- Technical Paper -

RHEOLOGICAL PROPERTIES OF CEMENT-BASED MATERIALS INCORPORATING SCMS WITH LOW-VISCOSITY TYPED SUPERPLASTICIZER

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ABSTRACT

The aim of this research is to provide a rheological performance of the low viscosity typed superplasticizer (SP) on the cement-based materials incorporating various supplementary cementitious materials (SCMs). As a newly introduced typed SP, the low viscosity typed SP should be used for high performance concrete mixture of low water-to-cementitious materials ratio or incorporating various SCMs. Despite the demand of this new-typed SP, the research about the influence of the low viscosity typed SP on the fluidity or rheological properties of the cement-based materials incorporating SCMs is not sufficient. In this research, hence, to evaluate the performance of low viscosity typed SP on rheological properties of cement-based materials, cement paste and mortar incorporating fly ash, blast furnace slag, and silica fume were tested with generic and low viscosity typed SPs. According to the experiment results, low viscosity typed SP successfully reduced plastic viscosity of the mixtures. Additionally, the low viscosity typed SP reduced yield stress or improved fluidity more than generic typed SP. Based on this research, it is expected to contribute on providing a data for further usage of low viscosity typed SP efficiently.

Keywords: Low viscosity typed SP, Rheological properties, SCMs, Fluidity, Plastic viscosity

1. INTRODUCTION

Introduction of superplasticizer (SP) caused a tremendous improvement on the performance of cement-based materials not only limited within workability improvement but also increasing strength and durability [1]. With powerful dispersing effect of SP, mainly polycarboxylate-based SP, concrete can be decreased water-to-cement ratio dramatically, contained various supplementary cementitious materials and reinforced with fibers [2-4]. In aspect of fresh state concrete performance, SP provides an advanced workability with a good formwork filling [5], a successful pumpability [6], and removing vibration [7]. Finally, the development of fresh state performance of concrete with SP developed into the self-consolidating concrete (SCC). The key of the SCC is satisfying both low yield stress and appropriate viscosity of the concrete mixture for a good fluidity and a sufficient segregation resistivity, respectively [8-11].

Although increasing viscosity of the concrete mixture is an issue for making SCC, decreasing viscosity of the concrete mixture can be an important subject for high performance concrete with high volume fraction or including fibers. As Wallevik et al.'s research [12], SP works to disperse the particles of the concrete, so it only decreasing yield stress but viscosity. Hence, high performance concrete, generally a low water-to-cement ratio, or including various supplementary cementitious materials (SCMs) of a powder-typed material with high fineness, suffers extremely high viscosity even though it contains SP.

With the demand of decreasing viscosity of the concrete mixture, there is no specific chemical admixture. Although an overdosed SP causes segregation of the concrete materials, it cannot be controlled. According to Hamada et al.'s research [13, 14], a new typed SP with denser polymer structure was introduced. This new SP has a performance of reducing plastic viscosity of the concrete mixture and increase the robustness of the mixture. Additionally, Matsumoto, et al. [15] also introduced viscosity reducing typed SP in their research. These new typed SPs were relatively a new product thus there was not enough research on the properties of the concrete mixture including this type of SP. From the author's previous proceedings, the properties change and performance of the high performance concrete mixture including low-viscosity typed SP after the pumping were presented.

However, the former research was focused on the properties evaluation of the superplasticized concrete mixture without SCMs or under the specific

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conditions of pumping. Therefore, in this research, cement paste and mortar mixtures including various SCMs were evaluated by comparing both generic SP and low viscosity typed SP. The high-performance concrete is generally used with SCMs. Additionally, depending on various SCMs, the fluidity or rheological properties of cement-based materials are changed. Hence, evaluating low-viscosity typed SP with cement-based materials incorporating SCMs can provide a fundamental information for safe and efficient usage of the low-viscosity typed SP.

2. EXPERIMENT

2.1 Experimental plan

The experimental plan of this research is summarized in Table 1. The prepared mixture phases were cement paste and mortar. To evaluate the performance of two different types SP, 0.35 of low water-to-cementitious materials ratio (w/cm) was prepared for both cement paste and mortar. For the mortar mixture, cement-to-sand ratio was fixed to 1 to 0.7. The SCMs prepared were fly ash, blast furnace slag, and silica fume. All SCMs were replaced to cement and replacement ratio was 10 % of cement weight. To evaluate the performance of the low-viscosity typed SP, generic typed SP was prepared as a control. The SPs were added to the mixture and the dosages were 0.4, 0.8, and 1.2. Since the tested mixtures were low w/cm mixture, it was impossible to mix without SP. The test was designed to evaluate the fluidity and rheological properties of the mixtures. Hence, modified mini slump flow test was conducted for general fluidity. Additionally, for rheological properties, rheological test with rheometer was conducted for cement paste. However, in the case of mortar mixture, the channel flow test was conducted to obtain the rheological parameters of yield stress and plastic viscosity.

2.2 Materials and sample preparations

Cement used was ordinary Portland cement from South Korea market. The information on the physical properties of cement is provided in Table 2. The mixing water used was tap water, and sand used was crushed sand. The crushed sand was same product for ready mixed concrete manufacturing and the physical properties are shown in Table 3. As a SCMs, fly ash, blast furnace slag, and silica fume used were same materials for ready mixed concrete manufacturing. The information on these SCMs were obtained from the providers and the brief information on the SCMs was provided in Table 4, 5, and 6, respectively. For SPs, all SPs were general products for ready mixed concrete manufacturing. Especially, the low-viscosity typed SP, since there is no alternative product in South Korean market, the product provided from a certain manufacturer. Two SPs were polycarboxylate-based SP and the brief physical properties were provided in Table 7.

The mortar was mixed with the planetary table-top mixer. The mixer capacity was 5 liters and the mixtures were mixed with 5 liters. The mixing protocol

Table 1 Experimental plan

Mixture	Phase		Cement paste, mortar
	w/cm*		0.35
	Cement : Sand (weight)**		1 : 0.7
	SCMs	Type	Fly ash, Blast furnace slag, Silica fume
		Replacement ratio (%, weight)	10
	Chemical admixture	Type***	Generic SP, Low-viscosity typed SP
		Dosage (%, weight)	0.4, 0.8, 1.2
Test	Modified-mini slump flow Rheological test (for cement paste) Channel flow (for yield stress, and plastic viscosity of the mortar mixture)		

*w/cm: water-to-cementitious materials ratio
** cement to sand ratio is valid for mortar mixture
*** SP: superplasticizer

Table 2 Physical properties of cement

Specific gravity	Blaine (cm^2/g)	Setting time (min.)		Compressive strength (MPa)		
		Initial	Final	3D	7D	28D
3.15	3,660	295	330	28.1	38.1	52.8

Table 3 Physical properties of fine aggregate

Density (g/cm^3)	Fineness modulus	Absorption rate (%)	Distribution of grain shape (%)
2.62	2.79	0.90	54.80

Table 4 Physical and chemical properties of fly ash

Density (g/cm^3)	Blaine (cm^2/g)	L.O.I (%)	SiO_2 (%)	Hygroscopic moisture (%)
2.21	3,520	4.6	52.3	0.13

Table 5 Physical and chemical properties of blast furnace slag

Density (g/cm^3)	Blaine (cm^2/g)	L.O.I (%)	Chemical composition of major chemical components (%)		
			SiO_2	CaO	MgO
2.91	4,378	0.32	26.50	45.6	5.26

Table 6 Physical and chemical properties of silica fume

Density (g/cm^3)	Blaine (cm^2/g)	L.O.I (%)	Chemical composition of major chemical components (%)		
			SiO_2	CaO	MgO
2.20	160,000	0.05	96.65	0.38	0.19

Table 7 Physical properties of SPs

Type	Phase	Color	Specific gravity	Solid content (%)	pH
Generic type	Liquid	Brown	1.048	20	5.4
Low-viscosity type	Liquid	Transparency	1.200	38	6.5

was followed with ASTM C305 [16] method. All fresh state tests were conducted right after the mixing.

2.3 Test methods

For modified mini slump flow test uses same apparatus of table flow test (ASTM C1437 [17]) without flow table. The mini slump cone was placed on the acrylic plate and flow distance of two diameters were measured after lifting the cone without dropping or external forces.

For rheological properties, cement paste was tested with commercial rheometer. The rheometer used was vane shaped impeller with 100 mm diameter and 200 mm height of cup system. The rheological parameter was obtained from Bingham model by flow curve. The flow curve was obtained by step-up and step-down shear rate protocol: the shear rate was increased up to 25 s^{-1} and decreased to 0 s^{-1} with 5 steps for each. For each step, constant shear rate was applied for 15 seconds and shear stress values at equilibrium state were collected and flow curve was obtained. (see Fig 1 for applied shear rate protocol and Bingham model from flow curve.) The channel flow test was designed to measure the rheological parameters of the mortar such as yield stress and plastic viscosity. Generally, rheological properties of the cementitious materials are measured by using rheometer. Due to the wide range of the particle size of the concrete, especially, coarse aggregate, there are specially designed rheometer is necessary to measure the rheological properties of the concrete mixture. In this research, since the mixture phase was a mortar, it was limited to use the commercial rheometer for cement paste. Therefore, based on the former research of Kim et al. [18], channel flow test was conducted. The used channel flow dimension was provided in Fig 2. Using the channel flow mold, the mortar mixtures were flown by its own weight and using certain equations, yield stress and plastic viscosity were calculated. For these conditions, the flowing distance and time until stop the flowing the mixture are C_f and T_f, respectively. Additionally, using all constants in Table 8, the yield stress value was calculated by equation (1), and parameters a and b for plastic viscosity from equation (2) can be calculated by equation (3), and (4). Finally, using a, and b, plastic viscosity value was obtained from equation (2).

$$\tau_y = k_1 exp(-C_f/k_2) \quad (1)$$

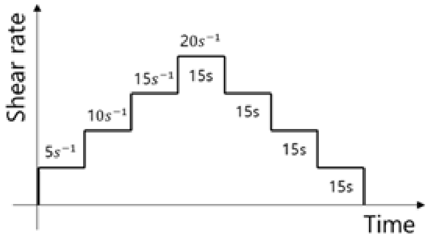
$$\eta_p = exp\left(\frac{T_f - b}{a}\right) \quad (2)$$

$$a = -k_3\, ln(\tau_y/k_4) \quad (3)$$

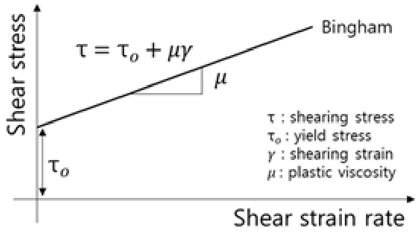
$$b = k_5 exp(-\tau_y/k_6) \quad (4)$$

where, τ_y is yield stress and η_p is plastic viscosity. the yield stress and plastic viscosity is based on the Bingham model.

As a limitation of the rheological test of channel flow test method, although both rheometer measurement and channel flow calculations provide yield stress and plastic viscosity, it is difficult to compare with same parameter of both cement paste and mortar.



(a) Applied shear rate protocol



(b) Bingham model from flow curve

Fig.1 Shear rate protocol for flow curve and Bingham model from flow curve

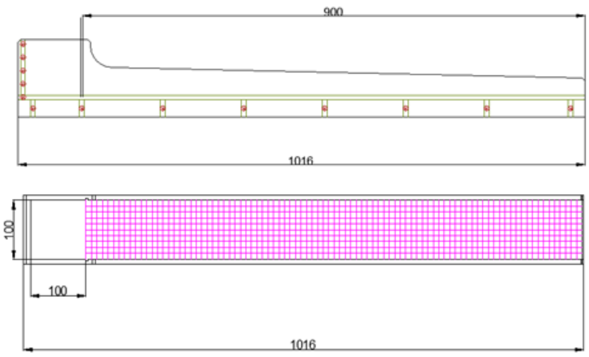


Fig.2 Dimension of the channel flow test mold

Table 8 Coefficients for the channel flow test's correlating model (equations)

k_1(Pa)	k_2(mm)	k_3(s)	k_4(Pa)	k_5(s)	k_6(Pa)
729	126	3.0	1568	46.1	11.6

3. RESULT AND DISCUSSIONS

3.1 Fundamental properties

(1) Modified mini slump flow

To check the general fluidity of the mixtures, modified mini slump flow test was conducted. As shown in Fig 3, generally flow values were increased with increased dosage of the SPs regardless mixture phases and types of SP. First, for cement paste, low viscosity typed SP increased flow of the mixture rather than generic SP. It means that although low viscosity typed SP is expected to decrease the viscosity, it also decreases the yield stress or improved fluidity of the mixture. Except silica fume, 0.4% of SP dosage was successfully improve the fluidity of the mixtures. Especially, flow of the cement paste incorporating blast furnace slag showed the highest flow value with low viscosity typed SP. The performance of low viscosity typed SP was presented for blast furnace slag and silica fume. This is considered that because of the improved fluidity of fly ash due to the spherical shape, the performance of low viscosity typed SP is decreased.

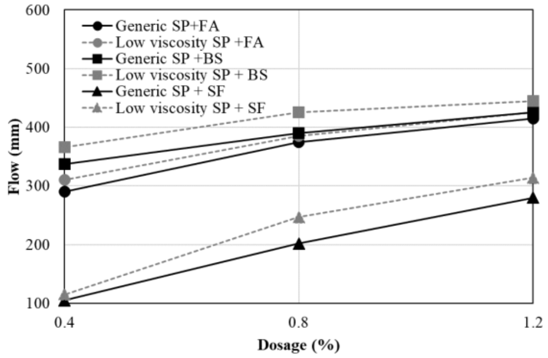
In the case of mortar, flow values were generally similar trend with the cement pastes. Additionally, the degree of flow values was also similar with cement paste except the mortar incorporating silica fume with low viscosity typed SP. This is considered that low viscosity typed SP contributes decreasing plastic viscosity and contributes on improving fluidity of the mortar with silica fume.

(2) Yield stress

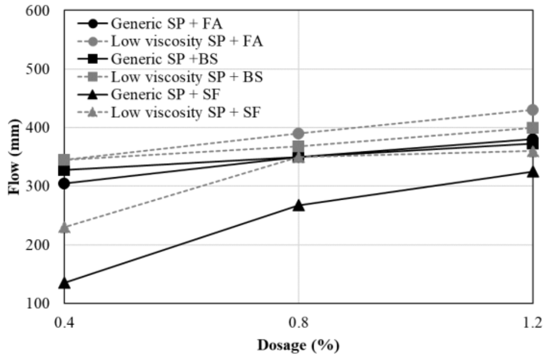
Yield stress is defined as a minimum stress of the material should be overcome to be started flowing or deforming. Therefore, decreasing yield stress of the cement-based materials is generally expressed with fluidity and closely related with slump or slump flow results. Rheologically, using SP is decreasing yield stress of the cement-based materials, and the results of this research also showed decreased yield stress with increasing dosage of SP regardless the types of the SP (see Fig 4). For the cement paste, 0.4% dosage of SPs showed already almost zero, regardless of the types of SP. On the other hand, for silica fume, low viscosity typed SP decreased yield stress rather than generic SP for all dosages range. As it was checked in modified mini slump flow test results, it means that the low viscosity typed SP decreases not only plastic viscosity but also yield stress of the mixture. Therefore, although it is not clearly defined from former research, it can be considered that the low viscosity typed SP has a similar dispersing action as well as the generic SP. For mortar mixture, low viscosity typed SP worked as an additional dispersing factor. Therefore, for the mixture with silica fume, low viscosity typed SP caused decreased yield stress. On the other hand, there was no specific difference between generic and low viscosity typed SP for fly ash and blast furnace slag contained mortar mixtures. It is considered that the yield stress is already close to 0 with 0.4% dosage of SP.

(3) Plastic viscosity

Plastic viscosity of the material is defined as the inverse of the velocity. Therefore, high plastic viscosity means slow fluidity of the material. Additionally, in aspect of segregation of cement-based materials, high plastic viscosity can help preventing segregation. However, as stated above, high plastic viscosity is also can be a problem for concrete construction. For suspension like cement-based materials, plastic viscosity can be determined by solid volume fraction, particle condition, and viscosity of medium. Therefore, low w/cm of cement-based materials or cement-based materials replaced with other materials with small particles have a high plastic viscosity. As shown in Fig 5, viscosity of the mixtures was decreased with

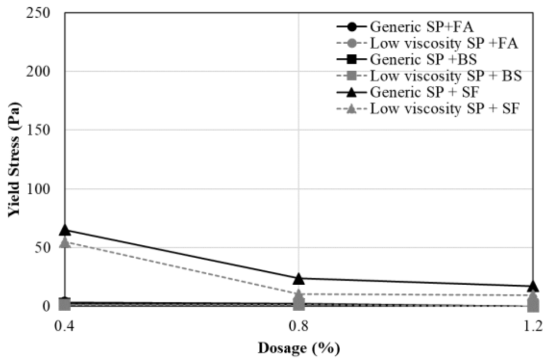


(a) Cement paste

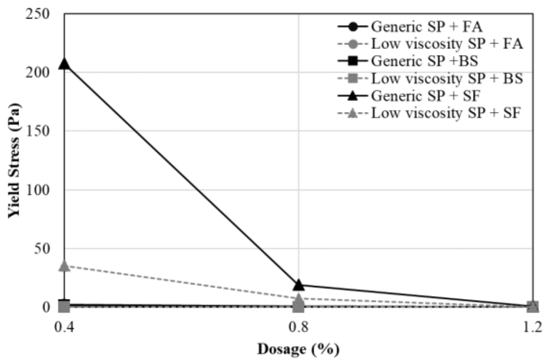


(b) Mortar

Fig.3 Influence of SPs dosage on modified mini slump flow of cement-based materials incorporating various SCMs depending on SP types



(a) Cement paste



(b) Mortar

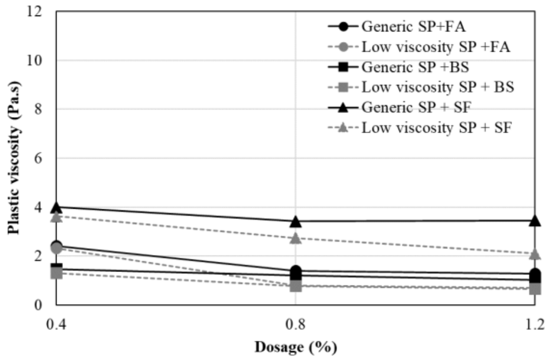
Fig.4 Influence of SPs dosage on yield stress of cement-based materials incorporating various SCMs depending on SP types increasing dosage of SPs, regardless of types. First, for cement paste, the mixture with silica fume showed the highest viscosity values. Comparing the mixture with generic SP, low viscosity typed SP decreased plastic viscosity of the cement paste incorporating silica fume. Interestingly, although the generic SP decreased plastic viscosity up to 0.8 % dosage and no further decreasing, the low viscosity typed SP continuously decreased plastic viscosity until 1.2 % dosage. For other SCMs of fly ash and blast furnace slag, commonly, low viscosity typed SP produced lower plastic viscosity than generic SP. For mortar, similar to cement paste, low viscosity typed SP showed decreased plastic viscosity for all mixtures regardless SCMs. For the mixture with silica fume, the viscosity decreasing performance of low viscosity typed SP was higher than any other mixture. The mixtures incorporating fly ash and blast furnace slag, the plastic viscosity of the mixtures was close to 0 when they contain low viscosity typed SP.

For viscosity reducing performance of the low viscosity typed SP comparing with generic typed SP, as shown in Fig 6, for the mixtures incorporating silica fume, the low viscosity typed SP decreased plastic viscosity more than yield stress. In the case of cement paste incorporating silica fume, the low viscosity typed SP reduced plastic viscosity more than yield stress at 1.2 % of SPs dosage. On the other hand, for mortar incorporating silica fume, the low viscosity typed SP reduced plastic viscosity more than the generic typed SP although both SPs reduced yield stress more than plastic viscosity. Therefore, it is considered that the low viscosity typed SP has a valid performance of reducing plastic viscosity of the cement-based materials although the low viscosity typed SP also decreases yield stress of it.

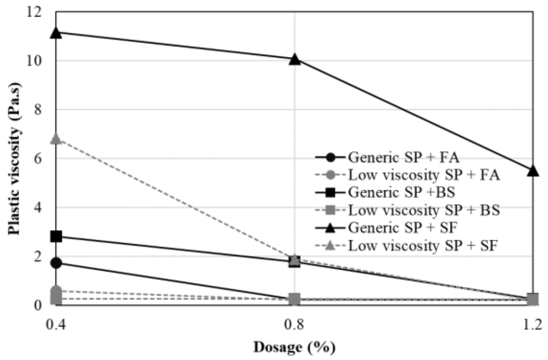
4. CONCLUSIONS

In this research, with a goal of providing a fundamental data on low viscosity typed SP, the fluidity and rheological properties of cement paste and mortar mixtures incorporating SCMs were evaluated with generic and low viscosity typed SPs. Although it was difficult to compare the rheological properties measured from cement paste and mortar because of different measuring methods, for each mixture phase, rheological properties depending on low viscosity typed SP were provided, and summarized as follow:

(1) The fluidity of cement paste, and mortar can be increased by adding both generic and low viscosity typed SPs. Comparing two different types of SPs, low viscosity typed SP provided higher fluidity than generic typed SP.
(2) Low viscosity typed SP decreased yield stress as well as generic typed SP. As shown in modified mini slump flow test results, low viscosity typed SP decreases the yield stress of the cement paste and mortar more than generic typed SP.
(3) Plastic viscosity of cement paste, and mortar can be decreased by using low viscosity typed SP. Depending on the SCMs conditions, when silica

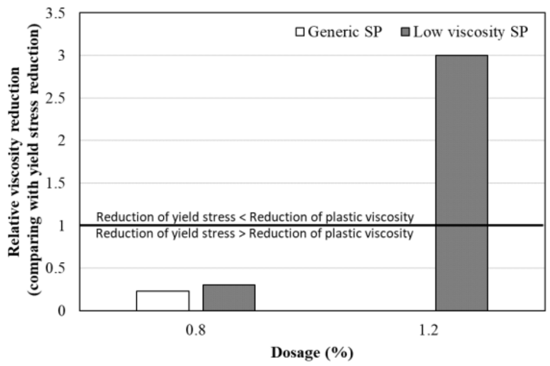


(a) Cement paste

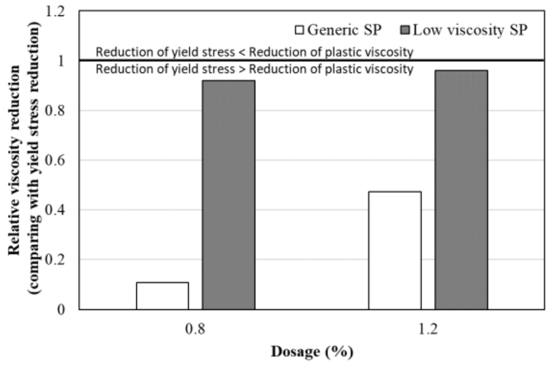


(b) Mortar

Fig.5 Influence of SPs dosage on plastic viscosity of cement-based materials incorporating various SCMs depending on SP types



(a) Cement paste (incorporating silica fume)



(b) Mortar (incorporating silica fume)

Fig.6 Relative viscosity reduction comparing with yield stress reduction (relative viscosity reduction was calculated by $\Delta \text{viscosity}/\Delta \text{yield stress}$)

fume is used, and the mixtures have a high viscosity, the viscosity reducing performance of low viscosity typed SP showed higher viscosity reducing performance than other SCMs.

(4) Although there is limitation on the decreasing viscosity mechanisms of low viscosity typed SP, it can be considered that the low viscosity typed SP has a higher capacity of dispersing the cement-based materials than generic typed SP and has a valid performance of reducing plastic viscosity.

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REFERENCES

[1] Aïtcin, P.C. "Cements of yesterday and today: concrete of tomorrow." Cement and Concrete Research, Vol. 30 (9), 2000, pp.1349-1359.

[2] Balaguru, P., and Shah, S.P. "Fiber-reinforced cement composites." McGraw-Hill, New York, 1992.

[3] Akkaya, Y., Shah, S.P., and Ghandehari, M. "Influence of fiber dispersion on the performance of microfiber reinforced cement composites." ACI Special Publication, Vol. 513, 2003, pp.1-18.

[4] Ozyurt, N., Mason, T.O., and Shah, S.P. "Correlation of fiber dispersion, rheology and mechanical performance of FRCs." Cement and Concrete Composites. Vol. 29 (2), 2007, pp.70-79.

[5] Alfi, M., Enarjee, N., Feys, D., and Park, J. "Simulation of Formwork Filling by Cement Fluid: the Effect of the Formwork Structure on Yield-stress Fluid." In proceedings 2013 COMSOL conference in Boston, Boston, 2013.

[6] Ngo, T.T., Kadri, E.H., Bennacer, R., and Cussigh, F. "Use of tribometer to estimate interface friction and concrete boundary layer composition during the fluid concrete pumping." Construction and Building Materials, Vol.24 (7), 2010, pp.1253-1261.

[7] Khayat, K. "Optimization and performance of air-entrained, self-consolidating concrete." ACI Materials Journal, Vol. 97 (5), 2000, pp.526-535.

[8] Bui, V.K., Akkaya, Y., and Shah, S.P. "Rheological model for self-consolidating concrete." ACI Materials Journal, Vol. 99 (6), 2002, pp.549-559.

[9] Khayat, K. "Workability, testing, and performance of self-consolidating concrete." ACI Materials Journal, Vol. 96 (3), 1999, pp.346-353.

[10] Kong, H.J., Bike, S.G., and Li, V.C. "Development of a self-consolidating engineered cementitious composite employing electrosteric dispersion/stabilization." Cement and Concrete Composites, Vol. 25 (3), 2003, pp.301-309.

[11] ACI Committee 237. "Self-Consolidating Concrete." Farmington Hills, MI, 2007.

[12] Wallevik, O.H., and Wallevik, J.E. "Rheology as a tool in concrete science: The use of rheographs and workability boxes." Cement and Concrete Research, Vol. 41 (12), 2011, pp.1279-1288.

[13] Hamada, D., Hamai, T., Shimoda, M., Shonaka, M., and Takahashi, H. "Development of new superplasticizer providing ultimate workability." ACI Special Publication, Vol. 239, 2006, pp.31-50.

[14] Hamada, D., Sagawa, K., Tanisho, Y., and Yamamuro, H. "Pursuance of Workability Retention by New Superplasticizer." ACI Special Publication, Vol. 262, 2009, pp.297-308.

[15] Sugiyama, T., Matsumoto, T., and Ohta, A. "Application Study of Viscosity Reducing Type Superplasticizers for Low Water-Binder Ratio Concrete." ACI Special Publication, Vol. 262, 2009, pp.177-186.

[16] ASTM International. "ASTM C305, Standard Practice for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency." ASTM International, West Conshohocken, PA, 2012.

[17] ASTM International. "ASTM C1437, Standard Test Method for Flow of Hydraulic Cement Mortar." ASTM International, West Conshohocken, PA, 2013.

[18] Kim, J., Lee, J., Shin, T., and Yoon, J. "Rheological method for alpha test evaluation of developing superplasticizers' performance: Channel flow test." Advances in Materials Science, Vol. 2017, 2017, pp.1-8.