SEPTEMBER 2018 M.Sc. in Civil Engineering AHMED JAAFAR ALI AGHA

REPUBLIC OF TURKEY

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GRADUATE SCHOOL OF

NATURAL & APPLIED SCIENCES

COMPARISONS OF CEMENT-BASED GROUT WITH GEOPOLYMERIZED (COLD-BONDED) STABILIZER INCLUDED METAKAOLIN AND SLAG

M.Sc. THESIS

IN

CIVIL ENGINEERING

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AHMED JAAFAR ALI AGHA

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Supervisor

Assoc. Prof. Dr. Hamza GÜLLÜ

by

Ahmed Jaafar ALI AGHA

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Ahmed Jaafar ALI AGHA

ABSTRACT

COMPARISONS OF CEMENT-BASED GROUT WITH GEOPOLYMERIZED (COLD-BONDED) STABILIZER INCLUDED METAKAOLIN AND SLAG

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The objective of this research was to investigate the rheological parameters, hardened properties, setting times of cement based grouts containing geopolymerized stabilizer (metakaolin (MK), slag(SL)) produced from geopolymerization process (cold bonded) and compare it with cement based grout containing (MK, SL). All mixes were made with water-to-binder ratio (W/B) of 1. The replacement of cement by different stabilizer was varied from 0% to 100% (by mass), and dosages of most stabilizers were varied according to rheological performances. The properties investigated in this study were: rheological properties (apparent viscosity, yield stress and plastic viscosity), fresh states (mini-slump, marsh funnel, bleed capacity and final setting time) and mechanical properties (compressive strength, Ultrasonic Pulse Velocity). The results showed that rheological behaviors were affected obviously by surface area, particle size distribution and hydration proses of stabilizers. While strength characteristic affected by dosage rate of MK and hydration process. All stabilizers suspensions behaved as the Bingham fluids and were stable for W/C = 1.0. Results indicate that the use of MK increased yield stress, plastic viscosity and flow time, reduced mini-slump, bleeding ratio and setting times due to the higher surface area associated with an increase in the water demand. MK improved compressive strength at 10% and by mixing with geopolymerized slag stabilizer (GSL). While geopolymerized metakaolin stabilizer (GMK) effect is less than MK and more sufficient fluidity. Whereas the SL and GSL has insignificant change on rheological properties but improved the mechanical properties at 10%, 30% for SL and GSL respectively.

Key Words: Grout, rheological, geopolymer, geopolymerized stabilizer, metakaolin, slag

ÖZET

JEOPOLYMERİZE METAKAOLİN VE CÜRUF (SOĞUK BAĞLANTILI) İÇEREN GROUTLARIN KARŞILAŞTIRILMASI

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68 sayfa

Bu araştırmanın amacı, jeo-polimerizasyon işleminden (soğuk yapıştırılmış) elde edilen jeo-polimerize stabilizatörü (metakaolin (MK), cüruf (SL)) içeren çimento esaslı harçların reolojik parametrelerini, sertleştirilmiş özelliklerini ve ayar zamanlarını araştırmak ve çimento bazlı olarak karşılaştırmaktır. harç içeren (MK, SL). Tüm karışımlar su-bağlayıcı oranı (W / B) 1 ile yapılmıştır. Farklı stabilizörlerle çimento değişimi% 0'dan% 100'e (kütle) değişmiştir ve reolojik performanslara göre çoğu stabilizatörün dozajı değiştirilmiştir. Bu çalışmada incelenen özellikler şunlardır: reolojik özellikler (görünür viskozite, akma gerilmesi ve plastik viskozite), taze haller (mini çökme, marş hunisi, sızma kapasitesi ve son ayar zamanı) ve mekanik özellikler (basınç dayanımı, Ultrasonik Darbe Hızı). Sonuçlar reolojik davranışların yüzey alanı, parçacık boyutu dağılımı ve stabilizatörlerin hidrasyon işlemi tarafından açıkça etkilendiğini göstermiştir. Mukavemet özelliği MK dozaj hızından ve hidrasyon işleminden etkilenir. Tüm stabilizatör süspansiyonları Bingham akışkanları olarak davrandı ve W / C = 1.0 için kararlıydı. Sonuçlar MK kullanımının akma geriliminde artış, plastik viskozite ve akış süresi, azaltılmış mini çökme, kanama oranı ve su ihtiyacında artışa bağlı yüksek yüzey alanından dolayı kalma sürelerini arttırdığını göstermektedir. MK,% 10 oranında sıkıştırma mukavemeti ve geopolimerize cüruf stabilizatörü (GSL) ile karıştırılarak geliştirilmiştir. Jeo-polimerize metakaolin stabilizatör (GMK) etkisi MK'den daha az ve daha fazla akışkanlıktır. SL ve GSL'nin reolojik özellikler üzerinde önemsiz bir değişim göstermesine karşın, SL ve GSL için sırasıyla% 10,% 30'luk mekanik özellikler geliştirilmiştir.

Anahtar Kelimeler: grout, reolojik, jeopolimer, jeo-polimerize stabilizalar, metakaolin, cüruf

To My Parents, I am grateful to God for having parents like you. Your unconditional love and support made my every achievement possible.

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First praise is to Allah, the Almighty, on whom ultimately we depend for sustenance and guidance. I am ever grateful to Allah, the Creator, and the Guardian, and to whom I owe my very existence.

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LIST OF SYMBOLS/ABBREVIATIONS

τ_0 (pa)	Yield stress
γ (s^{-1})	Shear rate
τ (pa)	Stress
μ_p (Pa.s)	Plastic viscosity
UCS	Unconfined Compressive Strength
ASTM	American Society for Testing and Materials
ACI	American Concrete Institute
SL	Blast Furnace Slag
MK	Metakaolin
GMK	Geopolymerized Metakaolin stabilizers
GSL	Geopolymerized Slag stabilizers
Na_2SiO_3	Sodium silicate
NaOH	Sodium hydroxide
C	Constant
W/b	Water per binder ratio

CHAPTER 1

INTRODUCTION

1.1 General

Grouting for ground engineering is a method for filling the voids, fissures or cracks standing in the soil and rock to improve water-tightness or mechanical characteristics of the grouted materials. Three modules of grouting materials are generally recognized: i) suspension-type grouts, ii) emulsion-type grouts and iii) solution type grouts. The suspension-type grouts contain clay, cement, and lime, while the emulsion-type grouts include bitumen and the solution-type grouts include a wide variety of chemicals. It includes the injection of appropriate suspension and/or solution into soil and rock to either decrease the permeability or improves the mechanical properties. The corrected choice of the grout mix dependent on rheological properties are considered significant parameters for layout the grouting effort. The grout mix selection is also dependent mainly on trial and error test; a balanced grout mixture was submit by (Lombardi and Deere, 1993), i.e., that means no separation in grout mixtuer between cement and water.

Grouting is commonly used to reduce permeability, improving the hardened characteristic of soil strength or rock foundation materials for structure or drilling upholding goal. Grout treatment is based on the fact that penetration into new or existing structures and foundations can lead to lower leakage rates, improved support capacity, improved subsidence-related properties such as elastic modulus and porosity, reduced hydrostatic forces acting on structures, The slope for achieving a specific design goal is a flow path, To control the water for excavation required to suppress the internal erosion of foundation and levee material and / or to promote the stability of dehydration or drilling. For important hydraulic applications, grouting is usually one of several lines of protection (Army U.S, 2017).

The basic categories of grouting (Figure 1.1) distinguished by the mode of entry into the soil or rock are:

i). Permeation grouting (intrusion, penetration)   ii). Displacement grouting

iii). Compaction grouting (including slab-jacking)   iv). Jet Grouting

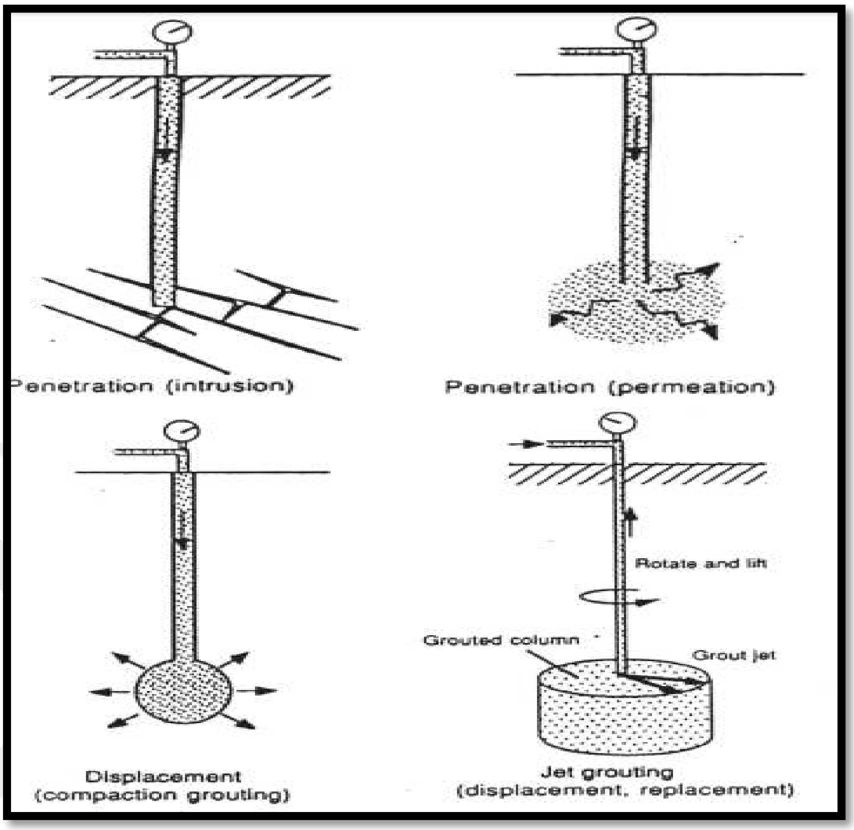

Figure.1.1 Basic Modes of Grouting

One of the main topical of this study was to verify the (cement-based grout + aluminosilicate sources and geopolymer stabilizers) characteristics of the grouts in fresh (rheological) and hardened states. Rheology (Banfill, 2014) (Westerholm, 2006) shows that rheological science focusing on material deformation and flow, flow confirmation is related to the relationship between strain, time, stress and velocity distortion. The rheological behavior of any grout mixture is thought to be very complicated due to the interaction of some physical and chemical properties of any mixture arising from both the solid and fluid phases (Hansson, 1993).

The use of geopolymer for grouting is limited, its results compared to cement-based grout can be considered beneficial. Fundamental differences have been found between geopolymer paste with metakaolin source and standard ordinary Portland cement on flow and rheological influences (Favier et al., 2014), where the viscosity of paste was mainly controlled by the high viscosity of geopolymer. Apart from using geopolymer for grouting surveyed above, some past studies have been employed a different application of the alkaline activators (i.e., sodium hydroxide and sodium silicate) and aluminosilicate sources (i.e., metakaolin, slag, fly ash, etc.) that are the main ingredients of geopolymer material in order for producing a cold bonded lightweight aggregate (say "geopolymer aggregate") through a geopolymerization process (Jo et al., 2017; Bui et al., 2012; Van Jaarsveld and Van Deventer, 1999) . The cold bonded aggregate by the geopolymerization process has been deserved a further research due to engineering performances and environmental concerns. Thus, it can be suggested that when passing the sieves of fine particle sizes such as fine sand, clay, silt, etc., the cold bonding aggregates could potentially become a good candidate for grouting as a non-traditional stabilizer (say "geopolymer stabilizer") in binder composition (i.e., cement+stabilizer) through a possibility of high-quality cement-based grout mixtures.

Water to cement ratio (w/c) is one of the important limitations in the cement-based grout. As they play an important role in the penetration ability of the grouts into the soil or penetrate the crack in concrete (Akbulut and Saglamer, 2002). Different ranges of w/c ratios can be express the used of grouting for different applications. For the restoration and integration of the constructing structure, the w / c ratio of the cement grout domain from 0.5 to 1.5 (Miltiadou, 1991). In addition, for jet-grouting applications it must be behave like a fluid to permeate in to granules or particles of soil and rocks, the w/c ratio varies between 0.6 and 2 (Moseley, 1992). That means that the water to cement ratio (w/c) 1 is used in most applications of grouting.

The rheological behavior of a fluid determines the relationship between pressure and flow for various geometries, and is therefore of fundamental importance when it comes to theoretical treatment and modeling of flow situation. Moreover, understanding and controlling the rheology of the used grouts is essential for their successful use in particle grouting operation (e.g choosing the right pressure or estimating the penetration length and grout volume).

By measurement of the rheological properties and by knowing how they are influenced by different factors (concentration, additives etc.). Measurements are also needed in order to achieve an optimization of the two conflicting mechanisms, stability, and favorable flow properties.

1.2 The aims of the research

For grouting applications in a part of a detailed study, this present paper aims to investigate: i) the rheological (shear stress versus shear rate, shear stress versus viscosity (i.e., apparent viscosity), yield stress, plastic viscosity), ii) fresh (mini-slump, flow time, bleeding, setting time) and iii) strength (unconfined compressive strength (UCS) and ultrasonic pulse velocity (UPV)) properties of non-traditional cement-based grouts included geopolymer stabilizers produced through the geopolymerization of metakaolin and slag, in comparison with traditional cement-based grouts.

1.3 Organization chart of the thesis

Chapter 1: general background and the introduction of the study were given. General

Information about the grouting techniques.

In Chapter 2: detailed literature information related to rheological and haedened properties of different grouts mixture was given and discussed.

In Chapter 3: an experimental study of rheological and fresh properties of cement-Based grouts mixing with (geopolymerized stabilizers, metakaolin, and slag) prepared for this study were presented. The properties of the materials that used, mixture design, mixing procedures, the preparation of samples and the testing procedures.

In Chapter 4: display the experimental results and discussion the fresh and hardened properties of different types of grout mixture. Results, figures, and evaluation are presented and discussed.

Chapter 5: contains the conclusions of the tests and recommendations.

CHAPTER 2

LITERATURE REVIEW

2.1 Grouting

Grout for subsurface construction is a process for filling voids, fractures or cavities in rocks and giving an improved water tightness or hardened properties to the grouted substance. Grout is most frequently used as an costly repair tool after a project problem has happen. As stated by the Federal Highway Administration Bureau, "Grout as a means to establish soil is an costly and time-consuming process for repairing shaft sinks and collapse, so even if extremely great attention is paid that can be trustworthy (www.fhwa.dot.gov).

2.2 Deep Soil Mixing

The Deep Soil mixing method (DSM) was invented in Japan and Scandinavia and subsequently further developed by authors and construction companies. Its use to improve the strength and sealing weak and permeable soils is growing around the World. This technique leads to a marked improvement in the mechanical and physical properties of the soil that mixed with cement or composite binder forming so-called soil mixture (or soil cement).The resulting stabilized soil material generally have a lower permeability, higher strength and lower compressibility than the original soil. DSM technology is based on simple principles, but on the one hand the accuracy of implementation including soil mixing and geotechnical design, and on the other hand a tool suitable for mixing soil with grout to meet the imposed specifications which is also required.

Basically, there are two different mixing methods. The standing soil to be improved can be mixed mechanically either with a dry binder dry deep soil mixing or slurry incorporating a binder wet deep soil mixing (wet DSM)

2.2.1 Wet deep soil mixing (wet DSM)

Wet soil mixing is an optional method of injecting fluid grout into the soil matrix with several means of homogenizing the matrix. The injection can be carried out under modest fluid pressure of 200 to 400 psi or very high pressure (e.g. 10,000 to 20,000 psi). The low pressure system uses a device similar to a drilled shaft rig, where a large diameter multi paddle tool slowly rotates and enters the soil during the injection of grout slurry (Figure 2.1). A mixing tool that can move up and down along the column length to improve the homogeneity of the soil mixture ensures thorough mixing of the slurry and the soil. This process will cause spoilage of up to 30 or 40% of the final column volume, which can be a problem if the soil is contaminated. Jet grouting is more commonly proceduer for wet soil mixing by High-pressure

2.2.2 Dry deep soil mixing (dry DSM)

In contrast to the wet method, Dry deep soil mixing (dry DSM) In contrast to the wet method, dry soil mixing is only can be applied in soils that have enough moisture content to allow chemical reaction with the soil and groundwater of stabilizing binders injected in dry form. The basic benefit of dry blending is that it is highly productive, has little damage, and has a stabilizing effect at low cost in deep sediments of very weak soils including organic soil. In addition, Operation at low temperature is possible. A typical device for dry DSM (Figure 2.2) is intended for a fixed or movable binder storage and supply plant and a drilling device for installation of columns with special mixing tools at the end of the mixing rod. As a principle, the plastic clay and silt are strengthened with cement containing lime or lime, and in organic soil a mixture containing blast furnace slag is used.

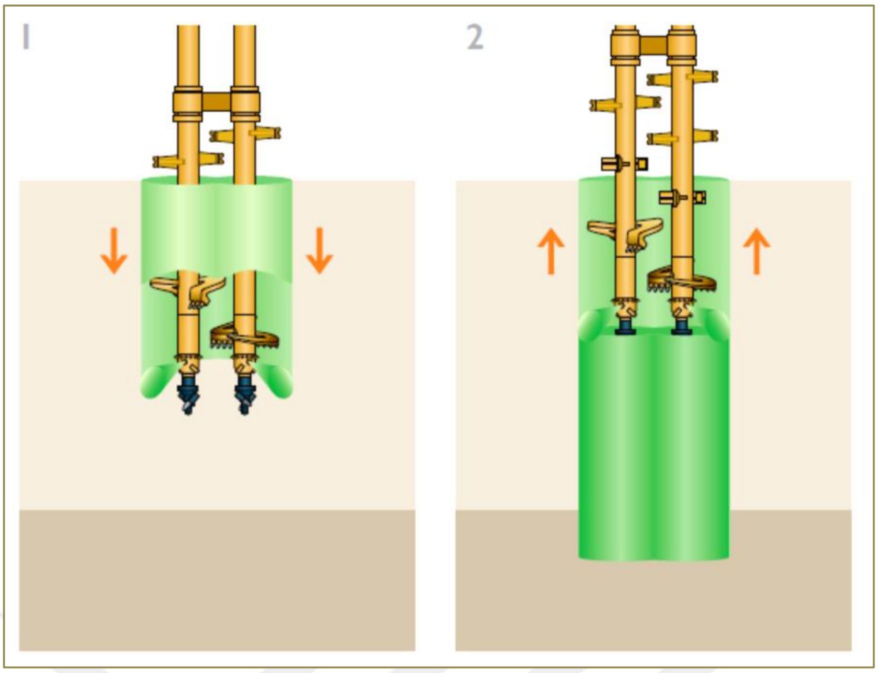

Figure.2.1 wet deep soil mixing

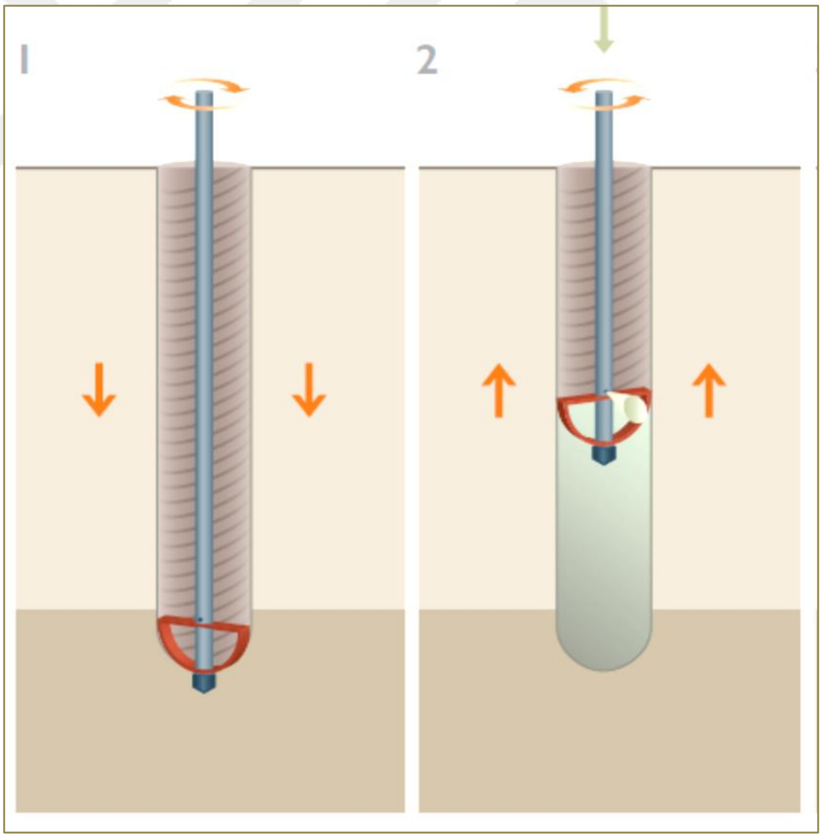

Figure.2.2 Dry deep soil mixing

2.3 Studies on Rheological Characteristics of Cement based Grout

Several researchers have studied the rheological properties (e.g. yield stress and viscosity) of grout including other influence factors such as mixing time, stability (bleeding), Flow time in pipes, the degree of saturation and additives since 1954. However, the information from all these studies are found more concentrated in the properties of solution grout, microfine cement grout or Portland cement grout with additives (e.g. Slag, fly ash etc.) Due to low permeability of pure cement grout, its high viscosity and short setting time. Some comments/findings extracted from the past research works are summarized as follows.

(Klein and Poloivka, 1958) taken schematically the stages of cement grout after mixing as dormant, setting and hardening with the strength of grout increasing with curing time almost in exponential or power function. (Raffle and Greenwood,1961) presented a relation (Figure 2.3) between the rheological properties of cement based grout and its permeation capacity to soil and indicated that injectability of grout can be controlled by arranging viscosity and shear strength in the beginning and last stage, respectively.

Obvious increases in the viscosity and shear strength were reported for the cement grout with (w/c) not exceeding 0.6 as shown in Figure 2.3 (Tattersall, 1955; Nessim and Wajda., 1965) Theoretical understanding attempts and the ultimate recognition of the importance of thixotropic or structural destruction and consequently the application range of flow curve / hysteresis loop is limited. (LittleJohn, 1975) confirm that the range of water to cement ratio is between 0.4 and 0.45, can be used in injection process, it is lightly placed in a small diameter borehole and has sufficient continuity after injection to act as a strengthening medium Grout with sufficient fluidity to hold.

(Deere et al., 1982) classified cement grout as stable grout based on bleeding no exceeding 5% after 2 hours from completion of mixing and commented that low range of bentonite usage seems to be convenient, to improve the pump ability and penetrability it must be sufficient reduce sedimentation and bleeding but not so great as.

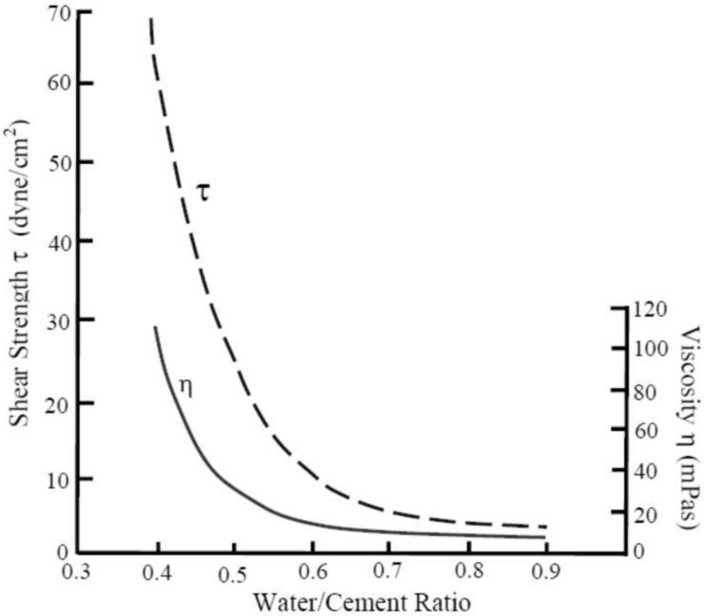

Figure. 2.3 Shear Strength and Viscosities for Cement Pastes with Varying Water/Cement Ratio (after Raffle and Greenwood, 1961)

(Branfill, 1983) showed that a mixing time of about 5 minutes is sufficient In order to obtain certain properties for both the yield stress value and the plastic viscosity, However, the volume of the mixed grout is not known.

(Paoli et al., 1992) discussed the fundamental observation on cement-based grout and commented that permeation is controlled by the size of the particles more than by viscosity and yield stress of the grout material. The grout penetrability could be improved by reducing the size of the cement grains and improving the grout's rheological properties, increasing the stability under pressure infiltration and reducing the yield stress values.

(Hakansson et al., 1992) studied the rheological properties of micro fine cement grouts with additives and concluded that as the specific surface area and bentonite increase, yield stress and plastic viscosity increase, and reduce with increasing plasticizing admixtures.

(Krizek et al., 1992) studied the anisotropic behavior of cement-grouted sand and concluded that the permeability of grouted sand depends on the direction of flow relative to the direction of sedimentation. For sands injected with grout at a water/cement ratio less than 3.0, the horizontal permeability is about two orders of magnitude higher than the vertical.

(Vipulanandan and Shenoy, 1992) studied the properties of cement-based grouts and the sands incorporated with grout and additives and indicated that the maximum particle size cannot be exceeded referenced with 1/3 to 1/10 the size of a void because of penetrating a formation at suitable pressure and rate.

(Helal & Krizek, 1992) studied arranging of void structure in the sand cooperated with cement base grout demonstrated that void structure grouted with a native cement grout can be considered an assignment of w/c ratio and precipitation behavior of grout mixture particles.

(Shroff et al., 1996) studied the rheological properties of micro fine cement dust (MCD) grouts and reported that MCD grout is able to penetrate medium sand having permeability, k= 7.89 x 10-3 cm/sec. He also commented that MCD grout possesses not only penetration ability in medium to fine sand comparable to many chemical grouts but also imparts higher adherent strength to the grouted mass.

(Hanehara and Yamada, 1999) studied the interplay between the chemical admixture types and cement with respect to the cement hydration point. The contribution of cement, chemical admixture, mixing method of admixtures and w/c ratio have an important effect on the rheological properties of mortars. The test results founded that the polycarboxylate type superplasticizer was influenced by the rate of alkaline sulfates amount in cement.

(Perret et al. 2000) studied the influence of sand saturation by grout ability and concluded that the spread of grout through porous media is affected not only by the particle size distribution of the soil and cement, the water permeability of the soil and rheological characteristics of the grout but also the degree of saturation of sand. Water dilution of the grout is greater in unsaturated soil than in saturated soil, where the grout can displace the water, resulting in a layer of grout intermixed and diluted with water. The suction resulting from capillary pressure in the case of unsaturated sand and the noncontinuous aqueous medium formed by the pore water in the soil is thought to have led to greater water dilution in the case of unsaturated sand.

(Svermova et al., 2003) conducted an experimental design, to evaluate the integrated influences of the different factors such as w/b ratio, amount of both viscosity agent and superplasticizer, and constitution of limestone powder replaced with cement. Rheological properties of the mixtures were investigated by using marsh flow time cone, mini-slump, Lombardi cohesion plate meter and also bleeding(stability) and viscosity. Test results showed that different rheological properties of cement based grouts obtained from the statistical method were directly related with the powder of limestone and the dosage of VA and SP.

(Eriksson et al., 2004) studied several tests whereby a number of estimation of grout features related with the rheology and penetrability of fresh mixtures were conducted. Test results showed that rheological characteristic of grout mixtures were more several than the penetrability related with parameters; thus, the other parameters could importantly affect the grout features such as cement condition, w/c ratio, and mixing materials.

(Axelsson and Gustafson, 2006) studied expansion of a method to evaluate the shear strength of cement for grout in the field in an easy and strength way. This method was made of a stick sinking in the grout because of direct estimating the shear strength. Test results indicated that estimation of the shear strength completed in the lab was repetitively comparing with field results.

(Banfill and Frias, 2007) investigated the influence of recycled metakaolin rheological properties and conduction calorimetric of cement pastes and compared the results with the influences of commercial metakaolin. Their test results showed that similar influences on the rheological properties of the cement base grout mixtures were observed for both calcined paper sludge and metakaolin.

(Mohammed et al., 2014).studied the rheological properties of cement based grout containing talc or palygorskite were investigated for fluidity during injection and rapid strengthening. Grout consists of 1) talc of fluidity and strength by reaction with cement, 2) palygorskite (attapulgite) for early gelling due to thixotropic and 3) powdered quartz for chemical integrity, where viscometer tests, mini-slump cone tests, pipe flow tests and marsh funnel tests have studied. And the freshly prepared grouts behaved as Bingham fluids. The results have shown that the grout's fluidity was positively influenced by talc and was negatively affected by hydration of palygorskite and early cement.

(Vikan et al., 2007) investigated the “flow resistance” of grouts with respect to the effect of cement features such as fineness of cement and composition of clinker and three vary of plasticizers types. This study was done with respect to The area under the shear stress-shear rate curve. Test results showed that either linear function or exponential function depending on type and amount of plasticizer could be obtained. It was concluded that the correlation between a mix of pure cement and cement with fly ash,(4%) limestone filler at a constant amount of silica fume was observed. Vikan and Justnes (2007) investigated the rheological properties of cement based grout. In their study, they replaced cement with raw silica fume (SF), densified (SF) and limestone. The flow resistance was obtained from the area under the flow curve to indicate the influence of SF. It directly depended on the dispersing ability of the plasticizer. It was concluded from the test results that increasing of SF replacing with cement as independently of plasticizer type increased the gel strengths of the mixtures.

(Yahia, 2011) investigated the influence of water/cement ratio, high-range water reducer (HRWR) cement proportions, and supplementary cementitious materials (SCM) on non–linear rheological behavior of several grout samples experimentally. The yield stress of samples decreased because of the constitution of high-range water-reducer and however increasing deformability, while silica fume improved the mechanical and durability properties of mixtures. Test results showed that shear thickening behavior of the grouts mixtures was observed for lower w/c ratios, while the shear-thinning behavior was seen at high w/c ratios.

(Bouras et al., 2012) investigated the effects of the amount of -modifying admixtures (VMA) on the stable condition rheological properties of cement based grouts experimentally. These features contain the yield stress, fluid consistency index and flow behavior index. It was concluded from test results that the rheological behavior of the grouts is generally dependent upon shear rate ranges, therefore shear-thinning behavior at lower shear-rates was observed for grout samples, while the grouts started to show shear-thickening behavior at higher shear-rates because of inverse interactions between the solid particles.

(Sonebi et al., 2013) Test results indicated that with increasing SP the fluidity and the setting times started to increase, the rheological properties, flow time and plate cohesion started to reduce. While the increase of the (VMA) caused a reduction in the fluidity and increasing plate cohesion, Marsh cone time yield stress, and plastic viscosity. It was concluded from the test results that Increase in flocculation plate, flow time, yield stress, plastic viscosity and improvement in compressive strength were observed. In addition, mini-slump and setting times started to reduce with respect to using MK. The rheological properties of grouts for the fresh condition influences of micro and Zapata et al. (2013) investigated nano-SiO2 under several amounts of carboxylated-polyether-copolymer-type superplasticizer. The test results showed that marsh cone test indicated a nonlinear behavior in samples with micro and nano SiO2 particles and at saturated condition chemical material incorporating, flow time and decreasing of fluidity was discussed with respect to control samples.

2.4 Metakaolin

the metakaolin is a pozzolanic material with a high pozzolanic activity that is composed of silicate or alumino-silicate elements produced from kaolinite type of clay (at temperatures of around 800 ± 100°C). When replaced with cement by small amounts (i.e., 5%, 10%), it has the effect of avoiding segregation, leading loss of fluidity (Santos et al., 2017) and increasing compressive strength (Frias et al., 2000; Santos et al., 2017).

(Curcio and DeAngelis, 1998) by a coaxial cylinder rotational viscometer, the cement pastes containing metakaolin has been studied. They show a dilatant behavior which is tightly dependent on the water/binder ratio, on the level of cement replacement by metakaolin and on the powderiness of the latter. The angular and tabular shapes of the metakaolin particles are causes dilatancy

(Bai and wild, 2002) studied the effects of FA and MK on heat evolved using embedded system thermocouples. Mixtures of mortar were placed in 150mm plywood cube molds and thermally isolated Plywood of expanded polystyrene with a thickness of 100 mm and other plywood. Increase in replacement level and reduction in temperature rise in FA system were noticed. It be also studied the using of MK and FA in ternary total cement exchange level blend to 40%. These seemed to have compensation influence on temperature increase as shown in Figure 2.4

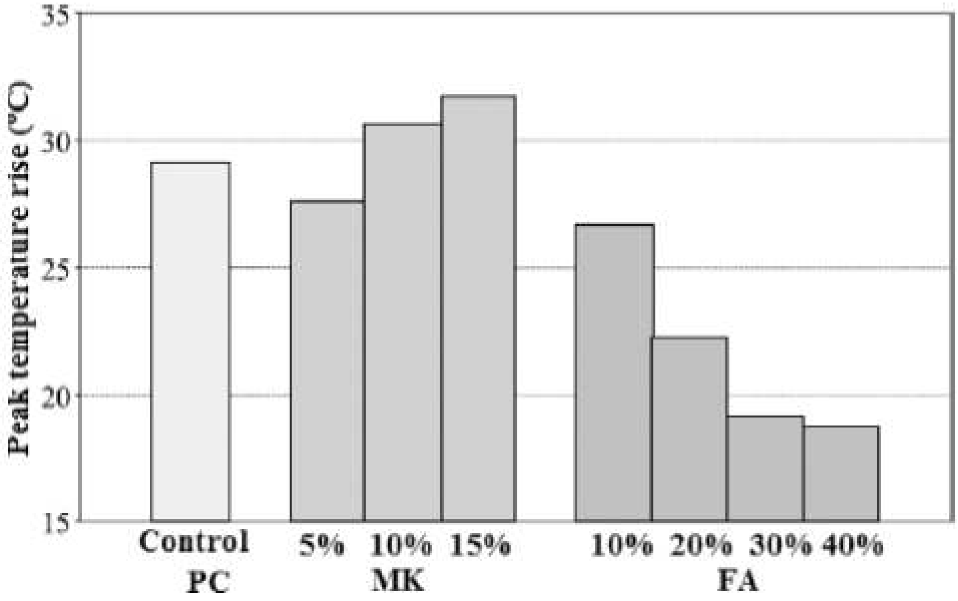

Figure 2.4 Peak temperature rise for PC, MK and FA blended mortar. Reprinted with permission from J. Bai and S. Wild

(Sonebi et al. 2013) Contain partially substituted cement in MK and cement grout mixture at various levels ranging from 6% to 20% by weight. The w/b ratio of 0.4 was fixed and various dosages of SP and viscosity-modifying agent were used. They reported that the MK inclusion, increased yield stress, marsh flow time and cohesion plate due to the higher surface area associated that increases in water demand. MK reduced the setting time and mini-slump.

(Mansour et al., 2015) Cement that was partially replaced with a paste mixture with MK at levels of%, 5%, 10%, 15% and 20%. They investigate that at the levels of 10%and 15% MK the fluidity of grout mixture have been improved, especially at indicated level of replacement.

From the study above on the literature in this section, the most of the authors reported that the MK inclusion in the mixture reduced the workability (Figure. 2.5). The decrease in workability process due to the attachment of MK was attributed to the MK high chemical activity, this leads to more exhaustion of water (both for acceleration in cement hydration and for initial pozzolanic reaction) than in the case of normal PC mixtures. The reduction in process ability produced by MK is a function of the high specific surface area that absorbs higher moisture.

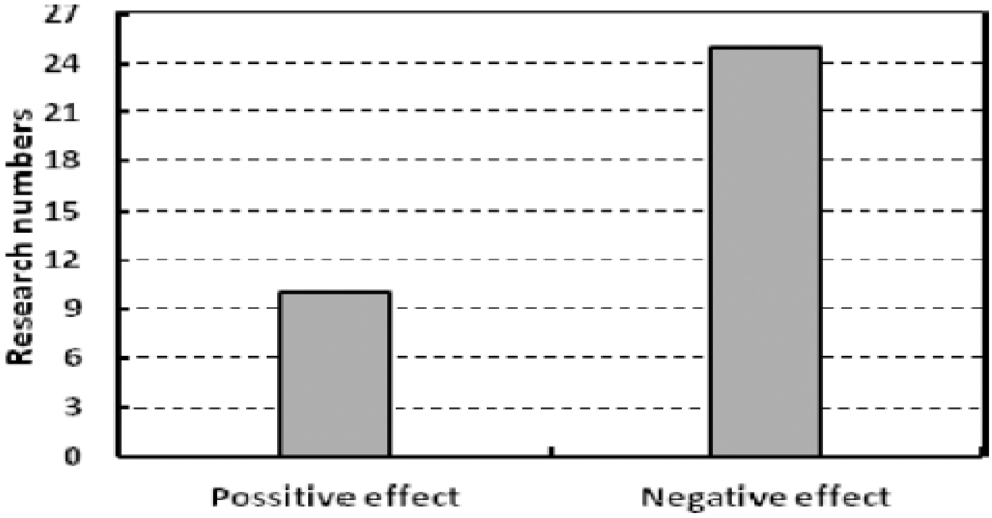

Figure 2.5 Effect of MK on the workability (Rashad, 2014)

2.5 Ground Granulated Blast Furnace Slag

By-product of the iron manufacturing industry, The Blast-Furnace slag was produced (Figure 2.6). At a temperature of about 1500 ^{0}C to 1600 ^{0}C, Iron ore, coke, and limestone are nourished into the furnace and the resulting melted slag floats above the melted iron. The melted slag has a composition of about 30% to 40% SiO2 and about 40% CaO that is closely parallel to the chemical composition.

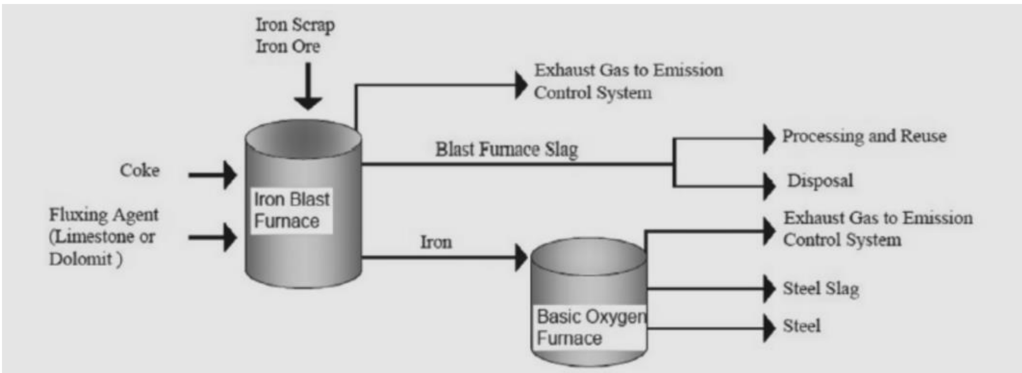

Figure 2.6 (Production process of slag Piatak et al., 2015)

Currently the most commonly used pozzolan is fly ash, silica fume highly reactive metakaolin, ground blast furnace slag and other materials are also used as pozzolans.

(Atezeni et al., 1985) Rheology studies were conducted on Portland cement and grout made of blended cement containing pozzolana, blast furnace slag, and fly ash at W/CM in the range of 0.30 to 0.45. The authors compared the degree of forecast of grout flow characteristics using Herschel-Bulkley, Casson, Eyring, and Vom Berg models. The authors found that the best fit was obtained with the Eyring and Herschel-Bulkley models, but the vom Berg model was suitable only at low shear rates.

(Shariq et al., 2008) The influence of the curing procedure on the compressive strength development of concrete blended with cement mortar and blast furnace slag was studied. The Development of compressive strength of cement mortar incorporating 20%, 40% and 60% replenishment of GGBFS for different types of sand and strength expression of concrete by 20, 40 and 60% replacement of GGBFS for two grades of concrete have been studied. The test results show that incorporating 20% and 40% GGBFS is very effected for increasing the compressive strength of the mortar after 28 days and 150 days.

(Park et al., 2005) carried out that by using a cylindrical spindle rheometer, the rheological properties of GGBFS contained within cementations materials were studied. As a parameter to replace PC with GGBFS, yield stress and plastic viscosity were investigated when substitution level was increased by 45% up to 45%, and the results are shown in Fig. 3. As the GGBFS in the mixture increases, the yield stress appears to decrease and increase; however, at GGBFS the plastic viscosity decreases. GGBSF behaves as a good flow adjuvant up to 30%. GGBSF has a larger specific surface area than cement and also has many spherical particles, and low hydration reaction capacity.

(Leung et al., 2010) they studied BS 15167-1, which needs to have a minimum specific surface area of 2750 cm 2 / g for GGBS (BS 15167 - 1: 2006). In China, GGBS is classified into three grades, That is, S 75, S 95, S 105. GB/T18046 requires a minimum surface area of 3000 cm2/g for grade S75 GGBS higher than BS EN requirements (GB/T18046-2008), 4000 cm2/g for grade S95 and 5000 cm2 / g for grade S105. It has been reported that slag having a specific surface area of 4000 cm 2 / g to 6000 cm 2 / g markedly improves the performance of GGBS concrete. (Kogbara et al., 2011) they investigated the possibility of GGBS activated with cement and lime for stabilization/solidification (S / S) treatment of mixed contaminated soil.

2.6 Rheological Test of Grouts

Both viscosity and yield stress are considered the of the most important determinants of grouting.and tested by using Viscometer instrument .where, there are many types of viscometer instrument .in this paper (proRheo R180 Instrument, Germany) has used. Is a portable rotary viscosity scale and stand-alone based on solid principles of operation. It's unique because it's easy to use and measure how wide. The Rheomat 180 works with all types of measurement systems: concentric cylinder, cone-plate, and measuring systems conform to DIN 53019 or ISO 2555 etc. All related rheological parameters are shown simultaneously and the results can be stored and put out to the computer or printer.

(Yahia and Khayat, 1999), they are studied the rheological properties of cement based grout by using two coaxial–cylinder viscometers (FANN 35and CHAN A35) having a gab size of 1.17 mm were employed. Their experience was focused on yield stress and by using multiple models of rheological the results were discussed.

2.7 Effect of the Mixer Type and Mixing Procedure

(Kantro, 1980) has been reported that the first use of a high speed shear mixer to produce cement paste in connection with the development of a mini slump cone test. As Kantro used high-speed shear mixers, many authors used this mixing technique for cement paste rheology in later years. Some researchers used a blender as a cement paste rheology study (Williams et al., 1999) for a Hobart paddle mixer as a high speed shear mixer (up to 10,000 rpm) and a low speed shear mixer (100 rpm). They found that when comparing the rheological parameters, yield stress results in both cement paste tests using two different mixers with the concrete slump test as shown in Figure 2.7, the cement paste mixed in the mixer is concrete He stated that it represents the performance more accurately. This is in particular a study (Helmuth, 1987) showing that cement paste is sheared at energy and speed in a mixer, in contrast to the low shear rate of the Hobart mixer during mixing. Therefore, it is important to prepare cement paste using a high shear mixer to measure representative rheological parameters (Yang and Jennings, 1995).

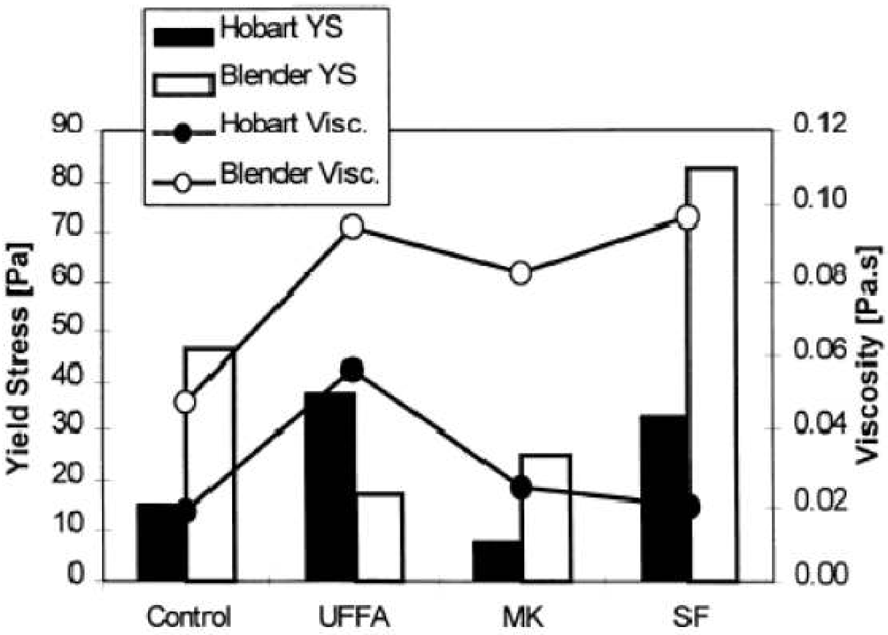

Figure 2.7 Effect of mixer on the Rheological Properties of Cement Paste.

(Ferraris et al., 2001).

CHAPTER 3

EXPERIMENTAL STUDY

This chapter reviews the research work conducted on the cement and different additives (fundamental research, field test, case study if available, etc.). It also presents a detailed review of the most used cementitious materials and admixtures (chemical and mineral).

3.1 Materials

3.1.1 Ordinary Portland cement (OPC)

Ordinary Portland cement kind CEM I 42.5 R similar to ASTM C150/C150M-17. Was utilized to using for grouting mixtures for determination the rheological and mechanical properties. OPC having Blaine fineness of 394 m^2/kg and 3.14 specific gravity. Table 3.1. Shows the chemical composition and physical properties of OPC.

3.1.2 Metakaolin (MK)

As a kind of mineral admixture, metakaolin (MK) is a model precursor because it usually contains a smaller amount of impurities than other precursors. The chemical composition of MK can vary but is close to 2SiO2·Al2O3. When metakaolin is added, it will contribute to the formation of additional C–S–H (calcium silicate hydrate), Metakaolin is processed by calcining kaolinite at elevated temperatures to remove water. The chemical compositions of different MKs provided by manufacturers are given in Table 3-1. with a surface area of 350-410 m2/g. The particle size distribution of MK is illustrated in Figure.3.2.

3.1.3 Ground Granulated Blast Furnace Slag (SL)

A by-product of the manufacturing process when steel and iron are melted down, ground granulated blast furnace slag (GGBFS) or (SL) is designated in ASTM C 989 and consists mainly of silicates and aluminosilicates of calcium. is most similar to Portland cement with respect to cementitious behavior, and is readily available.

Table 3.1 indicated to the physical and chemical compositions of SL. The particle size distribution of SL is illustrated in Figure.3.2.

3.1.4 Geopolymerized Fine Stabilizer (GMK & GSL)

Geopolymerized Fine Stabilizer (Geopolymer aggregate) used in this study as an additive in the cement-based grout and prepared by using (Metakaolin & slag) activated with alkaline solution (sodium hydroxide (NaOH)+sodium silicate (Na2SiO3). Its particles passed through a 1 mm sieve since mostly distributed in the range from 0.1mm to 0.5 mm, It was classified as (sand) according to the Unified Soil Classification System (USCS) (ASTM D2487–11).The particle size distribution of the geopolymer aggregate is illustrated in Figure.3.2.

3.1 Table Chemical and physical properties of materials

a) Chemical composition & Physical and index properties						
Constituent (%)	PC	MK	SL	Na_2SiO_3	GMK	GSL
CaO	62.58	0.2	34.19	-	-	-
SiO_2	20.25	50.62	40.42	29.4	-	-
Al_2O_3	5.31	45.7	10.6	-	-	-
Fe_2O_3	4.04	0.31	1.28	-	-	-
MgO	2.82	0.32	7.63	-	-	-
SO_3	2.73	-	0.68	-	-	-
K_2O	0.92	0.11	0.0128	-	-	-
Na_2O	0.22	0.02	-	14.7	-	-
H_2O	-	-	-	55.9	-	-
MS (modulus ratio)	-	-		2.0	-	-
Specific surface (m^2/kg)	326	13200	565	-	-	-
Specific gravity	3.15	2.6	2.9	-	1.64	2.1
Cu (Coefficient of uniformity)	-	-	-	-	8	4.33
Cc (Coefficient of curvature)	-	-	-	-	1.8	1.9
Classification (USCS)	-	-	-	-	SW	SW
GMK=geopolymer-based metakaolin stabilizer, GSL=geopolymer-based slag stabilizer						

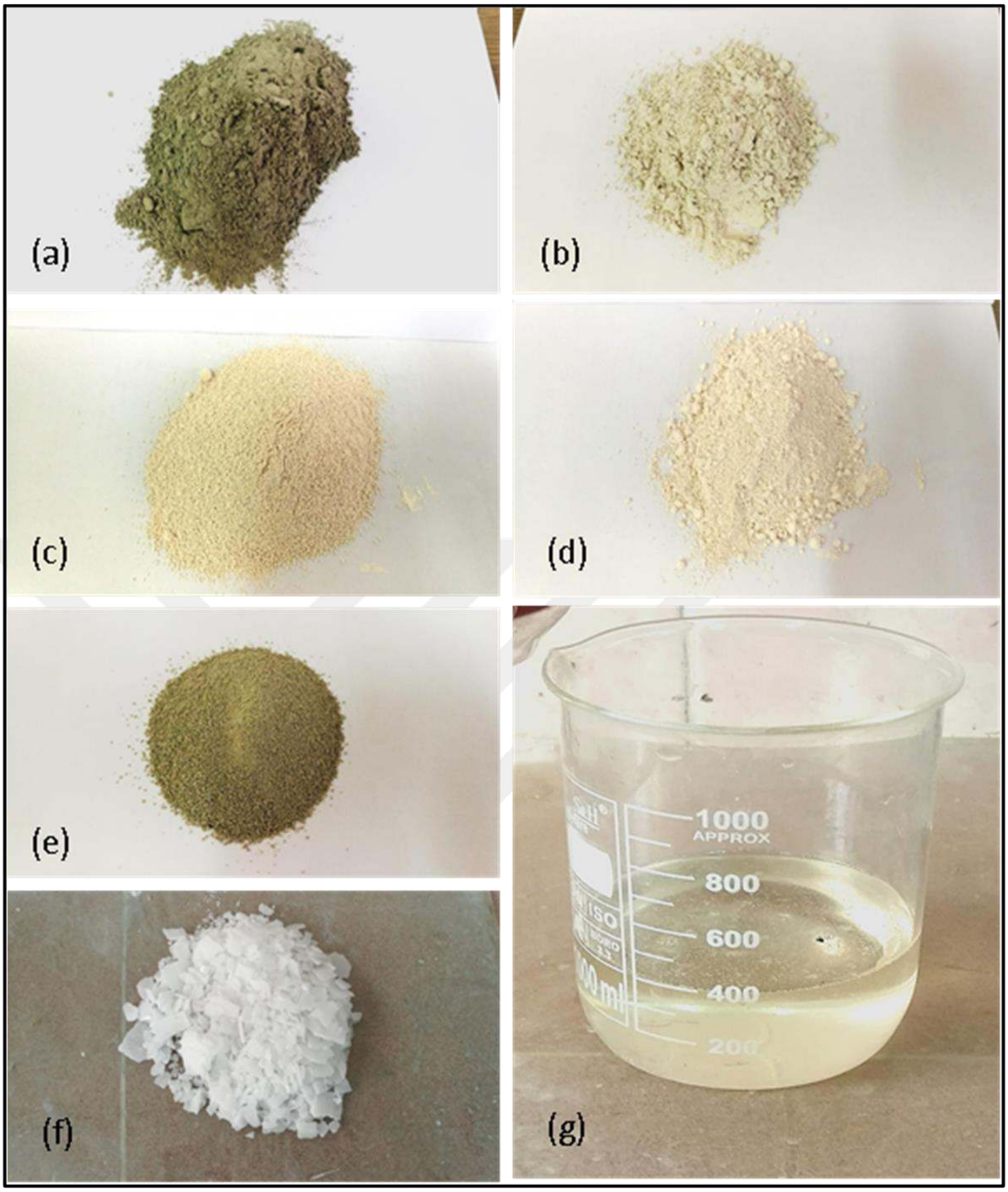

Figure 3.1 Materials used in grout mixtures: (a) Portland cement, (b) Granulated Blast Furnace Slag, (c) Geopolymerized Metakaolin Stabilizer (GMK), (d) Metakaolin, (e) Geopolymerized Slag Stabilizer (GSL), (f) sodium hydroxide, (g) sodium silicate (in gel state).

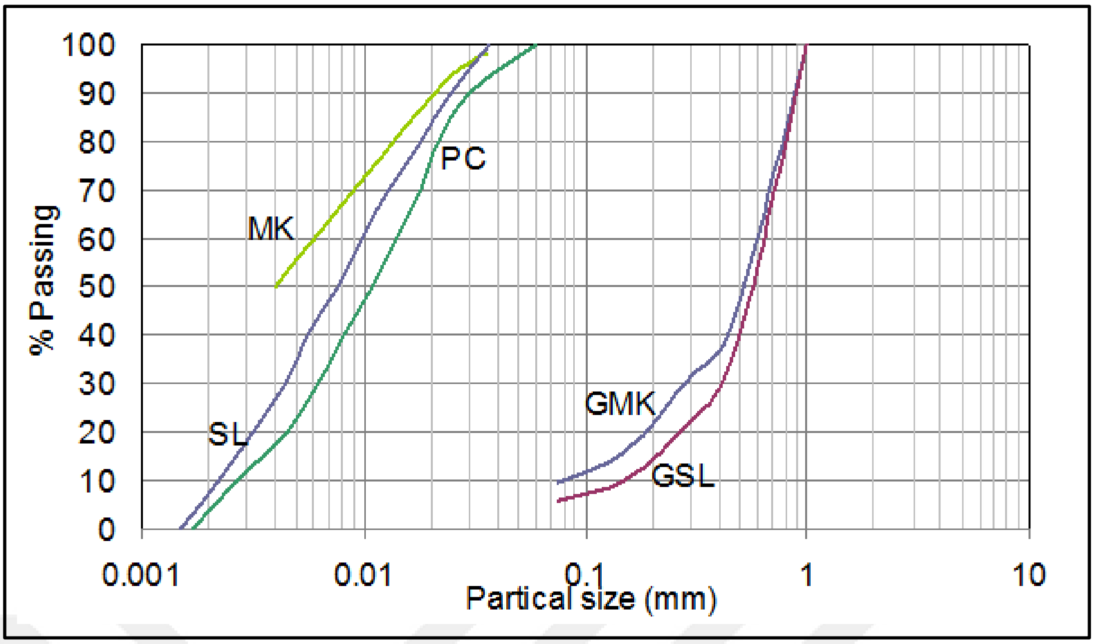

Figure 3.2 Particle size distributions (PC=Portland cement, MK=Metakaolin, SL= Slag, GMK= geopolymer-based metakaolin stabilizer, GSL= geopolymer-based slag stabilizer.

3.2 Sample preparation

In this study different types of grout have been investigated they are i) cement-based grout with metakaolin ii) cement-based grout with slag iii) cement–based grout with geopolymerized metakaolin stabilizer, iii) cement–based grout with geopolymerized slag stabilizer and iii) cement–based grout with geopolymerized slag stabilizer + metakaolin . Below, the method of preparing for each type with illustrated the proportions in the tables and figures.

All samples of grout mixtures have been prepared in a standard rotary-type of laboratory mixer (capacity of the 5L mixing bowl and 280rpm) at the rotation speed of 140rpm for 3min following similar mixing procedure at the room temperature of 23±3°C and humidity of 55–65%. Before rotation, binders (i.e., cement, stabilizers) in dry state were mixed together for 2min by hand, and then water was added to a binder for obtaining grout mixtures .The rheometer, mini cone slump, marsh funnel, bleeding and setting time tests were initiated within 1-3min, 4-6min, 7-9min, respectively as soon as mixtures were obtained. For the strength tests (i.e., unconfined compressive strength and ultrasonic pulse velocity), samples from mixtures were put into cylindrical molds (for 50mm diameter and 100mm height of specimen dimensions) and then allowed to cure 7 and 28-day for measurements.

3.2.1 Cement-based grout with Metakaolin (MK)

Consist of Portland cement and metakaolin as a binder substituted at 0%, 10%, 20%, 30%, 40%, 50%, by the dry weight of binder with various water to binder (w/b) ratio which has also measured by weight. the sample preparation has been performed in the laboratory at the room temperature of 23±30C and humidity of 55–65%. based on previous guides (Khayat and Yahia, 1998; Cristelo et al., 2013; Sonebi et al., 2006), since the cement and metakaolin were mixed in dry state by hand next for 1 minute, the water which was kept constant at a temperature of 20 ± 2 Co added to them. The grout mixtures were mixed at 150 rpm for 1 min and at 300 rpm for another 2 min until they were uniform. Thereafter, as soon as mixing was finished the grout mixture was poured into a test cup for viscometer testing, mini cone slump, marsh funnel, bleeding and setting time, for fresh properties. İn the other hand for hardened properties unconfined compressive strength (UCS) and ultrasonic pulse velocity(UPV). However, the proportions are illustrated in Table 3.2 and the samples prepared were shown in Figure 3.3.

3.2.2 Cement-based grout with Slag (SL)

Consist of Portland cement and slag (SL) as a binder substituted at 0%, 10%, 20%, 30%, 40%, 50%, 60%, 70%, 80%, 90%, 100%, by the dry weight of binder with various water to binder (w/b) ratio which has also measured by weight. And the same sample preparation referred to above have been performed.

3.2.3 Cement-based grout with Geopolimerized stabilizer (GMK & GSL)

The fine geopolymer aggregate has manufactured by the cold bonded method, since a special disc pelletizer as shown in (Fig 3.3), The angle of the disc with the disc diameter of 500 mm and the depth of 250 mm can be adjusted between 35 ° and 50 ° and a speed of 55 rpm was used. Initially the (metakaolin or slag) powder class was mixed for 2 to 3 minutes duration in the disc pelletizer, then sprayed by alkaline activator solution at the ratio of alkaline activator to fly ash 0.3 for slag and 0.35 for metakaolin by weight (Ma et al., 2012; Xie et al., 2009). Crushing strength and pelletization duration of 10 minutes and 20 minutes were adopted.(Gomathi and Sivakumar, 2012). After producing the fine aggregate it was put in plastic bags and stored for a day in humidity and temperature of the laboratory 55–65% and 23± 3, then dried in air for 7 days after the curing (air curing) of the aggregate has finished, it became present for use in the grout preparation. Since the Portland cement and geopolymer aggregate were mixed well in a dry state by hand for a minute then, the water was added to them, the blending continued for 1 min at 150 rpm and for 2 min at 300 rpm to obtain a uniform mixture. after the mixing was finished the grout mixture was poured into the testing cup for performing the viscometer tests mini cone slump, marsh funnel, bleeding and setting time, for fresh properties. İn the other hand for hardened properties of (UCS) and (UPV). The proportions are illustrated in Table 3.2

Figure 3.3 Disc pelletizer machine and geopolymerized stabilizer

Table 3.2 Mixture proportion of grout with different stabilizer

Cement-based grout with metakaolin (PC+MK)					Cement-based grout with slag (PC+SL)					Cement-based grout with geopolymer-based metakaolin stabilizer (PC+GMK)					Cement-based grout with geopolymer-based slag stabilizer (PC+GSL)					Cement-based grout with metakaolin+ geopolymer-based slag stabilizer (PC+MK+GSL)**				
PC (%)	MK (%)	PC (g)	MK (g)	W (g)	PC (%)	SL (%)	PC (g)	SL (g)	W (g)	PC (%)	GMK (%)	PC (g)	GMK (g)	W (g)	PC (%)	GSL (%)	PC (g)	GSL (g)	W (g)	PC (%)	MK+GSL (%) **	PC (g)	MK+GSL (g)	W (g)
100	0	800	0	800	100	0	800	0	800	100	0	800	0	800	100	0	800	0	800	100	0	800	0	800
90	10	900	100	1000	90	10	900	100	1000	90	10	900	100	1000	90	10	900	100	1000	90	10	900	100	1000
80	20	800	200	1000	80	20	800	200	1000	80	20	800	200	1000	80	20	800	200	1000	80	20	800	200	1000
70	30	700	300	1000	70	30	700	300	1000	70	30	700	300	1000	70	30	700	300	1000	70	30	700	300	1000
60	40	420	280	700	60	40	420	280	700	60	40	420	280	700	60	40	420	280	700	60	40	420	280	700
50	50	350	350	700	50	50	350	350	700	50	50	350	350	700	50	50	350	350	700	50	50	350	350	700
40	60	280	420	700	40	60	280	420	700	40	60	280	420	700	40	60	280	420	700	40	60	280	420	700
30	70	210	490	700	30	70	210	490	700	30	70	210	490	700	30	70	210	490	700	30	70	210	490	700
20	80	140	560	700	20	80	140	560	700	20	80	140	560	700	20	80	140	560	700	20	80	140	560	700
10	90	70	630	700	10	90	70	630	700	10	90	70	630	700	10	90	70	630	700	10	90	70	630	700
0	100	0	700	700	0	100	0	700	700	0	100	0	700	700	0	100	0	700	700	0	100	0	700	700

PC= Portland cement, MK= metakaolin, SL=slag, W=Water amount, GMK= geopolymer-based metakaolin stabilizer, GSL= geopolymer-based slag stabilizer

* Dependent upon the test type and mixture composition (i.e., stabilizer type), the replacement proportions (i.e., 0 to 100%) have been varied in different ranges.

** 90%GSL+10%MK was added in the stabilizer combination "MK+GSL" during the replacements with PC for all proportions 0% to 100%.

3.3 Rheological and Hardened Tests

3.3.1 Viscometer test

the rheometer test has done for all grout mixture at the conditional laboratory where the temperature of 23 ± 2 ºC and the relative humidity of 50 ± 5% by using Coaxial rotating cylinder rheometer (ProRheo R180 Instrument, Germany) Figure 3.4 . In order to obtain a consistent and uniform mixture the rheological properties of grout mixture was carried out by following the same mixing procedure, mixing speed and temperature. All mixture samples were taken by weight and prepared for 1000 grams. All mixture samples were taken by weight and prepared for 200 grams. After mixing at 150 rpm for 1 min and at 300 rpm for another 2 min (Güllü, 2016), as soon as mixing was finished the grout mixture was poured into the testing cup for performing the viscometer (rheometer) tests to obtain the shear stress and shear rate curve that throughout it the (plastic viscosity and yield stress) was obtained where, the shear stress was measured at the eight high shear rates of the rotations of (500 s-1, 571.43 s-1, 642.86 s-1, 714.29 s-1, 785.71 s-1, 857.14 s-1, 928.57 s-1 and 1000s-1). To achieve an equilibrium state each shera rate was maintained for a duration of 15s, and testing was completed in totaly 4min including the ascending (2min) and descending (2min) curves (i.e., shear stress versus shear rate, viscosity versus shera rate). The ascending curves in this study were used for investigation of rheological properties since their trend lines correspond to undisturbed state (Sahmaran, 2008). Regarding earlier practices from the curves (Yahia, 2001; Sahmaran, 2008; Yahia et al., 2016), the yield stress and plastic viscosity have been estimated via a modified Bingham (i.e., , where □=shear stress, □0=yield stress, □p=plastic viscosity, =shear rate, c=constant) correlated better the rheology of grout mixtures. The high shear rate led to all result of grout mixture shown in most cases, shear-thickening behavior, Which are described by the equation (Eq.A, Eq.B):

$$\tau = \tau_0 + \mu_p \dot{\gamma} \qquad \text{(A)}$$

$$\tau = \tau_0 + \mu_p \dot{\gamma} + c\dot{\gamma}^2 \qquad \text{(B)}$$

Where, stress represent it by τ (Pa), the yield stress represent it by τ_0 (Pa), the plastic viscosity represent it by μ_p (Pa.s), $\dot{\gamma}$ is the shear rate (s^{-1}), and c is a constant. The value of the constant c is typically about 10^{-3} and less, which is much smaller than the value of the plastic viscosity μ_p and the yield stress τ_0, so this constant is regarded as zero.

Figure 3.4 Coaxial rotating cylinder Rheometer

3.3.2 Mini-slump test

In the mini slump test, the mini slump cone is modification tool from the cone (ASTM C-143) for estimating the consistency of concrete, And it can be consedered alternative or supporting tools for rheology testing. By measuring the spread diameter of fresh grout (mm) on the Plexiglas, it can be obtain an idea and definition about the mini slump, and from Paste studies have shown that mini slump spreads give thought about yield stress (Ferraris, Obla, & Hill, 2001). The mini slump cone test for measurement of spread diameter of grout (in millimeter) was obtained using a mini cone with the dimensions that are upper diameter of 19mm, lower diameter of 38.1 mm and height of 57.2 mm (Sahmaran et al., 2008a; Sonebi et al., 2013). By using the horizontal plat, the mini cone was placed on the center of plate. After pouring the grout into the cone without overflow, the upper part of the cone was tamped lightly to bleed off any air pockets, and the continent cone was gently lifted.

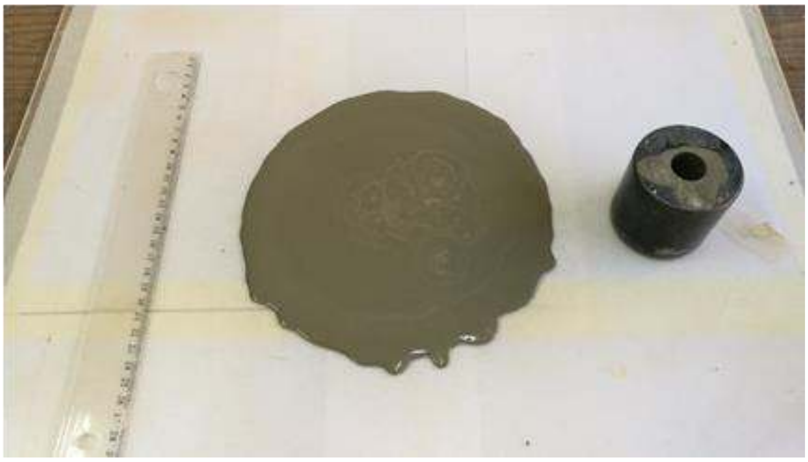

Figure 3.5 Mini Slump Cone Test

3.3.3 Marsh funnel cone test

The Marsh cone test is a workability test used for specification and quality control of cement pastes and grouts. It belongs to the family of the orifice tests as the V funnel. The Marsh funnel cone test for measuring flow time of grout (i.e., time elapsed in seconds until an amount of grout mixture flows) (Figure 3.6) was carried out using a steel funnel with a capacity of 1500mL and an outlet-pipe diameter of 4.76mm, modified from ASTM C939-10. For a sufficient response, the sample was pouring through a fine sieve to retain any cement particles. Flow time of grout was measured for a sample mixture of 1000mL filled into funnel based on earlier practices (Mirza et al., 2002). through a standard size cone, The Marsh flow cone could measures estimating the flow time of a known volume of a grout, and we could estimate plastic viscosity by measuring marsh cone flow time (Pitt, 2000). Since the time required by the grout sample flowing through the cone is proportional to its viscosity, the flow time increases with increasing viscosity.

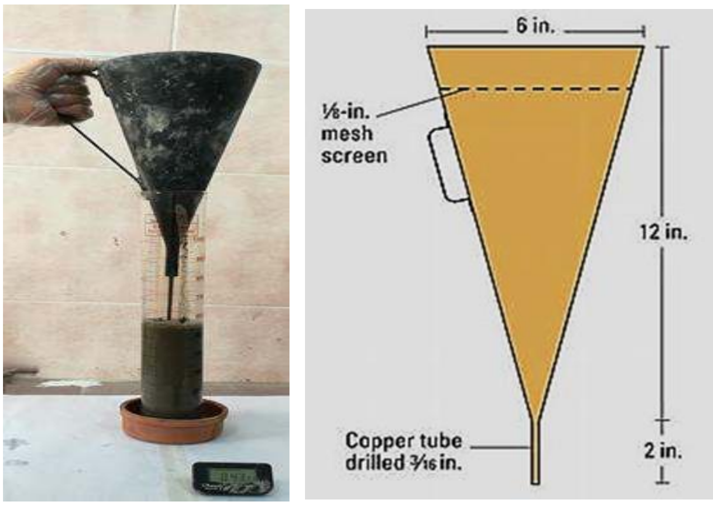

Figure 3.6 Marsh funnel cone test (flow time)

3.3.4 Bleeding (stability)

Bleeding is a an important property that shows how grout responds when left standing for a certain period (like a crack).The bleeding (or stability) tests (figure 3.7) were performed following the procedure given in ASTM C940-16. By putting the grout mixture in a graduated cylinder with a volume of 1000mL for 24h, the bleeding was measured 15-minute intervals for the first 60minute, and thereafter at 30-minute intervals until the completion of sedimentation (i.e., two successive readings show no further bleeding). If the final bleeding is less than 5% after 120 minutes, the grout is can be considered stable (Tan et al., 2005). For the grout to stabilize, the bleeding water secluded on the surface of the graduated 1000 mL cylinder it should be less than 5% of the total mixture volume after 2 hours after mixing. Final bleed water it can be consedred an important for determining the effective W/C of grout mixture, given by:

Effective W/C = $C_s f$ * Initial W/C

Where C_{sf} is the final coefficient, of the volume in suspension (%) = [(V0 _ Vf)/V0] *100, V0 is the initial volume (mL), and Vf is the final bleeding water (mL).

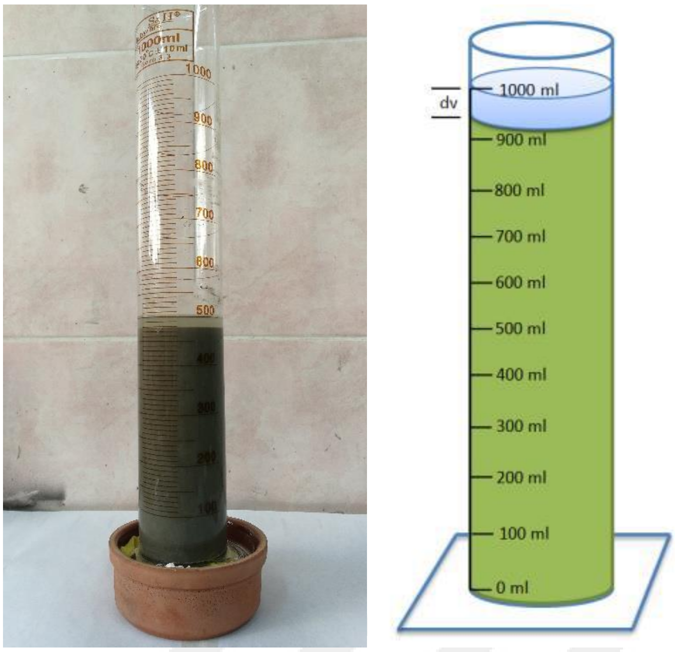

Figure 3.7 Bleeding test (stability)

3.3.5 Setting Time

The setting time is the last rheological characteristic ta be verified. The time that a freshly mixed grout takes to set is basic information, which is needed before injection takes place. For measuring setting times of grout mixtures, the Vicat needle penetration tests (Figure 3.8) were carried out by following ASTM C191. This test measures the setting time of grout by measuring the penetration depth of a standardized needle as a function of time. Initial setting time is measured at 25mm penetration, while 1mm penetration is recorded for final setting time. It should be noted that the Vicat mould in this study was filled with grout sample after completion of bleeding. It was noted that the Vicat mold filled with the hardened sediment. Only the results for final setting times were taken into discussion in this study.

Figure 3.8 Setting time

3.3.6 Compressive Strength

The mechanical characteristics of the hardened grout have a decisive effect on the selection of a suitable product, which must be compatible with the base concrete to avoid debonding due to tensile or shear stresses inside the crack. The compressive strength (fc ') is the maximum axial force that the grout specimen can withstand. After molding, all of the specimens in cylindrical (50 mm × 100 mm) figure 3.9 were used in compressive tests. The fresh grouts were placed in beakers and the dropper tools removed the bleeding water. Water evaporation was avoided by a plastic membrane were used to seal the grouts. The specimens were cured in a moist chamber (23 ± 3 °C and 100% R.H.) for 28 days. According to ASTM Standards C942 (ASTM C942-10, 2010) and (ASTM D2938, 2002), cylindrical specimens (50 mm × 100 mm) was used in compressive tests. In order to explain the various bleeding rates of different grouts, compressive strength is analyzed based on that change in the percentage of material addition.

Figure 3.9 compressive equipment test specimen, curing state

3.3.7 Ultrasonic Pulse Velocity (UPV)

The specimens before UCS measurements were tested for ultrasonic pulse velocity (UPV) performances that conform to ASTM C597-16. The ultrasonic pulse velocity (UPV) tests performed on grout (paste) samples (7 and 28-curing day). The UPV test is considered a non-destructive test method that can be applied to the display of the strength property of the cured material and which can typically be used to determine the dynamic properties of the material (Anon, 1979). As a nondestructive test, it is known that the UPV values are valuable for indication of stiffness performances of hardened material in different class of qualities. This technique is used to determine the hardened grout uniformity. Uniformity of the grout leads to faster velocities and usually better an improved strength.The procedure to complete this test is simple. A transducer emits an ultrasonic pulse through the grout specimen and this pulse is received on the other side of the specimen by another transducer (see Figure. 3.10).

Figure 3.10 Ultrasonic pulse velocity test instrument and view of the test in progress

3.4 Deep Soil Mixing

The stabilization technique by deep soil mixing is based on introduction and mixing of additives (stabilizer) as a powder or a suspension, as a grinding or a suspension using special equipment, and volume stability, to improve strength, permeability and durability of the soil. By replacing the fluid in the structure of the soil with the stabilizer used, a reduction in the initial void volume allows developments of strength superior to the initial void volume, so that the particles and agglomerates are mutually Approach, at the same time prevent swelling. This soil action can be used as a practical, economic and environmental key for an extensive range of engineering submissions. Applications for soil mixing include road/iron embankment for limiting excavation, lime, cement or lime/cement column, marine clay modification of submarine platform, shallow foundation, reinforcement of dam, improvement of stability of gradient, basis of soil.

Grout composition investigated In this study could be proposed for deep mixing. Also in this study a deep mixing device which has been designed by the supervisor and researcher of this thesis, has been illustrated in (figure 3.11). A deep soil-mixing device was developed by reverse engineering rules according to specifications of a full-scale soil mixing machines in the scale of 1 to 10 for using in

Laboratory condition. The central part of the device, counting the rotary motor, the auger and the slurry injection system, is mounted on a moving plate. This moving plate is associated to the support rod and allows vertical movement. Thus, when the device is turned on, the auger rotates clockwise at a speed of 58 rpm, penetrates the soil and injects a constant air pressure of 0.5 bar to prevent clogging of the nozzle. When the desired depth is reached, the auger rotates upward counterclockwise (to penetrate at the same speed). At this stage, the slurry is injected into the ground (stopped pneumatically), mixed with the surrounding soil via an auger blade, resulting in the formation of a column deeply mixed with the soil. This deep mixing device designed in this study is a preliminary effort that could be proposed for further adjustments and modifications.

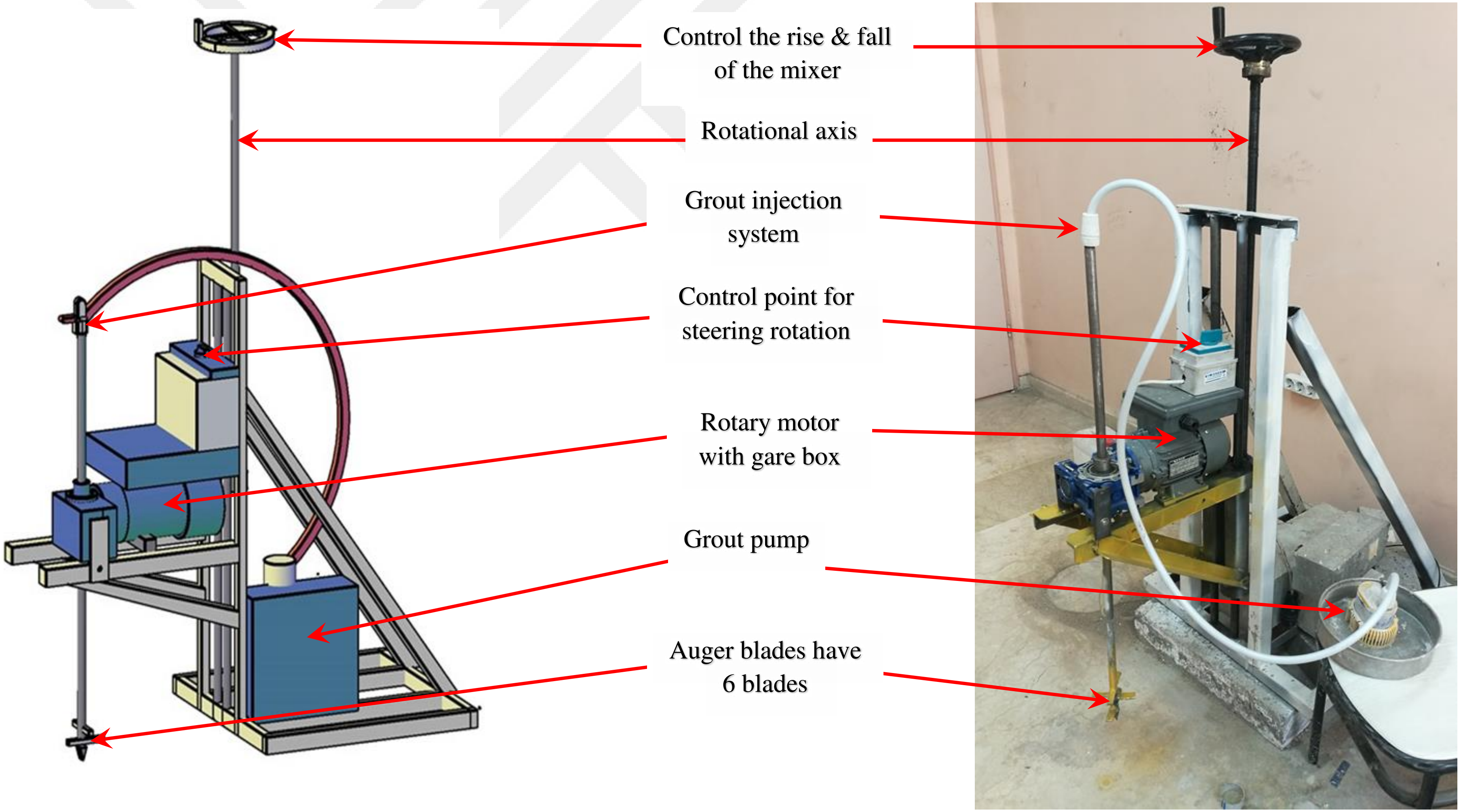

Figure 3.11 Deep soil mixing device designed at Gaziantep University

CHAPTER 4

EXPERIMENTAL RESULTS AND DISCUSSIONS

4.1 Rheological properties of cement-based grout with different stabilizer

4.1.1 Rheological properties of cement-based grout with metakaolin stabilizer

Through the flow, curves figure 4.1, which have been given for viscosity vs shear rate, & Shear stress vs shear rate of cement-based grout with metakaolin. It is observed that the all grout mixture result in (non-Newtonian behavior), and summarized by the corresponding behaviors (i.e., pseudoplastic (shear thinning), dilatant (shear thickening)) in Table 4.1. The flow curves have been drawn using an index value of power law that is most appropriate for corresponding rheological behavior (i.e., pseudoplastic when power index<1, Bingham when power index=1, and dilatant when power index>1) (Güllü, 2016) It should be noted that even though the trials were attempted for all dosage rates, but the replacement proportions 0-50%MK . Due to these reasons, the rotation of the cylindrical needle was unable during the rheometer test. Since it is observed that the apparent viscosity decreases with increased the shear rates to make the mixtures behave as (dilatant behavior) for (20-50%MK) dosage replacement. For the samples with MK showed lower apparent viscosity than the sample without MK (100% cement) and continues to decline with increasing MK dosage rates. As it is known that the water content play a significance role in grout mixtures through its effected on the fluidity of grout which consider the one of the most requirement of grout during injection processes, since it can be noted that the flowability or fluidity of grout could be easy in the high dosage replacement with cement at the pseudoplastic behavior due to the decreases viscosity thus, a large energy needs during injection the grout mixtures because it is already obtained high viscosity response. While, it should be offered a low amount of the metakaolin additions (GMK) for better grouting (flowability, workability).

It is obtained from the grout mixtures (Figure 4.1, Table 4.1) that the metakaolin is more able to change dilatant (shear thickening) behavior of cement to pseudoplastic (shear thinning). The pseudoplastic behaviors may be explained by the shear-induced ordering of particles acted by Brownian forces that cannot be reorganized into a random packing (Rastogi et al., 1996). The increase in the flow responses (i.e., high flow resistance) due to metakaolin may be attributed to the associated increase in water demond induced by the high surface area and finesses of metakaolin particles (Sonebi et al., 2013). Therefore, the dilatant behavior could have more disadvantages than advantages especially in the process (mixing, pumping, and extrusion, etc.) at high shear rates (Barnes, 1989; Feys et al., 2009).

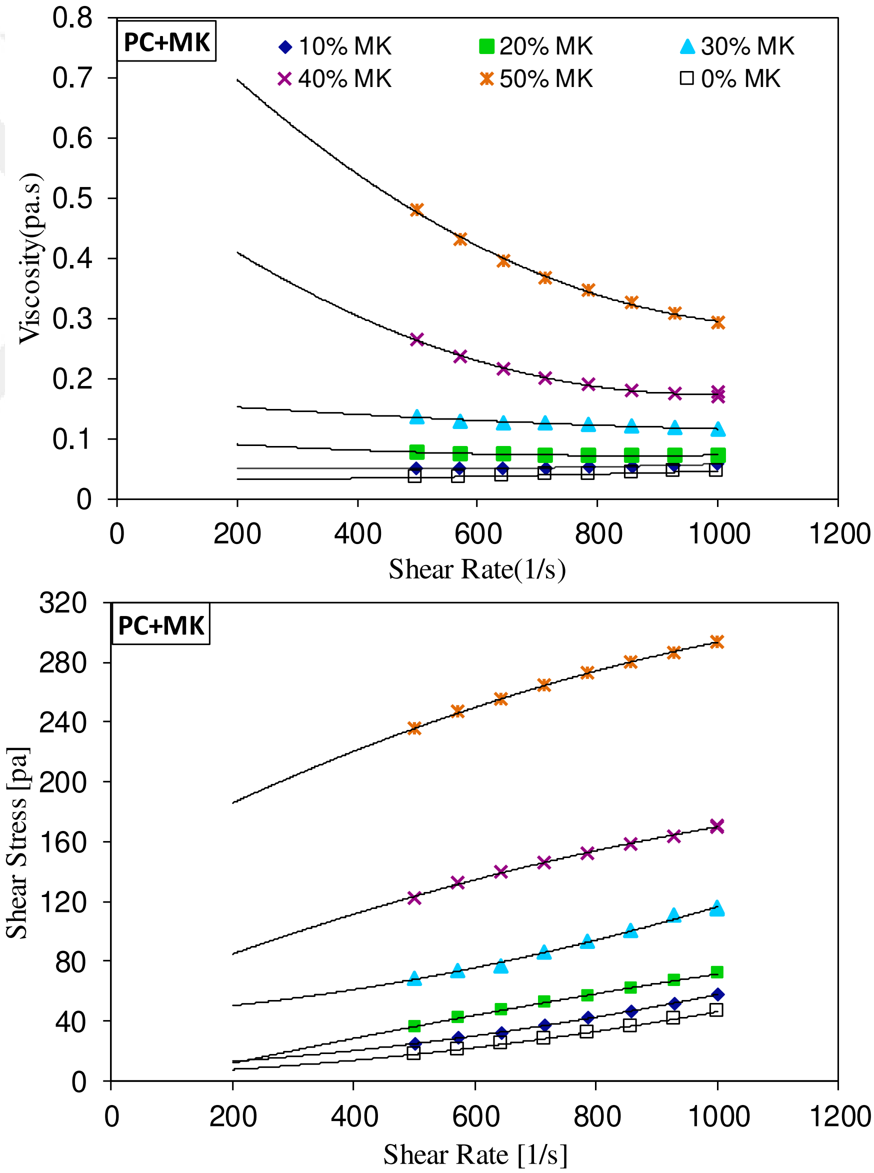

Figure 4.1 Viscosity vs shear rate & Shear stress vs shear rate of cement-based grout with metakaolin

4.1.2 Rheological properties of cement-based grout with slag stabilizer.

The rheological (flow) behaviors of grout mixtures with investigated stabilizers have been illustrated for the flow curves of shear stress versus shear rate and viscosity (apparent) versus shear rate in Figure 4.2. The corresponding behaviors (i.e., pseudoplastic (shear thinning), dilatant (shear thickening)) in Table 4.1. It is observed that the all grout mixture result in (non-Newtonian behavior), and the dilatant (shear thickening) was clear in all mixtures for water to binder (w/b) = 1 ratios. Generally it can be said that the dilatant behavior could be attributed to the clusters formation theory, since the hydrodynamic forces between the particles dominate the repulsive forces, and due to the inertia theory via the transfer of momentum transfer between suspended particles (PC+SL+water) where a part of the momentum is transferred directly between the particles (Bossis and Brady, 1989; Maranzano and Wagner, 2001; Feys et al., 2009). The dilatant behavior of slag conclusion obtained in this system could be specifically attributed to the lower solids volume fraction which affected by the inertial forces (Feys et al., 2009; Yahia et al., 2016). since it is seen that the increases inclusion of slag rates decrease or no affected the response of shear stress as compared to grout with native cement (100%PC) and there is a slight change in the apparent viscosity. Consequently, the dispersed grains decrease interparticle friction that results in a reduction in viscosity. As it is known that the water content play a significance role in grout mixtures through its effected on the fluidity of grout which consider the one of the most requirement of grout during injection processes, since it can be noted that the flowability or fluidity of grout could be difficult in the low water content and with the dilatant behavior due to the increasing viscosity thus, a large energy needs during injection the grout mixtures, therefore the dilatant behavior could be has more disadvantages than advantages especially in the process (mixing, pumping and extrusion, etc.) at high shear rates (Barnes, 1989; Feys et al., 2009). For the effect of slag obtained, it can be said that compared to metakaolin, the slag has poorer hydration reaction than native cement that results in causal to high flowability (Ozbay et al., 2016). This could make easier use of slag for grouting purpose. Even the lower hydraulic activity due to slag (Ozbay et al., 2016), increasing the slag replacement could lead to insignificant flow responses obtained in this study compared to native cement. Similar concerns given above could be valid for the effect of geopolymerized slag stabilizer on the native cement. The fluidity effects of slag may be mainly attributed to particle size distributions and chemical compositions that include SiO_2 and Al_2O_3. (Li et al., 2017a)

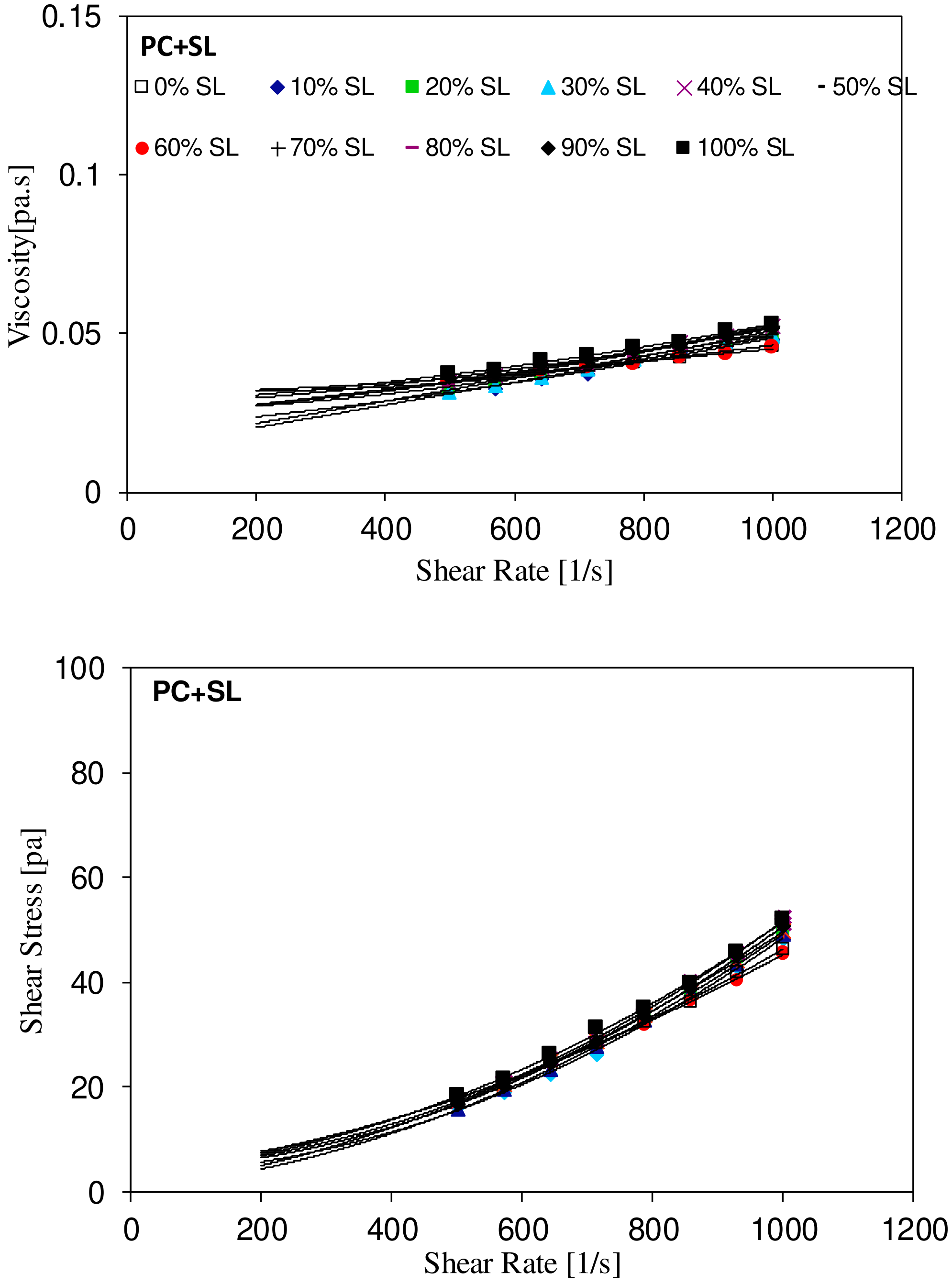

Figure 4.2 Viscosity vs shear rate & Shear stress vs shear rate of cement-based grout with Slag

Table 4.1 Rheological behaviors (i.e., pseudoplastic (shear thinning), dilatant (shear thickening)) of grout mixtures.

Stabilizer replacement with cement Stabilizer (%)	Cement-based grout with MK	Cement-based grout with SL	Cement-based grout with GMK	Cement-based grout with GSL	Cement-based grout with GSL+MK
0	Dilatant.	Dilatant.	Dilatant.	Dilatant.	Dilatant.
10	Dilatant.	Dilatant.	Dilatant.	Dilatant.	Dilatant.
20	Pseudoplastic.	Dilatant.	Dilatant.	Dilatant.	Dilatant.
30	Pseudoplastic.	Dilatant.	Dilatant.	Dilatant.	Dilatant.
40	Pseudoplastic.	Dilatant.	Dilatant.	Dilatant.	Dilatant.
50	Pseudoplastic.	Dilatant.	Dilatant.	Dilatant.	Dilatant.
60	-	Dilatant.	Pseudoplastic.	Dilatant.	Dilatant.
70	-	Dilatant.	Pseudoplastic.	Pseudoplastic.	Pseudoplastic.
80	-	Dilatant.	-	-	Pseudoplastic.
90	-	Dilatant.	-	-	-
100	-	Dilatant.	-	-	-

4.1.3 Rheological properties of cement-based grout with GMK & GSL

The flow (rheological) behaviors of grout mixtures with additives geopolymerized stabilizer have been presented as (shear stress vs shear rate) (apparent viscosity versus shear rate) curves in figure 4.4, figure 4.3 for geopolymerized metakaolin stabilizer (GMK) and polymerized slag stabilizer (GSL). The test limitations of proportions may be attributed to water demand for MK (i.e., beyond 50%) and high stiffness for geopolymerized stabilizers (i.e., beyond 70% of GMK and GSL, 80% of GMK+MK). Due to these reasons, the rotation of the cylindrical needle was unable during the rheometer test. Through the curves could be observed that most of the grout mixtures result in dilatant (shear-thickening behavior) (Table 4.1) as the cement-based grout with (GMK, GSL & GSL+MK) , while someones (20-50%MK, 60-70%GMK, 70%GSL, 70-80%GSL+MK) lead to pseudoplastic behavior. The additions of GMK (Figure 4.3) increase the responses with increased replacement rates. However, the GMK additions lead to less response compared to MK additions. This indicates that geopolymerized metakaolin stabilizer brings the about the better flow of grout than metakaolin. On the other hand, the responses due to the additions of SL (Figure 4.2), GSL (Figure 4.3) and GSL+MK (Figure 4.4) almost remain unchanged with replacement rates. It is observed that these additions (SL GSL and GSL+MK) insignificantly increase or decrease the flow responses regarding the corresponding stabilizer rates compared to native cement-based grout. The results indicate that while the additions of 10%-20%MK and 10%-40%GMK yield to similar behaviors and responses with native cement added grout, all additions of SL, GSL, and GSL+MK result in similarly with native cement. Thus, it should be offered a low amount of the metakaolin and geopolymerized metakaolin additions (GMK) for better grouting (flowability, workability), while all amounts of the slag and geopolymerized slag seem suitable.

The reasons of dilatant behavior, attributed to the high shear rate that applied in this study, since the apparent viscosity of the suspensions (PC+ geopolymerized stabilizer +water) increases at high shear rates this leads grout mixtures with GA behave as shear-thickening behavior (dilatant) (Roussel et al., 2010). As well as mentioned above there is more energy required to increase the material flow rate, influencing mainly on mixing and pumping (Yahia et al., 2016). Also could be said that the use of alkaline activator solution (NaOH+Na2SiO3) in manufacturing the GA has a significant role in increasing the strength, this leads to reducing flowability (Sashidhar et al., 2015).

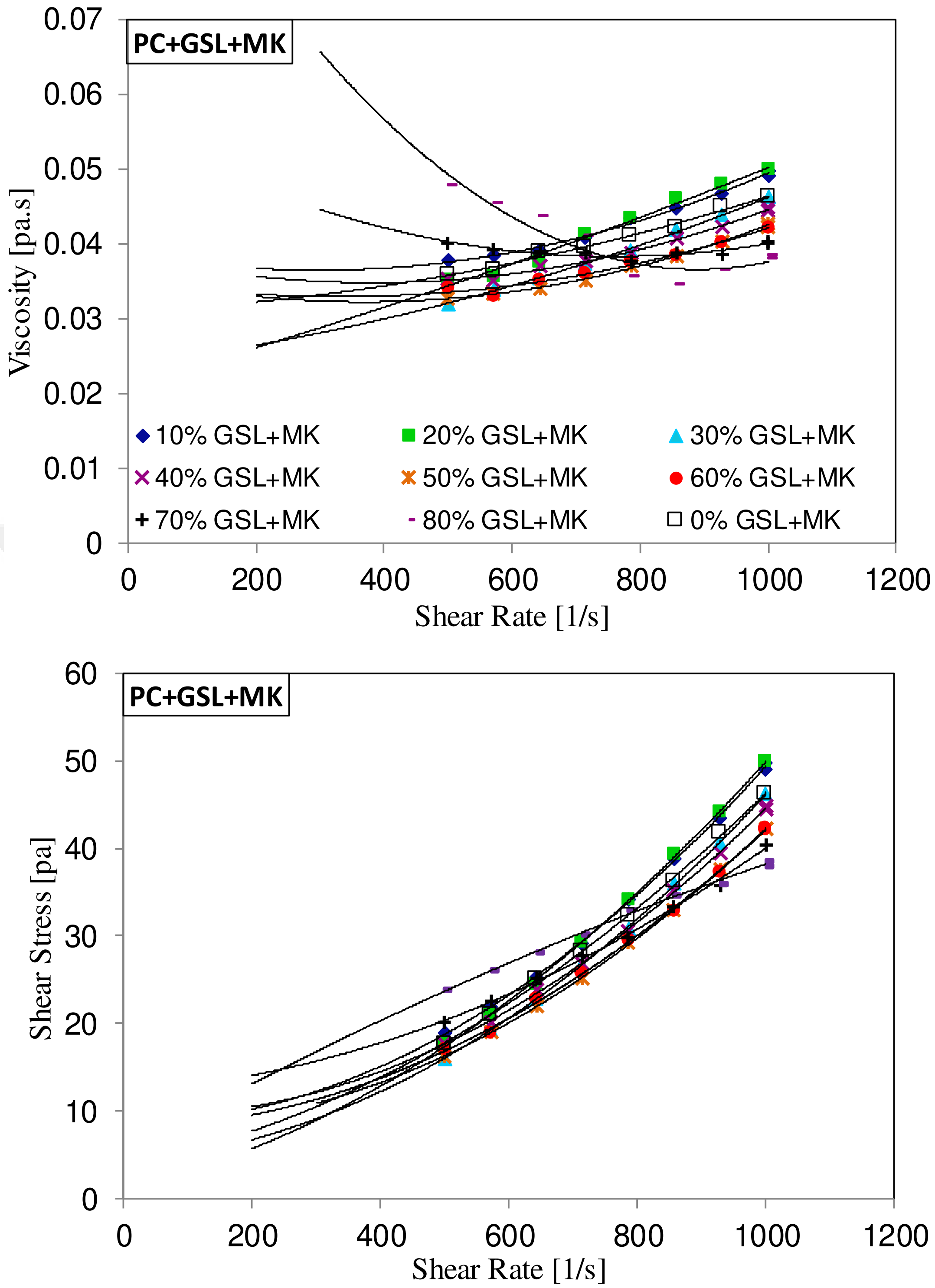

Figure 4.3 Flow curve of apparent viscosity versus shear rate & shear stress vs shear rate for GSL+MK with native cement

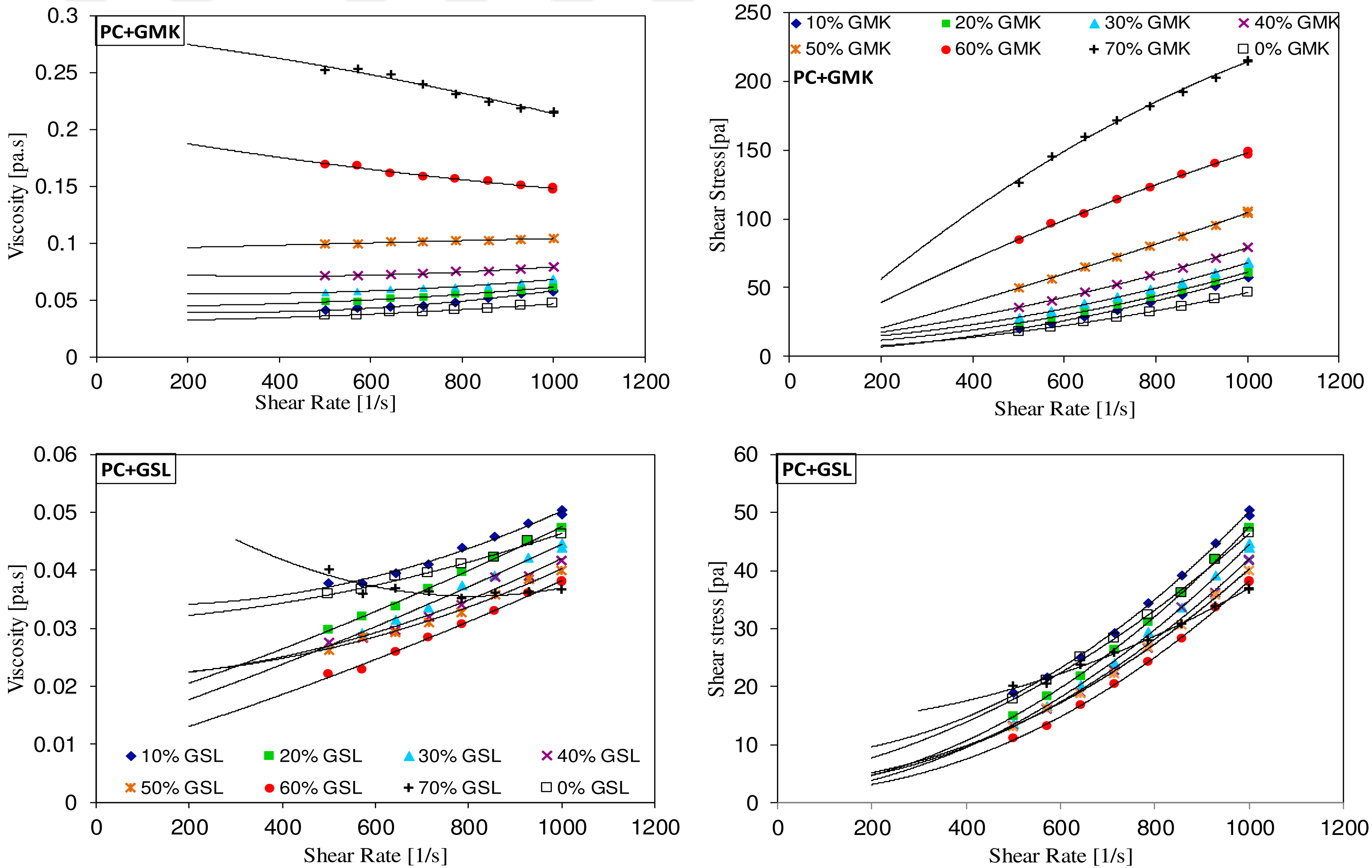

Figure 4.4 flow curve of apparent viscosity versus shear rate & shear stress vs shear rate for GMK & GSL

4.1.4 Rheological parameter (Yield stress and Plastic viscosity)

4.1.4.1 Yield stress and Plastic viscosity of cement-based grout with metakaolin

The rheological parameters including yield stress plastic and viscosity as a function of dosage rate of MK are shown in (figure 4.5 Table 4.2). for the samples with MK the yield stress is slightly increased up to 20% then it observed that the yield stress has been increasing dramatically with MK dosage is increased, it can be said that the changes in the rheological parameter specifically with increases the rate of metakaolin. May be attributed to the increase in volume fraction among the binder particles (Zhou et al., 1999; Zhou et al, 2001). Their results may be due to the water demand requirement resulting from the high surface area of metakaolin. (Sonebi et al., 2013) reported that an increased content of metakaolin modified (increased) yield stress and plastic viscosity of native cement. The metakaolin (0 to 20%) does not significantly change the plastic viscosity of grout but helped to rise yield stress.

For the plastic viscosity (Figure 4.4) as compared with the native cement (100% PC) it is observed that native cement has a plastic viscosity less than samples that contain MK. The additions MK increase plastic viscosity, particularly beyond the 30% dosage rate. Generally, it can be said that the rheological parameters are heavily influenced by the water/binder ratio, the amount of metakaolin, surface area and it fineness (Curcio and DeAngelis, 1998; Sonebi et al., 2013). Generally can be said that the increasing trend in the plastic viscosity could be due to An energy attractive of inter-particle between cement particles and fine particles of fly ash (Barnes,1997; Roussel et al., 2012).

4.1.4.2 Yield stress and Plastic viscosity of cement-based grout with Slag (SL)

In regard to the stabilizer effects on yield stress and plastic viscosity compared to native cement (Figure 4.5, Table 4.2). The sample without 0%SL (100% cement) shows higher yield stress value than the samples with SL. For the samples with SL (Fig.4.5) the yield stress is slightly decreased up to 30% then increases as the SL dosage is increased, it can be said that the changes in the rheological parameter specifically at the slag dosage rate 30%-40%. It also observed that native cement has a plastic viscosity higher than samples that contain SL. However, the samples with SL have slightly decreased with increases rate of replacement in the most dosage of mixtures with native cement. Generally can be said that the increasing trend in the plastic viscosity could be due to the attractive interparticle forces between the cement grains and fine particles of slag (Barnes,1997; Roussel et al., 2012), and the decrease could be attributed to the ball bearing effect of fly ash (Park et al., 2005). In addition, the water content effects on the fluidity, which is one of the most requirements of grout (Nguyen et al., 2011. Since the yield stress varies in the range 1.05Pa to 5.99Pa and the plastic viscosity varies in the range 0.0133Pa.s to 0.003Pa.s. The reason may be attributed to dependent upon the relation between the fineness of cement and slag (Adjoudj et al., 2014).

Some researchers (Wu et al., 1984; Shi et al., 1998) found that the yield stress and plastic viscosity values can increase or decrease with slag additions. The reason may be attributed to dependent upon the relation between the finiteness of cement and slag (Adjoudj et al., 2014). Since grout is able to present a thixotropic behavior (Yahia et al., 2016), it is reported (Feys et al., 2009) that time influences the yield stress and plastic viscosity of grout mainly due to hydration effects, related to physical and chemical properties of added (stabilizer) materials.

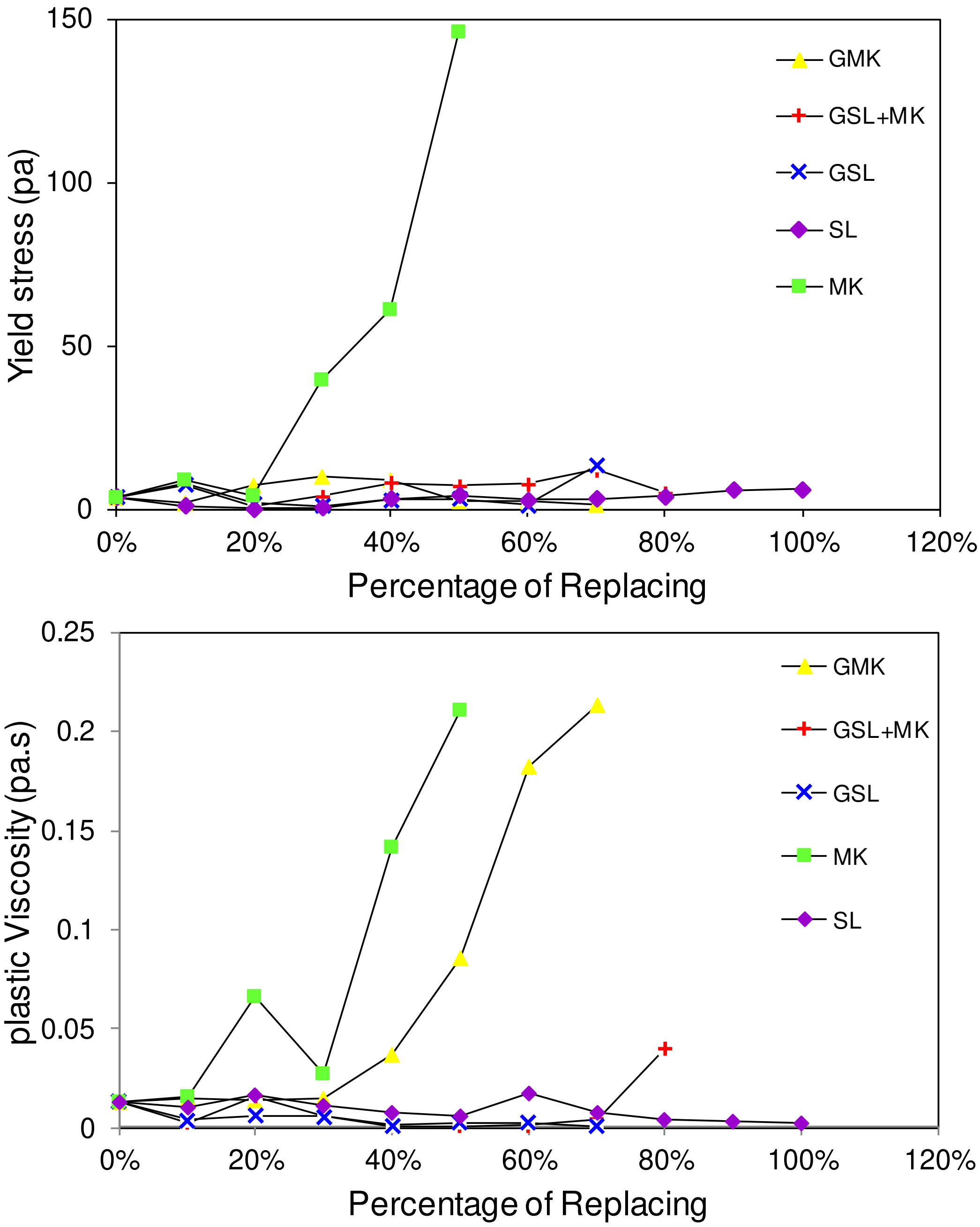

Figure 4.5. Yield stress and plastic viscosity versus dosage rates of stabilizers

Table 4.2. Basic rheological characteristics of grout mixtures (i.e., yield stress (τ_0), plastic viscosity (μ_P))

Percentage of replacement	PC+MK		PC+SL		PC+GMK		PC+GSL		PC+(90%GSL+10%MK)	
	τ_0 (Pa)	μ_P (Pa.s)	τ_0 (Pa)	μ_P (Pa.s)	τ_0 (Pa)	μ_P (Pa.s)	τ_0 (Pa)	μ_P (Pa.s)	τ_0 (Pa)	μ_P (Pa.s)
0	3.7	0.0131	3.74	0.0133	3.78	0.0132	3.75	0.0133	3.8	0.0133
10	8.97	0.0155	1.05	0.0108	2.131	0.0154	7.87	0.004	7.6	0.0029
20	4.42	0.0668	0.405	0.0166	7.369	0.0135	1.8	0.0064	1.18	0.0161
30	39.93	0.0278	0.57	0.0117	10.236	0.0151	1.16	0.0059	4.12	0.0058
40	61.2	0.142	3.26	0.0075	8.783	0.0368	2.91	0.0017	8.2	0.0011
50	146.23	0.2107	4.37	0.006	2.76	0.086	3.21	0.0028	7.17	0.0012
60	-	-	2.96	0.0174	1.77	0.1874	1.5	0.003	7.92	0.0016
70	-	-	3.25	0.0077	1.327	0.293	13.54	0.0011	12.2	0.0048
80	-	-	3.99	0.004	-	-	-	-	5.26	0.04
90	-	-	5.96	0.0032	-	-	-	-	-	-
100	-	-	5.99	0.0033	-	-	-	-	-	-

4.1.4.3 Yield stress and Plastic viscosity of cement-based grout with geopolymerized stabilizer (GMK, GSL, GSL+MK).

The rheological parameter is given in yield stress and plastic viscosity versus dosage rates of the geopolymerized stabilizers curves in (Figure 4.5, Table.4.2) respectively. Its observed through the curves it is clear that the samples (GMK, GSL, GSL+MK) have insignificant change (increase or decrease) on yield stress result. On the other hand, both the additions MK and GMK increase plastic viscosity particularly beyond the 30% dosage rate, again all other stabilizer replacements result in slight modifications with insignificant changes. The results (Figure 4.5) supports the findings obtained from the responses of flow curves (Figure 4.4) that offer low dosage rates of MK and GMK, as well as all rates of other stabilizers, for better grouting nearly following the native cement’s results.

The reason for insignificant change of yield stress and plastic viscosity in both could be attributed to the fact that the geopolymerized stabilizers have a rounded shapes which result in lower internal friction among the particles of cement and geopolymerized stabilizer, additionally it has a lower settable surface that leads to release of a larger amount of water flocculated between the cement particles consequently, both yield stress and plastic viscosity is decreasing that leads to enhances the grout fluidity (Khayat and Yahia, 1997; Priyadharshini et al., 2011; Yahia et al., 2016).

4.2 Workability (fluidity) properties of grout mixtures

4.2.1 Mini-slump test result

One important test for traditional grout, in this research, have been investigating for different stabilizer and discuss in (Figure 4.6 Table 4.3). The mini slump test was chaperon by very low shear. (Roussel et al., 2005) reported that the value of mini-slump is to characterize yield stress, therefore, a smaller slump value is could be given as a lower yield stress. According to the result that has been shown, Increasing the amount of MK in the grout mixture effected by decreases the diameter of the mini-slump spread. This is likely due to an increase in water demand caused by the high surface area of metakaolin (Sonebi et al., 2013). This result similar to other researches (Moulin et al., 2001).

For slag, the result in (Figure 4.6 Table 4.3) showed that an insignificant change in speared of the diameter of mini-slumps. That may due to the Use of high-grade mineral admixture that work to decreasing the water demand and increase spread of slump. (Lange et al., 1997) reported that the addition of right fine blast furnace slag is increasing demand for mortar water. He discovered that due to the specific flow, the top amount of blast furnace slag will reduce the water demand of mortar.

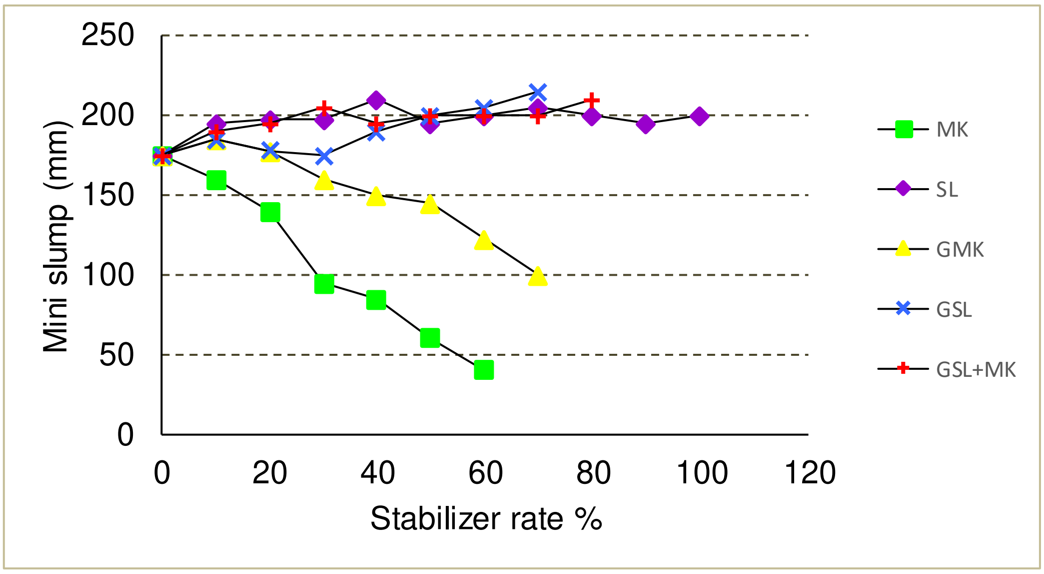

Figure 4.6 Mini-slump spread (mm)

For geopolymerized stabilizer (GMK, GSL, GSL+MK) the result carried out that geopolymerized metakaolin stabilizer reduces the mini-slump diameter with increases the dosage of GMK. This measurement may be due to there is a quantity of metakaolin stuck with granules after curing of geopolymerized metakaolin stabilizer. On another hand the grout mixture that includes GSL, GSL+MK have slightly decreased by up to 30% than insignificant change continues to the end of replacement operation. It can also be observed that the grout mixtures with the addition of geopolymerized stabilizer have very similar shapes each other that may consider an advantage to increases the workability and fluidity. Especially physical factors are Spherical particles movie roll easily to each other, which makes reducing friction between particles (Ramachandran, 1996).

Table 4.3 Mini Slump flow (mm)

Stabilizer rate (%)	PC+MK	PC+SL	PC+GMK	PC+GSL	PC+(90%GSL+10%MK)
0	175	175	175	175	175
10	160	195	185	185	190
20	140	198	177	178	195
30	95	198	160	175	205
40	85	210	150	190	195
50	60	195	145	200	200
60	40	200	122	205	200
70	-	205	100	215	200
80	-	200	-	-	210
90	-	195	-	-	-
100	-	200	-	-	-

4.2.2 Marsh funnel (flow time) test result

Measuring the flow times for the investigated stabilizers in this study could be prominent for fluidity that is an important parameter for cement-based grouts. According to (Figure 4.7 Table 4.4) an increase in the amount of MK in the grout, the mixture increases the time of the flow time. The decrease in the process ability of grout samples when MK is used can explain that MK has higher water absorption capacity. The surface area of the brain is for PC (3040 cm2/g) and MK (13200 cm2/g). As it is shown that grout mixtures including MK high dosage, decreasing fluidity and deform ability dependent on the W/C.

As for the effect of the addition of slag to grout mixture. The result is similar to the other workability test except a slight increases in flow time. This reduced the Water detention of spherical case, decrease of water demand for special processability after that. The SL particles are filled into the space formed by the larger cement particles (Zhang and Han, 2000; Ferraris et al., 2001), It has an average particle size of 18.09 nm, which decreases the internal frictional force of this OPC-SL material. The increases fluidity effects of SL may be attributed to their particle size distributions and chemical compositions, such as high amounts of SiO_2 and AL_2O_3.

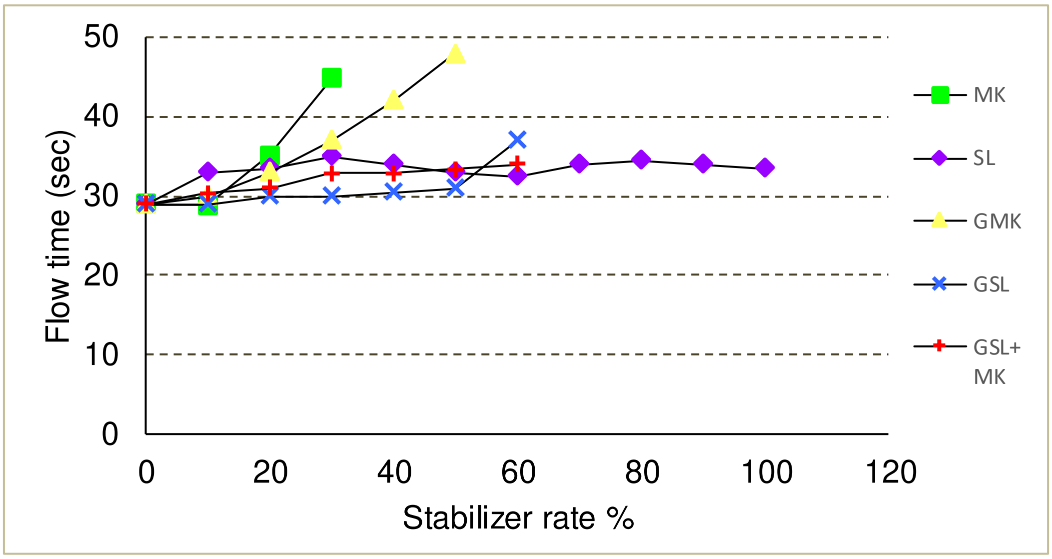

Figure 4.7 Marsh funnel test (flow time)

For geopolymerized stabilizer (GMK, GSL, GSL+MK) the result carried out that geopolymerized metakaolin stabilizer GMK increases the flow time from marsh cone with increases the dosage of GMK. The replacement process was up to 50% and did not continue to 100% because the granules began to accumulate and can not pass through the hole. The same reason was for the grout mixture that including GSL, GSL+MK. We conclude from this test that the mixing ratios for the geopolymerized stabilizer should be at a low rate to obtain higher efficiency and effect.

Table 4.4 Marsh Flow time (s)

Stabilizer rate (%)	PC+MK	PC+SL	PC+GMK	PC+GSL	PC+(90%GSL+10%MK)
0	29	29	29	29	29
10	29	33	30	29	30
20	35	34	33	30	31
30	45	35	37	30	33
40	-	34	42	31	33
50	-	33	48	31	33
60	-	33	-	37	34
70	-	34	-	-	0
80	-	35	-	-	-
90	-	34	-	-	-
100	-	34	-	-	-

4.2.3 Bleeding (stability)

Bleeding, particle size distribution and viscosity are the three important parameters that could controlling injectabilty of grout in cracks (Langevin, 1995). By this test, we can obtain or measure the effective W/C ratio which is limited to the reaction of grout mixture.

The grout mixture that included MK had shown a decreasing in bleeding ratio. Where we obtained a clear effect after the addition of 10% of metakaolin, and the bleeding ratio became 0% at 50% replacement with cement. That gives as an aide the optimum grout mixture can be obtained at 20% to 50% dosage of MK. Where the reason is to contain a percentage of fine powder that Demand of water an increased due to surface area increases (Ferraris et al., 2001). (Wainwright and Rey, 2000) reported the The delay in hydration and the reduction in the occurrence of hydration products is the primary cause of this main cause of bleeding. As for slag addition effect on cement based grout as shown in (figure 4.8, Table 4.5) the replacement process continuously to 100% dosage replacement with slightly increasing in bleeding ratio.

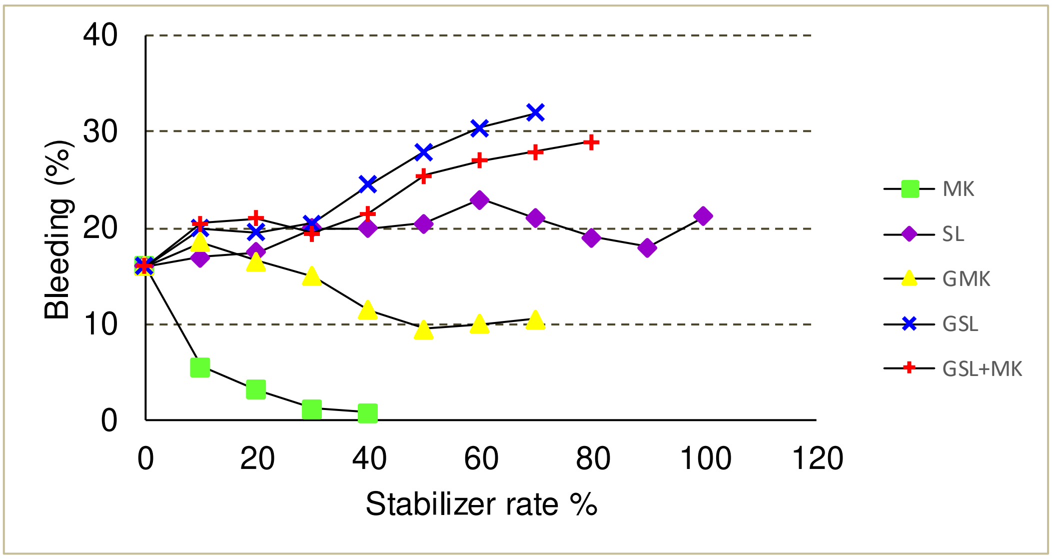

Figure 4.8 Bleeding (stability)

For geopolymerized stabilizer (GSL & GSL+MK) the result showed (figure 4.8, Table 4.5) that the bleeding ratio increased with increasing dosage rate of the geopolymerized stabilizer. However, GMK showed different behavior after 10% rate replacement, the mixture has been decreasing in a bleeding ratio up to end of replacing process. From that, we can conclude the GMK addition could give as an optimum mixture at high dosage of replacement.

The reason for reducing bleeding rate is due to the lack of hydration process of grout mixture that including (GSL, GSL+MK) addition with the low percentage of cement that used during the replacement process. Except (GSL+MK) showed less bleeding than (GSL), this due to its contained a small amount of MK.

Table 4.4 Final Bleeding (%)

Stabilizer rate (%)	PC+MK	PC+SL	PC+GMK	PC+GSL	PC+(90%GSL+10%MK)
0	16	16	16	16	16
10	6	17	19	20	21
20	3	18	17	20	21
30	1	20	15	21	20
40	1	20	12	25	22
50	-	21	10	28	26
60	-	23	10	31	27
70	-	21	11	32	28
80	-	19	-	-	29
90	-	18	-	-	-
100	-	21	-	-	-

4.2.3 Setting Time

The setting time mainly depends on the temperature, chemical composition, particle size and W / C (which is striped by the amount of crystal form of C3A and C3S) of the cement particles.

As for the setting time of fresh performances, it is seen that some stabilizer rates (>60%MK, 70%GMK, >70%GSL, >80%GSL+MK) were limited due to rheological performances similar to mini slump test. From (Figure 4.9, Table 4.5) we observed that Increase the proportion of metakaolin up to 50% the grout mixture lead to reducing the initial and final setting times, This may be due to increased hydration which is cause by the particulate nature of MK, As the fineness and surface area of MK was large, cement particles accelerated the rate of hydration and the strength developed rapidly as reported by authors (Wild et al., 1996; Frias et al., 2000). The mixture containing MK It showed quicker reaction rate than the control mixture. The higher peaks classically correspond to the reaction of the C3A. In the paste containing metakaolin, it indicates that hydration of calcium aluminate is promoted or some additional exothermic reaction is occurring in the MK mix. On the other hand, and by compared to the control mixture the MK in the manufacture of grout showed shortened the final setting time. Shortening of the setting time of the grout mixture with MK is due to the large surface area of the MK particles, and after the usual consistency conditions are satisfied at the beginning of the set time measurement, release water is continuously Adsorb from the mixture.

Back to the effect of slag on the grout mixture that showed (Figure 4.9, Table 4.5), we observed that with increases the addition of slag, the final setting time have been increased up to 1600 min. This is due to a reason that slag (SL) has roughly spherical particles and low hydration reaction ability than cement. (Gesoglu and Ozbay, 2007) found that as the replacement level of GGBFS increased to 60%, the initial setting time and the final setting time increased by 2.8 times and 2.31 times, respectively. Contributing to the high liquidity of this system to the same extent as SL replacement.

For a geopolymerized stabilizer, the result (Figure 4.9, Table 4.5) showed that the addition of (GMK, GSL, GSL+MK) has insignificant change on grout mixture. However, we observed that (GMK, GSL+MK) show different behavior than GSL, this is due to the (GMK, GSL+MK) contains particles or amount of MK that made the setting final time of grout mixture reduced to a lower level than GSL. The cement grain concentration decreases (the excess water slows down the hydration reaction) and it takes more time for the grout to set and harden.

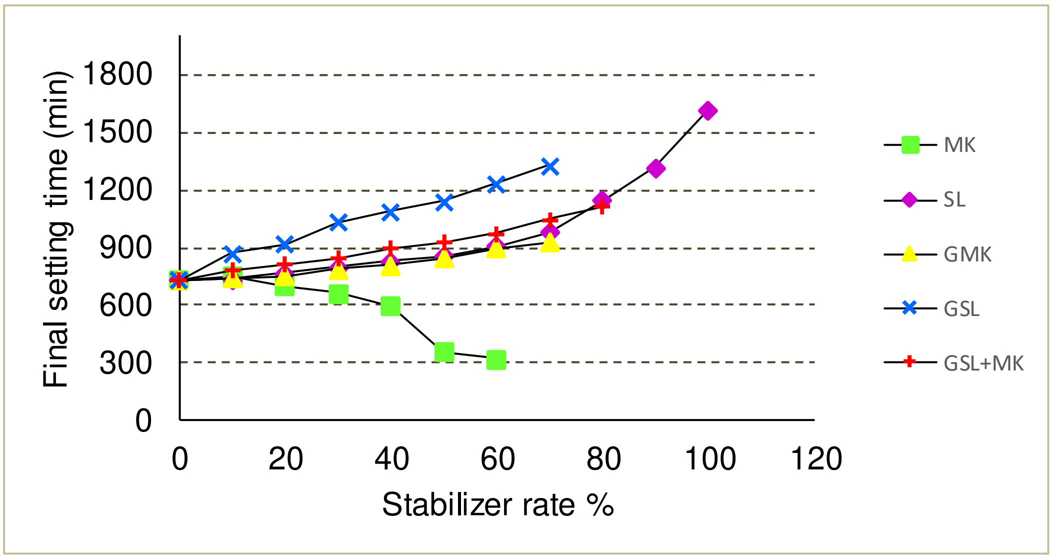

Figure 4.9 Final setting time (min)

Table 4.5 Final Setting time (min)

Stabilizer rate (%)	PC+MK	PC+SL	PC+GMK	PC+GSL	PC+(90%GSL+10%MK)
0	730	730	730	730	730
10	750	735	744	873	783
20	700	770	753	918	813
30	665	800	789	1035	846
40	595	830	810	1092	900
50	360	855	846	1143	927
60	322	910	900	1233	978
70	-	980	927	1326	1050
80	-	1145	-	-	1113
90	-	1320	-	-	-
100	-	1620	-	-	-

Due to the issues of setting time results also with considering the previous fresh tests (mini slump, Marsh flow time, bleeding), as well as the rheological results (i.e., shear stress and viscosity versus shear rate, yield stress and plastic viscosity) the stabilizer inclusions in low dosage rates 0% to 30% investigated in this study could be more offered for favorable flow and fresh performances.

4.3 Hardened properties of grout mixtures

4.3.1 Unconfined compressive strength

Unconfined compressive strength (UCS) performances of cement-based grout with the stabilizer inclusions (0% to 30% replacement) of MK, SL, GMK, GSL and GSL+MK have been illustrated all together in Figure 4.10 for the curing times of 7-day and 28-day. It is found from the results (Fig.8) that the UCS values due to the stabilizer inclusions are varied in the range from 5.5MPa to 10.2MPa for 28-day curing, and from 2.6MPa to 6.5MPa for 7-day curing.

On the responses of stabilizers particularly for 28-day curing (Fig.8), it is observed that the UCS performance of native grout due to the stabilizer MK increases slightly up to the rate of 10%, from 8.5MPa to 9MPa. Beyond that rate, the UCS decreases to 5.9MPa with increased MK. The increase and decrease trends of metakaolin can also be found in past studies (Sonebi et al., 2013; Cyr et al., 2014). However, the increases rat of MK up to 20% and 30% show a decreases in compressive strength in spite of grout mixture containing 20% MK also showed a significantly faster rate of reaction than others samples with 10% MK and reference mix. It was suggested (Wild et al., 1996) that main factors influencing the strength due to the use of metakaolin are i) the pozzolanic reaction of metakaolin with calcium hydroxide for producing more CSH, ii) acceleration of Portland cement hydration and iii) filler effect. A faster pozzolanic reaction rate and a greater acceleration of cement hydration could be occurred due to finer particles of metakaolin. Dependent upon an earlier effort (Cyr et al., 2014). In (Ramlochan et al., 2000) opinion that Even though the dilution effect of cement was taken into consideration, the alkali in the MK mixture was still small because of alkali fixing in pozzolan C-S-H structure.

For the effect of SL inclusions (Figure 4.10), the SL increases the UCS of native cement from 8.5MPa to 10.2MPa, at the rate of 10%. Beyond that rate, the UCS value decreases to 5.5MPa with increased SL rate.

Similar to the obtained decrease trend, it has been reviewed that (Rashed, 2018) that high amounts of slag additions (>50%) lead to a reduction or an insignificant enhancement in compressive strength found in most of researches. For the increase trend due to less amount of slag (i.e., 10% in this study), it can be said (Li et al., 2017b) that fine slag particles ensured the efficient hydration and enhanced the hydration rate of cement provide high strength by decreasing flaws in hardened grout. On the other hand, high amount of slag (20% to 30% in this study) could result in reduction of strength, likely due to the slow pozzolanic reaction, decreased amounts of effective oxides (CaO) and reduced concentration of hydration minerals (C3S) (Mirza et al., 2002).

As for geopolymerized metakaolin stabilizer (GMK), the result in (Figure 4.10) showed insignificant change or development in compressive strength. Where the highest value that obtains at 20% rate replacement with cement. However, this result due to the manufacturing stabilizer that obtained from the geopolymerized process has a low specific gravity (1.64) where it is considered a lightweight stabilizer or fine aggregate. On the other hand, by comparing with GMK, observed that the compressive strength of GSL increased with increasing the dosage of GSL. Where the grout mixture transformed from normal paste (cement + additional + water) to mortar that contains manufacture fine aggregate (sand). That means the compressive strength will increase with increasing the rate of stabilizer slag especially the specific gravity (2.4).

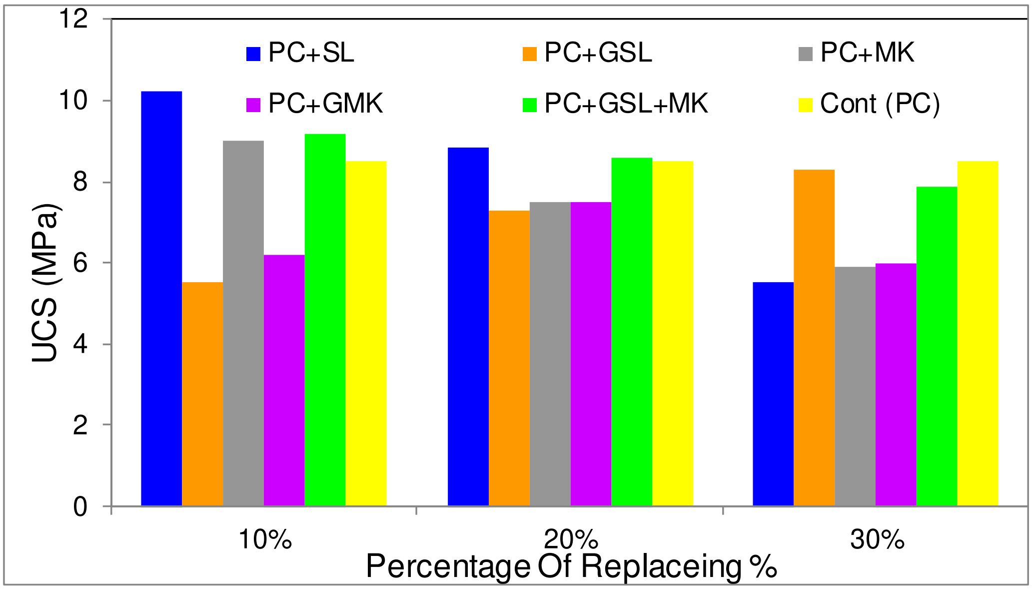

Figure 4.10 Compressive strength (MPa) result of grouts mixture with different additional

Cement replacement with MK in grout mixture at 10% of total (GSL+MK) showed (Figure 4.10) that observed a development in compressive strength. The maximum strength curries out at 10%, 20% (GSL+MK) and simple drop at 30% (GSL+MK) comparing to control (native cement). (Khatib et al., 2012) reported that The results show that the maximum strength of mortar happen at about 20% MK. When MK exceeds 30% MK as cement replenishment, compressive strength starts to decrease.

4.3.2 Ultrasonic Pulse Velocity (UPV)

The strength qualities of the stabilizer inclusions (0% to 30% replacement) of MK, SL, GMK, GSL and GSL+MK in the view of ultrasonic pulse velocity (UPV) tests have been presented all together in Figure 4.11 for the curing times of 7-day and 28-day. And this is what shows us through the results (figure 4.11) where it turns out through the rapid transmission of waves in the grout samples that include MK. Where there is a consensus between the result of UPV and UCS by giving high speed to wave transfer at the same sample that carries out high strength. The similar behaviors were observed at different binder content (SL, GSL, GMK, GSL+MK) by giving a high speed of wave transfer at high compressive strength with high accuracy.

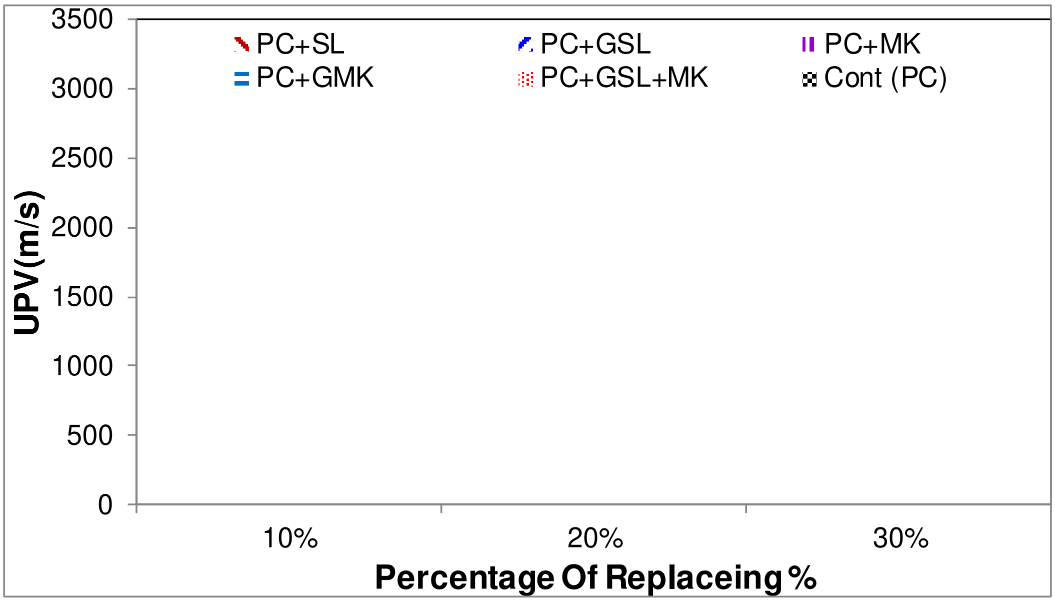

Figure 4.11 P-wave velocities of developed composites at different stabilizer percentage

Generally speaking for very low to low UPV performances of stabilizer inclusions, it can be said (Kramer, 1996) that the body waves (shear waves) propagate in low velocity through material when water content is high. Here, the low velocity could be due to the voids of grout specimens dependent upon the particle size and water content of specimen (Güllü et al., 2017). It is reported (Mandal et al., 2016) that UPV decreases with decreasing density of specimen. Characterization of changes in UPV can be permitted due to hydration (Rapoport et al., 1997; Ye et al., 2004). As implied above, water/air phase of specimens could be a dominant factor that determines UPV reported in a past study (Ye et al., 2004). The evolution of UPV was considered as a function of the volume fraction of each phase. However, different UPV behavior were observed for different stabilizers. This is because UPV increased linearly in 7 to 28 days. This is believed to be a sustained evolution of strength in these samples, i.e. formation of moisture products, is still in progress and is probably associated with an inherently slow pozzolanic reaction. These findings are in good agreement with the findings of (Ye et al., 2004) that declared that an increase in UPV is bounded after a solid frame fully coupled with cement based material occurs.

Chapter 5

Conclusion and recommendation

Perpetually, an invention for new materials is playing important role in the environment by reducing carbon emission due to Industry of traditional materials such as cement.

The use of different grout mixture cement-based grout and cement based geopolymerized stabilizer grout by use w/b ratio (1), has been investigated in this research based on the resulting obtained from viscometer test, mini-slump, marsh flow time, bleeding and setting time as a workability test. For hardened properties, compressive strength and ultrasonic velocity were investigated. The following concluding remarks could be proposed as following:

The rheological properties of cement-based grouts importantly have been affected by the addition of metakaolin to cement based grouts at different high MK dosage ratios. Strongly shear thinning regardless of the 20%-50% MK content. Therefore, the optimum dosage of MK is 10%, which make the grout mixture thickening, behave.

The increased MK fraction resulted in an increase in flow time, yield stress value, and plastic viscosity, and a decrease in mini slump. It is due to the related increase in water demand caused by the high surface area that is characterized by MK. The proportion of MK significantly decreased the bleeding and setting time. The substitution of MK had a great influence on the improvement of compressive strength on the 28th day. This may be due to the reaction of MK particles with calcium hydroxide to produce more CSH. Insignificant effect on P wave velocity in the ultrasonic test. From the test, the result of cement -based grout which component of slag as replacement of cement. The samples with SL show very similar shapes each other and almost was closest to the control group and have been behaved like thickening.

The SL-blended cement pastes had a longest initial setting time due to lower heat of hydration that means the SL is more active at a later age. That is what we observed when a result of compressive strength for grout mixture improved to 10%, 20% dosage of SL. With increasing amount of SL, the mini slump and flow time were increased, that gives an impression of the slag improving the workability and fluidity. Although the addition of SL increases the bleeding rate, it can be avoided by adding 10-15% of MK.

The high proportions of geopolymerized mixtures (60-70%GMK, 70%GSL, 70-80%GSL+MK) lead to pseudoplastic behavior, likely due to particle influences of stabilizers at high amounts. About the stabilizer (SL, GMK, GSL, GSL+MK) effects on yield stress and plastic viscosity, yield to insignificant changes (increase or decrease).

GMK result showed more workability and more replaceability than MK. As the mini slump spread is reduced by an acceptable rate. Better workability than MK was observed in flow time test. Moreover, gives the lowest percentage of bleeding from GSL & GSL+MK, lowest weight than GSL that it can be seen clearly through the specific gravity of both stabilizer. All of that due to the spherical shape of the granules and the containment of the outer shell on the powder of MK. insignificant improvement in compressive strength and UPV.

GSL replacement process with cement showed that the GSL could a good workability behavior at low dosage rate of replacement. It shows clearly in the tests of the mini slump, marsh funnel and setting time, and it could be indicate that the GSL more portability to segregation in the mixture, Showed it through bleeding and flow time tests test. On the other hand, by comparing with GMK, it observed that the compressive strength of GSL increased with increasing the dosage of GSL.

Returning to the effect of MK, add the amount of 10% of the total (GSL+MK) that did not show significant effect on grout mixture that showed through of viscometer and workability test. Compressive strength have been improved at low replacement rate in proses of grout mixture by mixing small amount of MK in (GSL+MK). Where the effect of MK is clearly observed in contrast to the effect GSL alone in grout mixture.

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