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### Investigation of the Rheological Properties of Microfine Grained Cement and Normal Portland Cement with Silica Fume

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#### ABSTRACT

In this experimental study the rheological properties of microfine grained portland cement and normal portland cement grouts with or without silica fume additives were to investigated. To estimate the injectability of the cement slurry, we need to know the rheological properties of the slurry. In the study, microfine portland cement (DMFC-800), normal portland cement and normal portland cement with 10% silica fume were used. Marsh funnel, sedimentation and vicat tests are carried out to determine the rheological properties of these grouts having water/cement (w/c) ratios of 0.5, 0.75, 1.0, 1.25, 1.5, 2.0, 2.25, 2.5. Obtained results were compared with between them. It was determined that grouts prepared microfine Portland cement and Normal Portland cement (NPC) with silica fume additives have better rheological properties and penetration performance when compared with the normal portland cements.

**Keywords:** Rheological properties, Normal portland cement, Microfine grained cement, Silica fume.

### INTRODUCTION

Chemical and cement suspensions grouts are widely used as a ground improvement technique especially for granular soils all over the world. These methods involve the injection of suitable cement suspension and/or chemical grouts into soil and rock to either improve the mechanical properties or reduce the permeability. The jet grouting technique has become very popular all over the world as a practical means for solving several geotechnical problems. Sometimes, however, the inappropriate use of jet grouting has led to unsuccessful results raising relevant concern on its efficiency. Nevertheless, these failures usually stem from ignorance about the actual possibilities and limitations of the method rather than from the value of the method itself.

In many geotechnical project design, it is aimed to provide impermeability besides increase of strength. For this; it is important to inject the grouts properly into the soil. The determination of the rheological properties of these grouts is very important in terms of their practical application. The properties and behavior of microfine grained cement, also referred as superfine cement or microfine cement, grouts have been a major research subject in recent years since they are regarded as an alternative to chemical grouts to some extent. One of the main challenges in the utilization of microfine grained cement is its grain size distribution, which is quite finer than that of Normal portland cement (NPC). Microfine cement grouts have also better flow properties and bleed characteristics than NPC grouts (Zebovitz et al. 1989; De Paoli et al. 1992; Schwarz and Krizek 1994; Warner 2003).

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(Henn and Soule, 2010) compared three international and U.S. standards definitions for ultrafine cement: The International Society for Rock Mechanics (ISRM) defines it as: microfine cement is characterized by greater fineness (D95 < 16  $\mu$ m) and made of the same materials as normal portland cement. British Standart defines it as: a microfine grained cement if the specific surface area is greater than 800 m<sup>2</sup>/kg and the corresponding 95% finer (D<sub>95</sub>) particle size is smaller than 20  $\mu$ m (BS EN 12715, 2000). Ultrafine cement grouts are primarily used for grouting medium-to-fine sands where grouting is almost impossible using NPC grout. Committee 552 of the American Concrete Institute (ACI) states that the particles must be less than 15 microns.

The cost of microfine grained cement is more than that of normal portland cement. However, it is important that microfine grained cement can be used especially where chemical suspensions need to be used and that the cost is cheaper than chemical suspensions. The grain size of the fine grained cement is very small, and the properties of the grain size distribution, hydration properties and setting time may vary widely. For this reason, the properties of each microfine cement products such as grain size distribution, rheology and setting time must be very carefully investigated before use in the field of application.

The main purpose of this study is to compare the rheological properties of microfine grained cement with those of NPC. In addition, silica fume, which has a good pozzolanic character, was added to portland cement at a rate of 10% for grouts, and the rheological properties of grouts were also determined. The study was repeated for different grouts of different water / binder ratios. Thus penetrability soil of the formed grouts was tried to compared for different situations. In the study Marsh Funnel, sedimentation and vicat test were performed and the results were compared.

# MATERIALS AND METHODS

# Microfine Cement (DMFC-800)

Microfine cement (DMFC-800) is portland cement based cements composed of ultrafine particles and are designed for injection into loose soils, rock and concrete. Because of microfine cement's small particle size (the fineness largest grain size passing  $D_{95}$  is < 16.0 µm and Blaine value > 600 m<sup>2</sup>/kg), it penetrates very well into tight joints, fissures and pore spaces to provide a water-tight grouted rock or soil mass. Size distributions of Microfine cement and Normal portland cement were also shown in Figure. 1. It can be used as grouting materials after adding water reducing agent.

As seen, Microfine cement has finer particles and higher specific surface area than Normal portland cement (Figure 2). The particle size distribution of microfine cement was determined by particle sizing instrument called Mastersizer. It uses the technique of laser diffraction to measure the size of particles. It does this by measuring the intensity of light scattered as a laser beam passes through a dispersed particulate sample. This data was then analyzed to calculate the size of the particles which created the scattering pattern.

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Figure 1. Particle Size Distributions of Microfine grained cement and NPC



Figure 2. Microfine cement



Figure 3. Silica fume

# Silica fume

Silica fume has a very good pozzolanic character and contributes to many properties of the cement slurry especially when it is used together with cement. It provides many important advantages such as increasing the strength values of the used soil, gaining early strength, gaining the impermeability to the soil. Silica fume, also known as microsilica or condensed silica, supplied from Antalya Etibank Fer-rochrome Factory was used in this experimental study. The average particle size of microsilica was around 3.21µm. It is usually added into cementations suspensions by about 10% of the dry mass of cement (Figure 3).

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## Normal portland cement (NPC) (42.5R)

Portland Cement is a fine powder mixture of various minerals. It is produced by mixing calcareous minerals such as chalk, limestone containing silica and alumina and heating it to 1450 <sup>o</sup>C when it cools a clinker is formed. This clinker is then finely ground and mixed with small quantity 3-5% of gypsum which has calcium sulphate in it. The amount of gypsum controls the physical properties of the eventual concrete. Sometimes very minute quantities of pigments are also added. Cement producing processes do however vary. Size distributions of Microfine cement and Normal portland cement were also shown in Figure 1.

## **Preparation of Suspension**

The microfine cement or NPC and water were mixed thoroughly in a container by means of high-speed propeller-type mixer at 1500 rpm for about five minutes. On the other hand third mixtures, 10% silica fume by dry mass of NPC were added to the suspensions and the suspensions were mixed for two additional minutes to ensure the dispersion of solid particles in the suspension.

## Marsh funnel test

Marsh Funnel is the simplest device commonly used for the measurement of viscosity of cement grout in term of time (i.e. second) which is usually co-related with the viscosity values of various grout mixes for quality control at the site because of its simplicity and easy operation. Flow times from Marsh cone (ASTM C 939, 2003) and the viscosities from Lombardi's 1985 approach were obtained respectively devised a cohesion meter that can be used in conjunction with the Marsh funnel to determine the viscosity of the grout (Figure 4).

Accordingly, (ASTM C939, 2003):

• The temperature of the dry materials and mixing water shall be such that the temperature of the freshly mixed grout is  $23.0 \pm 1.7$  °C ( $73.4 \pm 3.0$  °F), unless otherwise specified.

• Mixed and the temperature of the injection material to the 1000ml measuring cylinder transferred.

• The mixture was required to flow through the funnel time is determined by the stopwatch.

### Sedimentation test

The stability of a grout is very important such that the lack of stability of a grout results in the particles dropping from suspension and clogging the lines, also the voids of soil mass may not be adequately filled from the bleeding of unstable grout (Figure 5). Grouts with applicable range of water / cement (w/c) ratios were placed in a 1000 ml graduated cylinder and the volume of bleed liquid on top of grout to the total volume of the suspension at the end of two hours were recorded (ASTM C 940, 2003).

### Vicat test

The setting times for all the studied mixtures were measured by vicat apparatus. A specimen of fresh cement paste was prepared with high shear mixer at  $24^{\circ}C$  (75°F) constant room temperature (Figure 6). Immediately after mixing, the paste is placed in a frustum of 40 mm

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(1.57 in.) in height. Initial set is considered as the time when the needle penetration is 25 mm  $\pm$  0.5 mm (1.53 in.  $\pm$  0.019 in.). The final set corresponds to less than 0.5 mm (0.019 in.) penetration (Wong, G.S et al.,2001). The begin and final setting times of mixtures were determined ASTM C 191, 2003.



Figure 4. Marsh funnel test



Figure 5. Sedimentation test

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Figure 6. Vicat test

# **RESULTS AND DISCUSSION**

According to the Marsh funnel tests, when the water / cement (w/c) ratio increased, the viscosities of ultrafine cement suspensions increased compared to the viscoties of the normal portland cements. While the addition of silica fume to NPC suspensions increased their viscosity, (Figure 7) (Kanat, 2018). Similar results for viscosity regarding dispersive agent effect were also reported in literature (Hakansson et al. 1992).

Up to the Figure 7, we can say that;

- The normal portland cement suspensions when the w/c=1.25 have minimum viscosity.
- Adding 10% silica fume to NPC increased the viscosity, which is most effective when the w/c ratio is 0.75, minimum viscosity is limited to around 26 seconds when the w/c ratio is 1.5 (Table 1).
- Up to Table 1 at the same water cement ratios, the viscosity of the DMFC-800 microfine cement suspension is greater than NPC and NPC + 10% silica fume suspension, with a minimum flow time is at 26 seconds when the w/c ratio of 2.0 (Kanat, 2018).
- Up to Table 2 ,accordingly to ASTM C940,for the normal cement grouts used in the w/c ratio is 0.5 sedimentation capacities smaller than 5% for 2 hours. We can indicated that microfine cement and NPC grouts with 10% silica fume; w/c ratios of 0.5, 0.75, 1.0 and 1.25 were stable. The NPC grout stable only when the w/c ratio 0.5.

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Marsh Funnel Test					
	DMFC-800	NPC	NPC + 10% Silica Fume		
W/C ratio		Time			
0.45		360			
0.55		68			
0.6		44			
0.65		40			
0.75	88	34	45		
0.8	60	32	40		
0.85	44	31	35		
0.9	38	30	32		
1	34	27	30		
1.25	29	26	28		
1.5	27	26	26		
2	26		26		
2.5	26				

### Table 1. Flow time capacities of grouts



Figure 7. The Marsh funnel test result of different grouts

Up to the Figure 8; we can say that;

- The Normal portland cement grouts with w/c ratio 0.5 were stable.
- While the addition of silica fume to normal portland cement grouts increased their stability.
- The normal portland cement with silica fume and the microfine grained cement grouts are with w/c ratios of 0.5, 0.75, 1.0 and 1.25 were stable (Kanat, 2018).

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w/ c	NPC	NPC +10% Silica fume	DMFC-800		
	Percent(%)				
0.5	4.8	0	0		
0.75	14	0	0		
1	18	0	1.2		
1.25	31	1.5	2.1		
1.5	36	8	10		
2	44	39	20		
2.25	55	43	23		
2.5		54	28		
2.75			30		
3			35		
3.5			45		
4			55.5		

Table 2.	Sedimentation	capacities	of grouts
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Figure 8. The sedimentation test results of different grouts

The begin and final setting times of NPC, NPC + 10% silica fume and microfine grained cement suspensions were given in Table 3 and Figure 9. It was seen that as the w/c ratio of grouts increased, the setting time increased too. Adding the silica fume to normal portland cement grouts increased the setting times. Test specimens were prepared for mixtures with a w /c ratio greater than 1.0. However, due to the extreme sedimentation values, experiments were not carried out according to the ASTM C 191, 2003 standard.

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Up to the Figure 9;

- Addition of the silica fume to NPC increased the setting time of NPC.
- When the same w/c ratio microfine grained cement setting fastest.
- As the w/c ratio increases, the setting time is increases too (Kanat, 2018).

w/c	DMFC-800		NPC		NPC+%10 Silica fume	
	Begin	End	Begin	End	Begin	End
0.5	230	300	410	490	470	570
1.0	300	460	720	1050	810	1200



Figure 9. Grout setting times

# CONCLUSIONS

In this study, rheological properties of the microfine grained cement (DMFC-800), normal portland cement and normal portland cement + 10% silica fume grouts were investigated. Marsh funnel, sedimentation and vicat tests are carried out to determine the rheological properties of grouts having w/c ratios of 0.5, 0.75, 1.0, 1.25, 1.5, 2.0, 2.25, and 2.5. Obtained results were compared with between them.

- It was determined that grouts prepared microfine grained cement have better rheological properties and penetration performance when compared with others.
- At the same w/c ratios, the viscosity of the DMFC-800 microfine cement grout greater than NPC and NPC + 10% silica fume, with a minimum viscosity 26 seconds when

**Table 3.** Grout setting times of the different grouts

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the w/c cement ratio of 2.0

- The microfine grained cement and NPC + 10% silica fume grouts are with w/c ratios of 0.5, 0.75, 1.0 and 1.25 were stable.
- At the same w/c ratios, the setting of the DMFC-800 microfine grained cement grout is the fast.
- As the w/c ratio increases, the setting time is increases.
- While the addition of 10% silica fume to normal portland cement grout get the same stable with DMFC-800.
- Adding 10% silica fume to NPC increase the viscosity, which is most effective when the w/c ratio is 0.75, minimum viscosity is limited to around 26 seconds when the w/c ratio is 1.5.
- Adding 10% silica fume to NPC increase the grout stable time.

The results of the study generally show that the viscosity, sedimentation and setting of the microfine grained cement DMFC-800 grout are best compared to other grouts.

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