

THE EFFECT OF DIFFERENT CURING TEMPERATURES ON THE STRENGTH OF MICROFINE CEMENT GROUTED SANDS

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In this experimental study, the effects of different curing temperatures and curing conditions on the unconfined compressive strength of microfine cement injected sand samples were investigated. The experiments started with determination of the rheological properties of the microfine cement suspensions prepared in different Water/Cement (W/C) ratios. With the increase in the W/C ratio, setting times and bleeding percentage decreased and viscosity values increased. After determination of rheological properties, injection experiments were carried out microfine cement suspensions with W/C ratios of 1.5, 2.0, 2.5, 3.0. While the injectability was increased with the increase of W/C ratio, it also decreased with the increase of the fine sand content. The samples that were successful in injection were kept in different curing temperatures and conditions. Then, the unconfined compressive strength tests were carried out on the 3rd, 7th, 14th, 28th, 56th and 120th days. The unconfined compressive strengths of the grouted sand samples were increased with time and the rate of increase started to slow down after a certain value. Due to the increase of temperature, the grouted sand samples were gained faster strength. The unconfined compressive strength values of the injected samples increased with increasing temperature. The unconfined compressive strength of the sand samples that were grouted and kept in the air-dried environment was higher than the unconfined compressive strength of the sand samples that were injected and kept in the wet-curing conditions.

Keywords: Curing condition; Curing temperature; Microfine cement; Unconfined compressive strength

1. Introduction

Injection is defined as the injection of solution or suspension materials into the voids in a soil or in a rock. There are many injection techniques that used during the practices of geotechnical engineering. The "permeation injection" is the most widely-known one amongst these techniques. Permeation injection is the process of injecting a low viscosity injection material into the voids in the soil, under low injection pressure conditions without any hydraulic fracturing, repulsion and displacement. The strength and permeability properties of the soil improve after injection [1-4].

Suspension and solution injection materials are used in permeation injection. Many injection studies have been carried out with Portland cements which are classified as suspension injection material. A certain success in injection experiments was achieved up to the coarse sands, because of the higher particle size of Portland cement. Application areas of solution type chemical injection materials stayed limited because of their low durability, toxic effects in environment and high costs. Microfine cements were found as a result of the search for materials that can be injected into the soil with smaller particle sizes. Microfine cements are defined as cements which have a particle size less than 15 μm by ACI (American Concrete Institute) [5]. Microfine cements which have different chemical and physical properties are produced by many companies in the market with many different names. Some of these are MC-100, Micromix, Microdur RX, Finosol X, Microcem B, Superfine L, Spinor A12 and Allofix MS.

Experiments for determination of rheological properties were carried out on Microfine cements by some researchers. In these experiments, it was observed that the rheological properties of Microfine cements show better properties in comparison to Portland cements [4,5-8]. Also, many studies have been conducted on the injectability of Microfine cements onto sands [1,2,9,10]. Studies have shown that the injection can be injected into fine sands with Microfine cements. In addition, engineering properties of the grouted sand samples were also investigated by the researchers. It was observed in the studies that the strength properties increased and the permeability properties decreased. [4,7,10-15]. The effects of different temperatures on the rheological properties of Microfine cements suspensions were investigated by Mirza et al. [16]. There is not any performed study on the effects of different curing temperatures and curing conditions on the engineering properties of the microfine cement grouted sands among the studies conducted so far.

In this experimental study, the effects of different curing temperatures and curing conditions on the strength values of sand samples injected with microfine cement were investigated.

2. Materials and Methods

2.1 Sand Properties

The sands to be used in injection experiments was taken from Kızılırmak River which is passing through Osmancık township in the city of Çorum. Obtained sands were divided into two groups with the use of sieves. Sieve analysis test

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Table 1

Sands to be used in injection experiments

Specimen No	Particle size content %		$\gamma_{dry(max)}$ kN/m ³	$\gamma_{dry(min)}$ kN/m ³	e_{max}	e_{min}
	Fine	Medium				
1	100	0	15.82	12.65	1.145	0.716
2	80	20	15.74	12.73	1.132	0.724
3	60	40	15.61	12.78	1.125	0.736
4	30	70	15.38	13.05	1.079	0.762

Note : $\gamma_{dry(max)}$: maximum dry density ; $\gamma_{dry(min)}$: minimum dry density ; e_{max} : maximum void ratio ; e_{min} : minimum void ratio

Table 2

Chemical and Physical properties of microfine cement

Chemical Properties	SiO ₂	30.0
	Al ₂ O ₃	9.5
	Fe ₂ O ₃	1.25
	CaO	45.0
	MgO	5.6
	SO ₃	—
Physical Properties	Composition	80%Slag+20%Portland
	D ₅₀ (μm)	1.7
	D ₉₅ (μm)	4.0
	Specific Gravity	2.94
	Fineness (cm ² /g)	11,800

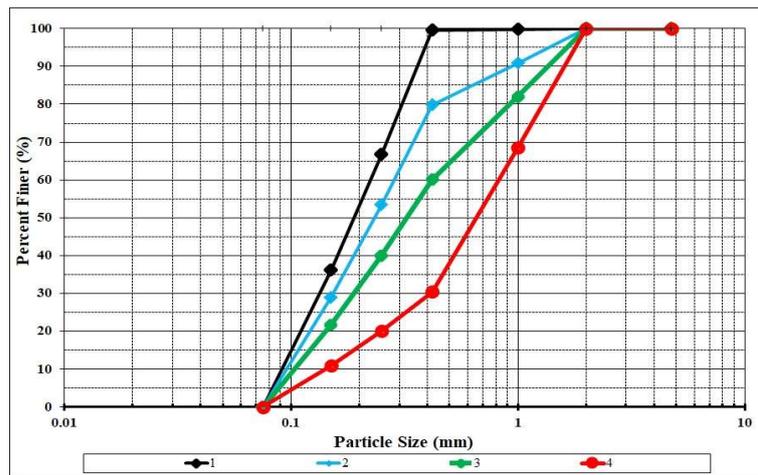


Fig. 1- Particle size distribution of sands to be used in injection experiments.



Fig. 2- Optical microscope scanning of fine and medium sands: a) fine sand, b) medium sand.

was carried out according to ASTM D6913M-17 standard [17]. The materials which passed from the Sieve 10 (2.00 mm) and remained on the Sieve 40 (0.475 mm) were classified as medium sands. Also, the materials which passed from the Sieve 40 (0.475 mm) and remained on the Sieve 200 (0.075 mm) were classified as fine sands. The fine and medium sands were mixed in different dry weight ratios as to form four different gradations to be used in the injection experiments. Mixing ratios of sands used in injection experiments were given in Table 1 and the particle size distributions were given in Fig. 1. In addition, the maximum and the minimum dry unit weights and void ratios of the sands used in the experiments were also determined [18,19]. Optical microscope scanning of fine and medium sand was shown in Fig. 2.

2.2 Properties of Microfine Cement

In this experimental study, Spinor A6 microfine cement produced by Holcim was used. D_{100} value of this microfine cement is 6 μm and its specific surface area is 11,800 cm^2/g . Some physical and chemical properties of microfine cement used in the experiments were given in Table 2. During the obtain of particle size distributions curve of the microfine cement used in the experiments particle sizing instrument tool was used. The particle size distribution of microfine cement that used in injection experiments was given in Fig. 3.

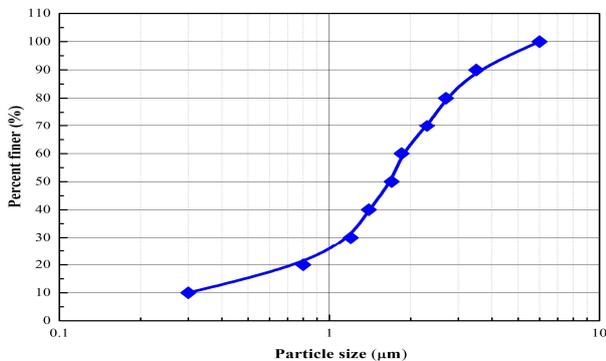


Fig. 3- Grain size distributions of Microfine cement.

2.3 Rheological Properties of Grout

The rheological properties determination tests were carried out on microfine cement injections with W/C ratios of 1.5, 2.0, 2.5 and 3.0. The experiments were carried out in $+20^\circ\text{C}$. No dispersive agent was used in microfine cement injections.

The bleeding test is based on the sedimentation of post-mixture cement grains in time and the percentage formed after sedimentation of particles in the prepared suspension. After two hours, if the volume of the water which became clear on the upper part of the mixture of cement and water is less than 5% in relative to the initial volume, then the suspension is defined as "stable". However, if the volume of water which became clear on the

1,000-mL graduated cylinder measure is greater than 5% of the initial volume of the suspension, then the suspension is defined as "not stable". The bleeding tests were performed according to ASTM C940-16 [20] standards. The bleeding tests results were given in Fig. 4.

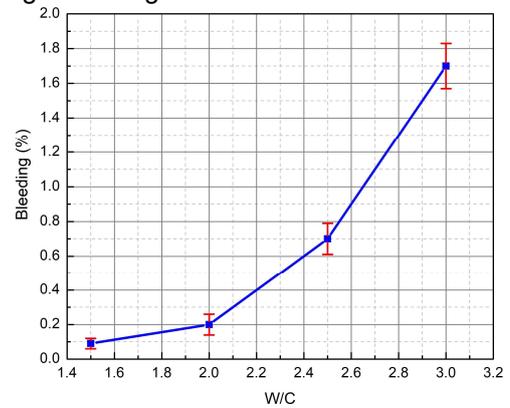


Fig. 4- Variation of bleeding percentages of microfine cement suspensions with W/C ratio.

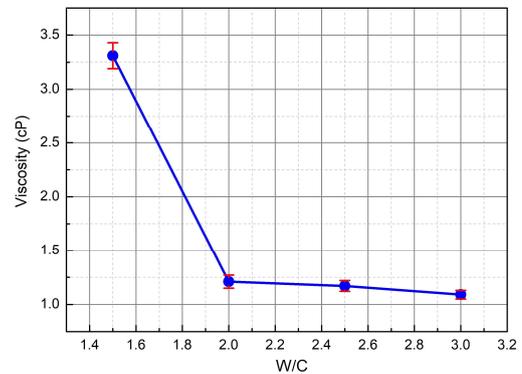


Fig. 5- Variation of viscosity of microfine cement suspensions with W/C ratio.

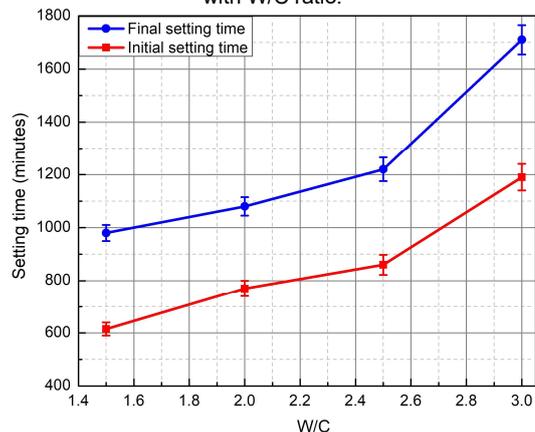


Fig. 6- Variation of setting times of microfine cement suspensions with W/C ratio.

In order to determine the fluidity properties on microfine cement suspensions which prepared in different W/C ratios, viscosity tests were performed according to ASTM D2196-18e1 [21] standards. The Brookfield DV-III Rheometer device was used to determine the viscosity values. The Brookfield DV-III Rheometer device manufactured by the Brookfield Engineering Laboratories Inc.

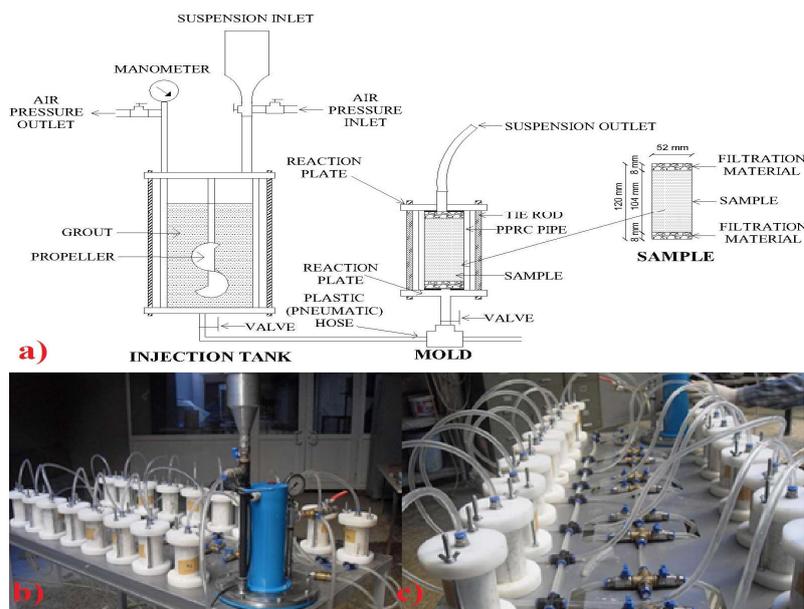


Fig. 7- Injection setup; a) schematic diagram, b), c) photos.

The shear rate was taken as 100 (1/sec.) while measuring the viscosity values. The viscosity values of microfine cement suspensions prepared in different W/C ratios were given in Fig. 5.

The Vicat Needle test were performed on microfine cement suspensions according to ASTM C191-19b [22] standards to be able to find the setting times of microfine cement suspensions prepared in different W/C ratios. The setting times of microfine cement suspensions prepared in different W/C ratios were given in Fig. 6.

2.4 Grout Tests

An injection test set-up has been made for injection experiments. The test set-up is designed by us. The photograph and schematic diagram of the injection test set-up is given in Fig. 7. Injection test set-up includes injection table, injection tank with a mixer, 80 injection molds, compressor, manometer and interconnecting elements (Fig. 7). The injection mold was made of a PPRC (Polypropylene Random Copolymer) material which has 52 mm diameter with 120 mm height. In addition, its bottom and the top caps were made of castermid material. A design vibratory hammer was used to adjust the relative density of sand mixtures which will be placed in molds. In the injection set-up, there exist a reduction gear motor that rotates with 100 rpm and pedals connected to the motor inside. In order to provide a main connection of injection material between the injection tank and the molds, dismantle (pneumatic) pipes which were easily plugged in were used. Connections of each molds to the main plastic pipe were provided with valves. The injection material coming out of the valves passes into the molds through plastic (pneumatic) pipes.

The samples were placed in the mold at 30%

relative density in 3 layers. Before the placement of sand samples in the mold, inside of the mold and the caps were lightly lubricated to ensure the release of the samples from the mold. Before the placement of sand samples in the mold, filter material consisting of 0.8 cm thick coarse sand was placed under the mold. The calculated amounts of samples were placed on the filter in 3 layers with the defined relative density and compressed with a vibratory hammer. During compression, it was measured that if the layers have reached the desired relative density or not. After sand mixtures and the filtration material consisting of coarse sand with a layer thickness of 0.8 cm were placed in the mold and the top of the mold was closed and fixed (Fig. 7). For the calculation of the amounts of fine and medium sand mixtures to be placed in the mold, the required void ratio (e_0) that was calculated with maximum and minimum void rates, was used. For the specified volume (V) and void ratio (e_0), the amount of the samples (M) that will be placed in the mold were calculated via related formulas. After the samples were placed in the molds, the molds were connected to the injection test set-up. Before injection, 0.030 MPa pressure water was given to the samples. In this way, the samples were saturated and the drainage of the water in samples was allowed up the point of any air bubbles were formed.

The microfine cement and the water were mixed in a cement mixer running at 3000 rpm for about three minutes and then this mixture was poured into the injection tank. Then, the reduction gear motor in the injection tank was operated at 130 rpm, to be able to prevent any sedimentation in the suspension during the process, until the injection was completed. The injection experiments

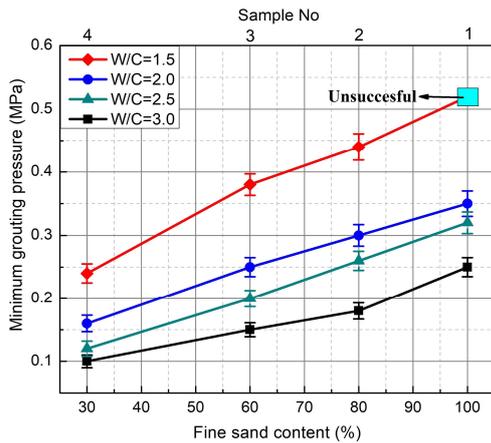


Fig. 8- The variation of minimum injection pressure values with the percentage of fine sand.

were carried out by putting pressure on the injection tank via compressor. The results of the injection experiment were given in Fig. 8. The injection pressures for the samples were carried out on a trial basis, and injectability limit pressures were determined based on these trials.

2.5 Unconfined Compressive Strength Tests

The samples that were successful in the injection experiments were taken into the curing rooms where they will be kept during curing process after injection. After setting time was completed, the samples were removed from the molds and kept in the curing environment until the unconfined compressive strength tests were performed. Samples were kept in three different

curing temperatures as 10°C, 20°C and 30°C and two different curing conditions as the air-dried and the wet-cured. Some of the injected sand samples extracted from the molds were kept in the open air in the laboratory, while the others were kept in the water tank until the test time. In this way two different conditions were represented. One of them represents the excavation faces exposed to open air and the other represents the grounds under groundwater. During the curing period, the temperature setting of the curing room was provided with Digital Fan Coil Room Thermostat. The temperature setting of the curing tank during was provided by a thermostat control heater and a water circulating pump. The unconfined compressive strength tests were carried out on the samples that became successful in injection and kept in curing environment at their 3th, 7th, 14th, 28th, 56th and 120th days according to the standards specified in ASTM D4219-08 [23]. The unconfined compressive strength tests were performed in the unconfined compressive strength test device manufactured by Ele International Company. Before the UCS tests, the granular filters placed at the top and the bottom of grouted specimens were trimmed by a diamond blade saw. Then, these filters were capped with the plaster of Paris. The unconfined compressive strength tests were repeated three times for each curing times and curing conditions and the mean values were calculated. The unconfined compressive strength test results of the samples kept in the air-dried and the wet-cured were given in Fig. 9, Fig. 10 and Fig. 11.

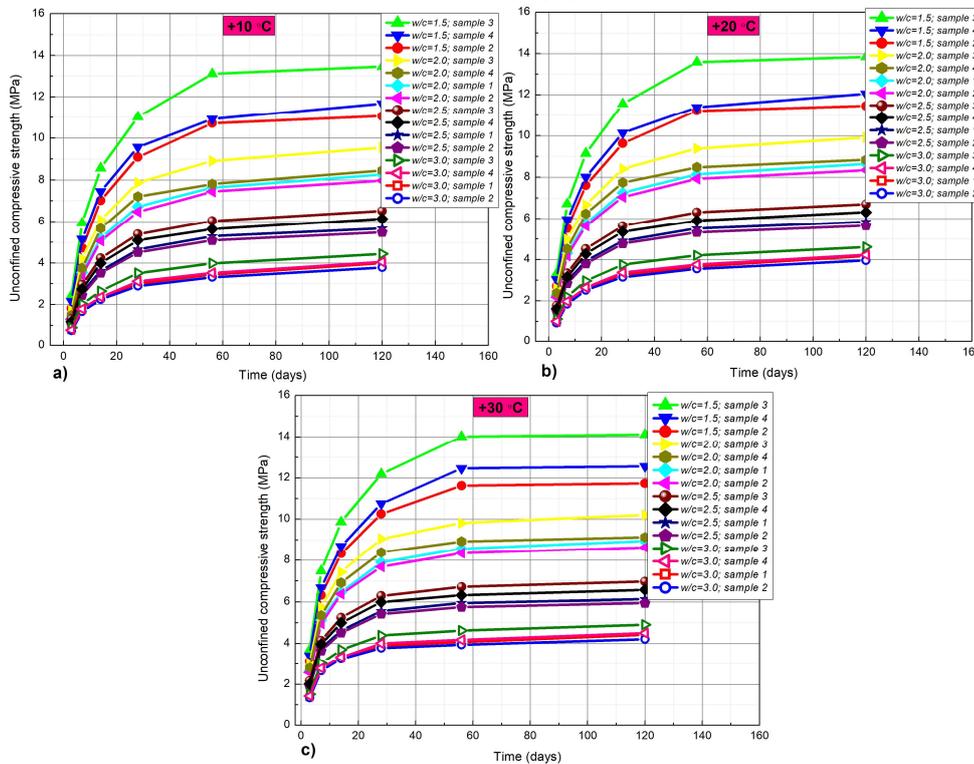


Fig. 9- Variation of the unconfined compressive strength of the samples kept in the wet-cured with curing time.

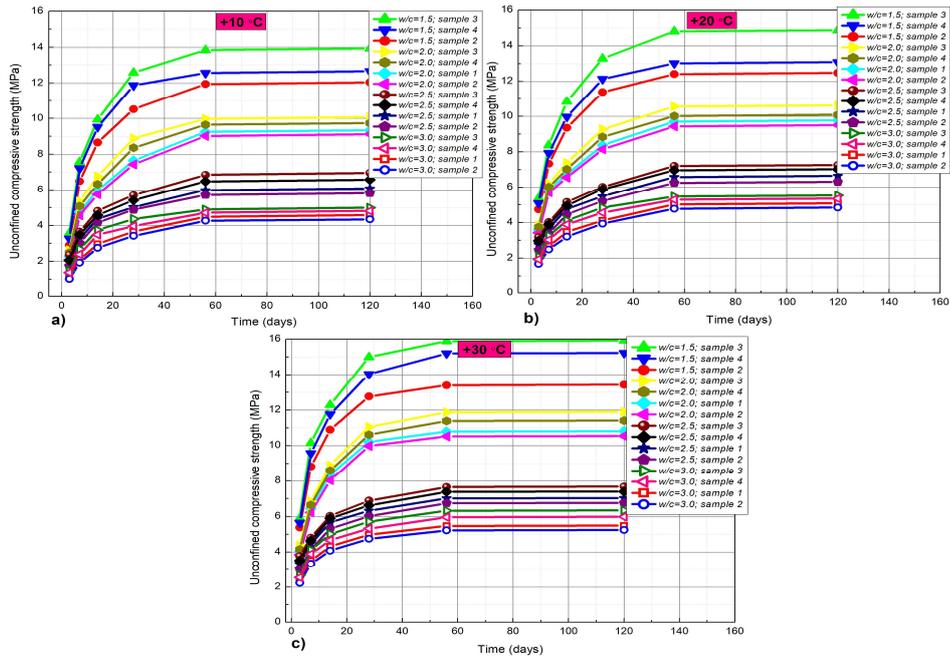


Fig. 10- Variation of the unconfined compressive strength of the samples kept in the air-dried with curing time.

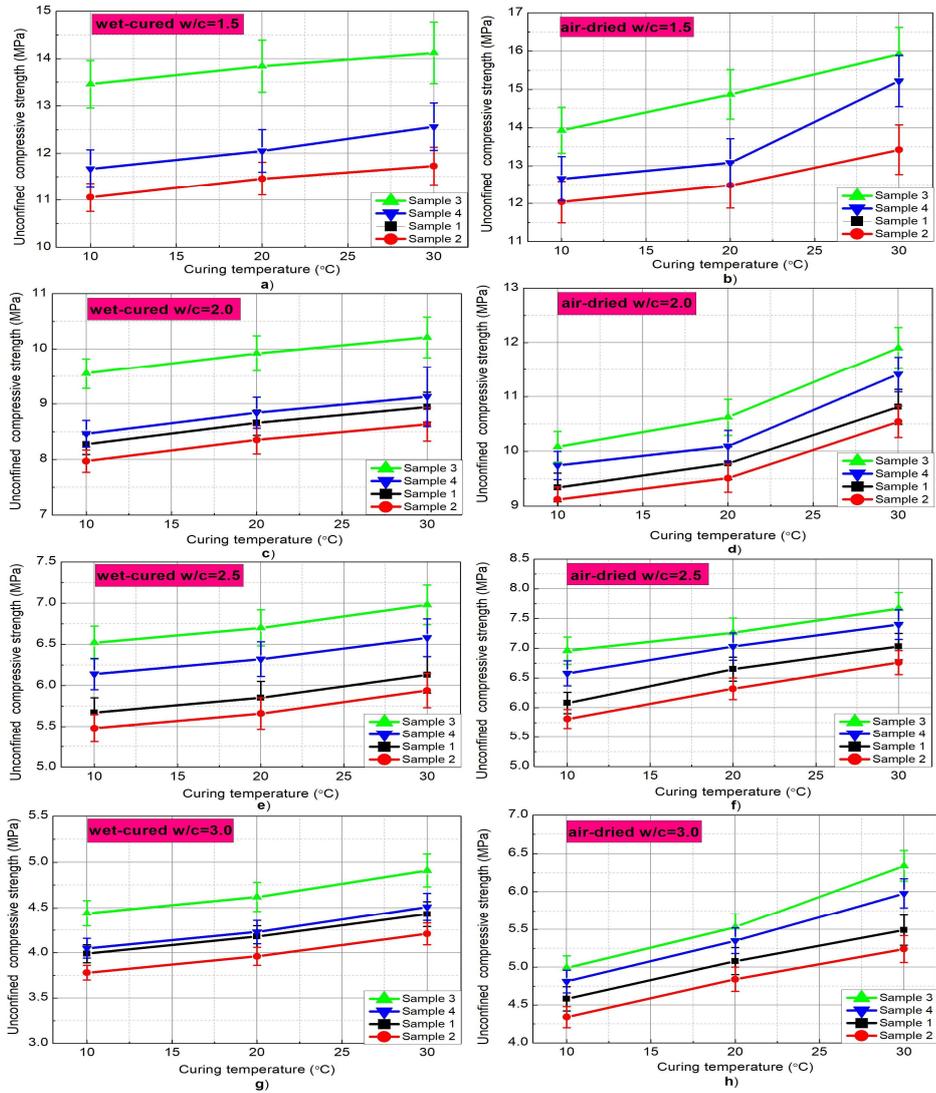


Fig. 11- Variations of the unconfined compressive strength .

3. Results and Discussion

3.1 Bleeding, Viscosity and Setting times of Grouts

Fig. 4. It is seen that bleeding percentages of all samples were stable and below 5%. Bleeding percentages of microfine cement suspensions for the W/C ratios of 1.5, 2.0, 2.5, 3 were 0.09, 0.2, 0.7 and 1.7 in order. The bleeding percentages were increased with the increase of W/C ratios.

As a result of viscosity tests, viscosity values of the microfine cement suspensions were reached to 3.31, 1.21, 1.17 and 1.09 cP in order, for the W/C ratios of 1.5, 2.0, 2.5 and 3.0. (Fig. 5). The increase of W/C ratio decreased the viscosity values (Fig. 5).

The initial and the final setting times of microfine cement suspensions that prepared in different W/C ratios were determined. Initial setting times for W/C ratios 1.5, 2.0, 2.5 and 3.0 were calculated as 615 min, 770 min, 860 min and 1190 min, respectively. In addition, final setting times were calculated as 615 minutes, 770 minutes, 860 minutes and 1190 minutes, respectively. With the increase in the W / C ratio, the first and final setting times increased (Fig. 6).

3.2 Groutability

In the injection experiments performed with microfine cement suspension, the success was provided in all samples except for the sample 1 in the sand samples injected with W/C ratio 1.5. The penetration of microfine cement suspensions with W/C ratio of 2.0, 2.5, and 3.0 were successful for all samples. The minimum injection pressure values for injections with a W / C ratio of 1.5 were 0.44, 0.38 and 0.24 MPa for 2, 3 and 4 samples, respectively (Fig. 8). In addition, for injections with W/C ratio of 2.0, the minimum Injection pressure values for 1, 2, 3 and 4 samples were 0.44, 0.38 and 0.24 MPa, respectively. In injections with a W/C ratio of 2.5, the minimum injection pressures were 0.32, 0.26, 0.20 and 0.12 MPa, respectively, while in injections with a W/C ratio of 3.0, the minimum injection pressures were 0.25, 0.18, 0.15 and 0.10 MPa respectively. Injectability increased with the increase of W/C ratio, and decreased with the increase of fine sand content. (Fig. 8).

3.3 The Unconfined Compressive Strength of Grouts

The unconfined compressive strength tests were carried out on the 3rd, 7th, 14th, 28th, 56th and 120th days of the samples injected at different W/C ratios and kept under different curing conditions.

The unconfined compressive strengths of the samples which were injected with the W/C ratio of 1.5 and kept in +10°C wet-cured environment, were resulted as 11.06, 13.46 and 11.67 MPa in order at the end of the 120th day, for the samples

2, 3 and 4. The unconfined compressive strengths of the samples kept in 20 °C wet-cured environment were 11.46, 13.84 and 12.05 MPa for samples 2, 3 and 4, respectively. In addition, the unconfined compressive strengths of the samples kept in a wet-cured environment at +30 °C were 11.73, 14.12 and 12.56 MPa for samples 2, 3 and 4, respectively (Figs. 9 and 11).

The wet-cured strength of the specimens grouted with W/C 2.0 and stored at +10 °C were 8.27, 7.97, 9.55 and 8.46 MPa for specimens 1, 2, 3, and 4 at the end of 120 days, respectively. The wet-cured strength of the specimens kept at +20°C were 8.66, 8.35, 9.92 and 8.94 MPa for specimens 1, 2, 3, and 4, respectively. The wet-cured strength of the samples stored at +30 °C were 8.94, 8.63, 10.21 and 9.13 MPa for samples 1, 2, 3, and 4 at the end of 120 days, respectively (Figs. 9 and 11).

The unconfined compressive strength values of wet-cured specimens injected with W/C 2.5 and kept at +10°C at the end of the 120th day of curing period were 5.67, 5.48, 6.52 and 6.14 MPa for samples 1, 2, 3 and 4, respectively. The wet-cured strength of the samples kept at +20°C were 5.85, 5.66, 6.70 and 6.32 MPa for the samples 1, 2, 3 and 4, respectively. The unconfined compressive strength values of the samples which were kept in +30°C wet-cured environment were resulted as 6.13, 5.94, 6.98 and 6.58 MPa in order at the end of the 120th day, for the samples 1, 2, 3 and 4 (Figs. 9 and 11).

The unconfined compressive strength values of the sand samples grouted with W/C ratio 3.0 and kept at +10°C under wet-cured conditions ranged from 3.99 to 4.05 MPa at the end of the 120th day. In addition, the wet-cured strength of the samples which were kept in +20°C varies between 4.18 to 4.23 MPa and the wet-cured strength of the samples which were kept in +30° varies between 4.43 and 4.51 MPa (Figs. 9 and 11).

The unconfined compressive strength of the specimens 2, 3 and 4 which were injected with the W/C ratio 1.5 and kept in +10°C air-dried condition for 120 days were resulted as 12.04, 13.93 and 12.66 MPa in order. For the samples kept in +20 °C, it resulted as 12.48, 14.87 and 13.08 MPa in order. For the samples kept in +30°C, it resulted as 13.42, 15.92 and 15.22 MPa in order (Figs. 10 and 11).

The air-dried strength of injected sand samples with W/C ratio 2.0 at the end of the 120th day were 9.34, 9.12, 10.08 and 9.74 MPa for the samples 1, 2, 3 and 4 in the order. The air-dried strength values were 9.78, 9.51, 10.62 and 10.09 MPa for samples kept at +20 °C, and 10.81, 10.54, 11.90 and 11.41 MPa for the samples kept at +30 °C, respectively (Figs. 10 and 11).

The air-dried strength of grouted sands with W/C ratio 2.5 varies between 6.08 and 6.58 MPa for samples kept at +10°C and varies between 6.65 and 7.03 MPa for samples kept at +20 °C at

the end of the 120th day. In addition, the air-dried strength of the samples kept at +30°C varies between 7.03 and 7.40 MPa (Figs. 10 and 11).

The air-dried strength of the specimens 1, 2, 3 and 4 which were grouted with W/C ratio 3.0 and stored at +10°C were 4.58, 4.34, 4.99 and 4.81 MPa, respectively. In addition, the air-dried strength of the grouted specimens 1, 2, 3 and 4 kept at 20 °C and +30°C were 5.08, 4.84, 5.53 and 5.35 MPa, and 5.49, 5.24, 6.34 and 5.98 MPa, respectively (Figs. 10 and 11).

The unconfined compressive strength of wet-cured and air-dried samples were also time-dependent. The unconfined compressive strength of the samples which were injected and kept in the wet-cured condition were increased due to time. This increase slowed down after the 56th day for the samples kept at +10°C, after the 40th day for the samples kept at +20°C and after the 28th day for the samples kept at +30°C. At the end of the 28th day, the grouted samples kept at +10°C, +20°C and +30°C reached 80%, 83% and 89% of their ultimate unconfined compressive strength, respectively (Fig. 9).

The air-dried strength of the injected sand samples also increased swiftly with time. This increase slowed down after the 28th, 14th and 10th day in the samples kept at +10°C, +20°C and +30°C, respectively. The grouted samples that were kept at +10°C, +20°C and +30°C reached 84, 87 and 92 percent of their ultimate unconfined compressive strength on the 28th day, respectively (Fig. 10).

Due to increase of the temperature, the unconfined compressive strength values of the injected samples were also increased (Fig. 10). While this increase was 3% between +10°C and 20°C in the samples kept in the wet-cured conditions, also it is 4% between +20°C and 30°C. For the samples kept in the air-dried conditions, the increase rate was 7% between +10°C and 20°C and 10% between +20°C and 30°C. With the increase of temperature, hydration was increased and the samples injected with microfine cement were gained faster strength. The increase in unconfined compressive strength was owing to the matric suction which usually seen in unsaturated soils. The process of drying resulted with the loss of moisture content which set up additional tension forces among the particles in order. As a result, the friction between particles was raised (Fig. 11).

The unconfined compressive strength values of the samples that were injected and kept in the air dried conditions was higher than the unconfined compressive strength values of the samples that were kept in the wet-cured conditions. This increase was 10% for the samples kept in 10°C, 14% for the samples kept in +20°C and 20% for the samples kept in +30°C. The increase in

unconfined compressive strength values occurred because more water was lost from the injections through drying (desiccation), the injection matrix shrank. Shrinkage of the injection material between soil grains resulted in the occurrence of forces that were similar to capillary tensile forces, but generally quite stronger. This led to an increase in the resistance to relative motion between grains, and the soil mass gained additional strength (Figs. 9, 10 and 11).

In the injected samples, gradation of the sand samples affects the unconfined compressive strength values. While the highest unconfined compressive strength values in the injected sand samples were seen in the sample 3, the lowest unconfined compressive strength values were seen in the sample 2 (Figs. 9, 10 and 11).

The W/C ratio was also affected the unconfined compressive strength in injected samples. The unconfined compressive strength values of the injected samples were increased with the increase of W/C ratio (Figs. 9, 10 and 11).

4. Conclusions

The main conclusions can be extracted from this study are:

- With the increase in the W/C ratio, setting times and bleeding percentage decreased and viscosity values increased.
- While the injectability was increased with the increase of W/C ratio, it also decreased with the increase of the percentage of fine sand content.
- The unconfined compressive strength of the sand samples which were injected with microfine cement and kept in both conditions of the curing (air-dried and wet-cured) was increased depending on time and the rate of increase started to slow down after a certain value.
- Due to the increase of temperature, the samples injected with microfine cement were gained strength faster.
- The unconfined compressive strengths increased with the increase of temperature in the injected samples.
- The unconfined compressive strength of the sand samples that were injected and kept in the air-dried gave higher results than the unconfined compressive strength of the samples that were kept in the wet-cured.

The experimental study has shown that both the curing temperature and the curing conditions affect the strength values of microfine cement grouted sands.

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