

UNIVERSITY OF HASAN KALYONCU GRADUATE SCHOOL OF NATURAL AND APPLIED SCIENCES

EFFECT OF RICE HUSK POWDER CONTENT WITH DIFFERENT WATER-CEMENT RATIOS ON RHEOLOGICAL PROPERTIES OF CEMENT-BASED GROUTS

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Muhammet ÇINAR

ABSTRACT

THE INVESTIGATION OF EFFECT OF RICE HUSK POWDER CONTENT WITH DIFFERENT WATER-CEMENT RATIOS ON RHEOLOGICAL PROPERTIES OF CEMENT-BASED GROUTS

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It is appear that a lot of waste materials producted during industrial production in all over the world. There is a difficulty for storing and removing these products. Moreover, they cause pollution of the environment. One of the huge waste products is rice husk. Every year nearly 400 million tons of waste rice husk produced in the world.

The rice husk has been used for different purposes such as a fuel in brick kilns, in furnaces, as an cleaning or polishing agent in metal and machine industry, in the manufacturing of building materials. In addition, rice husks themselves are a class A thermal insulating material because they are difficult to burn and less likely to allow moisture. Its ash is also replaced with cement as a cementitious material in concrete production

In this study, feasibility of use of rice husk powder (RHP) as filler in cement grout was investigated. For this purpose rice husk was grinded to certain grain size and mixed with cement grouts at 4, 8, 12, 16 and 20% respectively. The mixtures were prepared at different water to cement ratio (0.75, 1.00, 1.25 and 1.50). Effect of RHP on fluidity and rheological properties of the cement grout mixtures were studied. Test results showed that at water cement ratio (w/c) of 0.75, 1.00, 1.25 mixture showed shear thinning behavior. On the other hand, this behavior changes to shear thickening behavior at w/c = 1.50 at all RHP content. Experimental results indicated that this waste product can be utilized as filler in cement grout for geotechnical application such as filling large voids.

Key Words: Grout, Rheological properties, Rice husk powder, Grouting, Waste materials

ÇİMENTO ESASLI ENJEKSİYON HARCINA FARKLI SU-ÇİMENTO ORANLARI İLE FARKLI ORANLARDAKİ PİRİNÇ KABUĞU TOZU KATILARAK REOLOJİK ÖZELLİKLERİNİN ÜZERİNDEKİ ETKİSİNİ İNCELEME

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Tüm dünyada endüstriyel ürünlerle beraber yan atık ürünlerde ortaya çıkmaktadır. Bu ürünlerin depolanması ve uzaklaştırılması gibi zorluklar vardır. Ayrıca çevre kirliliğinede neden olmaktadır. Yüksek miktarda oluşan atık malzemelerden biri de pirinç kabuğudur. Her yıl dünyada üretilen atık pirinç kabuğu yaklaşık 400 milyon tondur.

Pirinç kabuğu çok farklı amaçlarda kullanılmıştır, örneğin; tuğla fırınlarında yakıt olarak kullanımı, metal ve makina endistürisinde temizleme parlatma işlemi için, inşaat alanında yapı malzemeleri üretiminde kullanılması. Ayrıca hemen tutuşmadığı için birinci sınıf termal yalıtım malzemesi olarakda kullanılır. Beton üretiminde çimento ile yer değiştirilerek de külü kullanılmaktadır.

Bu çalışmada, pirinç kabuğu tozu çimento bulamaçlarında dolgu maddesi olarak kullanılabilirliği incelendi. Bundan amaçla, pirinç kabuğu belirli bir inceliğe kadar öğütüldü ve çimento ile beraber karışama 4, 8, 12, 16 ve %20 oranlarında katıldı. Bu karışımlar farklı su/çimento oranlarında (0.75, 1.00, 1.25 ve 1.50) hazırlanarak karışımın akışkanlık ve reolojik özellikleri incelendi. Su-çimento oranının 0.75, 1.00 ve 1.25 olduğu karışımlarda kayma incelmesi / psüdoplastik akışkan özelliği tüm pirinç kabuğu oranlarında gözlenmiştir. Ayrıca, su- çimento oranı 1.5 olan karışımlarda kayma kalınlaşması / dilatant akışkan özelliği görülmüştür. Deney sonuçları, bu atık ürünün jeoteknik uygulamalarda büyük boşlukları doldurmak için enjeksiyon harcı içinde dolgu maddesi olarak kullanılabilir olduğunu göstermiştir.

Anahtar Kelimeler: Enjeksiyon, Reolojik Özellikler, Pirinç Kabuğu Tozu, Enjeksiyon Harcı, Atık Malzeme



To My Parents...

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LIST OF SYMBOLS/ABBREVIATIONS

ASTM	American standard for testing materials
ASTM C-143	Standard Test Method for Slump of Hydraulic Cement Concrete
С	Constant
D _{flow} K	Mini-slump flow diameter. coefficient of permeability to water (m/sec)
k _G	coefficient of permeability to grout (m/sec)
n	effective porosity of aggregate media
Pas	pascal-second = $1 \text{ N} \cdot \text{s/m}^2$
PC	Portland cement
RH	rice husk
RHP	rice husk powder
R ²	The coefficients of correlation
T _{flow}	Marsh- cone flow time
t _{cohession}	Plate cohession thickness
w/c	water-cement ratio
μρ	plastic viscosity (Pas)
τ	shear stress (Pa)
το	yield stress (Pa)
γ	shear rate (s ⁻¹)
ρ	density of fluid (kg/m ³)

CHAPTER 1

INTRODUCTION

Cement-based grouts are widely used in many construction applications [Nonveiller E. 1989]. Jet grouting, permation grouting, compaction grouting, coating pre-stressed cables, tunnels applications and rock pre-stressing anchors are the examples of the cement-based grout applications [Stille B. 2010, Yeon KS. 1997, Baltazar LG. 2012]. The rheological properties of the grouts are directly related with the pumpability and ability while penetrating to voids and cracks.

Grouting is a geotechnical process, which involves injection of cement or chemical grout for the purpose of filing cracks or voids in the rock mass or soil. Cement is the most common grout used in rock treatment. Before treatment, it is important to understand the rock condition and properties. For choosing the proper grout, both the soil formation and grout characteristics should be considered [Kamal H. 2011]. Cement-based grouts are called as a fluid composite of water, cement and, possibly, admixture. Different ranges of w/c ratios can be used for grouting applications. While doing the coating of prestressed cables, w/c ratio is ranged between 0.35 and 0.42 [R.J. Woodward 1990]. For the repair and consolidation of masonry structures w/c ratio of cement grout is ranged between 0.5 and 1.5 [A.E. Miltiadou, 1991]. As jet grouting applications need to behave like fluid to penetrate into soil or rock, w/c ratio changes between 1 and 2 [O. Benhamou, 1994]. Sealing cement grouts should have w/c ratios between 0.5 and 1 [C. Danot, N. 2001]. Figure 1 shows the schematical view of the different areas of use of cement grout and regarding of w/c ratio.



Figure 1.1. Summary scheme of the different fields of use of cement grouts [F. Rosquoe, 2011].

The most commonly used grout consists of cement and water with additives that reduce the cost or improve workability, rheological, durability and applicability. When the voids are large and penetration is easy, filers for bulking out are mixed with the grout. They weaken the grout but strength, however, is not an important issue in this type of application. Sand is a cheap filer but requires care to avoid segregation. Clay, such as Bentonite, could be used as grout filer or as a grout on its own, but it is more expensive and difficult to use than sand [C. Danot,2001].

There is not extensive research work present on RHP related with rheological properties of cement-base grout produced by rice husk. RH, as an organic waste, is a significant problem in rice-cultivating areas because it is not used profitably and is generally burned after harvest, which causes environmental problems [Kamal H. 2011]. The annual world rice production amount is approximately 400 million tons [Maeshima T, 2003], in which more than 10 percent is husk. More than half of the rice production in Turkey occurs in the Thrace region. Farmers claim that after rice production has finished, RH is their main problem in preparing seed beds for future crops to be grown [Sisman C.B, 2011]. This environmental problem may solve and provide an advantage use of RHP in cement based grout.

Whereas, limited research studied to generate cement-based grout with RHP is available to investigate the possible effects of grout on the fluidity or workability properties of grouts. Therefore, the important point is to observe the effects of RHP on the rheological properties of grout mixtures. In other words, it is attached importance to the preparing of grout matrixes including RHP, and effects of RHP on grout mixtures should be defined experimentally to be applied in site applications. Because of that the aim of this study is to show the potential usage of RHP in preparing of grouts. 24 grout composites including different amounts of RHP by percentage of total cementitious materials weight (%4, %8, %12, %16 and %20) were prepared at different water–cement ratios (w/c) of 0.75, 1.00, 1.25 and 1.50 respectively. A serie of workability and rheology tests including Lombardi Plate cohesion test, modified-marsh cone flow time test, mini-slump flow diameter test and a coaxial rotating cylinder rheometer were conducted to observe the fresh properties of grout mixtures.

CHAPTER 2

LITERATURE REVIEW

2.1 Definition and purposes of grouting

Grouting is defined as the injection of fluidized materials into voids of the ground or space between the ground and adjacent structures. The main objectives of grouting are to produce a stronger, denser, and/or less permeable soil or rock; it may also simply serve to fill voids, which are otherwise inaccessible and may prevent adequate stress transfer within the ground or from a structure to the ground.

Grouting for ground engineering is a process for filling the voids, fissures or cavities existing in the soil and rock to improve water-tightness or mechanical characteristics of the grouted materials. Three (3) classes of grouting materials are generally recognized: i) suspension-type grouts, ii) emulsion-type grouts and iii) solutiontype grouts. The suspension-type grouts include clay, cement and lime, while the emulsion-type grouts include bitumen and the solution-type grouts include a wide variety of chemicals. With the various pressures and operations applied in the grouting process, the improvement can be achieved in various forms, e.g. permeation or penetration, compaction or controlled displacement and hydrofracturing or uncontrolled displacement (Figure 2).

Due to the need for underground developments (e.g. basement, subway, etc.) in the past two decades, application of grouting technique in solving problems associated with groundwater seepage, incompetent foundation soil and sensitive existing structures have been widely used in the substructure construction works in Turkey. Permeation grouting by injecting cement grout into soil via a pressure system, e.g. pump, was found commonly used in the construction industry for reducing seepage effect induced by excavation in porous media, e.g. sand of high permeability and improving the stability and bearing resistance of ground in excavation and foundation works, respectively.

Due to the complexity of the rheological properties (e.g. yield stress and viscosity) of cement grout and its unclear flow behavior (i.e. groutability or injectability) in porous media underground, especially in the local sandy soil commonly found with high content of fines usually treated by using superfine cement grout or chemical grout overseas, the effectiveness of permeation grouting using ordinary Portland cement with high water cement ratio exceeding 3.0 by some local practitioners is not clear. Therefore, it appears that it is still largely a trial and error process in the current

practice, especially in the local construction industry. If it is not satisfactorily done, it could lead to wastage or unsatisfactory performance (e.g. poor water tightness) of the soil improvement work.



Figure 2.1. Various Forms of Improvement in Soil and Rock Grouting : (a) Permeation Grouting (Penetration), (b) Compaction Grouting (Controlled Displacement), (c) Hydrofacturing (Uncontrolled Displacement) [Koerner, 1985]

In the current state of the art of grouting, the motion of a viscous fluid injected from borehole into soil was analyzed by considering the laminar flow (i.e. Newtonian fluid) from inside a spherical or cylindrical cavity into the mass of granular soil perfectly homogeneous. According to Tomiolo (1982), these two available flow models (Raffle and Greenwood, 1961) consider the flow of viscous fluids through the soil follows the same laws ruling the flow of water, all values (e.g. coefficient of permeability to grout, k_G) being amplified proportionally to the ratio of grout viscosity to water viscosity as shown in Eq. (1-1). It is the opinion of the author that such consideration may not be appropriate for cement grout with water/cement ratio (W/C, by weight) below 1.5 in view of the significant Bingham's fluid characteristics possessed by these cement grout mixes and also the very high injection pressure applied in the cement based permeation grouting works, which may influence the validity of Darcy's law.

 $\frac{k}{k_G} = \frac{n}{n_w}$ (after Muller – Kirchenbauer, 1968) (Eq 1.1)

Where k_G = permeability of soil to grout, m/s

k= permeability of soil to water, m/s

n = viscosity of Newtonian grout, Pas (N .s/m²)

n_w= viscosity of water, Pas

For enhancing the application of cement based permeation grouting using the existing flow models in the local construction industry, proper understanding of the properties of cement grout, including the influences of the handling process to the measurement of viscosity of cement grout and grout flow characteristics taking into consideration of the influence of high injection pressure on the coefficient of permeability for cement grout (k_G) in porous media, i.e. the validity of constant k_G value assumed based on Darcy's law in the existing flow models, is needed.

2.2 Historical Development

The history of cement-based grouts commonly used in permeation grouting including grouting of fractures in rock masses as well as pores in soil deposits has been described in detail by Houlsby (1990), Weaver (1991) and Litteljohn (2003), from whose research much of the following data are drawn.

The concept of injecting a self-hardening cementitious slurry was first exploited in 1802 in Dieppe, France, to improve bearing capacity under a sluice. Over the next 40 years or so, various French engineers followed suit, concentrating on locks, docks, canals and bridges. In the United States, Worthen grouted the foundations of a flume in 1845, and nine years later had graduated to sealing a masonry pier on the New Haven Road at Westford.

From 1856 to 1858 in England, Kinipple who regarded himself as the inventor of cement grouting carried out experiments in creating in-situ concrete. Application did continue internationally and in 1876 the first dam grouting project was completed by T.Hawksley in Rochdale, England, and successful application in French and German mines, London tunnels, and Maltese and Scottish docks.

By 1915 the first technical paper devoted to the grouting of a rock foundation under a dam (Estacada, Ore.) was published (Rands, 1915), and much interest resulted. The grouting at Hoover Dam between 1932 and 1935 is said to mark the beginning of systematic design of rock treatment in the United States (Glossop, 1961).

Since then, development in rock fissure grouting have continued apace, with research into drilling and grouting technologies, water testing, and materials developments being well documented by Simonds (1947, 1958) and Leonard & Grant (1958) and in the proceedings/publications issued by USCE (1956), ASCE (1982, 1985, 1992), ICE (1963, 1992) and ACI (1984).

By 1933, Ischy had invented the tube-a-manchette system, a grout injection method ideally suited to the controlled treatment of soils with great operational flexibility. Thereafter, the approach to soil permeation was progressively enhanced and rationalized due to theoretical research (e.g. Maag, 1938), and materials developments. These latter focused on lowering viscosity, increasing gel time control, increasing strength, and improving durability. In 1963, the ICE Conference in London reviewed the contemporary state-of-the-art.

Since then, developments have continued principally into new materials, including those that are water reactive, elastic after gelling, highly durable, and environmentally compatible. Indeed, by 1983, Karol was able to list eight major research and review documents prepared directly by, or commissioned for, government agencies. These documents were in addition to fundamental, classic works by Cambefort (1977) and Caron (1982), as prime examples. In the early nineties, renewed attention has been devoted to the microfine cement-based grouts (e.g. DePaoli et al., 1992a, b) and the whole concept of grout rheology as related to efficiency of injection (Deere and Lombardi, 1985).

Regarding processes, the Japanese in particular have been active, bringing to commercial use a series of drill and grout systems (Bruce, 1989a), which have enjoyed considerable success in soft ground tunneling projects in the Far East, although they have received little attention elsewhere.

The interest shown in the some literature (e.g. Karol, 1983, 1990, etc.) and at conferences (e.g. ASCE, 1992; ICE, 1992, IS-Tokyo, 1996 and ASCE, 2003) confirms that permeation grouting remains a very dynamic, challenging, and evolving topic. Typically, developments originate with specialty contractors or materials suppliers, and are then explored further by universities and governmental agencies before entering general usage.

2.3 Classification of Grout Materials

Three (3) basic types of grout are differentiated according to composition as

follows :

• Suspension: Small particles of solids are distributed in a liquid dispersion medium, e.g. cement and clay in water, having a Bingham's fluid characteristics.

• Emulsions: A two-phase system containing minute (colloidal) droplets of liquid in a disperse phase, e.g. bitumen and water that are evolutive Newtonian fluids in which the viscosity increases with time.

• Solutions: Liquid homogeneous molecular mixtures of two or more substances, e.g. sodium silicate, organic resins, and a wide variety of other so-called chemical grouts, nonevolutive

Newtonian solutions in which the viscosity is constant until setting, within an adjustable period.

The type of grout material (i.e. cement) involved in the present study belongs to the suspension type of grout. The solution grouts are evolutive Newtonian liquids during their period of practical injectability, when permeation occurs in accordance with Darcy's law. However, the applicability of Darcy's law to the complicated Bingham's fluid characteristics of suspensions (e.g. cement grout) was not well investigated and verified.

2.4 Grout Material Parameters

The permeability of particulate grouts in porous media depends on the following factors.

- Stability (i.e. bleed capacity)
- Pressure filtration (i.e. loss of water under the applied pressure)
- Rheology (principally yield stress and viscosity)
- Grain size concentration (i.e. grout dislodges fine particles from soil matrix, which in turn become part of the suspension grout and reduce penetration)

As mentioned above, the solution grouts are evolutive Newtonian liquids during their period of practical injectability, when permeation occurs in accordance with Darcy's law. The principal controls over penetration distance and grout characteristics are therefore,

- Ground permeability and porosity
- Initial grout viscosity and its evolution. Deere and Lombardi (1985) noted that cohesion (i.e. yield stress) determines distance of travel and viscosity
- determines the flow rate.

- Pressure (related to flow rate)
- Practical duration of injection

2.4.1 Fillers

Fillers in portland-cement grout are used primarily for reasons of economy as a replacement material where substantial quantities of grout are required to fill large cavities in rock or in soil. Almost any solid substance that is pumpable is suitable as a filler in grout to be used in nonpermanent work. For permanent work, cement replacements should be restricted to mineral fillers. Before accepting any filler, tests should be made in the laboratory or in the field to learn how the filler affects the setting time and strength of the grout and whether it will remain in suspension until placed. All aspects of the use of a filler should be carefully studied. The economy indicated initially by a lower materials cost may not continue throughout the grouting operation. Additional personnel and more elaborate batching facilities may be needed to handle the filler. Some fillers make the grout more pumpable and delay its setting time. Such new properties may add to the costs by increasing both the grout consumption and the grouting time.

Sand is the most widely used filler for portland-cement grout. Preferably it should be well graded. A mix containing two parts sand to one part cement can be successfully pumped if all the sand passes the No. 16 sieve and 15 percent or more passes the No. 100 sieve. The use of coarser sand or increasing the amount of sand in the mix may cause segregation. Segregation can be avoided by adding more fine sand or using a mineral admixture such as fly ash, pumicite, etc. Mixes containing up to 3/4-in. aggregate can be pumped if properly designed. Laboratory design of such mixes is recommended. Sanded mixes should never be used to grout rock containing small openings and, of course, should not be used in holes that do not readily accept thick mixes of neat cement grout (water and portland cement only).

Fly ash is a finely divided siliceous residue from the combustion of powdered coal, and may be used both as a filler and as an admixture. Most grades of fly ash have about the same fineness as cement and react chemically with portland cement in producing cementitious properties. The maximum amount of fly ash to be used in grout mixtures is 30 percent by weight of the cement, if it is desired to maintain strength levels comparable to those of portland-cement grouts containing no fly ash.

Diatomite is a mineral filler composed principally of silica. It is made up of fossils of minute aquatic plants. Processed diatomite is an extremely fine powder resembling flour in texture and appearance. The fineness of the diatomite may range from three times to as much as15 times that of cement. Small amounts of diatomite may be used as admixtures to increase the pumpability of grout; however, large amounts as fillers will require high water- cement ratios for pumpability. As a filler, diatomite can be used where low strength grouts will fulfill the job requirements.

Pumicite, a finely pulverized volcanic ash, ashstone, pumice, or tuff, is also used as a filler in cement grout. Like fly ash and diatomite, it improves the pumpability of the mix and has pozzolanic (hydraulic cementing) action with the portland cement.

Other fillers is silts and lean clays not contaminated with organic materials are sometimes used as fillers. Leess, a windblown silt containing from 10 to 25 percent clay, is a suitable filler. Rock flour, a waste product from some rock-crushing operations, is also used as a filler. Rock flour produced during the manufacture of concrete sand is very fine but not always well graded. Grouts containing poorly graded rock flour are frequently highly susceptible to leaching. Most finely divided fillers increase the time required for the grout to set. It may be expedient to add an accelerator, described subsequently, to compensate for this.

2.5 Categories of grouting

Depending on the method used to introduce the grout into the ground grouting techniques can be subdivided into permeation grouting, fracture grouting, compaction grouting, jet grouting. Comprensation grouting is not a grouting process but may include grouting processes such as fracture and compaction grouting. The type of grouting are illustrated in Figure 3



Figure 2.2. Types of basic grouting techniques

2.5.1 Permeation Grouting

The first known application of "Permeation" Grouting dates from 200 years ago. The French Engineer Charles Berigny in 1802 used a suspension of water and puzzuolana cement to fill up the cavities in the foundation of a sluice in Dieppe, that had been damaged by settlement. In this way alluvial deposits were simultaneously sealed and stabilised. He named this treatment 'procédé d'injection'. After that, in the 19th century, injection was mainly used in mining applications (Glossop, 1960). The first application

of Portland Cement Injection dates from 1839, when Collin used injection to fill fissures in the masonry of the Grosbois Dam in France (Nonveiller, 1989). From 1900 onwards grouting equipment was constantly improved. The introduction of hydraulically driven, high pressure pumps and manometers led to increasing control over pumping pressures and grout flow.

The first known application of permeation grouting using a sodium silicate (gel) dates from 1886 (Jeriorsky patent). In 1909 Lemaire en Dumont patented a single-shot system based on a dilute silicate and acid solution, which however, gave too many practical problems to be applied (Karol, 1983). The Dutchman Joosten solved the practical problems in 1925 by developing an ingenious method for the treatment of sands, in which small volumes of concentrated sodium silicate were injected in stages through a perforated pipe as the pipe was driven to required depth (Littlejohn, 1985). During withdrawal of the pipe a strong calcium chloride solution was injected transforming the soil into "impermeable" sandstone. This system was extensively used when constructing the Berlin under ground in 1930.

The next milestone in grouting history was the invention of the tube-à-manchette (TAM) by Ischy in 1933. His invention permitted grouts of different properties to be injected in any order and at any interval of time from the same borehole. Shortly after that, Mayer (1934) developed a one-shot silicate, procedure. From the 20th century onwards, engineers also started to approach grouting in a more scientific manner. In 1902 a congress on grouting was held during, which engineers discussed the effect the number of injection points and the grouting pressure have on the results. The first large-scale permeation grouting stabilisation using cement was applied during the construction of the Hoover Dam from 1932 to 1936. This project made a large contribution to knowledge on grouting.

During the 1939-45 war the development of practical grouting applications was largely halted. However from then on there was a dramatic increase in the development of all sorts of new chemical formulations (mostly resins). Most important was the patent of Mello, Hauser en Lambe in 1953, concerning the acrylate of polyvalent metal (AM-9). This soft gel grout had a viscosity only slightly higher than that of water and thus could also be used in silty soils and it possessed excellent gel time control. In about 1980, this system was replaced by less toxic grouts like AC-400. The disadvantage of these resins is that they are very expensive compared to cement at silica gels. Therefore they are

usually only used for the renovation of masonry. Soletanche in 1957 developed a hard silicate gel, using an organic ester, producing strengths of 2-3 MPa in sands.

From the 1980s, the widespread use of permeation grouting for stabilisation purposes was largely discontinued in favour of other techniques, mainly because of problems arising from public opinion about the pumping of chemicals into the ground (because of environmental concerns). This contributed to the development of jet grouting and also to the use of fine cements (micro-cements) for permeation grouting. Important research concerning micro-cement properties has been performed by De Paoli (1992b).

Nowadays both silicate gels and as micro-cements are used on a large scale to stabilise soil and create grouted soil layers with low permeability.

Permeation grouting equipment usually comprises:

- \checkmark the drilling rig;
- \checkmark the injection unit (mixing and grouting plant);
- ✓ double packers and tubes-à-manchette.

The injection unit consists of storage facilities for grout and other materials, a mixing plant and several high-pressure grout pumps. Usually four or six pumps, the controls and the monitoring equipment are placed in a container

Depending on the intended function of the grout, the ideal grout should live up to the following criteria:

- ✓ be non toxic/non aggressive/non explosive, not harmful to personnel and material
- ✓ give a stable reaction/react stable and not cause any environmental pollution when hardened;
- ✓ be soluble in water (so the grout can be mixed with water on site and transport costs can be
- ✓ reduced);
- \checkmark keep well for an unlimited time independent of storage conditions;
- \checkmark have chemical reactions that are independent on soil conditions;
- \checkmark have low viscosity (equal to water) before hardening;
- \checkmark have a controllable gel time
- \checkmark be easy to mix;
- \checkmark have no degeneration with time;
- \checkmark have good strength and stiffness properties.

2.5.2 Compaction Grouting

Compaction grouting originated in the USA (California) in the 1950s. The first publication, on the compaction grouting procedure, describes a theoretical model of radial densification of the soil based on a spherical injected mass (Graf, 1969). The first Dutch application of compaction grouting was the lifting of a pneumatic caissons that subsided 30mm owing to a flooding accident (Gelderloos et al., 1969). The caisson was returned to its original position by pumping concrete in the under ground channel that originated under it owing to the flooding. Brown and Warner (1973) were the first to conduct tests examining the actual grouting mechanism. These tests included injections and the excavation of grouted elements. Since then many different tests have been conducted to examine the grouting procedure, including measurements of lateral forces, CPTs before and after grouting and the monitoring of structures.

The first application of compaction grouting to control ground movements and to prevent possible damage to structures* during tunnelling was made during the construction of the Bolton Hill Tunnel (Baker, Cording & MacPherson, 1983). Grouting and monitoring were successfully used to compensate settlements caused by a 5.8 m diameter steel-lined TBM tunnel in very dense sand and gravel. Since this successful application, compaction grouting has been extensively used for soil densification and tunnelling projects, mainly in the United States. The technique was first used on a large scale in Europe during the construction of the new London Metro connections, like the Jubilee Line Extension and the Docklands Light Rail.

Compaction grouting can also be used for soil densification. Baker (1985) describes the use of compaction grouting to compact in-situ liquefiable soils below two existing dam embankments. Boulanger & Hayden (1995) describe the use of compaction grouting combined with dynamic compaction to densify deep, loose fill deposits.

Over the last 10 years the important influence of the properties of the grout on the grouting results has been increasingly recognized. This has led to some developments, which are discussed in the next subsection.

There are many different opinions on the definition of an acceptable composition for compaction grout. Compaction grout has often been described as a "zero slump" grout. However the definition of slump is not often mentioned*. The Geotechnical Engineering Division of the ASCE (Warner, 1992) defined compaction grout as: "Grout injected with less than 1 inch (25 mm) slump, normally a soil-cement with sufficient silt size to provide plasticity together with sufficient sand size to develop internal friction.

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The grout generally does not enter the soil pores but remains in a homogeneous mass that gives controlled displacements to compact loose soils, gives controlled displacements of structures or both.".From the examined literature (Warner 1972, 1978, 1992, 1997, Warner et al. 1974, 1992 and Bandimere, 1997) it can be concluded that the mobility of the grout is more important than the slump.

The grout has to remain mobile until it has reached the intended destination, after which it will preferably become immobile and have adequate internal friction properties. Grouts that are too mobile may result in fracturing of the soil and subsequent loss of control of the grouting process. The effectiveness of compaction grouting will cease and the density of the overlying soils may even be reduced (Warner, 1992).

The slump, the mobility and the pumping characteristics are not directly related. Grouts with high slump characteristics may have low mobility and vice versa. Therefore it would be wise to replace stipulations relating to slump with stipulations relating to mobility.

Compaction grouting equipment usually comprises a drilling rig (which may be combined with a crane to pull the grout tubes), an injection unit and grout tubes. The drilling rig that is used to install the grout pipes, which are usually made of steel, is similar to the rig used for permeation grouting or jet grouting. The grout pipes are installed by means of rotary flush drilling which optionally can be cased. Casing may be preferred when grouting horizontally and/or under foundations. Casings typically have a 50 mm diameter, although casings up to 150 mm are used (Bandimere, 1997). To accommodate a single pass insertion the drilling rig is usually also used for grouting. When a long extraction of the tube is necessary, it is necessary to use a crane with long pulling capacity.

The injection unit consists of a grout mixer and a pump. Because of the high shear that has to be overcome to mix the grout, the grout mixer used must be a batch pug or screw mixer. Preferably equipment capable of recording the quantities of the added grout components should be used. Because of the risk of fracturing the soil when too much water is added, the use of an automatic water pump provided with a water-meter is especially recommended. The pump that is used must be able to displace grouts with low mobility and low slump at pressures up to 15 MPa and grouting rates of 0.1 m³/min. Ideally, it should be possible to change the rate of displacement without interrupting the pumping process. When a piston pump is used the quantity of grout injected can be

assessed by counting the pump strokes. More specific information on grouting equipment can be found in Müller and Bruce (2000).

Types of soils, although it should be noted that as a result of the increase in pore water pressure, consolidation effects might occur in cohesive soils. Much information can be found on the application compaction grouting to compensate for settlements of shallow foundations induced by tunnelling. However, the information on compaction grouting to compensate for the settlements of pile foundations as well as for increasing the bearing capacity of pile foundations is very limited.

2.5.3 Jet Grouting

Jet grouting was first applied around 1950 by Cementation Co. in Pakistan (Lunardi, 1997). from 1965 onward, further development was carried out by the Yamakado brothers in Japan (Ichise, 1974). In the seventies two jet grouting concepts were developed simultaneously. The first method developed by Nakanishi N.I.T. (Nakanishi, 1974) was named Chemical Churning Pile or CCP jet grouting. As the chemical or cement grout was being injected at ultra high pressures through 1-2mm diameter nozzles located at the bottom of a single drill rod, the rod was pulled and rotated, thus creating a soil-cement column. The second method, named jet grouting by its creator Yahiro (1973), is based on cutting, replacing and cementing the soil, typically by using three concentric rods supplying water, air and cement grout.

In Japan, many subsequent modifications of the jet grouting system have since appeared. The most important was the use of an air-encapsulated cement grout, creating the so called Jumbo Special Grout (JSG) (Ichise, 1974). The JSG is capable of providing columns with diameters 1,5 - 2 times larger than the CCP Columns.

Following the initial development in Japan, jet grouting was introduced and further optimised in the 1980s in Germany (Company: Keller), France (Company: Soletanche-Bachy), Brazil (Company: Novatecna) and especially Italy (Company: Rodio and C. And Pacchiosi). The most recent optimisation of the jet grouting process concern a system called the X-jet (Cross-jet) system. This system, which is capable of obtaining greater jet grouted column diameters at greater accuracy, was developed in Japan by the Chemical Grouting Company and licensed for Europe by Keller. The system is based on using two jets, which maintain high cutting energy until they cross. Relevant information on this process has not yet been published.

Jet grouting is a process involving disaggregation of a soil and its mixing with and partial replacement by a cementing agent. The disaggregation of the soil is achieved by a high-energy jet.

Before explaining the process some definitions are given:

- the rig is a rotary rig able to automatically regulate the rotation and translation of the jet grouting
- \checkmark string and monitor.
- ✓ the string is used to convey the grouting materials down-hole to the required depth.
- ✓ the monitor is a device attached to the end of the jet grouting string, comprising a drill
- \checkmark bit and nozzles.
- ✓ a nozzle is a device especially manufactured and fitted into the monitor, which is
- ✓ designed to transform a high pressure fluid flow in the string into a high speed jet directed at the soil.

Several different jet grouting systems are available, permitting jet grouting to be used in a variety of types of deposits and the creation of various diameter elements.

The main jet grouting systems that can be distinguished are:

- \checkmark the single fluid system;
- \checkmark the double fluid (air) system;
- \checkmark the double fluid (water) system;
- \checkmark the triple fluid system.

Each of these systems is explained in the following text.

The single fluid system is a jet grouting process in which the disaggregation and cementation of the soil are obtained by using a high energy jet of a single fluid, usually a cement grout. One or more circular nozzles* are used to allow the jetting of grout. For the high pressure supply of the cement mix only one rod is used.

The double fluid air system is a jet grouting process in which the disaggregation and the cementation of the soil are obtained by using one fluid (usually a cement grout) assisted by an air jet shroud as a second "fluid". One or more double nozzles are used to allow the simultaneous jetting of air and grout. The air nozzle is an annulus around the circular nozzle for grout. Two rods are used, separately conveying the two fluids (air and cement mix or water and cement mix respectively.

The double fluid water system is a jet grouting process in which the disaggregation of the soil is obtained by using a high energy water jet and the cementing is simultaneously obtained by a separate grout jet. One or more nozzles* are used for high pressure jetting of water and one or more deeper nozzles for jetting or grouting of cement mix.

The triple fluid system is a jet grouting process in which the disaggregation of the soil is obtained by using a high energy water jet assisted by an air jet shroud, and the cementing is simultaneously obtained by a separate grout jet. In special cases the water can be replaced by other appropriate liquids or suspensions. One or more double nozzles are used to allow the simultaneous jetting of air and water and one or more simple nozzles located at a deeper level are used to allow the grout injection. Three rods are used to supply the high pressure water, the compressed air and the pressure cement mix.

Pre-jetting is optionally used in cohesive soil layers. The jet grouting of an element is then facilitated by a preliminary disaggregation phase with a water jet only (single fluid system) or a water and air jet (double fluid system).

The equipment has to be able to perform the jet grouting operation by assuring the translation and rotation displacement of the string at the designed speed. It is also necessary to ensure that the string is supplied with the fluids coming from the plant, at the required pressure and rate of flow.

Jet grouting can be used for different purposes in either temporary or permanent works, for example:

- \checkmark to provide foundations for new
- ✓ structures;
- ✓ to underpin existing foundations;
- ✓ to create low permeability barriers;
- \checkmark to create retaining or supporting
- ✓ structures;
- \checkmark to complement other geotechnical
- \checkmark works (for instance temporary
- ✓ stabilisation);
- \checkmark to reinforce a soil mass.

2.6 Rheological Characteristics of Cement Based Grout

The rheological properties (e.g. yield stress and viscosity) of grout including other influence factors such as mixing time, stability (bleeding), degree of saturation and additives have been studied by various researchers 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. bentonite, etc.) because of poor permeation of pure cement grout due to its high viscosity and short setting time, and the grout mixes considered in these studies are found not to cover the practical range of cement grout mixes, i.e. W/C = 0.6 to 1.5, for effective application of permeation grouting in sand using ordinary Portland cement (i.e. Portland Type I) as adopted in the experimental program of the present research. Some comments/findings extracted from the past research works are summarized as follows.

Cambefort (1964) explained that cement grout has a well-defined shear stress that develops immediately after mixing and is characterized by its viscosity function.

Klein and Poloivka (1958) interpreted schematically the stages of cement grout after mixing as dormant, setting and hardening with strength of grout increasing with curing time approximately in exponential or power function.

Caron (1959) classified cement grout as Bingham's grouts, as possessing rigidity and viscosity simultaneously, both increase with time and displacement can only begin beyond a certain pressure or so called yield stress.

Raffle and Greenwood (1961) developed a graphical relation (Figure 4) between the rheological characteristics of grout and its capacity to permeate soil and indicated that injection of neat cement grout is controlled by viscosity and shear strength in the early and later stages, respectively. Obvious increases in the viscosity and shear strength were reported for the cement grout with water/cement ratio (W/C) not exceeding 0.6 as shown in Figure 4

LittleJohn (1975) emphasized that a water/cement ratio between 0.4 and 0.45 gives a grout with sufficient fluidity to be pumped and placed easily in a small diameter borehole and yet retain sufficient continuity and strength after injection to act as a strengthening medium. He reported a rapid increase in viscosity and shear strength for cement grout with water/cement ratio less than 0.9 which is different from the ratio of 0.6 reported by both Burgin (1979) and the author.



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

Deere et al. (1982) classified cement grout as stable grout based on bleeding not exceeding 5% after 2 hours from completion of mixing and commented that small amount of bentonite appears to be preferable, sufficient to reduce sedimentation and bleeding but not so great as to improve the pumpability and penetrability.

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

Lombardi (1985) analyzed the flow conditions of a mix through a smooth rock fissure and concluded that the yield stress determines the maximum distance the grout can reach and the viscosity determines the flow rate and therefore the time necessary to complete the injection.

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 microfine cement grouts with additives and concluded that yield stress and plastic viscosity increase with increasing specific surface and bentonite, 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 et al. (1992) studied the properties of cement grouts and grouted sands with additives and indicated that the maximum particle size must not exceed 1/3 to 1/10 the size of void in order to penetrate a formation at reasonable pressure and rate.

Helal & Krizek (1992) studied the orientation of pore structure in cement-grouted sand indicated that pore structure injected with a cement grout is a function of water/cement ratio and sedimentation behavior of the suspended particles.

Shroff et al. (1996) studied the rheological properties of microfine cement dust (MCD) grouts and reported that MCD grout is able to penetrate medium sand having permeability, $k= 7.89 \times 10-3$ 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.

Perret et al. (2000) studied the effect of degree of saturation of sand on groutability and concluded that the propagation of grout through porous media is influenced 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 are though to have led to greater water dilution in the case of unsaturated sand.

2.7 Rice Husk

Rice husk is major by-product of paddy processing. The rice husk accounts for about one fifth of the paddy produced, on weight basis. Estimated annual production of rice husk is about 180 thousand tonnes. Economic utilization of this by-product is highly desirable.

The main constituents of rice husk are cellulose, pentosan, lignin and silica. All these constituents have valuable industrial applications. Pentosan is considered a good source for furfural production. Amorphous silica is a very good source for preparation of pure silicon and a number of silicon compounds such as silicon carbide, silicon nitride, cement, ceramic and other silicate materials. Cellulose is raw material for pulp and

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paper making. Rice hull has thermal conductivity of about 0.034 W/(m.°C), which is comparable with thermal conductivity of excellent insulating materials. Energy content of rice hull is 11.9-13 MJ/kg at 14% moisture content. In terms of nutrients, it has less than 10% total digestible nutrients. Rice hull has a bulk density of 100-160 kg/m3.

Uses of Rice Husk

- ✓ Fuel
- ✓ Building material (Fibre boards)
- \checkmark Ash and pure silica
- ✓ Production of furfural
- ✓ Cattle feed

2.7.1 Fuel Use of Rice Husk

Traditionally, it has been mostly used as a fuel to provide energy for rice mill operation. However, most of the furnace designs convert only about half of the available energy in husk to the use. The remaining heat potential remains in its fixed carbon portion, which creates a disposal problem also. Several researchers have conducted various studies related to combustion of rice husk to utilize maximum energy and to produce better grade residue.



Figure 2.4 Fuel Use of Rice Husk

2.7.2 Production of white ash

Rice husk-ash is a unique source of high-grade amorphous silica. The silica present in rice husk, being of biogenic origin, is inherently amorphous. Normally, controlled combustion below 700°C yields white ash with amorphous silica. Amorphous silica obtained from rice husk is chemically active and hence a very useful product. At higher temperatures, it undergoes a phase change resulting into crystalline forms of silica.



Figure 2.5 Production of white ash

2.7.3 Production of furfural

Furfural from rice husk can be prepared by acid hydrolysis using sulphuric acid (<0.5M) and super heated water at 185°C. Furfural products can replace formaldehyde in phenol-formaldehyde resins and can form chemically inert materials useful in a number of products such as the manufacture of corrosion resistant pipes. Furfural resin is also used to dissolve the undesirable constituents of lubricating oil.

2.7.4 Fibre boards

For binderless boards, the powdered rice husk (0.25-0.39 mm) mixed with 5-8% (by weight) concentrated sulphuric acid should be sun dried. The dried powder can be pressed at 60-70 kg/cm² for 20-25 min at 165°C or 12-15 min at 175-200°C to form the board.

Board using binder can be made by mixing rice hull powder (0.78-1.68 mm at 4-6% moisture content) with 7-8% (by wt.) phenol-formaldehyde resin, dried in the sun, and molded in a hydraulic press into 1 mm thick board by pressing 1 min at 175-200°C.



Figure 2.6 Fibre boards

2.7.5 Rice husk as feed

Rice hull forms the major part of the concentrate fed to the cattle. However, it is low in total digestible nutrient level. It is suggested that the level of husk should not exceed more than 15% in cattle, and 25% in lambs. To improve the digestibility of husk, some treatments like treatment with alkali (NaOH), fermentation is suggested

CHAPTER 3

EXPERIMENTAL PROGRAM

3.1.Materials

3.1.1 Rice Husk

The Rice husk was obtained from a rice producing factory in Mersin, Turkey. The RH used in this study is produced and sold as a commercially waste material by this factory. The firstly rice husk was grind. Particle size distribution curve of RHP is given in Figure 3.1. The physical and chemical properties of RHP and cement are given in Table 3.1.

Chemical analysis (%)	Portland cement (PC)	Rice Husk Powder				
CaO	61 94	0.22				
SiO ₂	18.08	20.17				
Al ₂ O ₃	5.58	0.04				
Fe ₂ O ₃	2.43	0.12				
MgO	2.43	0.07				
SO ₃	2.93	-				
K ₂ O	0.99	0.61				
Na ₂ O	0.18	0.02				
ZnO	-	0.01				
MnO	-	0.05				
CuO	-	0.01				
P_2O_5	-	0.13				
Ti_2O_2	-	0.01				
Loss on ignition	4.40	78 54				
	4.40	/0.34				
Pysical properties						
Specific gravity	3.17	0.43				
<i>Fineness (Blaine)(cm²/g)</i>	3750	10657				

Table 3.1: Properties of Portland cement and RHP



Figure 3.1.Grain-size distribution curve

According to grain size distribution curve, RHP values are $D_{10} = 0.09 D_{30} = 0.15 D_{60} = 0.28$. Also, C_U and C_C values are 3,11 and 0.89. All sizes of particles from the No. 4 to No. 200 sieve.

3.1.2 Cement

Portland cement (CEM1), grade 42.5R conforming to ASTM C150 Type-I was used in the production of cement paste and grout. The cement was produced by Maraş Çimento Company.

3.1.3 Water

Damla spring water was used to manufacture cement paste grout. Because, there are so much lime inside school network water.

3.2 Methods

3.2.1 Mixture proportions

One of the significant parameters that has remarkable effect on hardened and fresh properties of grout mixtures is water-cement (w/c) ratio. Therefore, in this study different w/c ratios (0.75, 1.00, 1.25 and 1.50) were selected to investigate the effect of RHP on the grout matrixes. This range for w/c ratio is used for geotechnical applications such as cement grouts for soil or rock injection, jet-grouting and sealing cement grouts [O. Benhamou 1994, C. Danot]. Twenty-four grout mixtures at various

water-cement ratios were prepared to observe the effect of RHP at different proportions mixed to grout (Table 3.2). Both the amount of RHP and water-cement ratio were selected as two important variables. Different amount of RHP were added to grout mixture as a proportions of 4, 8, 12, 16 and 20% by total cementitious material, respectively. In one grout mixture (control mix), produced at each w/c ratio, any RHP was not added for control purposes. As shown in Table 3.2, all proportions of RHP and w/c ratio and mix design parameters are tabulated.

Mıx ID	w/b	RH percent (%)	PC (kg/m ³)	RH (kg/m ³)	Water (kg/m ³)	Density (g/cm ³)	D _{flow} (mm)	T _{flow} (s)	t _{cohession} (mm)
M1	0.75	0	927	0	696	1.62	175	34	0.23
M2	0.75	4	875	61	656	1.59	120	57	0.42
M3	0.75	8	829	116	621	1.56	99	145	0.52
M4	0.75	12	787	165	590	1.54	65	302	0.91
M5	0.75	16	749	210	562		-	-	-
M6	0.75	20	714	250	536	-	-	-	-
M7	1.00	0	751	0	751	1.50	200	27	0.15
M8	1.00	4	712	57	712	1.48	167	29	0.26
M9	1.00	8	677	108	677	1.46	155	36	0.27
M10	1.00	12	645	155	645	1.44	123	80	0.35
M11	1.00	16	616	197	616	1.42	86	0	0.65
M12	1.00	20	589	236	589	1.41	80	0	1.15
M13	1.25	0	632	0	789	1.42	230	26	0.09
M14	1.25	4	600	54	750	1.40	210	28	0.24
M15	1.25	8	572	103	715	1.39	194	31	0.31
M16	1.25	12	546	147	683	1.37	157	38	0.33
M17	1.25	16	523	188	653	1.36	140	67	0.59
M18	1.25	20	501	225	626	1.35	99	271	1.18

Table 3.2: RHP mixture proportions and fresh properties of the grout mixtures

3.2.2 Test Procedures

The same mixing methods were used for the all grout mixtures. To obtain grout mixtures, 5 L laboratory mixer that has a standart rotary type was used. The standart procedure applied in the study is given following: the binders including portland cement and RH were prepared with mixing water for one minute and then the mixtures were mixed by hand for one minutes also. Finally, the mixing process was gone on at 240 rpm speed for 3 minutes. While preparing mixtures and doing all tests, humidity and temperature of the laborotory were 55-65% and 23 ± 3 , respectively. As soon as mixing was finished, plate cohesion, marsh cone flow time and mini slump flow diameter were determined on the fresh grouts. Coaxial rotating cylinder rheometer (proRheo R180 Instrument, Germany) was used for carrying out the rheological properties of grou mixtures. To check the tets results all tests were conducted in twice with new grout mixtures and test results show that tests were recurrent.



Figure 3.2. a. Mixer b. Rheometer (ProRheo R180 Instrument)

3.2.2.1 Mini Slump Flow Diameter, Plate Cohesion and Marsh Cone Flow Time Tests

In order to estimate workability or fluidity of fresh grout mixtures mini slump flow diameter, plate cohesion and marsh cone flow time tests were conducted. All tests were completed at 8-12 minute as soon as water contacted with cement. Doing these tests are easy methods to define the workability and fluidity performance of the grouts prepared at site works. The mini slump test can be defined as spreading of grout mixture on a plate after grout was placed into cone shaped mould. Diameter of the spreaded grout gives the mini slump diameter as milimeter. The mini slump cone is similar to the slump cone (ASTM C-143) used for concrete. Their dimensions are proportional with height, bottom diameters and top diameters (38, 19 and 57 mm, respectively) [D.L. Kantro 1980]. The flowing of a defined volume of grout through a flow cone at measuring time can be calculated by marsh cone test. A metalic funnel that has an internal orifice diameter of 5 mm and 1500 ml volume capacity was used to calculate the marsh cone flow time in this study. The marsh cone was filled with 1250 ml grout mixture and bottom outlet was opened and then grout started to flow. After opening the bottom outlet, the elapsed time was measured as long as 1000 ml of grout had flowed. As a result of that the elapsed time gived the marsh cone flow time. The marsh cone flow time of water was estimated as 24 second to compare to that of fresh grout. Cohesion was calculated by using Lombardi plate cohesion meter. The plate cohesion meter does not have any ASTM standard, but some studies describe the dimensions of plate cohesion meter [Weaver, K, (1991)]. Dimensions of the plate cohesion meter produced by a thin steel plate is 10x10x3 mm. Both sides of the plate must have rough surfaces. When the plate immerses to grout mixture, grouts sticks to the plate because of cohesion. The meter obtained from plate cohesion does not have any standart. Therefore, the cohesion calculated from the experiments is compared with control mixture. Because of that there is no exact value, the results obtained from the tests are compared each other.

All rheological parameters including apparent viscosity, plastic viscosity and yield stress of groutmixtures were calculated by using rheometer. Sliding is usually seen while grouts are flowing in coaxial cylinders because of decreasing of the cement particles [H.A. Barnes,1995]. An existance of slip layer can be shown with the yielde stress that was largely independent of rotational speed [A.W. Saak,2001]. Gravity may increase the slip effects for grout mixtures that cause sedimentation with time [H.A. Barnes,1995]. Slip effect was not noted in this study. While rheology tests were being conducted, shear rates were ranged from 25 s⁻¹ to 500 s⁻¹ for each grout mixture. The apparent viscosity is a function of the shear rate. Therefore, the shear-thickening behavior of the grouts were observed with respect to the apparent viscosity of grouts.

Both the ascending and descending curve were ploted from flow curve depending on the shear stress-shear rate curve. Fig. 3.2 presents the a ordinary flow curve of a grout composite. Ascending curve was shown in Fig.3.2 as blue curve because of the undisturbed state of the grout. Ascending curve was obtained with shear rate increasing. Similar behavior was shown in descending curve (dashed red line in Fig. 3.2). The ascending curve was used to obtain the shear stresses conducted from the test in this study.



Fig. 3.3. Typical flow behavior of grouts obtained from the coaxial rheometer.

Different types of analytical models have been proposed to estimate the rheological parameters of cement-based grouts. Among these, the Bingham model $(\tau=\tau_0+\mu_p\gamma)$ that is usually used for defining the rheological properties of cement based grouts [A. Yahia]. Plastic viscosity (μ_p) and yield stress (τ_0)are obtained from the shear stress versus shear rate curve. On the other hand, if highly pseudo-plastic and shear thickening behaviors disappear the yield stress calculated by using the Bingham model is lower than true yield stress (Fig.3.3). Because of this situation, yield stress and plastic viscosity are estimated by using modified Bingham model (Fig.3.3). Modified Bingham model is described as second order polynomial equation and following equation is given below [A. Yahia]:

$$\tau = \tau_0 + \mu_p \dot{\gamma} + c \gamma^2$$

where τ =shear stress (Pa), τ_0 =yield stress (Pa), μ_p =plastic viscosity(Pa s), $\dot{\gamma}$ =shear rate (s⁻¹) and c=constant. The modified Bingham model presents a more certain solutions than the traditional Bingham model for the similar mixtures [K.H. Khayat,1997].



Fig. 3.4. Typical flow behavior of grouts obtained from the coaxial rheometer.

CHAPTER 4

RESULTS AND DISCUSSIONS

Table 3.2 presents the workability properties (mini-slump flow diameter, Lombardi plate cohesion and marsh cone flow time) of all grout mixtures. On the other hand, Table 4.1 presents the rheological properties expressed as the yield stress and plastic viscosity of all the grout mixtures. The coefficients of correlation (\mathbb{R}^2) of the modified Bingham model are also included in that table.

Mıx ID	τ_0 (Pa)	μ _p (Pa.s)	Grout Temperature (⁰ C)	R ²	
M1	2.470	0.006	25.0	0.991	
M2	4.748	0.018	25.0	0.991	
M3	7.710	0.079	25.0	0.988	
M4	12.66	0.417	25	0.991	
M5	-	-	24.0	-	
M6	- / /	- /	24.0	-	
M7	0.111	0.005	23.0	0.987	
M8	0.386	0.005	22.0	0.994	
M9	1.073	0.009	23.0	0.996	
M10	5.060	0.031	23.5	0.991	
M11	7.225	0.016	25.0	0.978	
M12	13.37	0.338	24.5	0.994	
M13	0.027	0.002	22.5	0.994	
M14	0.146	0.001	23.5	0.996	
M15	0.422	0.003	23.0	0.987	
M16	1.662	0.004	23.5	0.996	
M17	4.058	0.022	24.5	0.986	
M18	6.828	0.160	24.5	0.976	
M19	0.013	0.004	24.2	0.995	
M20	0,083	0.015	23.2	0.991	
M21	0.226	0.012	22.2	0.993	
M22	0.665	0.042	22.5	0.994	
M23	2.002	0.037	22.5	0.993	
M24	4.686	0.439	22.5	0.981	

Table 4.1 Rheological properties of the grout mixtures

4.1. Workability (Fluidity) properties of grout mixtures

Deformability and fluidity (workability properties) of the grout mixtures are discussed in Fig. 4.1. According to Fig. 4.1-a, increase of the RHP amount in the grout mixtures decreases the diameter of mini slump flow. On the other hand, at a constant RHP content, increase of w/c ratio increase the diameter of mini slump flow due to the water effect. According to Fig. 4.1-b, at w/c=0.75 and w/c =1.00, increase of the RHP amount in the grout mixtures cause an increase on the marsh cone flow time and grout mixture could not mix % 12, % 16 and % 20 percent of RHP amount for w/c =0.75. Also grout mixture couldn't mix % 20 percent of RHP amount for w/c=1.00. But it does not have so much effect on marsh cone flow time at other w/c ratios. Moreover, at a constant RHP content, increase of w/c ratio decrease the marsh cone flow time due to the followability effect of water. As it is shown that grout mixtures including RHP reduce fluidity and deformability when w/c is particularly less than 1.00 with compared to w/c \geq 1.00. In other words, an increase of RHP content in grout mixtures remarkably reduced the workability properties of the grout samples at low w/c ratios. This reduction in workability of grout samples when RHP is used can be explained that RHP has a higher water absorption capacity.

The Blaine surface areas are given for PC ($3030 \text{ cm}^2/\text{g}$) and RHP ($10657 \text{ cm}^2/\text{g}$) in Table 3.1. According to Table 3.1, the blaine surface area of RHP is notably greater than Portland cement's. As a result of that high surface area causes to absorption of more water. An other reason can be explained that volume of the mixture increases with addition of RHP when density and total weight of cementitious materials with same w/c ratios are kept constant (Table 3.1). An increase in volume of paste reduces the fluidty of the grout mixtures because of an increase on the plasticity and cohesiveness [Sahmaran M , 2006- Yahia A,2005]. However, according to Fig. 4.1, increase of w/c ratio in grout mixtures incluiding RHP effectively changes the workability behavior of grout mixtures. The workability of the grout mixtures can be adjusted by optimizing the w/c ratio of the grout samples prepared with the addition of RHP.



Fig. 4.1. (a) Effect of RHP on mini-slump flow; (b) Effect of RHP on marsh cone flow time

4.2. Rheological properties and Compressive Strength

The effects of w/c ratio and RHP amount on rheological properties of grout mixtures can be discussed on the flow curve drawn by different shear rate versus shear stress. The apparent viscosities of the grout samples at different shear rates are listed in Table 4.2

for RHP addition at different w/c ratios. Flow curves of grout mixtures including RHP at various w/c ratios are shown in Fig. 4.1, respectively. The shear stress versus shear rate curves are examined by using modified binghm model for grout mixtures. The correlation coefficients(R^2), which were obtained from the graphics, are almost equal to unit Table 4.1 summarises the rheological properties and R^2 values of the grouts mixtures. Typical variation of plastic viscosity with RHP content is shown in Fig. 4.4.

Mıx	Shear rate (s ⁻¹)									
ID	50	100	150	200	250	300	350	400	450	500
M1	0,069	0,037	0,029	0,025	0,020	0,020	0,019	0,021	0,022	0,021
M2	0,135	0,075	0,056	0,048	0,042	0,040	0,039	0,038	0,037	0,040
M3	0,255	0,172	0,135	0,115	0,100	0,092	0,087	0,084	0,082	0,078
M4	0,378	0,318	0,300	0,308	0,302	0,288	0,255	0,225	0,203	0,190
M5	-	-	-	- /		- / -	- / /	-	-	-
M6	-	-	- / /	-	- /	- /	-	-	-	-
M7	0,012	0,005	0,005	0,006	0,007	0,008	0,008	0,007	0,007	0,007
M8	0,018	0,011	0,007	0,008	0,009	0,010	0,011	0,012	0,013	0,012
M9	0,037	0,026	0,020	0,018	0,017	0,018	0,019	0,019	0,019	0,019
M10	0,155	0,092	0,072	0,059	0,052	0,048	0,047	0,047	0,046	0,045
M11	0,302	0,246	0,205	0,172	0,149	0,133	0,118	0,108	0,098	0,092
M12	0,388	0,327	0,307	0,283	0,249	0,222	0,196	0,175	0,155	0,139
M13	0,002	0,004	0,004	0,004	0,004	0,005	0,005	0,005	0,006	0,006
M14	0,006	0,006	0,007	0,005	0,005	0,005	0,005	0,007	0,007	0,008
M15	0,020	0,018	0,008	0,007	0,007	0,008	0,009	0,010	0,010	0,010
M16	0,048	0,028	0,020	0,018	0,016	0,017	0,017	0,018	0,018	0,019
M17	0,120	0,072	0,055	0,048	0,040	0,038	0,037	0,034	0,036	0,035
M18	0,290	0,226	0,195	0,168	0,145	0,125	0,112	0,100	0,095	0,088
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M19	0,003	0,002	0,003	0,004	0,004	0,005	0,005	0,005	0,006	0,006
M20	0,001	0,002	0,003	0,004	0,004	0,005	0,005	0,006	0,006	0,007
M21	0,011	0,006	0,005	0,004	0,006	0,006	0,007	0,007	0,007	0,008
M22	0,025	0,014	0,012	0,010	0,013	0,013	0,014	0,014	0,015	0,015
M23	0,053	0,032	0,026	0,022	0,020	0,020	0,020	0,023	0,024	0,025
M24	0,148	0,099	0,078	0,067	0,058	0,050	0,053	0,048	0,047	0,048

Table 4.2: Apparent viscosities (Pa s) of grout mixtures at various shear rates

As seen from Fig. 4.3, the higher reduction in the apparent viscosity of the grout mixtures for a given range of shear rate indicates that the degree of pseudo-plasticity of grout increases with an increase in the amount of RHP. Grout mixtures (w/c= 0.75, w/c= 1.00 and w/c= 1.25) exhibit significant shearthinning behavior whereby apparent viscosity decreases with increasing shear rate (Table 4.2). Such mixtures are typically

thixotropic where the viscosity buildup is accelerated at a low shear rate that can further inhibit flow and increase viscosity. RHP increases the water demand, and increased pseudo-plasticity response in the presence of RHP may be due to the high water retention capacity of RHP, which increases the agglomeration of particles. Grout is a concentrated suspension composed of cementitious material particles (cement+RHP) suspended in water and mixing breaks down the flocculent structure responsible for thixotropic behavior of grout [L.J. Struble,1991]. Thus, a highly thixotropic behavior grout mixture with natural zeolite is a result of combination of reversible coagulation, dispersion and then re-coagulation of the RHP particles [J. Wallevik,2003].



(b)



Fig. 4.2. Flow curves of grouts containing RHP ranged between 4% and 20 %, with w/c ratio of (a) 0.75; (b) 1.00; and (c) 1.25; (d) 1.50.

As it is shown in Fig.4.2, shear thickening behavior was observed from rheological proeperties of the grout mixtures obtained by the flow curves at the w/c =1.50 in all RHP percentages [Cyr M, 2003]. Shear thickening behavior may be explained by the order–disorder transition theory of Hoffman [Hoffman RL,1998]. According to his theory, the flowing state, defined as ordered, turns to disordered state at critical shear rate. In less-ordered structures a big amount of the applied energy revealed by collisions between particles is distributed to obtain suspension flow. Hence, the experimental study showed that increase of the shear rate exponentially increase or nearly constant the apparent viscosity of grout mixtures in shear thickening behavior. For instance, a grout mixture made with a RHP content of 20 % at w/c=1.50 has apparent viscosity

value changing from 0.058 to 0.055 Pa.s with respect to shear stress ranging from 4.63 to 23.22 Pa. In addition, grout mixture contained RHP content of 4 % at w/c=1.50 has apparent viscosity value changing from 0.002 to 0.007 Pa.s with respect to shear stress increasing from 0.150 to 2.820 Pa. It can be concluded that increase of shear rate increase the disorder state between the cement and RHP particles in the free liquid. As a result, it can be said that disorder state can cause an increase in apparent viscosity of grout mixtures. And also, increase of water amount can be resulted as a partial flocculation of cement particles.



Fig. 4.3. Effect of RHP content at different w/c ratios on the yield stress of grouts

Yield stress can be explained by a limit stress value that must be exceeded to start to flow. If yield stress is lower this means that the resistance will be less to start flowing. As it is known that yield stress is directly related with slump property of grouts [Ferraris C.,1998]. When the yield stress value of grout mixture exceed the gravitational force, the grout mixture is prohibited from fully crumpling to the plate surface. Hence, the yield stress value is partially linked with the penetrability of grout mixture into cracks and voids [Şahmaran M,2008]. The yield stress values of grout mixtures conducted with different amounts of RHP at different w/c ratios are shown in Fig. 4.3. The increase of amount of RHP content at w/c=1.00, w/c=1.25 and w/c= 1.50 increases the yield stress. There are small effects on the yield stress. As it is known that the increasing of w/c ratio greater than 1.00 slightly affect the yield stres. This can be explanied that the grout mixtures that have more amount of water show high-fluidity behavior like water.



Fig. 4.4. Effect of RHP content at different w/c ratios on the plastic viscosity of grouts. Plastic viscosity is one of the rheological properties of a fresh mix. After the yield stress is exceeded, the flowability of fresh mix can be explained by plastic viscosity. The spreading rate of a flow is also inspected by it. The segregation, which is seen in inhomogeneous flow between gravitational sedimentation and the substances of grout mixture, is prevented by viscosity [Şahmaran M,2008]. Fig. 4.4 shows how the plastic viscosity of grout mixtures obtained from the coaxial rheometer is affected by the RHP content at various w/c ratios. According to Fig.4.4, increase of RHP amount in the grout mixture increases the plastic viscosity with respect to all w/c ratio. In addition that, for constant RHP amount, an increase of w/c ratio increase the plastic viscosity of grout mixtures. The plastic viscosity of the grout mixtures producted at w/c ratios are needed to decrease the plastic viscosity of grout samples, if RHP is used at lower percentages.

4.3. Correlation between rheological and workability properties of grout

Mini slump flow diameter, plate cohession meter and marsh cone flow time were correlated with yield stress and plastic viscosity to ensure easy and serviceable methods for adjusting rheological control of grout mixtures. In order to define the correlation between workability and rheological properties of grout mixtures, some graphics were plotted between test results of grout mixtures and R^2 value between any of two grout mixtures were estimated. If there is a perfectly correlation between two test results, this means that coefficients of correlation (R^2) will be 1.00. On the other hand, if weak correlation is observed, R^2 value will decreases and move away from 1.00.



Fig. 4.5. Correlation between rheological and workability properties of grout.

As it is known that the slump flow diameter give some informations about the deformability of concrete which the yield stress has a relation [Ferraris C.,1998]. Therefore, there is a well correlation between the yield stress observed from the modified Bingham model and mini slump flow diameter of fresh mixes. The results are shown in Fig. 4.5. As it is shown in Fig. 4.5-a a reduction in the yield stress increases the mini slump flow diameter of grout mixtures. There is a good linear correlation between the mini slump flow diameter and the yield stress obtained from grout mixtures with respect to R^2 =0.851 (Fig. 4.5-a). As a result of that a well defined demonstration of the yield stress value could be estimated from the mini slump flow diameter test. The results are compatible to the literature [Lachemi M]. There is relation between the plastic viscosity and mini slump flow diameter test.

As soon as the yield stress is exceeded, flowing of grout mixture starts in the marsh cone test. Because of this reason, there is a direct relation between the marsh cone flow time measured and the viscosity. While the marsh cone flow test is being done, the shear rate obtained from the marsh cone flow test is higher than the mini slump flow test's. The marsh cone flow time test is a relatively simple test method to estimate the plastic viscosity value of grout mixture in the field applications [Şahmaran M,2008]. As it is shown in Fig. 4.5-b, an increasing of plastic viscosity increases the marsh cone flow time and the plastic viscosity obtained from grout mixtures with respect to R^2 =0.994 (Fig. 4.5-b). Therefore, a well defined demonstration of the plastic viscosity value could be estimated from the marsh cone flow time test.

There is a relation between the Lombardi plate cohesion meter test and yield stress. The cohesion can be calculated by the Lombardi plate cohesion meter test. When the plate is immersed into grout mixture in the Lombardi plate cohesion meter test, some of the grouts is caused to stick to plate. For this reason, the yield stress value is directly related with the measured value from the Lombardi plate cohesion meter test. Also, there is a correlation between the Lombardi plate cohesion meter test and the yield stress (Fig. 4.5-c). The reason can be explained that other factors such as friction and shear rate may influence the cohesion of grout.

CHAPTER 5

CONCLUSION

The addition of RHP to cement base grouts at different w/c ratios was studied in this experimantal work. The aim of the work is to investigate feasibility of use of RHP as a filler material in soil and rock grout. Following results were found from the study;

- Shear thinning behavior was observed from rheological proeperties of the grout mixtures obtained by the flow curves at the all RHP content and w/c= 0.75, 1.00 and 1.25 ratios. Also, shear thickening behavior was observed when the w/c =1.50 in all RHP percentages.
- The increase in the amount of RHP content at w/c=1.00 increases the yield stress. However, RHP has a very little affect on the yield stress at the w/c ratios greater than 1.00. The increas in w/c ratio reduces the yield stress at constant RHP content.
- Increase of RHP amount in the grout mixture increases the plastic viscosity with respect to all w/c ratio. However, the increase of w/c ratio increase the viscosity of grout for any given RHP contents.
- Increase of the RHP amount in the grout mixtures decreases the diameter of mini slump flow. On the other hand, at a constant RHP content, increase of w/c ratio increase the diameter of mini slump flow due to the water effect. Moreover, at a constant RHP content, increase of w/c ratio decrease the marsh cone flow time due to the followability effect of water. The grout mixtures including RHP reduce fluidity and deformability when w/c is particularly less than 1.00 with compared to w/c greater than 1.00.
- A good linear correlation between the mini slump flow diameter and the yield stress was obtained from grout mixtures with R²=0.85.
- There is a good linear correlation between the marsh cone flow time and the plastic viscosity obtained from grout mixtures with R²=0.99. Therefore, a well defined demonstration of the plastic viscosity value could be estimated from the marsh cone flow time test.

• There is a correlation between the Lombardi plate cohesion meter test and the yield stres obtained from grout mixtures with $R^2=0.84$.



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