum value of W/S ratio (1.27). This is possibly due to its high consistency resulted from the high thixotropic palygorskite and lower water content which non-excessive in the hydration process as compared with the values of the other two grout mixes.



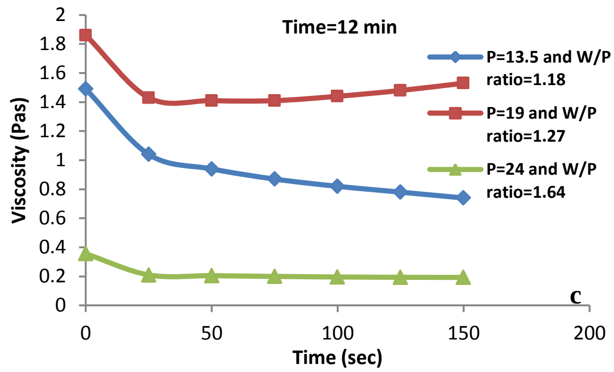


Fig. 10. Evolution of viscosities with time, a= 4 min, b=8 min and c= 12 min.

At the end of each test, the viscosity was measured after about 150 seconds for various grout mixes prepared with 4, 8 and 12 minutes stirring time as shown in Fig. 11. The test grout mixes were found to be not sensitive to stirring time except for the grout of MPG2, which showed higher viscosities values under all the stirring time. Mixing time of 8 minutes was found suitable for viscosity test in the laboratory and grout sample volume did not exceed 600 cm^3. To ensure that complete mixing for uniform grout mix is achieved in the field scale, mixing time for grout should be adjusted and verified by other test, Marsh Cone for example (Sio-Keong, 2005).

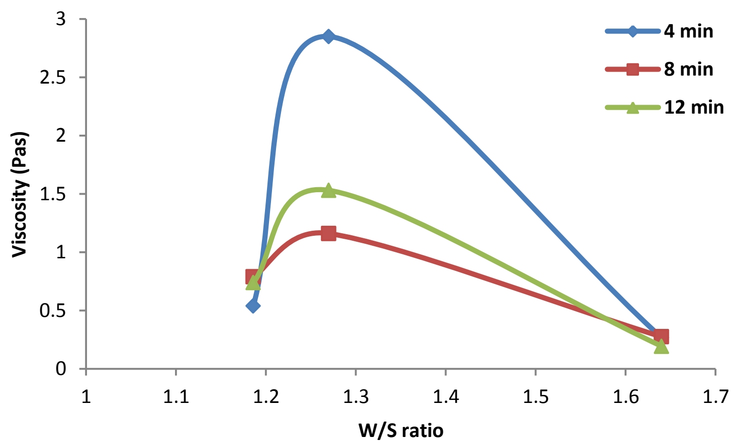


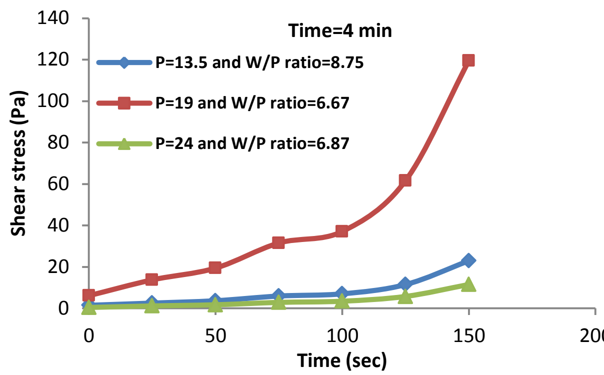
Fig. 11. Plot shows viscosity as a function of water/solid ratio, at different stirring time.

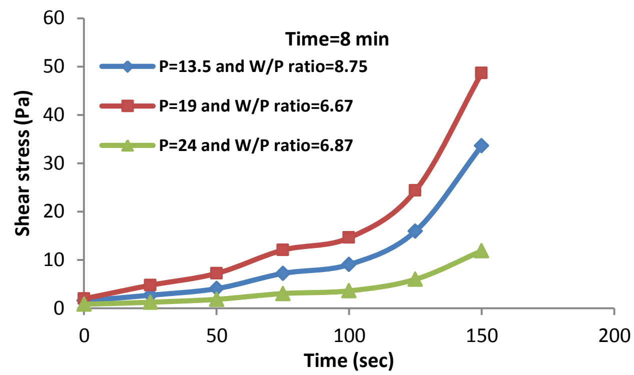
4.3.2 Stirring time and the shearing stress

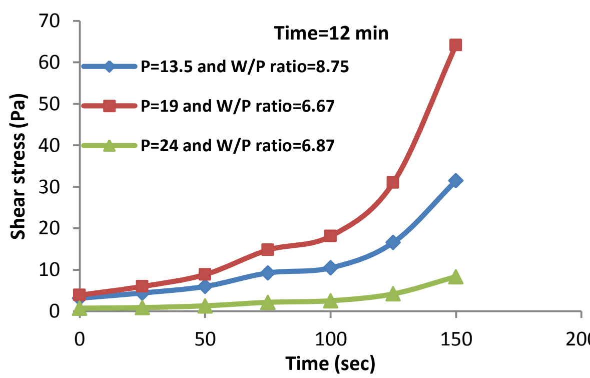
Typical Bingham's fluid characteristics for all of the cement-palygorskite grout were recognized during the development of shear stress measured during the tests where the shearing rate is increasing with time (Fig. 12). Shearing stress decreasing with time for the same grout, which simulate best, the flowing of grout condition in the permeation grouting progress is considered to be the most representative measurements for the viscosity of cement-palygorskite grout.

The yield stress and the plastic viscosity showed an increasing trend with decreasing W/P ratio (Fig. 13 and table 6). The measured values were found slightly reduced with increasing stirring time possibly due to more complete destroy of structural bonding in the longer stirring process.

Furthermore, it was found that the grout candidate 3, gave lower yield stress and plastic viscosity which are (0.1 Pa and 0.269 Pas) followed by grout recipe 1 (0.288 Pa and 0.531 Pas) respectively. However, candidate grout 2 showed stiffer than the others and the yield stress and plastic viscosity calculated were (1.507 Pa and 2.84 Pas). This is due to the higher palygorskite proportion and lower water content compared with the grouts 3 and 1 (Table 6).







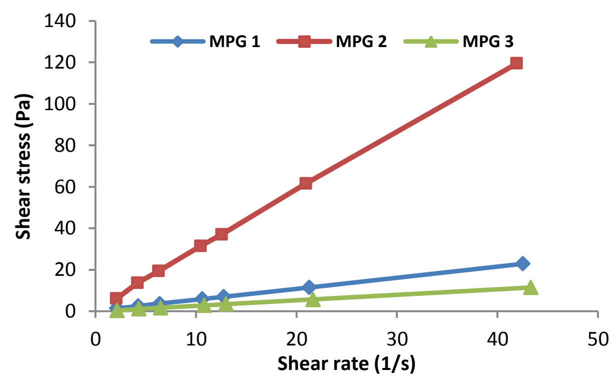


Fig. 12. shear stress versus time and different stirring time, 4, 8 and 12 min.

Fig. 13. Plot shows shear stress as a function of shear rate.

Table 6. Plastic viscosities and yield stresses according to the Bingham model of grouts recipes after 4 min time of mixing.

Grout	Plastic viscosity η_p, Pas	Yield stress τ_o, Pa
MPG1	0.531	0.288
MPG2	2.840	1.507
MPG3	0.269	0.100

4.3.3 Influence of time dependency

The time dependency of viscosity of cement-palygorskite grout could be important before injection under mixing disturbance from subsequent agitation. While, the time dependency of viscosity of grout is free from further mixing disturbance after injection in the process of permeation grouting (Sio-Keong, 2005). Figs. 14 to 16 show the “time effect”, i.e grout left for time (20, 40 and 60 min in this study) after mixing and before injection, as presented in that all cement-palygorskite grout recipes are found to be not sensitive to elapsed time due to the long setting time of these light cement slurries. Slightly increase in viscosity values with elapsed time were found in, as shown (cf. Figs. 14-16). The high viscosity values are believed to be attributed to the effect of sedimentation accumulated in the long time left. The very obvious drop in viscosity of the grouts with increased agitation suggests effective injection of dynamic type. The process of permeation grouting started with high shear rate at the beginning of injection to low shear rate at end of the injection with avoiding the effect due to setting and sedimentation of grout (Sio-Keong, 2005). Fig. 17 show the Bingham behaviors of cement-palygorskite grout with different “time effect” of 0, 20, 40 and 60 min.

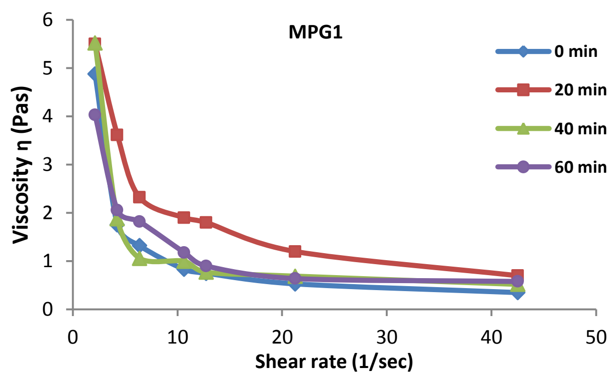


Fig. 14. Viscosity of MPG1 grout versus shear rate and elapsed time

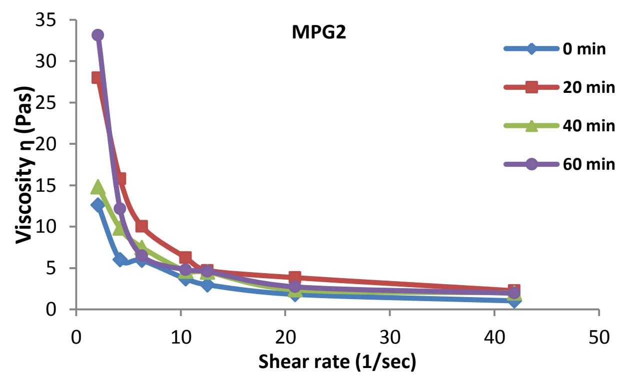


Fig. 15. Viscosity of MPG2 grout versus shear rate and elapsed time

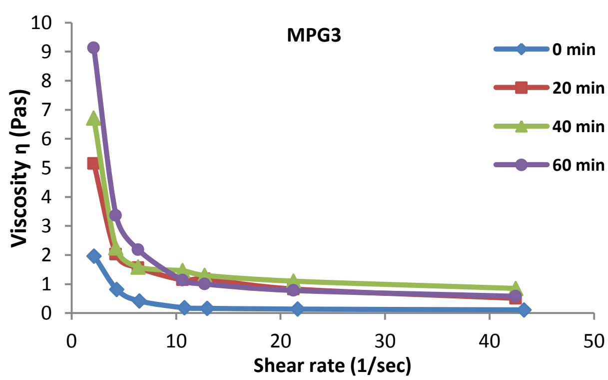


Fig. 16. Viscosity of MPG3 grout versus shear rate and elapsed time

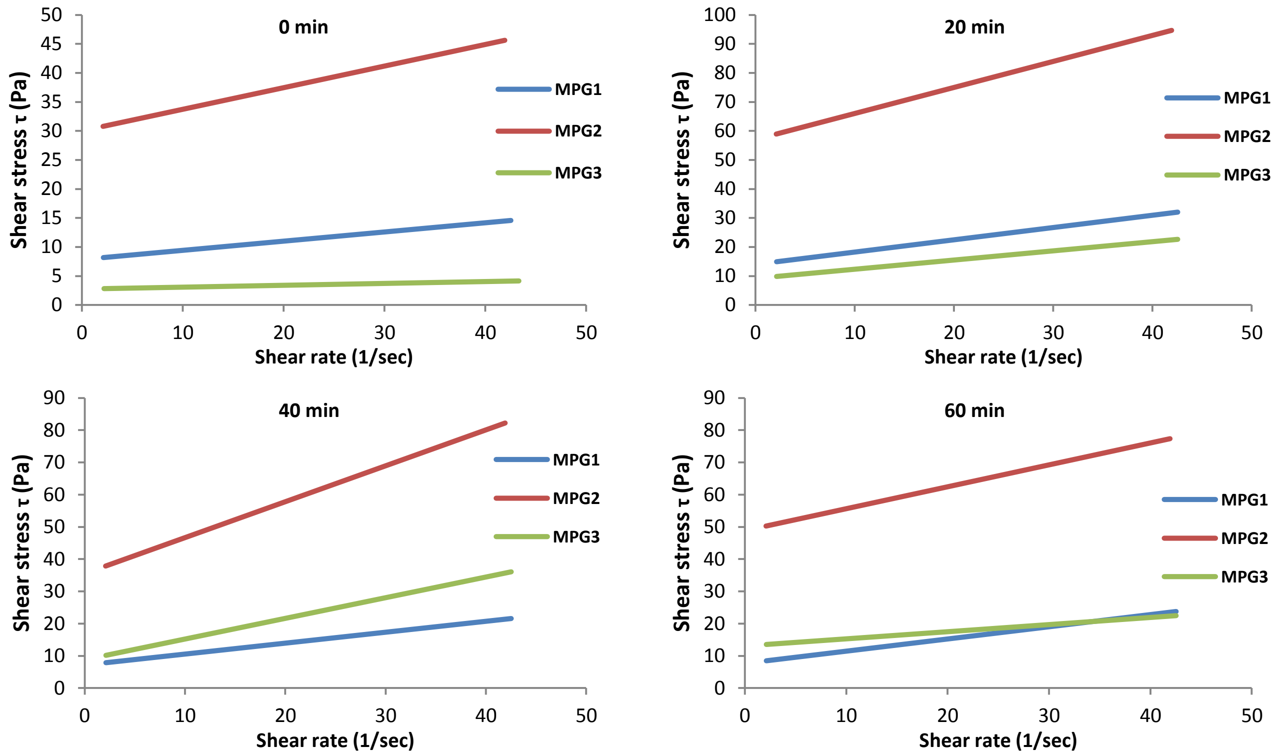


Fig. 17. Shear stress plotted with shear rate of grouts with different time effect 0, 20, 40 and 60 min.

The estimated yield values which were obtained by extrapolating the shear stress-shear rate data using the three different recipes of grouts with different elapsed times were adopted in this study. The plastic viscosity which represents the slope of the shear stress-shear rate relation (cf. Fig. 17) and the yield stresses for the various mixes of cement-palygorskite grout determined from the experimental works are plotted in Figs. 18 and 19.

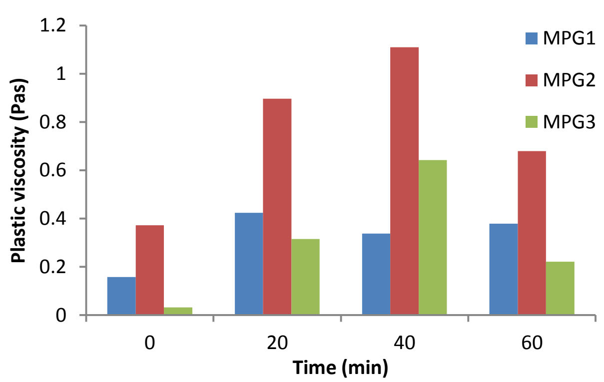


Fig. 18. Plastic viscosity of cement-palygorskite grouts versus time effect.

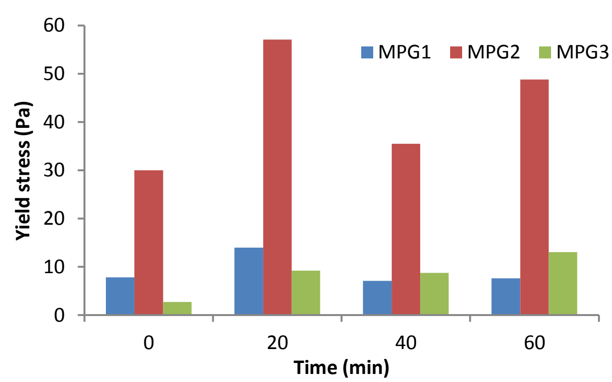


Fig. 19. Yield stress of cement-palygorskite grouts versus time effect.

It would be possible to calculate the penetration length of cement-based grouts by determining the shear strength and hence establish a design of the grouting procedure (Axelsson and Gustafson, 2006). Fig. 20 shows the max shearing strength of palygorskite-cement grouts as a function of "time effect". The results of measurement show significantly high value for grout mix (MPG1 and 2) at 20 min and then it was slightly decreasing because they have a lower amount of water content compared with the grout recipe (MPG3) which get the maximum value at 40 min. This possibly is due to its high consistency resulted from the thixotropic palygorskite and lower water content which non-excessive in the hydration process.

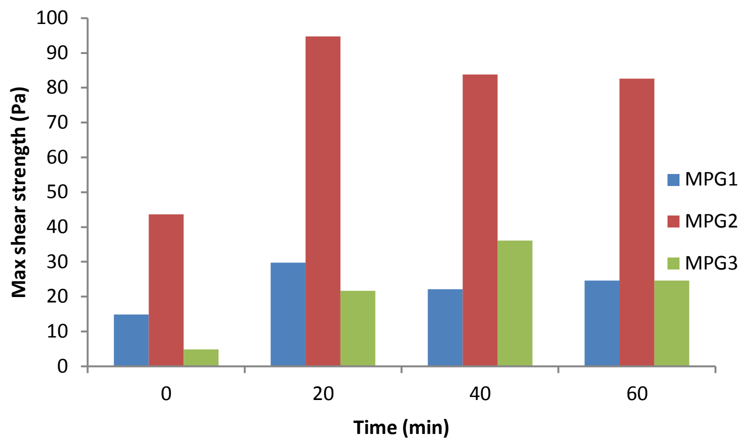


Fig. 20. Shear strength as a max values plotted against time for different grout recipes.

4.4 **Proposed theoretical model of shear stress**

Theoretical model constructed based on experimental work lead to having Eq. 1 by using of Minitab 16 for statistical treatment (Minitab). This model refers to the relationship of an individual effect of independent variables (yield stress, plastic viscosity and the shear rate) on the shear stress value. The analysis via regression of the statistical software was applied on the results behaved as Bingham's (cf. Fig. 17) at time effect 0 min to find out the relationship of cement-palygorskite grout candidates behavior. The correlation factor was R^2 = 95.4%.

$$\tau = - 2.66 + 0.994\tau_o + 14.8\eta_p + 0.185\gamma \qquad (1)$$

Where, τ is the shear stress (Pa), τ_o is the yield stress (Pa), η_p is the plastic viscosity (Pas) and γ, is the shear rate (s^{-1}).

5. Conclusions

Rheology plays an important role for defining the permeation of grout. Permeation grout work is largely a trial and error process and the desirable properties of the hardened materials can't be achieved without satisfactory fresh properties. An experimental study on the rheological properties of various cement-palygorskite grouts mixes with low-pH cement and quartz powder was carried out in the present research for the determination of the important grout parameters, such as viscosity, yield stress, density, stability and compressive strength of cement-palygorskite grout for enhancing the application of such candidate grouts for permeation grouting works. Based on the results of the present study, the following conclusions are drawn:

As a result, cement-palygorskite based candidate grouts conform closely to the Bingham model and in many practical situations, their behavior can be explained by reference to that model. The very obvious drop in viscosity of the grouts with increased agitation suggests effective injection of dynamic type.

Results indicated that the mineral palygorskite can be used to give some slight early strengthening by thixotropic action and some filtering capacity. Based on these experimental findings, it is therefore assumed that thixotropic stiffening was more important than the cementation bonds for the compressive strength.

The grouts recipes examined in this study with quartzite aggregate of different granulometry, are chemically compatible and fulfill the requirement of containing no organic matter.

All the examined grout recipes were found with sedimentation rate less than 1%. This indicates that all grout mixes can be satisfy the stable sedimentation criteria, which are <5% after 2 hours. Shearing stress showed that it was decreasing with time for the same grout, which can best simulate the flowing of grout condition in the permeation grouting progress which is considered to be the most representative measurements for the viscosity of cement-palygorskite grout.

Regarding the yield stress measured through this study, it was slightly reduced with increasing stirring time, this is possibly due to the more complete destroy of structural bonding in the longer stirring process. Also, the impact to the engineering characteristics of grout is not expected to be significant. The results indicated that by using palygorskite increases the yield stress and plastic viscosity due to the higher thixotropic associated with an increase in the water demand. Finally, summary of effects of mix design proportion changes on the rheological and physical properties as shown in table 7.

Table 7.Influence in the rheological and physical properties of cement-palygorskite grout by different mix design proportions

Change by	action	Effect on			
		Stability	Compressive strength	Plastic viscosity	Yield stress
Palygorskite	increased	++	+/-	++/-	++/-
Water content	increased	++	+/-	--	--
Age of test	increased		++		
Temp.	decreased			+	+
Mixing time	increased			-	-
Time dependency	increased	+		+	+

++ Increase +/- indifferent -- Decrease
+ Slightly increased - slightly decreased

Important factors that influences the flow properties of the candidate grouts are the proportions of palygorskite and water in the mix. An increase in the palygorskite content demands an increasing in water content will lead to a decrease in both the yield stress and plastic viscosity, as shown previously, grout recipe 3 compared with 2.

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