Dispersion Effectiveness of Organic Plasticizers

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Abstract. Centrifugation method to study rheological effectiveness of organic plasticizers in cement suspensions is presented. Cement suspensions modified with different doses of two types of superplasticizers were tested. It was determined that the critical shear stress of tested suspensions depends largely on plasticizer type and dose.

INTRODUCTION

A key point in improvement processability of cement suspensions is the dispersion of agglomerated cement particles within a mixture. Dispersion efficiency largely depends on superplasticizer (SP) playing a role of cement dispersant. Regardless significant effort focused on this topic (most currently in [1]), there is still uncertainty in reproducibility and appropriateness of analytical methods in this area of research [2] resulting in discrepancies among results reported. This study aims to evaluate the efficiency of SP with centrifugation method, which represents an alternative to traditional rheological approaches.

EXPERIMENTAL

Two types of suspensions were prepared (Tab 1) and combined with two SPs: sulphonated melamine-formaldehyde condensate Melment F10 X (MF) and polycarboxylate ether Gecedral Fluid 10.1 (PC). Using Size Exclusion Chromatography it was possible to indentify two main fractions of the dispersants: 55 % of fraction with a molecular weight of 350 000 g/mol and 30 % of fraction with a molecular weight of 9000 g/mol for MF, 50 % of fraction with a molecular weight of 80 000 g/mol and 50 % of fraction with a molecular weight of 14 000 g/mol for PC plasticizer.

TABLE 1. Basic parameters of cemented mixtures

Cement	Mineralogical composition (%)				Specific surface area (m^2/g)	
	C_3S	C_2S	C_3A	C_4AF	Blaine	BET
Cement S	65.31	8.7	9.1	16.89	0.516	1.80
Cement M	70.10	12.60	2.8	14.50	0.499	1.71

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The suspensions for the measurement were prepared in the following way: 13.5 grams of water/SP solution and 13.5 grams of cement were placed in a glass cell. The cell with suspension was immersed in an ultrasonic bath for 15 minutes in order to secure the interaction of organic molecules with the surface of solid particles, dispersion of agglomerates of fine particles and even distribution of all fractions of grainy particles throughout the suspension. Centrifugation of the cells with suspensions was conducted immediately after homogenization (Centrifugal machine Rotofix 32, Hettich Zentrifugen). The time of centrifugation was three minutes and different rotation speeds were used. After centrifugation, the separated fraction of water was carefully poured into a Petri dish. The weight of the cell with the remaining amount of the specimen and that of the Petri dish with separated solution (containing a small amount of ultra fine particles) were determined. The weight of separated water and the content of solids in it were calculated from the weights of the Petri dish before and after 25 minutes of drying at 105 °C. Accordingly, the water/solid ratio (*W/S*) was calculated

$$W / S = \frac{m_l - m_d}{m_s} \quad (1)$$

m_d – mass of separated water/additive solution after centrifugation (cm^3)
m_s, m_l – mass of solid and liquid phases in suspensions before centrifugation (cm^3).

To determine the critical shear stress (shear stress on contact surface) a penetration test was used. The cone of especially developed equipment was pushed into the mixture after centrifugation and the penetration depth was measured. The following Equations (2-3) and parameters were employed to determine efficiency of SP:

$$P = F\sigma_x \quad (2)$$

$$\tau = \frac{P}{v^2}\frac{1}{\pi}\cos^2\alpha \cot g\alpha \quad (3)$$

P – compacting force (N)
σ_x – normal stress on the frontal surface of specimen element (N/cm^2)
τ - shear stress on contact surface (N/cm^2)
F – cross-section of cell (cm^2)
v – penetration depth of the cone in a mixture after centrifugation (cm)
α – angle of the penetration depth of the cone in a mixture after centrifugation (°).

RESULTS

Efficiency of two types of superplasticizers in cement S is compared via critical shear stress as a response variable in Fig. 1. The decrease of the shear stress to the minimum (critical) value indicates the composition/condition, where the additive (SP) is the most effective. The obtained results reveal that decrease in shear stress is affected more pronouncedly if polycarboxylate ether (PC) superplasticizer is used. Figure 1 indicates that a maximum drop in shear stress is possible when PC dose reaches 0.6 %. This concentration is related to the equilibrium between external and repulsive forces of charged particles originated as a consequence of sorption of additive molecules on their surfaces.

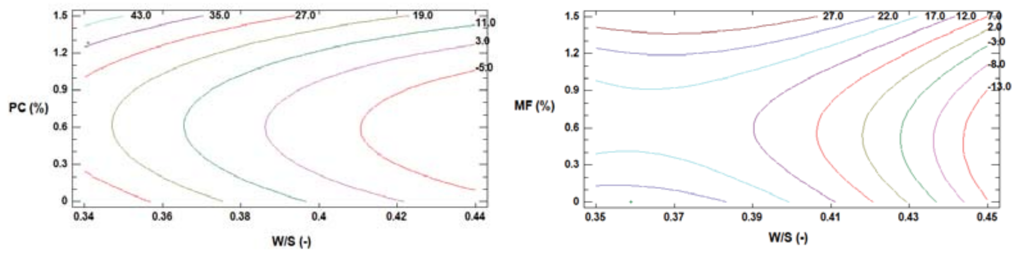

FIGURE 1. Influence of the factors W/S and PC (a) and MF (b) dose on the response variable τ (numbers at lines) for cement S.

Rheological effectiveness of SPs on various cements can be depicted from Fig.2. As can be seen, lower critical shear stress is achieved with PC additive for suspensions based on cement M as well.

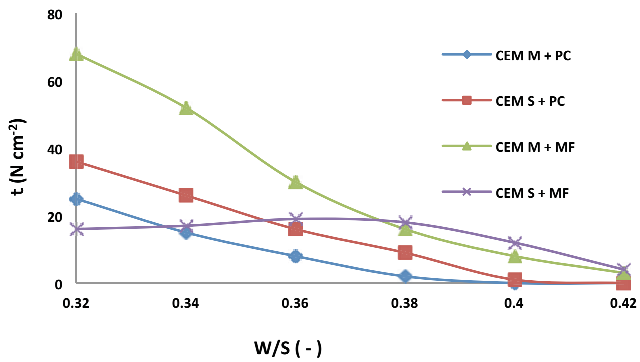

FIGURE 2. Relationship between τ and W/S for different types of additives and cements

CONCLUSIONS

Centrifugation method was used for determining the rheological effectiveness of polymer superplasticizers in cement suspensions. An abrupt increase of additive molecules in the liquid phase of suspension after the maximum sorption on the surface of cement particles changes the proportion between internal and external forces after centrifugation. Shear stress is reduced to rather low values due to the dispersion of the flocculated cement particles. In this respect, polycarboxylate ether plasticizer is more effective than sulphonated melamine-formaldehyde.

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