



## Effect of Waste Marble Powder and Fly Ash on the Rheological Characteristics of Cement Based Grout

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### Abstract

This paper shows the results of an experimental study conducted to research the impact of waste marble powder (WMP) and fly ash (FA) on the fluidity (marsh cone flow time (MCFT), mini-slump diameter and plate cohesion meter (PCM)) and the rheological properties (viscosity and yield stress) of cement based grout (CBG) mixtures. The experiments were applied with the CBG mix including 1.00 water-binder (w/b) ratios and combined use of WMP (5-25%), FA (5-25%) and WMP+FA (10-30% + constant 25%). Test results illustrated that the rheological properties of the CBGs importantly have been improved by the addition of WMP, FA and WMP+FA to grout mix at w/b=1 ratio. Strongly shear thickening behaviour was got from the CBG mixtures the all WMP, FA and WMP+FA content. The increase in the percentage of WMP (5-25%) amount reduced the mini-slump flow diameter. Moreover, the increment in the percentage of FA (5-25%) amount increases the mini slum flow. According to the control sample, the increment in the percentage of WMP (10-25%) amount increased the mini-slump flow diameter for constant FA (25%) content. Also, the increase in WMP amount in the CBG mix, there is no effect on MCFT. Also, MCFT decreased with the increase of FA amount. Especially, FA increased the fluidity of CBG, when the WMP showed negative effect in increase the MCFT in WMP+FA content.

*Keywords:* Waste Marble Powder; Fly Ash; Grout; Rheology; Fresh Properties; Waste Materials.

### 1. Introduction

Cement Based Grout (CBG) is a widely used method for many applications in the geotechnical area [1]. Some examples of CBG applications are suspension grouting, emulsion grouting, solution grouting, compaction grouting, permeation grouting, displacement grouting and replacement grouting [2-5]. The rheological and permeability properties of the CBG are straightly involved with the penetrability and pumpability in cracks and soil voids.

CBG is mixed of water, cement and admixture. For CBG mix design, different range of water-cement (w/c) ratio can be utilized. For the applications of permeation grout, w/c ratio of CBG range between 0.5 and 1 [6]. The w/c rate of injectable grout ranges from 1 to 2. Also, the grout should be similar to the liquid that can be injected into the rock and soil [4]. CBG mixes have usually water-cement ratios of 1.00 by volume.

Mineral and chemical admixtures are used to develop the properties of CBG like durability, permeability, rheological and fresh properties. Adding mineral admixtures to the CBG at different amount modify the rheological and fresh properties of injections. For various types of grout applications, various additions (Cement kiln dust, silica fume, rice husk ash, metakaolin and bentonite) have been applied. [7-9].

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Some recent studies showed that additive, like rice husk ash, have increased the durability, long-term performance and workability of CBG mix [10-11]. Consequently, the additives usage in CBG applications reduce the charge of the application but increment the flowability and the strength. Also, Singh et al. (2019), Khan et al. (2019) and Singh et al. (2017) investigated the use of Portland cement with FA and WMP in concrete. They pointed out that the binary composition of the binders provided economic and environmental advantages by reducing Portland cement (PC) production, thus CO<sub>2</sub> emission [12-14].

Marble is a very important material for construction, particularly for decoration reasons. %25 percent of marble turns to powder and dust due to shaping, polishing, and sawing. The wastes of the marble industry have become a major environmental problem since the beginning of the 2000s. This issue is not just a local problem, and it is of direct concern to dozens of countries. While many countries, such as Turkey, Iran, Italy, China, India, Spain, Brazil, South Africa, Portugal, Pakistan, USA, Egypt and Finland, export the marble products, many other countries, such as Japan, Germany, Taiwan and South Korea, import them [15]. Turkey owns 40% of the world's marble reserves, and storing the waste released during production is of utmost importance. Turkey has nearly  $3.872 * 10^6$  m<sup>3</sup> of marble reserves, of which close to  $125 * 10^3$  t/year are generated in Afyon City alone [12]. Five thousand processing factories are used for marble production in Turkey. It is clear that these waste products reach millions of tons; so it is too difficult to store this amount of wastes. [16, 17]

FA is a divided siliceous tailing from the burning up of powdered coal and can be used an admixture. FA has the same fineness like PC. It can also react easily chemically with cement. Also, the average annual FA production in Turkey has about 13 million tons and half of it was utilized.

The disposal of WMP and FA are one of the most environmental concerns all over the world nowadays. On the other hand, WMP and FA can be used to increase the fresh properties of cementitious grout.

WMP is used like a complementary cement based material in the concrete. Earlier researches concluded that maximum 10-15 percent of WMP can be conveniently mixed into the concrete without negatively impacting the durability, hardness and strength of the resulting concrete [18-20]. Another researches investigated the usage of WMP as a cement substitution. Also, Study showed that combining of WMP in self-compacting concrete (SCC) with concrete reduce the splitting tensile strength, compressive strength, bulk weight, slump flow, ultrasonic pulse velocity, porosity and cost [20].

Usually, the percentage of FA usage is 10-35% in concrete [21]. The usage of FA in concretes and SCC have also been investigated and have been well developed in the last decade [22]. On the other hand, there is limited knowledge available concerning the fresh characteristics of cement based grout containing FA. The replacement of Portland Cement with FA is important for decreasing the viscosity, increasing passing ability and flowability of cement based grout materials [23].

The aim of this study is to show the potential usage of WMP and FA in preparing the grout mixture and to observe the effects of WMP and FA on the workability, fluidity, and rheological properties of CBG. The experiments were performed on 16 grout mixes including different amounts of WMP, FA and WMP+FA with the percentages of 5-25% (WMP), 5-25% (FA) and 10-30% + constant 25% (WMP+FA) of the total cementation materials weight and with 1.00 ratio of w/c. A series of workability and rheology tests including Lombardi Plate cohesion test, Marsh cone flow time (MCFT) test, mini-slump flow diameter test and a coaxial rotating cylinder rheometer were conducted to observe the fresh properties of CBG mixtures.

## 2. Materials and Methods

### 2.1. Materials

In this research Portland cement (CEM I-42.5R) was used complying with ASTM C150 Type-I cement. Class F FA was used an additive in CBG according to ASTM C 618. Also, WMP was used as an additive. Waste marble sludge (WMS) was provided in the form of a wet slurry from marble factory in Gaziantep-Turkey. The WMS was dried in an oven at  $100 \pm 10$  ° C for 24 hours and then sieved with 150 µm sieve Figure 1. Table 1 demonstrates the chemical and physical properties of PC, WMP and FA. Moreover, particle size distributions of PC, WMP and FA are presented in Figure 2.



Figure 1. Waste marble powder, cement, fly ash

Table 1. The chemical and physical properties of PC, WMP and FA

Chemical Composition (%)	Portland Cement (PC)	Waste Marble Powder (WMP)	Fly Ash(FA)
SiO <sub>2</sub>	20.27	3.86	62.35
Al <sub>2</sub> O <sub>3</sub>	5.32	4.62	21.14
Fe <sub>2</sub> O <sub>3</sub>	3.56	0.78	7.35
CaO	60.41	28.63	1.57
MgO	2.46	16.9	2.35
SO <sub>3</sub>	3.17	–	0.10
Loss on ignition	3.55	43.3	2.07
Physical Properties			
Specific gravity	3.15	2.71	2.30
Specific surface (Blaine) (cm <sup>2</sup> /g)	3030	4190	3870

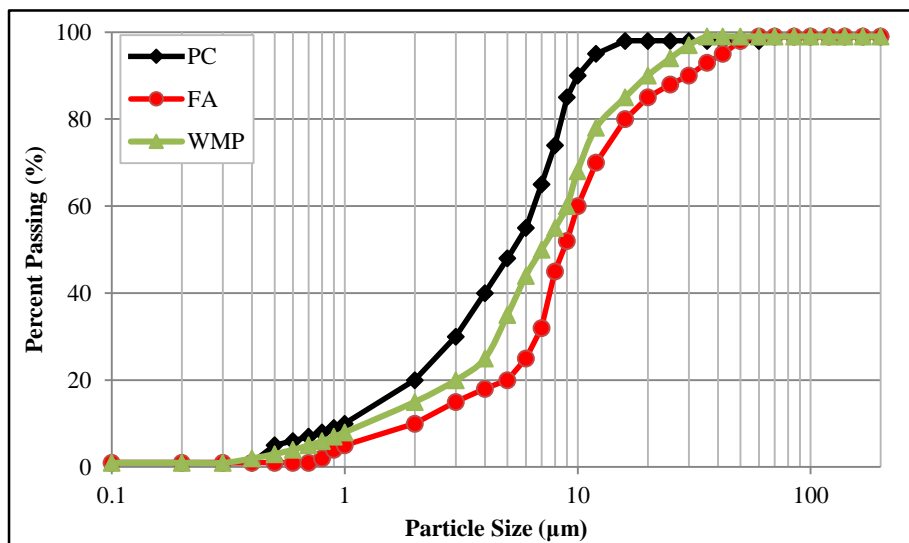


Figure 2. Particle size distributions of PC, WMP and FA

**2.2. Methods**

Water-binder (w/b) ratio is one of the important factors that has a prominent impact on the fresh and hardened characteristics of CBG mixes. So, in this experimental work w/b= 1.00 rate were chosen to research the effect of WMP, FA and WMP+FA on the CBG. 16 injection mixtures with various WMP, FA and WMP+FA amount were prepared to the impact of WMP, FA on the characteristics of CBG mixtures. (Table 2). WMP, FA and WMP+FA was replace with cement substitutions of 5-25%, 5-25% and 10-30% + constant 25% by volume. The control mix was prepared with only PC and water at w/b=1.00 rate. WMP and FA weren't added to the control mix. The mixture proportions are illustrated in Tables 2.

Table 2. WMP, FA mix amount for 1.5 L and fluidity characteristics of the CBG mixtures

Mix No.	Mix ID	Mix System	CEMENT (kg/m <sup>3</sup> )	WMP (kg/m <sup>3</sup> )	FA (kg/m <sup>3</sup> )	WATER (kg/m <sup>3</sup> )	w/b	FA (%)	WMP (%)	Mini Slump (mm)	Marsh Cone (s)	Plate Cohesion (mm)
M1	WMP0FA0	Control	1131	0	0	1131	1	0	0	202,5	27	3,08
M2	WMP5FA0	Binary	1072	0	56	1129	1	0	5	201,5	26,88	3,35
M3	WMP10FA0	Binary	1014	0	113	1127	1	0	10	204	26,91	3,3
M4	WMP15FA0	Binary	956	0	169	1124	1	0	15	205	26,54	3,31
M5	WMP20FA0	Binary	898	0	224	1122	1	0	20	199	26,89	3,67
M6	WMP25FA0	Binary	840	0	280	1120	1	0	25	201,5	27	4,37
M7	FA5WMP0	Binary	1069	56	0	1126	1	5	0	215	27,19	3,56
M8	FA10WMP0	Binary	1008	112	0	1120	1	10	0	218	27,12	3,33
M9	FA15WMP0	Binary	948	167	0	1115	1	15	0	218	27,72	2,92
M10	FA20WMP0	Binary	888	222	0	1110	1	20	0	219	27,22	2,89
M11	FA25WMP0	Binary	828	276	0	1104	1	25	0	220	27,03	2,78
M12	WMP10FA25	Ternary	715	275	110	1100	1	25	10	218	26,5	2,8
M13	WMP15FA25	Ternary	659	274,5	165	1098	1	25	15	218	26,6	3,15
M14	WMP20FA25	Ternary	603	274	219	1096	1	25	20	219	26,8	3,22
M15	WMP25FA25	Ternary	547	273,5	273	1094	1	25	25	211	26,8	3,04
M16	WMP30FA25	Ternary	491	273	328	1092	1	25	30	211	27,3	3,09

In preparation for the grout mixtures, five-liter laboratory mixer was used (Figure 3). The mixing step was applied in the experiment as following; WMP, FA, cement and water were mixed at slow speed (120 rpm) for 1 min. The mixer was paused and the remaining grout mixture on the sides of the container was stripped. Also, grout mixes was blended by hand for one minute. Lastly, the mixer was restarted at high speed (240 rpm) and the grout mixture mixed for three minutes. Temperature and humidity of the laboratory for all tests were checked and measured as 50-60% and 20±3 respectively. All CBG mixtures were obtained by using the same mixing procedures.



Figure 3. Laboratory mixer

Rotational viscometer (rheometer) was used to find the yield stress and the plastic viscosity (proRheo R180 Instrument, Germany) at  $20 \pm 3$  °C (Figure 4). The viscosity of the grout can be measured at various rotational speeds. The rheometer determines viscosity according to the searle-principle; The proRheo R180 is a standard rotational model viscometer which uses a motor-driven bob turning in a fixed measuring tube. The specimen is sheared in the gap between the bob the tube and the measured shear stress is used with the shear rate to compute the viscosity [24].



Figure 4. Rotational viscometer (Rheometer)

Ascending and descending flow curves in the shear stress–shear rate curve were gotten. The shear rates were varied between from 50 to 1000 s<sup>-1</sup> for every CBG mixture. The apparent viscosity is regarded as a function of the shear rate; therefore, the shear-thickening (dilatant) behaviour of the CBG mixes is obtained with according to the apparent viscosity of GBG [25].

To find the rheological properties of CBG, several types of analytical models exist. Plastic viscosity and yield stress are got by matching shear stress–shear rate curve values into Modified Bingham model (Equation 1)  $\tau_0$  is yielding stress and  $\mu_p$  is plastic viscosity.

$$\tau = \tau_0 + \mu_p \times \gamma + c \times \gamma^2 \tag{1}$$

The modified Bingham model gives a preferable solution than the Bingham model (Equation 2) for the same mixes [25].

$$\tau = \tau_0 + \mu_p \times \gamma \tag{2}$$

Figure 5 demonstrates the shear stress–shear rate curve values of MP5FA0 grout mixture by using both modified Bingham and Bingham model.

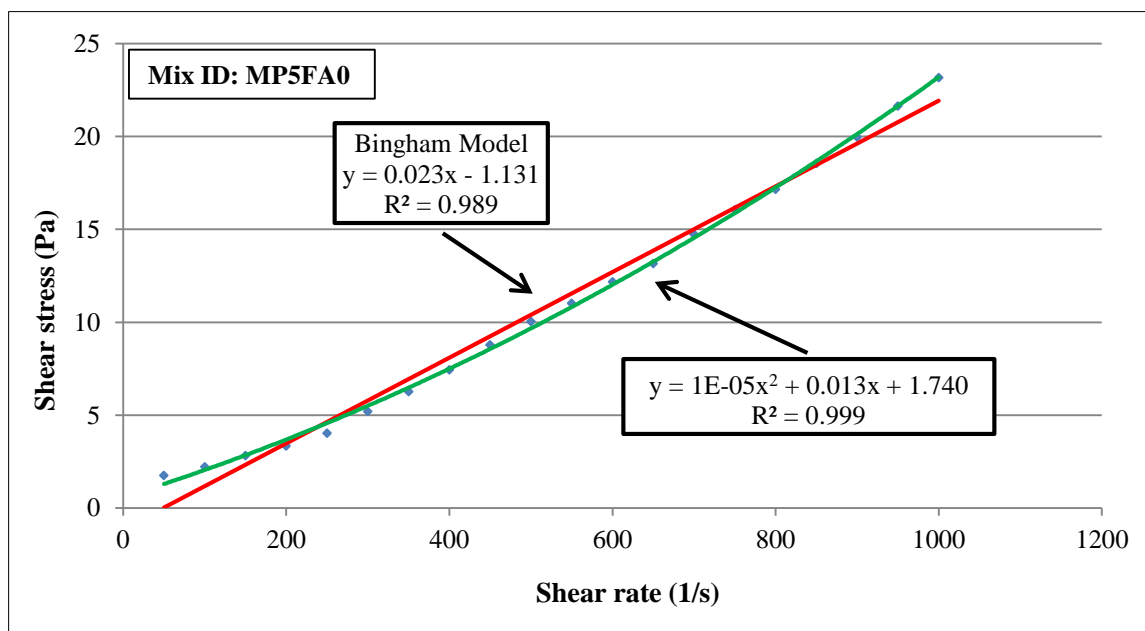


Figure 5. The characteristic flow properties of CBG obtained from Modified Bingham and Bingham Model

The mini-slump test, MCFT and PCM test were used for evaluating the fluidity or workability of fresh grout mixtures. These tests were very easy to identify the flowability and workability characteristics of the grout prepared in the construction area.



The mini-slump test was used to find the spread of grout mixtures. The apparatus shape is same to the slump cone described by ASTM C-143 [21]. Dimensions of mini-slump apparatus; 38 mm high with at the top diameters of 57 mm and 19 mm at the bottom (Figure 6) [25-27]. In the mini slump test, CBG mix is poured into a cone until it's full. Then, the mixture is allowed to spread by removing the mini slump cone. It is transfer to on a flat glass plate. In the vertical direction the spread diameters are measured and the average spread diameter is calculated.

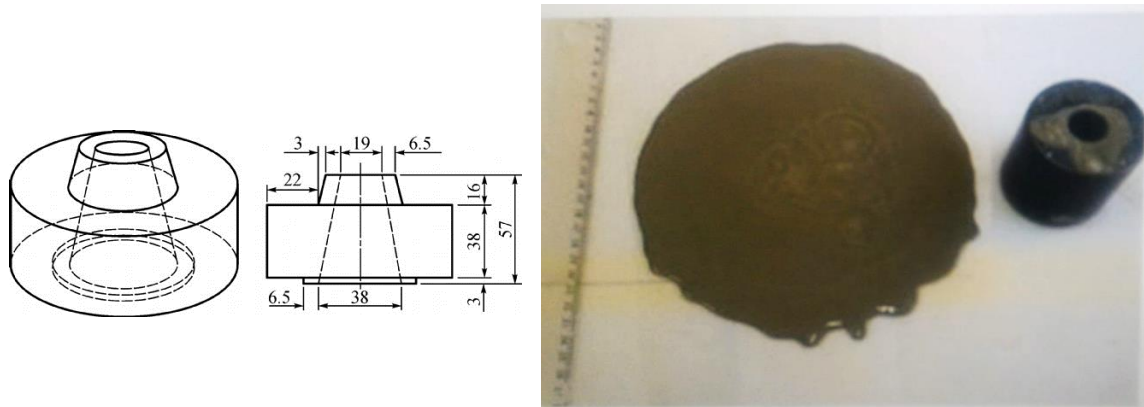


Figure 6. Mini-slump test apparatus

The MCFT is a flowability test used for the properties and quality check of CBG and cement mortars which developed by Bartos et al., [28]. The volume of this device has 1500 ml and 5 mm inner cap (Figure 7). The MCFT is used to detect the CBG volume through flow cone at a measured time. The CBG mix was proud in a cone (1250 ml) and bottom outlet was turned on. After that, CBG mix begins to flow and elapsed time was measured providing that 1000 ml of CBG had flowed. Consequently, the elapsed of time gave the MCFT time. The MCFT time of water was found as 24 s in comparison with cement based grout.



Figure 7. Marsh cone flow test apparatus

Lombardi PCM was used to find the cohesion. PCM consists of a steel plate with rough surfaces on both sides. 10×10×3 mm is a dimension of the PCM (Figure 8). PCM apparatus was used for calculating the cohesion. The plate was immersed in the CBG mixture. Due to cohesion, the CBG mixture sticks on the plate. After that, the cohesion measured from the control mix was compared with that measured from the CBG mixes [29-30]. All tests were done two times by preparing a new mixture for control purposes. The test results were similar and were not indicated in the tables. All tests were made at 8-12 minutes after contact with cement and water.

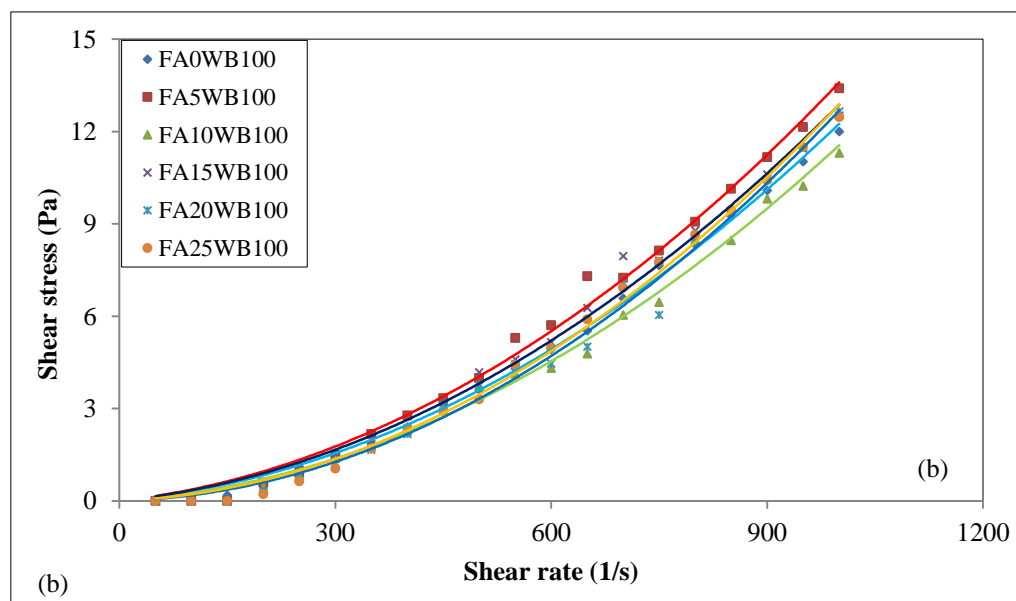
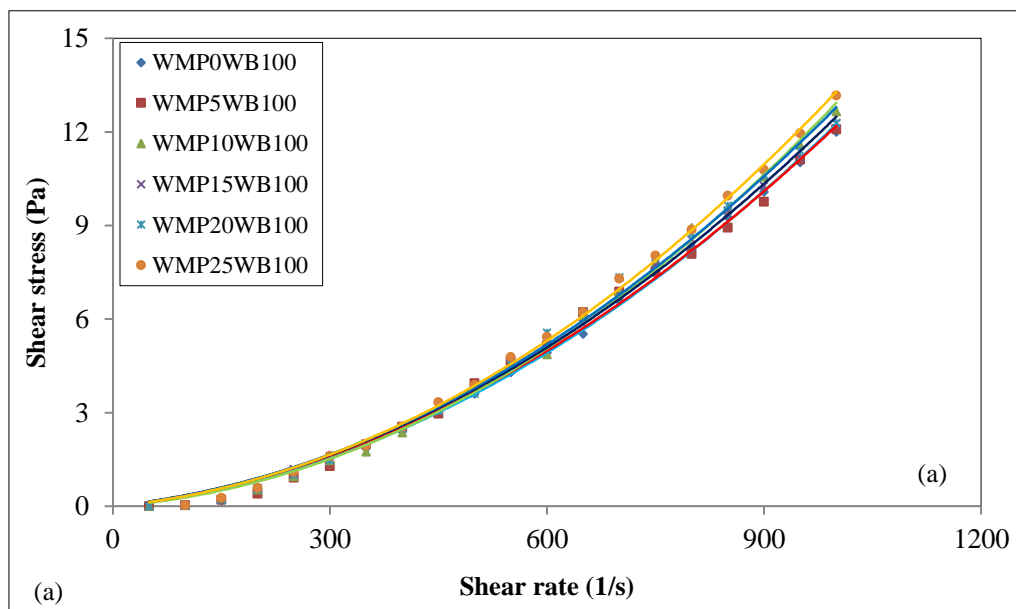


Figure 8. Plate cohesion test apparatus

### 3. Results and Discussions

#### 3.1. Rheological Properties of CBG

The influence WMP and FA content on the rheological properties of CBG mixture can be related with between the shear rate and the shear stress. The flow curves of the CBG mixes containing WMP, FA and WMP+FA with w/b = 1 rate are shown in Figure 9.



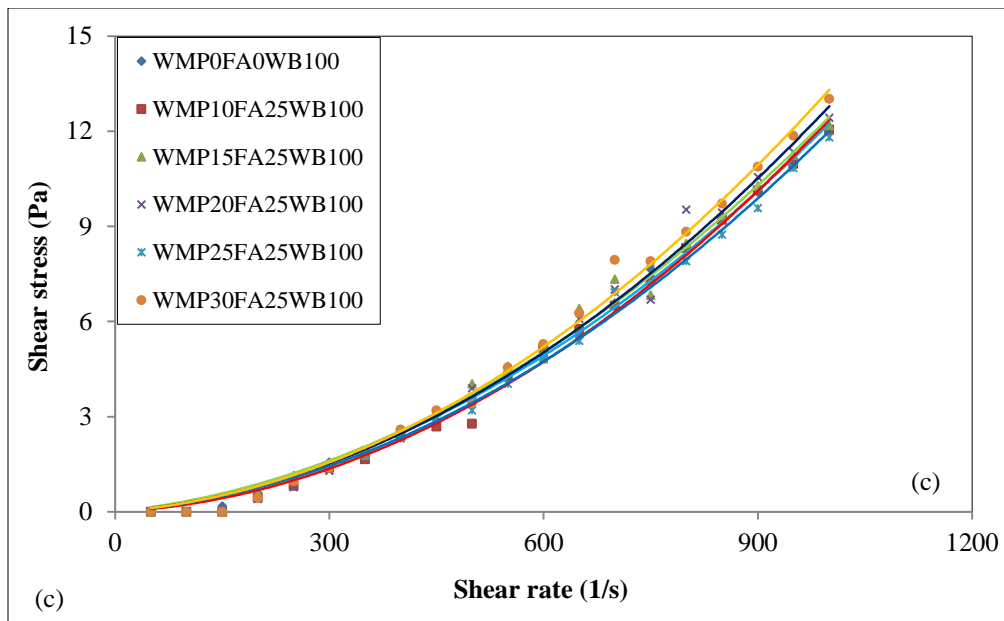


Figure 9. Flow curves of CBG containing, (a) WMP (5-25%); (b) FA (5-25%); (c) WMP(10-30%)+FA(25%)

The shear stress versus the shear rate curves were obtained by using modified Bingham model for CBG mixes. Table 3 shows the calculation of modified Bingham Model parameters. Authors used the values obtained from ascending and descending flow curves because it gives more accurate value. In Figure 9, the CBG mixtures illustrated shear-thickening behavior at all WMP, FA and WMP+FA contents.

Table 3. Rheological properties of the CBG mixture.

Mix ID	$\tau_0$ (Pa)	$\mu_p$ (Pa.s)	Grout Temperature (°C)	R <sup>2</sup>
WMP0FA0	0,76	0,003	19,0	0,998
WMP5FA0	0,72	0,003	18,5	0,997
WMP10FA0	0,70	0,003	18,0	0,998
WMP15FA0	0,76	0,004	18,5	0,999
WMP20FA0	0,46	0,004	18,5	0,997
WMP25FA0	0,58	0,004	18,5	0,999
FA5WMP0	1,00	0,003	19,3	0,995
FA10WMP0	0,95	0,002	18,1	0,981
FA15WMP0	0,90	0,001	18,5	0,994
FA20WMP0	0,80	0,001	18,5	0,982
FA25WMP0	0,75	0,001	18,8	0,997
WMP10FA25	0,89	0,003	19,0	0,996
WMP15FA25	0,92	0,003	18,1	0,994
WMP20FA25	0,97	0,003	18,5	0,992
WMP25FA25	0,98	0,003	18,5	0,995
WMP30FA25	0,99	0,004	18,8	0,995

Table 3 demonstrates how the plastic viscosity ( $\mu_p$ ) of w/b=1.00 ratio of CBG mixtures containing WMP and FA are affected. It was observed that the increment in WMP amount in the CBG mix increased the plastic viscosity. It can be concluded that the high water holding capacity. Similar result was reported by Singh et al. [14]. On the other hand, the increment in FA percentage in the CBG mix decreased the plastic viscosity, because of the spherical shape of the particles of FA.[31-32] Sha F. et al.[33] found an decrease viscosity, this was mainly because that the high amounts of SiO<sub>2</sub> and the “Ball Effects” of FA can improve rheological properties of grout.



### 3.2. Fluidity Properties of GBG

Figure 10(a) shows the mini-slump flow diameter of CBG prepared with the different amount of WMP, FA and WMP+FA contents. It indicates that the increase in the percentage of WMP (5-25%) amount reduced the mini-slump flow diameter because of the angular particle shape of WMP [34]. Moreover, the increase in the percentage of FA (5-25%) amount increments the mini slum flow. The spherical shape of FA decreases frictional force between the angular particles of PC owing to the ball bearing effect. According to the control sample, the increment in the percentage of WMP (10-25%) amount increased the mini-slump flow diameter for constant FA (25%) content.

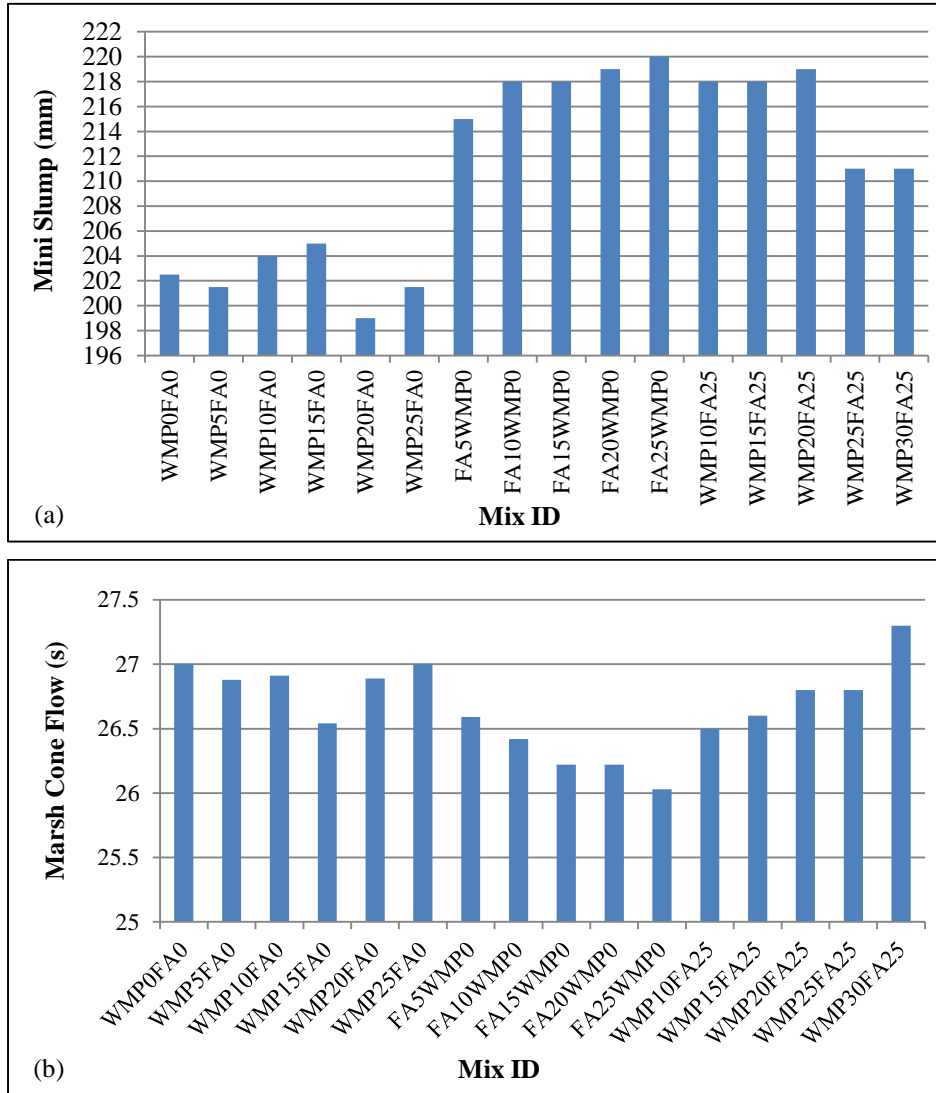


Figure 10. (a) Effect of WMP and FA on mini-slump flow; (b) effect of WMP and FA on MCFT

Figure 10(b) illustrates the impact of WMP and FA amount on MCFT at w/b=1.00 ratio. It seems that the increase in WMP amount in the CBG mix, there is no effect on MCFT. Also, MCFT decreased with the increase of FA amount. It can be said that increasing FA is able to improve the flowability of CBG. Similar results were reported by Güllü, H. et al. [35] and Sha. F. et al. [33]. Especially, FA increased the fluidity of CBG, while the WMP showed negative effect in increase the MCFT in WMP+FA content. Sing et al. [14] found a decrease flowability on increasing the replacement percent of cement by WMP which was attributed to high percentage of fines in WMP.

### 3.3. The Comparisons between Fluidity and Rheological Properties of CBG

To describe the relation between two rheological values (plastic viscosity and yield stress) and the flowability and the workability properties (mini-slump flow, MCFT and PCM) of the CBG, the curves were drawn between the test outcomes and the R<sup>2</sup> value. If correlation coefficient (R<sup>2</sup>) values are above 0.80, then it can be considered that there is a good correlation between two test outcomes. If R<sup>2</sup> values are below 0.80, it indicates that there is a poor correlation [5].

Figure 11 displays the relationship between the rheological and the flowability properties, meantime Figure 11(a) illustrates the relationship between the yield stress and the mini-slump flow. From the graph, the relation between the yield stress and the mini-slump flow was a very good correlation (R<sup>2</sup> = 0.868). Furthermore, it seemed that the reduction

in the yield stress incremented the mini-slump flow diameter. 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 [5, 25].

Figures 11(b) and 11(c) shown the relationship between plastic viscosity and MCFT and yield stress and Lombardi PCM test. In the Figure 11(b), it can be obtained that there is a good correlation ( $R^2 = 0.84$ ). Figure 11(b) shown that the increment in plastic viscosity increased the MCFT of the CBG mixture. Also, It seemed that there is weak correlation between yield stress and Lombardi PCM test ( $R^2 = 0.27$ ).

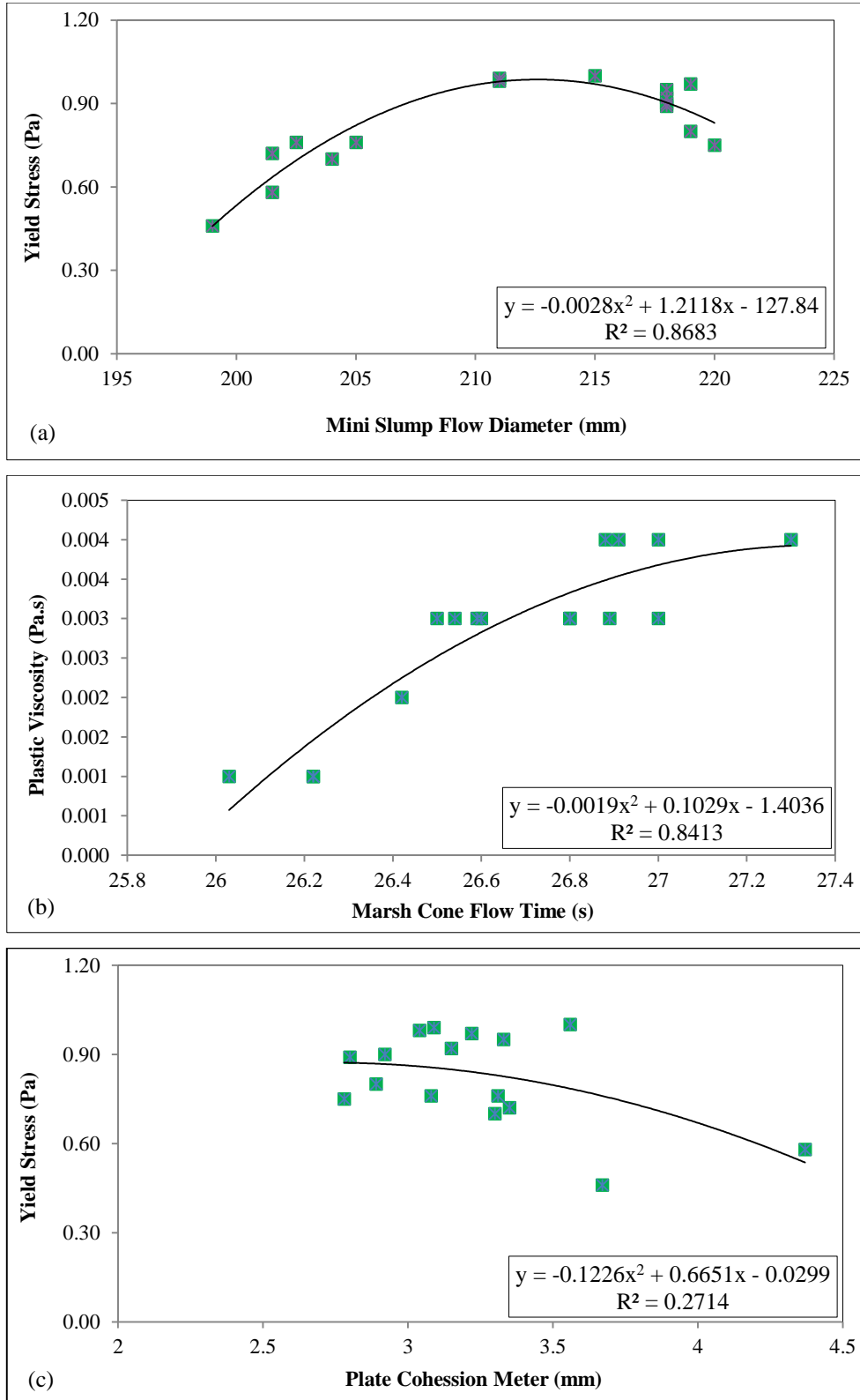


Figure 11. Correlation between workability and rheological properties of a yield stress –mini-slump flow, b plastic viscosity – MCFT and c yield stress –PCM

## 4. Conclusions

- The rheological properties of the CBGs importantly have been improved by the addition of WMP until %15 and all percentage of FA to grout mix at w/b=1.00 ratio. Strongly shear thickening behavior was got from the CBG mixture all mix WMP, FA and WMP+ FA contents
- The increment in the percentage of WMP in the CBG mixture incremented the plastic viscosity. On the other hand, the increment in FA percentage in the CBG mix decreased the plastic viscosity.
- The increase in the percentage of WMP (5-25%) amount reduced the mini-slump flow diameter. Moreover, the increment in the percentage of FA (5-25%) amount increments the mini slum flow. According to the control sample, the increment in the percentage of WMP (10-25%) amount increased the mini-slump flow diameter for constant FA (25%) content. Also, the increase in WMP amount in the CBG mix, there is no effect on MCFT. Also, MCFT decreased with the increase of FA amount. Especially, FA increased the fluidity of CBG, when the WMP showed negative effect in increase the MCFT in WMP+FA content.
- The rheological properties (the plastic viscosity and the yield stress) of the CBG got from the modified Bingham model also illustrates a good correlation with the MCFT and mini-slump flow of the CBG ( $R^2 = 0.86$ ,  $R^2=0.84$ ). On the other hand, no correlation was found between yield stress and Lombardi PCM test.

## 5. Conflicts of Interest

The authors declare no conflict of interest.

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