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Optimizing polycarboxylate based superplasticizer dosage with different cement type

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Abstract

The use of polycarboxylate ether (PCE) as superplasticizer (SP) in the manufacture of high strength concrete is increasingly common. Each brand of SP available on the market has different compositions, causing differences in dosage requirement and the resulting characteristics. Beside SP type, cement type and composition also affect the fresh and hardened concrete properties. In this study, the optimum dosages of several brands of PCE superplasticizer in making mortar were investigated. Two different cement types were used. The effect of SP on flowability, setting time, and resulting compressive strength were evaluated. The results show that with the increase of SP dosage in mortar mixture, the flowability increased. However, there is an optimum value for each brand and for each water cement ratio. The increase of flowability is accompanied by an increase in compressive strength until it reaches the optimum level. Nevertheless, excessive use of SP could lead to bleeding and segregation, and reduce the compressive strength. It was found that ordinary Portland cement (OPC) requires higher SP dosage than Portland Pozzolan cement (PPC) for the same flowability. Longer setting time was observed for all mixtures employing SP, at different degrees of extension. It correlates with the slump retention time. Simple method to determine the optimum dosage is suggested in this paper. © 2017 The Authors. Published by Elsevier Ltd. This is an open access article under the CC BY-NC-ND license (<http://creativecommons.org/licenses/by-nc-nd/4.0/>).

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Keywords: polycarboxylate; superplasticizer; setting time; dosage; cement type; OPC; PPC.

1. Introduction

High strength concrete essentially need to use superplasticizer to reduce cement interparticle force and to disperse the particle evenly in the concrete mix. High compressive strength could only be achieved when low water to

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cement ratio (w/c) is ensured in the concrete mix design, while maintaining adequate workability of the fresh mixture. Superplasticizer addition reduces the cohesiveness of the cement particles by electrostatic repulsion, in the case of naphthalene or melamine-based superplasticizer; and by a combination of electrostatic and steric repulsion mechanism in the case of polycarboxylate based superplasticizer [1,2].

Different brand of polycarboxylate ether (PCE) superplasticizer has different molecular structures, affected by the manufacturing process. Its chemical structures consist of main chain and side chains with different length and density, and will have different effectivity in increasing the workability of concrete mixture [3]. Several researches have been done on the dispersion mechanism based on the molecular structures of the materials, showing that it could cause changes in the dispersion behavior (slump flow), performance on slump retention (slump loss), delay on the reaction rate (setting time) and the particle packing improvement (compressive strength) with different chemical structures [4-8].

The effect of superplasticizer in concrete fresh mixture depends on its dosage and distribution in the mixture. Very low dosage will not affect the rheological behavior of the fresh mixture, and on the other hand very high dosage may cause detrimental effect such as bleeding and segregation. Yamada et al. [9] remark that there are critical dosage and saturation dosage of SP in the concrete mixture. Critical dosage is defined as minimal dosage needed to cause overall effect of SP in the mixture. Below critical dosage, the mixture will behave as if no SP is added. Saturation dosage implies that further addition of SP will not lead to improvement of rheology behavior of the concrete mixture. However, the SP dosage must also have an upper limit value, as higher dosage reduces cohesion of the mixture due to excessive bleed water, lowers the viscosity of the cement paste, and hence induces segregation. Interaction of SP in the concrete mixture is a complex process, as it has to compete with the dissolution of cement compound. Dissolution of sulfate ions from gypsum to control the setting time of cement occurs at the beginning of the process. The presence of gypsum, as well as other compounds, affects the effectivity of SP [9]. Direct addition of SP into mixing water may cause different SP dosage requirement compared to delayed addition. Lower SP dosage requirement was observed for delayed addition, however, delayed SP addition is not always possible when considering the mixing equipments and production cycle.

Different cement types may alter the critical and saturation SP dosages, because of the differences of the chemical compositions of cement. Variation of chemical composition and physical properties of one brand of cement between shipments may occur, and thus the optimum SP dosage needs to be adjusted for a good and consistent result. The addition of supplementary cementitious material can also reduce or increase the SP dosage requirement. Adding fly ash tends to reduce the SP dosage required to achieve the same workability, because of its chemical and physical properties [10].

The PCE-based SPs currently available in the market in Indonesia, are supplied by several manufacturers, both local and international, competing one to each other. Each brand of PCE-based SP comes with different behavior and characteristics, aside from the availability and price range. The customization of the SP by adding other ingredient, such as retarder, accelerator, foam buster, causes further confusion on the dosage requirement of the SP to produce a good, homogeneous and predictable fresh concrete. The objectives of this research are to study the different characteristics of polycarboxylate-based (PCE) superplasticizer commonly available in the market and to evaluate the proposed simple testing method to determine its properties. Simple testing method is proposed to simplify the optimization process. Two cement types were used to show the influence of cementitious mixture on the optimum dosage needed.

2. Experimental methods

2.1. Materials and mixtures

Five brands of PCE-based SP that currently available in the local market in Indonesia, produced by four different manufactures, were used in this study. The coding for the SPs used are (a) CC, (b) SV, and (c) AS, produced by three different producers; (d) BA and (e) BS produced by the same producer. Two cement types, i.e. Ordinary Portland cement (OPC) and Portland Pozzolan cement (PPC) from two different cement producers were used in this research.

Distilled water was used throughout the experiment to avoid any contamination. Sand was obtained from local quarry in Lumajang, East Java, Indonesia. The sand gradation was controlled by sieving to conform to graded sand according to ASTM C778 [11] to avoid any unnecessary variation in sand gradation that may affect the workability of the fresh mortar mixture. This controlled gradation is necessary to avoid variation of flowability.

The effective SP dosage was investigated in mortar mixes with sand to cement weight ratio of 2. Three series of w/c ratio of 0.25, 0.30 and 0.35 was used to investigate the effect of water content in the mixture. Superplasticizer dosage intended was from 0 to 2% by cement weight at 0.1% increment, the test series was terminated when the mixture was showing sign of bleeding and/or segregation.

2.2. Test Methods

Workability of the fresh mixture was measured using flow table test apparatus in accordance to ASTM C230 [12]. SP was added directly into the water. Dry material was mixed thoroughly before adding water in the mixture, then the mortar mixture was mixed using small hand drill for two minutes. Afterwards, the mixture was placed in a converted cone on the flow table test apparatus. The initial diameter or static flow (D1) was measured after the cone was removed from the table, and final diameter or dynamic flow (D2) was measured after applying 25 drops. Fig. 1(a) shows an example of a flowability test result. The mortar mixture was then cast in 50×50×50 mm cube molds. Demolding was carried out on the following day and specimens were kept submerged in clear fresh water until taken out one day prior to test.

Compressive strength test were conducted on the mortar cube specimens at 7 and 28 days, on three specimens for each variable. Determination of setting time was performed from temperature measurement of the mortar, on 200 ml mortar that was placed in a sealed polystyrene container. Monitoring the temperature evolution was done for 48 hours. Initial setting time was determined as the time at the median of the temperature rise, while final setting time as the time when the maximum temperature was reached, as shown in Fig. 1(b). This measurement was to simplify testing method based on ASTM C1679 [13].

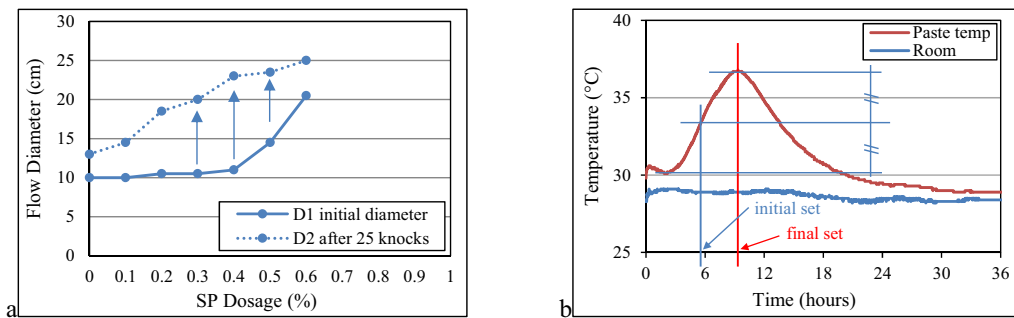


Fig. 1. (a) Result of flow table test, showing D1 and D2 with increase of SP dosage; (b) Measurement of initial and final setting time of the mortar mixture from temperature evolution of the mixture.

3. Results and discussions

3.1. Effect of superplasticizer type and water-to-cement ratio

Mortar mixture was tested for its flowability using flow table test. Initial diameter (D1) and final diameter (D2) were measured to determine the workability of the mixture. Fig. 2 to 6 show the correlation between the dosage of five SPs with the flowability of fresh mortar and the 28-day compressive strength. The water-to-cement ratios of mortar were varied, i.e. 0.25, 0.30 and 0.35, while maintaining the cement content, to investigate the influence of water content in the mixture. Adding water into the mixture increases the distance amongst the cement particles and reduces the viscosity. On the other hand, adding SP reduces the inter-particle attractive force, with slight change in

its viscosity. This difference causing intricate interaction between the mixture composition and the SP dosage required to achieve the targeted workability.

Water-to-cement ratio influences the static flowability of the mixture. Very low water-to-cement ratio ($w/c=0.25$) does not cause any static flowability of mortar mixture, even when SP is continually added. The increase of workability was observed at the dynamic condition, when the table was dropped for 25 times. When the SP dosage was increased, it was observed that there was an inflection point. The mixture showed low flowability at low SP dosage. Increase in SP dosage linearly increased the flowability until the optimum SP dosage was reached, where only slight increase of flowability was acquired with the increase of SP dosage.

From the inflection point, the critical and saturation dosages can be identified for the specific mixture composition. Different optimum SP dosages observed are depending on the SP brand. This can be attributed to the concentration of the active component in the SP. Therefore, the active component of SP should be also known for better estimation of the dosage needed.

The 28-day compressive strength was also shown in the Figures, correlating with the SP product used, SP dosage and w/c ratio of the mortar mixtures. It is shown that the presence of SP definitely has positive impact in increasing the compressive strength of concrete with the increase of workability and dispersion of cement particles. The increase is more pronounce for mixtures with lower w/c . Fig. 3(b) shows an example of the increase of compressive strength for mortar with SV superplasticizer. There is an optimum dosage of 0.3-0.4% to achieve higher strength. Further dosage increment reduces the strength, indicating the occurrence of bleeding and segregation.

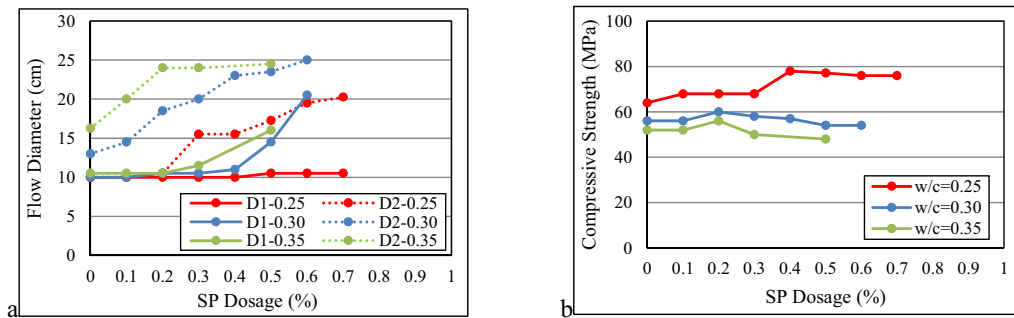


Fig. 2. (a) Flow diameter and SP dosage with different w/c ; (b) 28-day compressive strength of mortar with CC superplasticizer.

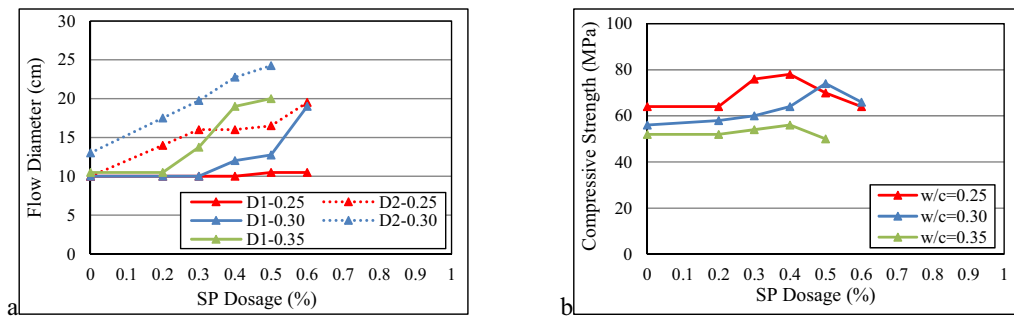


Fig. 3. (a) Flow diameter and SP dosage with different w/c ; (b) 28-day compressive strength of mortar with SV superplasticizer

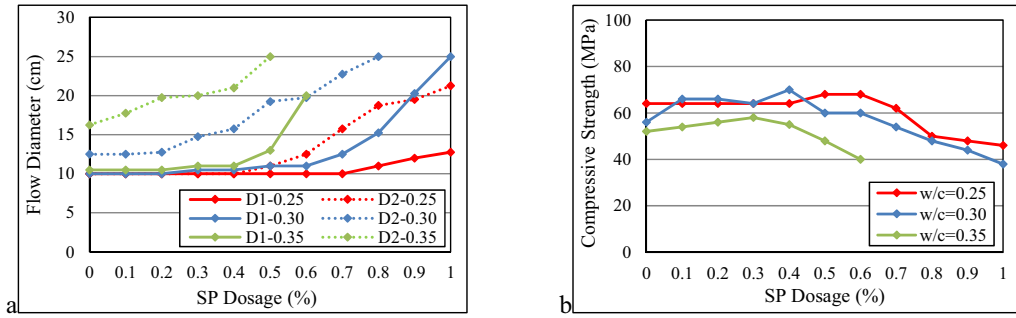


Fig. 4. (a) Flow diameter and SP dosage with different w/c; (b) 28-day compressive strength of mortar with AS superplasticizer

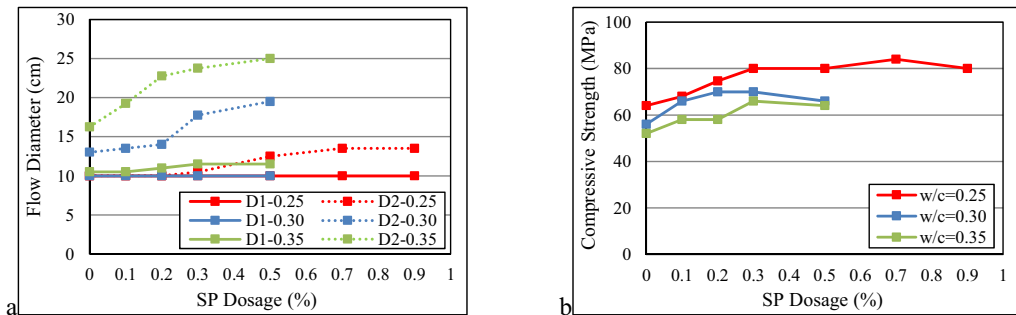


Fig. 5. (a) Flow diameter and SP dosage with different w/c; (b) 28-day compressive strength of mortar with BA superplasticizer

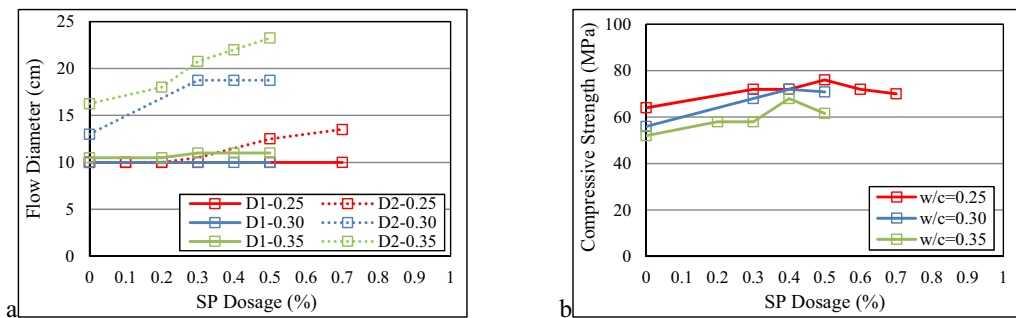


Fig. 6. (a) Flow diameter and SP dosage with different w/c; (b) 28-day compressive strength of mortar with BS superplasticizer

3.2. Effect of cement type

The SP dosage increment on mixture using Ordinary Portland cement (OPC) and Portland Pozzolan cement (PPC) are shown on Fig. 7-11. Distinct results are shown for mixtures with OPC and PPC, where PPC mortar mixture show better workability. Water-to-cement ratio was set constant at 0.3 in this experiment series. SP demand is lower for PPC mortar because in its composition there is additional pozzolanic material that contributes to lowering the inter-particle attraction force, and reduces the water demand of the mixture. OPC mortar is more benefitted from SP addition, because cement particles are distributed more evenly in the mixture. Its compressive strength is increased compared to the one without SP addition. Higher increase of strength with the increase of testing age is also observed on mortar mixture using OPC.

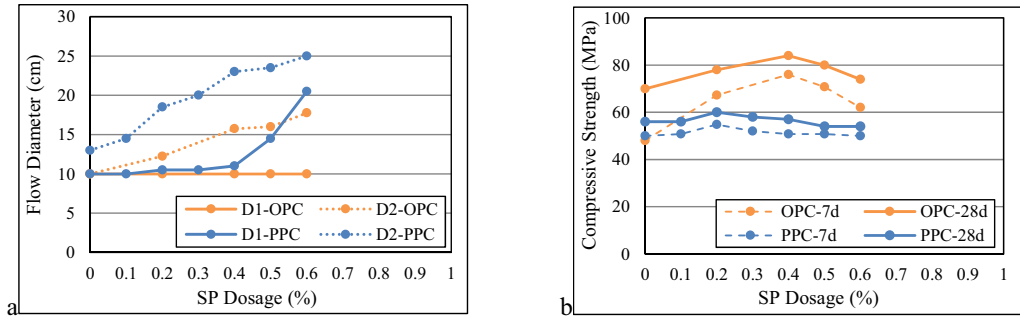


Fig. 7. (a) Flow diameter and SP dosage with OPC and PPC cement; (b) 7-day and 28-day mortar compressive strength with CC superplasticizer

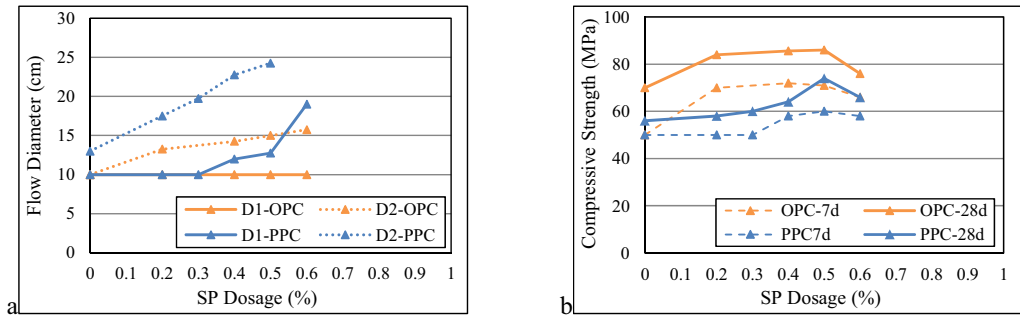


Fig. 8. (a) Flow diameter and SP dosage with OPC and PPC cement; (b) 7-day and 28-day mortar compressive strength with SV superplasticizer

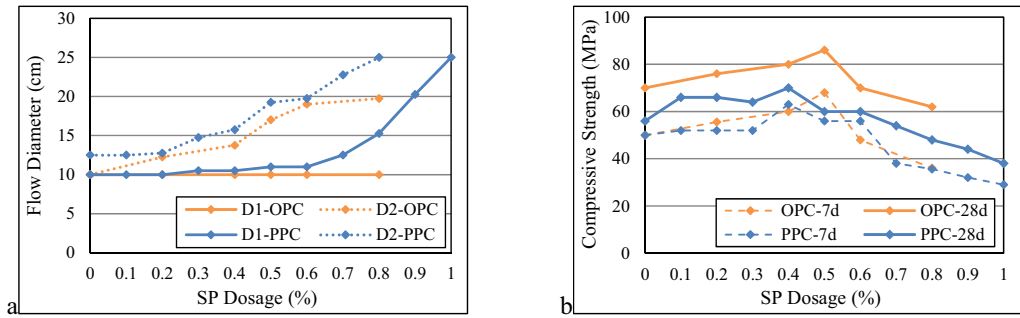


Fig. 9. (a) Flow diameter and SP dosage with OPC and PPC cement; (b) 7-day and 28-day mortar compressive strength with AS superplasticizer

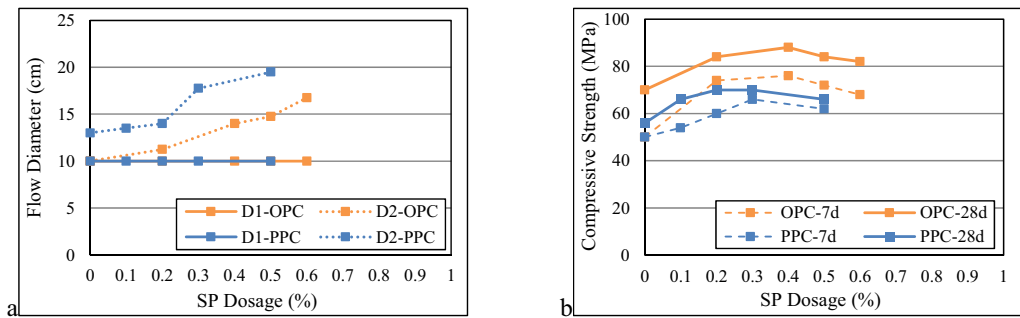


Fig. 10. (a) Flow diameter and SP dosage with OPC and PPC cement; (b) 7-day and 28-day mortar compressive strength with BA superplasticizer

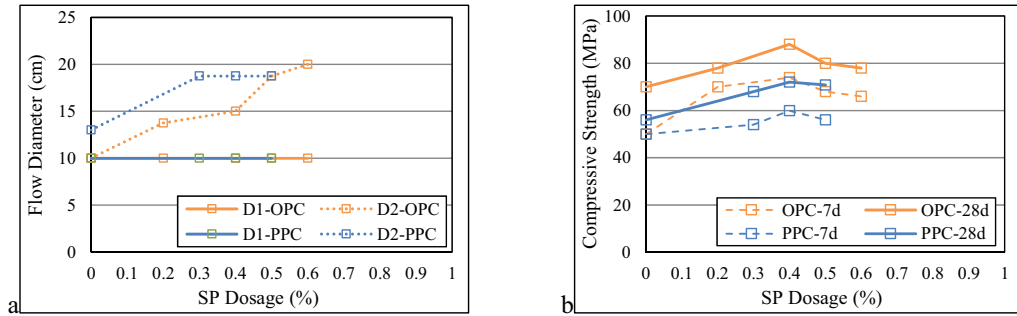


Fig. 11. (a) Flow diameter and SP dosage with OPC and PPC cement; (b) 7-day and 28-day mortar compressive strength with BS superplasticizer

3.3. Slump retention and setting time

The slump retention of mortar mixture with different SP was measured by dynamic flow (D2) at 30 minutes interval. Water-to-cement ratio was set constant at 0.3, with SP dosage was determined from previous step. Fig. 12 shows that there is reduction on slump flow for three SPs (CC, SV and AS) starting from 60 minutes after water addition. The other two SPs (BA and BS) show good slump retention even after 120 minutes.

The initial and final setting time measured from temperature evolution of the mixture is shown in Table 1. There is good correlation between setting time and slump retention time. The setting time is faster for OPC mortar compared to the one with PPC, as shown in the control mixture. However, the SP addition is also shown to retard the chemical reaction rate of the mixture with the longest retention time occurs for BS superplasticizer.

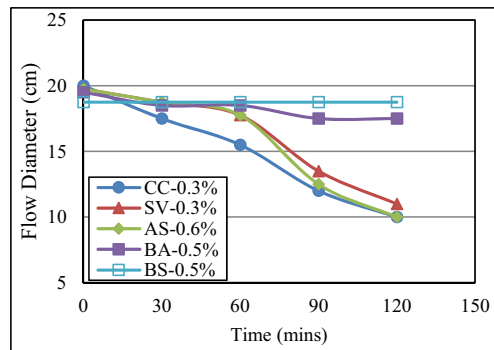


Fig. 12. Slump retention of PPC mortar mixture with SP dosage

Table 1. Initial and final setting time of PPC and OPC mortar with various SP addition for w/c=0.3

Superplasticizer	Type	PPC			OPC		
		Dosage (%)	Initial set (h)	Final set (h)	Dosage (%)	Initial set (h)	Final set (h)
Control	-	0	3.60	9.00	0	3.33	7.20
CC	PCE	0.3	5.75	11.50	0.4	5.75	8.78
SV	PCE	0.3	6.67	12.00	0.6	8.78	12.00
AS	PCE	0.6	6.00	11.20	0.6	6.00	10.00
BA	PCE	0.5	8.50	13.00	0.5	6.70	10.30
BS	PCE	0.5	12.70	18.00	0.4	12.00	16.67

4. Conclusions

From this study, the following conclusions can be summarized:

1. The addition of superplasticizer improves the flowability of mortar mixture. However, there is an optimum dosage for each water content. Excessive use of SP causes bleeding and segregation.
2. The increase of flowability increases the compressive strength up to a point. Optimum dosage is depending on the superplasticizer and cement type. Compressive strength will be reduced with excessive usage of SP.
3. The difference in flowability and strength gain are also affected by the type of cement used. PPC mortar has higher flowability compared to the one with OPC, but it has lower compressive strength for the same water-to-cement ratio.
4. Optimizing the superplasticizer dosage using flow table test and temperature evolution measurement give satisfactory information on the different performance amongst various SP brands.
5. Each SP brand has different behavior with regards to the optimum dosage, setting time, strength development and slump retention time. Hence, the use of different cement type needs to be carefully considered.
6. The variation of SP and cement type should be considered when doing the material selection for better prediction on the behavior of the fresh and hardened concrete mixture.

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References

- [1] R. Rixom & N. Mailvaganam, Chemical Admixtures for Concrete. E & FN Spon Publication, III Edition (2003).
- [2] H. Okamura & M. Ouchi, Self-Compacting Concrete. J. of Adv. Con. Tech., 1 (1) (2003) 5–15.
- [3] E. Sakai, K. Yamada, A. Ohta, Molecular structure and dispersion-adsorption mechanisms of comb-type superplasticizers used in japan, J. of Adv. Con. Tech., 1 (1) (2003) pp 16-25, <http://doi.org/10.3151/jact.1.16>.
- [4] F. Kong, L. Pan, C. Wang, D. Zhang, N. Xu, Effects of polycarboxylate superplasticizers with different molecular structure on the hydration behavior of cement paste, Con. & Build. Mat., 105 (2016) 545-553. doi:10.1016/j.conbuildmat.2015.12.178.
- [5] A. K. H. Kwan & W. W. S. Fung, Effects of SP on Flowability and Cohesiveness of Cement-Sand Mortar. Con. & Build. Mat., 48 (2013) 1050–57. doi:10.1016/j.conbuildmat.2013.07.065.
- [6] J. Gołaszewski, & J. Szwabowski, Influence of superplasticizers on rheological behaviour of fresh cement mortars. Cem. & Con. Res., 34 (2004) 235–248. doi:10.1016/j.cemconres.2003.07.002.
- [7] V. Morin, F. Cohen Tenoudji, A. Feylessoufi, & P. Richard, Superplasticizer effects on setting and structuration mechanisms of ultrahigh-performance concrete. Cem. & Con. Res., 31 (2001) 63–71. doi:10.1016/S0008-8846(00)00428-2.
- [8] S. Srinivasan, S. A. Barbhuiya, D. Charan & S. P. Pandey, Characterising cement-superplasticiser interaction using zeta potential measurements. Const. & Build. Mat., 24 (12) (2010) 2517–2521. doi:10.1016/j.conbuildmat.2010.06.005.
- [9] K. Yamada, S. Ogawa, S. Hanehara, Controlling of the adsorption and dispersing force of polycarboxylate-type superplasticizer by sulfate ion concentration in aqueous phase, Cem. & Con. Res., 31 (3), 2001, Pp 375-383, [http://dx.doi.org/10.1016/S0008-8846\(00\)00503-2](http://dx.doi.org/10.1016/S0008-8846(00)00503-2).
- [10] E. Tkaczewska, Effect of the superplasticizer type on the properties of the fly ash blended cement. Const. & Build. Mat., 70 (2014) 388–393. doi:10.1016/j.conbuildmat.2014.07.096.
- [11] ASTM C778. Standard Specification for Standard Sand. ASTM International, 2002.
- [12] ASTM C230 / C230M-14, Standard Specification for Flow Table for Use in Tests of Hydraulic Cement, ASTM International.
- [13] ASTM C1679 – 14, Standard Practice for Measuring Hydration Kinetics of Hydraulic Cementitious Mixtures Using Isothermal Calorimetry, ASTM International.