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INFLUENCE OF VISCOSITY ENHANCING AGENT ON RHEOLOGY AND COMPRESSIVE STRENGTH OF SUPERPLASTICIZED MORTARS

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Abstract. The results of investigation into the influence of Viscosity Enhancing Agents (VEA) on the rheological properties of mortars, different in cement and superplasticizer properties, are presented and discussed. Rheology results have been evaluated according to the Bingham model, which describes the rheology with 2 parameters: yield value and plastic viscosity. Rheological parameters were measured using Two-Point Workability Test (TPWT). Additionally, the influence of VEA on compressive strength of mortars is presented. It was found that effects of VEA addition on rheological properties of mortars, as well as on compressive strength of mortars, significantly depend on cement and superplasticizer properties. Basic relation of influence of VEA on rheological properties and compressive strength of mortars in respect to cement and superplasticizer properties were presented and discussed. It is concluded that addition of VEA influences compatibility between cement and superplasticizer. Because addition of VEA may cause decrease in compressive strength, the assessment of compatibility of VEA - cement - superplasticizer system should be performed in relation not only to rheological properties, but also to compressive strength.

Keywords: cement, compressive strength, concrete, mortar, rheology, superplasticizer, viscosity enhancing agent.

1. Introduction

The definition of workability of cement binder mixtures, such as fresh mortars and concretes, should be considered in terms of the system's state (i.e. the composition of the mixture and the method of processing). This state is determined by the relationship between 2 factors: the rheological properties of a given cement binder mixture and the forces acting on it during processing (Szwabowski 1999). The rheological properties are determined by the mixture's composition. They characterize its stress-strain behaviour. Therefore, workability of cement binder mixture is determined by the reaction of this mixture on the forces acting on it during transport and mechanical processing as the resistance of its structure to these forces. A required workability can be achieved in 2 ways: rheological properties of mixture are adjusted to the given method and conditions of processing or method and conditions of processing are adjusted to given rheological properties of cement binder mixture. In practice the first method is used. Thus, the condition sine qua non in workability of cement binder mixtures designing is the knowledge of the relationships between composition and constituents characteristics, as well as rheological parameters of mixture.

The main objective of using viscosity enhancing agents (VEA) is to eliminate or to reduce washout, settlement, segregation and bleeding of cement-based materials (Domone 2006; EFNARC 2006; De Schutter *et al.* 2008). Their usage is commonly recommended for increasing stability of high performance and self-compacting mortars and concretes. Incorporating the VEA in mortar or concrete can enhance cohesiveness and stability of these mixtures without interfering in their base proportioning.

Until now the number of the systematic experimental data on influence of VEA on properties of both fresh and hardened mortars and concretes has been increased and main effects of VEA addition are generally defined (Ed. Wallevik and Nielsson 2003; Holschemacher 2004; Lachemi *et al.* 2004; Skripkiūnas and Daukšys 2004; Ed. Yu *et al.* 2005; D'Aloia Schwartzentruber *et al.* 2006; Ed. Malhotra 2006; Sonevi 2006; Leemann and Winnefeld 2007; De Schutter *et al.* 2008; Paiva *et al.* 2009). However, in literature still is felt the insufficiency of systematic research on influence of VEA on rheology of fresh cement mixtures with different cements and superplasticizers

In this paper the methodology and the results of investigation into the influence of VEA type and content on the rheological properties and on the compressive strength of mortars with different cements and superplasticizers are presented and discussed. It was stated (Gołaszewski 2006) that the nature of rheological behaviour of fresh mortar and concrete is similar, and that mortars can be considered to be a model concrete. The knowledge of influence of chemical admixtures on mortars rheology may contribute to the understanding of rheological behaviour of fresh concrete, and also lead to the possibility of predicting the flow properties of the latter from small scale tests on mortars. Thus, the general relationships presented in this paper may relate to concrete, too.

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2. Rheological model and measurements of rheological parameters of fresh mortars

It is well documented that fresh mortar and fresh concrete behave as Bingham material, whose properties can be expressed by 2 rheological parameters, the yield stress and the plastic viscosity, according the formula

$$\tau = \tau_o + \dot{\gamma}\, \eta_{pl}, \quad (1)$$

where τ (Pa) is the shear stress at shear rate $\dot{\gamma}$ (1/s), τ_o (Pa) – the yield value and η_{pl} (Pa.s) – the plastic viscosity (Szwabowski 1999; Li 2007; Petit *et al.* 2007; Feys *et al.* 2008). The physical interpretation of yield value is that of the stress needed to be applied to a material in order to start flowing. When the shear stress is higher than a yield value, the mix flows and its flow resistance depends on plastic viscosity.

Rheological parameters of fresh mortar, like those of fresh concrete, can be measured using Two Point Workability Test (TPWT), by applying a given shear rate and measuring the resulting shear stress. Because of the nature of rheological behaviour of cement mixtures, the measurements should be taken at no less than 2 considerably different shear rates. The rheological parameters are determined by regression analysis according to the relation:

$$T = g + N\, h, \quad (2)$$

where T is the shear resistance of a sample measured at rotation rate N and *g* (N.mm) and *h* (N.mm.s) are constants corresponding respectively to yield value τ_o and plastic viscosity η_{pl}. By a suitable calibration of the rheometer, it is possible to express *g* and *h* in fundamental units. According to (Banfill 1991), in the apparatus like used in this work, $\tau_o = 7.9\, g$ and $\eta_{pl} = 0.78\, h$, but all results are given below in terms of parameters *g* and *h*. The principles of TPWT and rheological properties of fresh cement mortars and concretes are presented in the existing literature (Tattersall and Banfill 1983).

It should be noted that rheological properties of cement pastes and cement binder mixtures (mortars and concretes) differ from each other. During the flow test cement paste reveals plastic characteristics with a high degree of non-linearity and with high ticsotropic effects (Szwabowski 1999). As far as Bingham model is adequate to characterize rheological properties of fresh mortar and concrete, characterization of rheological properties of cement paste demands more complex models. It was demonstrated (in Atenzi *et al.* 1985) that rheological properties of cement paste are best described by the following models: Herschel-Bulkley, Robertson-Stiff and Ellis model. At the same time it was stated that Bingham model may be used for characterizing the properties of cement paste only in a narrow range.

3. Experimental

3.1. Testing program

The research program is presented in Table 1. In the first part of research cement type was kept constant (cement #1 was used in this part of research) and influence of VEA on properties of mortars with different the superplasticizers was investigated. In the second part, superplasticizer type was kept constant (SP1 was used in this part of research) and influence of VEA on properties of mortars with different cements was investigated. In this research, the following factors were taken into consideration:

- VEA type — VEA1, VEA 2 (Table 2);
- VEA content — 0, 0.2, 0.4, 0.6 %;
- cement properties — cements #1, #2, #3 different in C_3A and Na_2O_{eq} (Table 3);
- SP type — polyether superplasticizers SP1, SP2, SP3 different in molecular structure (Table 4).

Superplasticizers moment of adding (with water), w/c ratio (0,40), sand grading and type (CEN standard sand), cement to sand ratio (1/3) and temperature (20 °C) were kept constant in the tests. Superplasticizers were selected because of their different influence on rheological properties of cement mixtures (Gołaszewski 2006). Content of superplasticizer was kept constant in each part of research and established experimentally to obtain flowable mortars. It was 1% in first part and 3% in the second part of research (to obtain flowable mortar with 12% C_3A cement a considerably higher dosage of superplasticizer was necessary than in case of mortars with 2% C_3A cements). VEA dosage was adopted according to the manufacturer's recommendation.

3.2. Materials and mixes

Properties of viscosity enhancing agent, cements and superplasticizers used for the investigations are presented in Tables 2, 3 and 4. Cements were laboratory prepared by Mineral Building Materials Institute in Cracow. In order to eliminate the influence of type and grading of sand on rheological properties of mortars, the sand used was EN 196-1:1994 CEN model sand (2 mm max). The mixture proportions of mortars were based on standard mortar proportioning according to EN 196-1:1994 but with w/c ratio changed to 0.40. Mixture proportions are shown in Table 5.

3.3. Mortar mixing and testing procedures

The mixer and mixing procedure were in accordance with PN EN 196-1:1996; superplasticizers were added with water and VEA 15 s before the mixing end. After mixing, the samples of mortars were transferred to Viskomat PC rheometer (Fig. 1) and tested according to the procedure

Table 1. Research program

Part of research	VEA type	VEA content	SP type	Cement type
VEA – superplasticizer interaction	VEA 1, VEA2	0.2, 0.4, 0.6%	SP1, SP2, SP3	#1
VEA – cement interaction	VEA1	0.2, 0.4, 0.6%	SP1	#1,#2,#3

Table 2. Properties of VEA

VEA	Base component	Form	[g/cm^3]
VEA1	High molecular weight polysaccharide	powder	Bulk density – 0.80 g/cm^3
VEA2	High molecular weight polysaccharide	liquid	Density – 1.12 g/cm^3

Table 3. Properties of cements

Cement	Constituents [%]						Specific surface [m^2/kg]
	C_3S	C_2S	C_3A	C_4AF	SO_3	Na_2O_{eq}	
Cement #1 (NA)	59.6	19	2	16.6	3.0	0.3	370
Cement #2 (HA)	59.6	19	2	16.6	3.0	1.1	370
Cement #3 (NA)	57.9	15	12	9.5	3.0	0.3	370

Table 4. Superplasticizers properties

SP	Base component	Density [g/cm^3]	Concentration [%]
SP1	polyether (low molecular weight, short side chains)	1.09	18
SP2	polyether (low molecular weight, long side chains)	1.05	34
SP3	polyether (high molecular weight, long side chains)	1.05	36

Table 5. Mortar composition (g/batch)

Cement	Sand	w/c	SP
450	1350	0.45	4.5 or 13.5

Table 6. Measuring procedure used in tests

- Start of measurement.
- Speed 120 rev/min held constant for 10 min.
- Measurement of M at decreasing N = 120-100-80-60-40-30-20 1/min. Test time 70 s.
- Speed held constant for 39 min at 60 rev/min.
- Increase of speed to 120 rev/min.
- Speed 120 rev/min held constant for 10 min.
- Measurement of M at decreasing N = 120-100-80-60-40-30-20 1/min. Test time 70 s.
- End of measurement.

Total test cycle time 3620 s.

Fig. 1. Viskomat PC and its measuring element

in Table 6. This procedure roughly simulates the process of transport of concrete mixture in a truck concrete mixer. Because the measurement at the constant velocity of the impeller rotation enables only the investigation of shear resistance (which at a given speed consists of yield value and plastic viscosity), at 10 and 60 minutes the rotation speed was changed from 120 to 20 1/min to define the rheological parameters from flow curves.

The rate of sample segregation during measurement was checked testing differences in grading between upper, bottom, inner and outer parts of a sample. It was stated that differences in sample grading was in most cases lower than 5% and never exceed 10%. Thus, basing on the obtained results, it is possible to state that stability of mortar is high.

The correlation coefficients calculated from the flow curves used to determine rheological parameters of the mixes was in range of 0.95–0.99. The mean relative errors of yield value g and plastic viscosity h determination were respectively 4.7% and 7.3%. That proves that Bingham model is adequate to describe rheological properties of mortars. In the research, effects of segregation were not observed.

Samples for compressive strength testing were moulded according to PN EN 196-1:1996 after the end of rheological test. These samples were stored and tested according to PN EN 196-1:1996.

4. Test results and discussion

Relations of influence of viscosity enhancing agent (VEA) content on yield value *g* and plastic viscosity *h* of fresh mortars different in superplasticizer and cement type are presented in Figs 2, 3 and 4.

It can be seen that an addition of VEA significantly influences yield value *g* and plastic viscosity *h* of mortars and range of its changes with time.

In general, increasing VEA1 content causes linear increase of yield value *g* of mortars. However, for mortars with cements containing 2% C_3A and SP1, yield value *g* increases only until a certain maximum. Further increasing of VEA1 content may cause slight decrease of yield value *g* of these mortars.

Yield value *g* of mortars without VEA1 increases with time. It is worthy to underline, that addition of VEA1 may change direction of changes of yield value *g* with time. In case of mortars with cements #1 and #2 (containing 2% C_3A), independently on type of superplasticizer, yield value *g* clearly decreases with time. The range of this changes is generally independent on VEA1 content in mortar. Such nature of changes in yield value *g* with time is generally unbeneficial from point of view of mixture stability, however, one can say that it improves workability retention with time of the material. In case of mortars with cement #3 containing a 12% C_3A, addition of VEA1 does not influence direction and range of changes of yield value *g* with time.

Nature and range of influence of VEA1 content on plastic viscosity *h* of mortars also depends on the cement properties and on the superplasticizer type. In the case of mortar with SP1 superplasticizer, which is characterized by lowest value of plastic viscosity *h*, addition of VEA1 causes directly proportional to VEA1 content increase of plastic viscosity *h*. The exception of the rule is mortar with cement #2 (2% C_3A, 1.1% Na_2O_e); in that case plastic viscosity *h* slightly decreases with increasing VEA1 content. In the case of mortars with SP2 and SP3 superplasticizers, addition of VEA1 causes initially decrease in plastic viscosity *h*. Fall in plastic viscosity *h* is especially high in the case of mortars with SP3 superplasticizer. Further increasing of VEA1 content no longer changes plastic viscosity *h*. It is worthy to note, that very significant differences in plastic viscosity *h* of mortars with SP1, SP2 and SP3 superplasticizers and without VEA almost disappear after VEA1 addition.

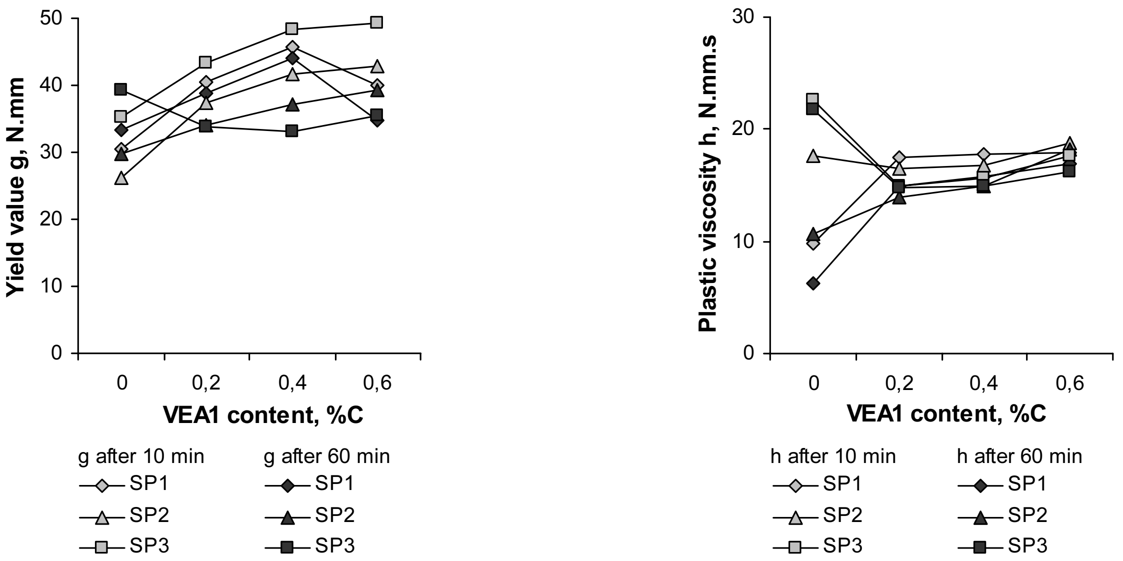

Fig. 2. Influence of VEA1 on rheological properties of mortars with cement #1 (C_3A = 2%, Na_2O_{eq} = 0.3%) and addition of 1% SP1, SP2 or SP3 superplasticizer (properties acc. Table 4)

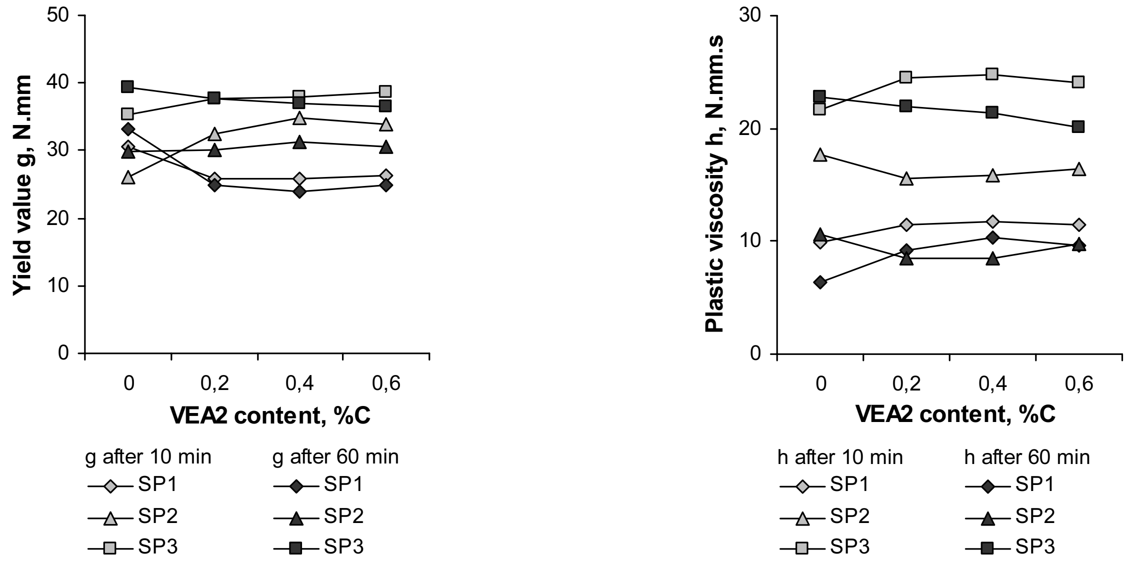

Fig. 3. Influence of VEA2 content on rheological properties of mortars with cement #1 and addition of 1% SP1, SP2 or SP3 superplasticizer

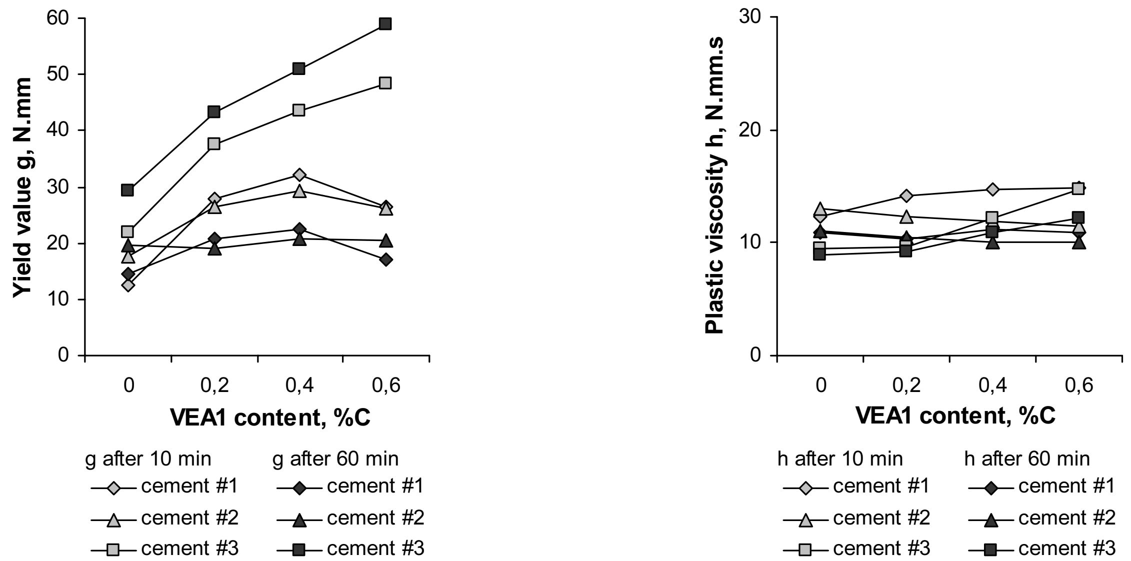

Fig. 4. Influence of VEA1 content on rheological properties of mortars with cement #1(C_3A = 2%, Na_2O_{eq} = 0.3%), cement #2 (C_3A = 2%, Na_2O_{eq} = 1.1%)or cement #3 (C_3A = 12%, Na_2O_{eq} = 0.3%) and addition of 3% of SP1 superplasticizer (properties acc. Table 4)

The range of changes in plastic viscosity *h* with time is significantly influenced by the VEA1 content, cement and superplasticizer properties. However, on the ground of obtained results it is difficult to point out unequivocal tendencies.

The influence of VEA2 admixture on yield value *g* is distinctly lower than the one of VEA1 admixture. This value *g* of fresh mortars slightly increases (mortars with SP1 and SP3 additives) or decreases (mortars with SP2 additives) with the increase of VEA2 content. Introducing VEA2 admixture causes yield value *g* to have a distinct tendency to decrease with the passing time, yet lower than in case of mortars with VEA1 additives.

In case of fresh mortars with SP1 and SP3, adding VEA2 admixture causes the increase of plastic viscosity *h*. In case of fresh mortars with SP2 superplasticizer, the usage of VEA2 admixture causes slightly decrease of plastic viscosity *h*. VEA2 addition does not influence the changes of plastic viscosity *h* in time of mortars with SP1 and SP2, but accelerate in case of mortar with SP3 superplasticizer.

Relations of influence of VEA content on 7 and 28 days compressive strength of these mortars are presented in Fig. 5, 6 (VEA1) and Fig. 7 (VEA2).

The obtained results show that character and range of influence of VEA on compressive strength of mortars depend on VEA type, cement properties and superplasticizer type, yet in this case it is difficult to determine systematic trends. Addition of VEA1 usually causes a decrease in compressive strength of mortars both after 7 and

28 days. Decrease in compressive strength increases with increasing VEA1 content and extremely may reach even 50%. However, that unbeneficial effect of VEA addition was not found in the case of mortars with cement containing low C_3A and high alkali amount (cement #2). In that case addition of VEA1 increases compressive strength of mortars. Influence of VEA2 on compressive strength is clearly lower; addition of VEA2 only slightly decreases or does not influence compressive strength of mortars.

5. Conclusions

Stabilizing effect of VEA on fresh mortar (and concrete) depends on increase in yield value *g* and plastic viscosity *h* of these mixtures. The presented above results show, that rheological effects of addition of high molecular weight polysaccharide-based VEA are strongly dependent on cement and superplasticizer properties. Depending on these factors, VEA shows different effectiveness of action, producing different range of yield value *g* and plastic viscosity

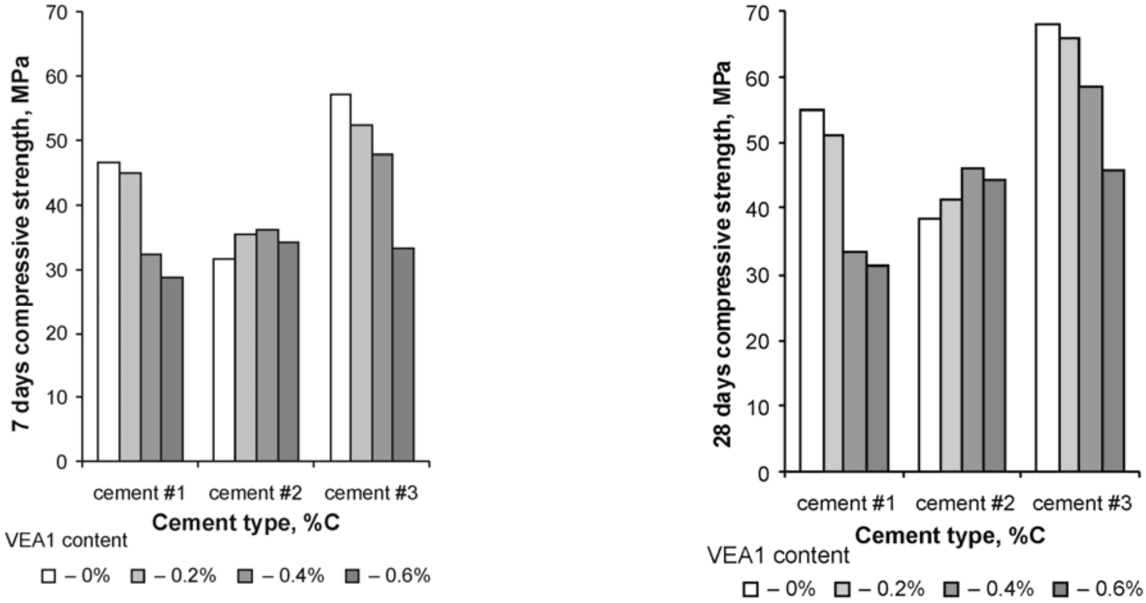

Fig. 5. Influence of VEA1 content on 7 and 28 days compressive strength of mortars with cement #1(C_3A = 2%, Na_2O_{eq} = 0.3%), cement #2 (C_3A = 2%, Na_2O_{eq} = 1.1%) or cement #3 (C_3A = 12%, Na_2O_{eq} = 0.3%) and addition of 3% of SP1 superplasticizer (properties acc. Table 4)

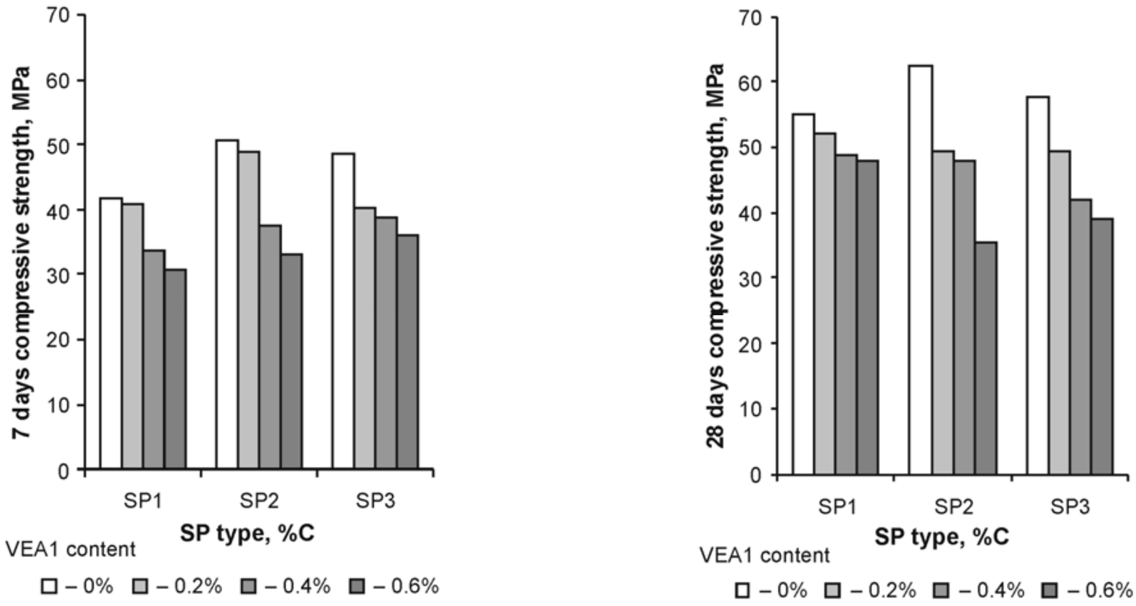

Fig. 6. Influence of VEA1 content on 7 and 28 days (b) compressive strength of mortars with cement #1 (C_3A = 2%, Na_2O_{eq} = 0.3%) and addition of 1% SP1, SP2 or SP3 superplasticizer (properties acc. Table 4)

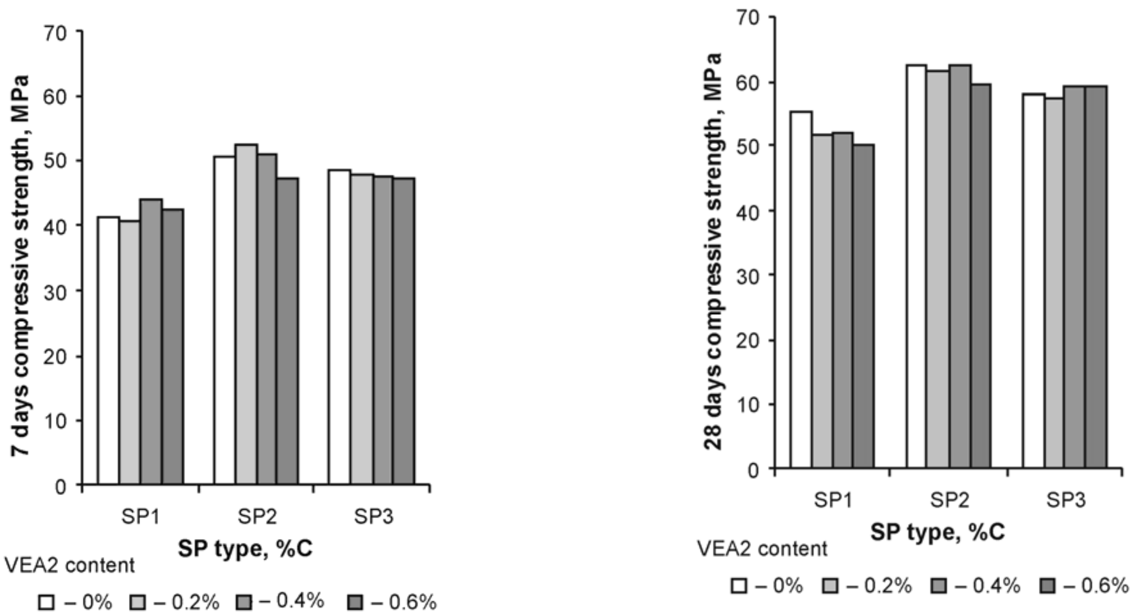

Fig. 7. Influence of VEA1 content on 7 and 28 days (b) compressive strength of mortars with cement #1 (C_3A = 2%, Na_2O_{eq} = 0.3%) and addition of 1% SP1, SP2 or SP3 superplasticizer (properties acc. Table 4)

h changes. It is also very important to notice, that sometimes effects of VEA addition may be different from the expected ones and a decrease in plastic viscosity *h* may occur after VEA addition.

Addition of VEA influences the rate and direction of changes of rheological parameters of mortars with time; however, it is difficult to point out equivocal trends. It is worthy to note, that in most cases yield value *g* of superplasticized mortars with VEA decreases with time passing. Therefore the flowability of these mortars increases with time. On the other hand, it should be underlined, that this effect is generally unbeneficial from the point of view of the main demanded effects of VEA action.

Influence of VEA on compressive strength of mortars varies with VEA type and superplasticizer and cement properties; in that case it is difficult to point out unequivocal trends. However, it should be noted that addition of VEA may cause substantial decrease of compressive strength of mortars after 7 and 28 days.

Addition of VEA has a significant impact on assessment of the compatibility between cement and superplasticizer. Thus, compatibility between cement and superplasticizer should be tested taking into account VEA type and content.

The presented results show, that it is necessary to experimentally define effects of VEA on properties of fresh and hardened mortars. In case of rheological effects of VEA, such investigation should be performed by the rheometrical method.

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KLAMPĄ DIDINANČIŲ PRIEDŲ ĮTAKA SUPERPLASTIFIKUOTŲ SKIEDINIŲ REOLOGIJAI IR STIPRIUI GNIUŽDANT

J. Golaszewskij

Santrauka

Straipsnyje pateikta ir išanalizuota klampą didinančių priedų įtaka skiedinių reologinėms savybėms. Skiediniai buvo ruošiami su įvairių savybių cementais ir superplastikliais. Reologijos tyrimų rezultatai įvertinti remiantis Binghamo modeliu. Reologinės savybės buvo tiriamos dviejų parametrų reometru (TPWT). Taip sudarytas naturalus šviežio skiedinio ir betono mišinio reologinės elgsenos būvis. Straipsnyje taip pat pateikti klampą didinančių priedų įtakos betono stipriui giuždant rezultatai. Nustatyta, kad klampą didinančių priedų poveikio efektas skiedinių reologinėms savybėms ir stipriui gniuždant iš esmės priklauso nuo cemento ir superplastiklių savybių.

Reikšminiai žodžiai: cementas, stipris gniuždant, betonas, skiedinys, reologija, superplastiklis, klampą didinantis priedas.

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