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THE EFFECT OF SYNTHETIC ZEOLITE ON THE VISCOSITY OF CEMENT PASTE

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Abstract. The paper reports the effect of the synthetic zeolite admixture on the viscosity of cement paste. Materials include Portland cement CEM I 42.5 R, synthetic zeolite obtained through low-temperature synthesis under laboratory conditions, polycarboxylate ester based supeplasticizer Muraplast FK 63.30 and tap water. Three compositions of cement paste were made adding 0%, 5%, and 10 wt% of synthetic zeolite. A rotating cylinder viscometer was used for measuring the consistence of the paste; viscosity was measured employing vibrational viscometer SV–10. The consistency test on cement paste showed that spread with no addition of synthetic zeolite made 149 mm, containing 5% of synthetic zeolite the slump was 85 mm and containing 10% of synthetic zeolite spread was 70 mm. The viscosity test also indicated that for the period of 30 minutes, changes in the viscosity of cement paste were as follows: 81.11 mPa s with no addition of synthetic zeolite, 746.5 mPa s containing 5% of synthetic zeolite and 827.1 mPa s containing 10% of synthetic zeolite. Data on the viscosity of cement paste were statistically processed applying software Statistica. Dispersion diagrams demonstrate changes in the viscosity of cement paste adding from 0 to 10 wt% of synthetic zeolite. Taking into account synthetic zeolite, the following correlation coefficients were calculated: 0% – 0.938, 5% – 0.967, 10% – 0.988. The coefficients of determination made 0% – 0,880, 5% – 0,935, 10% – 0,976. Equations for a dependent variable were produced by regression analysis.

Key words: synthetic zeolite, cement paste, viscosity, Portland cement, binder, super plasticizer.

1. Introduction

In the last decade, great attention has been paid to the re-use of industrial waste such as ash, silica micro-particles, etc. Along with other countries, Lithuania has been developing industrial technologies for recycling waste. The appropriate re-use of waste increases the economy of raw materials in the production of construction materials.

Modern cementing materials contain various additives, plasticizers and modifying admixtures, which in some cases may improve the properties of construction materials.

Researchers have found that the incorporation of zeolites, together with super plasticizers, affect rheological properties of concrete (Sahmaran *et al.* 2008). Feng *et al.* (1988) state that the incorporation of 5–10 wt% of zeolite, including super plasticizer, improves the strength of concrete up to 15%. The experiment described in this paper employed synthetic zeolite (modification A) obtained in the laboratory of Kaunas University of Technology by means of low-temperature synthesis (below 105 °C). We found that synthetic zeolite A contained 28.6% of SiO_2, 37.9% of Al_2O_3 and 14.1% of Na_2O.

The first attempts to synthesize zeolite were made in 1756 by Swedish mineralogist A. F. Cronstedt. In 1862, Saint Claire de Ville tried to synthesize zeolite imitating natural geological conditions that enabled the formation of zeolites. In 1948, Barrer declared his successful attempts to obtain a synthetic analogue identical to natural zeolite. In 1950, Milton representing Union Carbide Corporation synthesized zeolites using alkali silica alumina gels. In 1956, synthesized zeolite A became commercially available. In the early

60's, Barrer and Denny were the first to start synthesizing zeolites using organic instead of alkali metal cations. In 1962, Mobil Oil introduced synthetic zeolite X for cracking catalysers. In 1978, Grose and Flanigen patented zeolyte synthsis (Houssin Christophe 2003). According to the data presented by International Zeolite Association, the number of synthesized zeolites has been rapidly increasing. In 1970, there were 27 synthesized zeolites, in 2001 – the number reached 133 and today we can count more than 180 of those (Georgiev *et al.* 2009). Synthesized zeolites are used more frequently compared to natural zeolites because they are cleaner and have a more uniform particle size. It should be also noted that synthetic zeolites are obtained from industrial waste and therefore meet requirements for recycling waste. Synthetic zeolite has the following structure (Georgiev *et al.* 2009):

$$Me_{x/n}\,[(Al_2O_3)_x(SiO_2)_y]\cdot wH_2O, \quad (1)$$

where Me is an element from the periodic table groups 1 or 2 having valence n, x and y – numbers from 1 to 5 depending on zeolite structure w is the number of water molecules.

Manufacturing construction materials with the incorporation of zeolites is developed in the following directions: binders, concrete and non-autoclaved silicate products (Nan *et al.* 2000).

Rheological properties of concrete depend on the amount of liquid phase (water) in mixes and on the form of links between water molecules. Free water has a great effect on rheological properties of the mix, whereas chemically and physically bound water has no effect (Skripkiūnas 2007).

The addition of super plasticizer in cement paste without changing the consistence of the paste significantly reduces water content (12%), or, if water content is not changed, improves the workability of the paste or both (Naujokaitis 2007). Palycarboxylate ester based superplasticizer was used in the experiment described in this paper.

This paper reports the effect of the synthetic zeolite admixture on the viscosity of cement paste.

2. Research Methodology

Three compositions of cement paste were used for testing the viscosity of cement paste adding 0%, 5% and 10% of zeolite. Superplasticizer Muraplast FK 63.30 was used for improving the workability of cement paste.

The superplasticizer was added to water and mixed for about 60 seconds. The mixed mass was added to the cement mixer together with Portland cement and synthetic zeolite. The mass was mixed in the planetary mixer Bear Veramixer until the required consistence was obtained. After mixing cement paste, consistence was measured applying Suttard viscometer made of a hollow metal cylinder and glass plate. The hollow metal cylinder is moistened and placed in the centre of the glass plate. The tested cement paste is poured into the cylinder. The surface of the paste is evened by a spatula. Then, the cylinder is lifted and the paste slumps on the glass plate. Spread is measured in two perpendicular directions and the mean value of both measures is calculated. The slump value in millimetres indicates the consistence of the paste.

The viscosity of cement paste was measured by Vibro Viscometer SV–10 (Fig. 1).

Fig. 1. Vibro viscometer SV-10

40 grams of cement paste was placed into a glass container fixed on the spring plate that lifted the container with the sample so that sensor plates immerse in the sample. The vibro viscometer was connected to a PC and software monitored progress in changes in viscosity and temperature over real time referring to graphs and numerical values. The test lasted for 30 minutes.

3. Materials

The following materials were used for conducting the test:

- Portland cement CEM I 42.5 R manufactured by *AB Akmenės cementas* and complying with

LST EN 197–1:2001 requirements; cement composition and characteristics include 95 – 100% of clinker and up to 5% of admixtures, specific density of 2.75 makes 3.20 g/cm^3, bulk density of 0,9 – 1,5 g/cm^3 and particle size of 5 – 30 μm;
- synthetic zeolite obtained from production waste of aluminium fluoride (AIF_3) produced by *AB Lifosa*; the composition of modified synthetic zeolite contains 29.3% of SiO_2, 36.5% of Al_2O_3, 7.3% of Na_2O and 7.5% of CaO; modified zeolite is a mix of zeolite A ($Na_{96}Al_{96}Si_{96}O_{384} \cdot 2164H_2O$) and calcium zeolite (Ca, Na) $Al_2Si_{2,5}O_9 \cdot 6,4H_2O$;
- polycarboxylic ester based superplasticizer Muraplast FK 63.30;
- water complying with LST EN 1008:2002 technical requirements.

Table 1 illustrates three compositions of cement paste.

Table 1. Compositions of cement paste

Synthetic zeolite content,%	0	5	10
Portland cement, g	2800	2660	2520
Water, g	756	756	756
Superplasticizer, ml	14	14	14
W/S	0.27	0.27	0.27
W/C	0.27	0.28	0.30

4. Analysis of Tested Results

Figure 2 illustrates the results of the slump test.

The results of the slump test were as follows: the average spread of the paste without zeolite additive was 149 mm, containing 5 wt% of zeolite – 85 mm

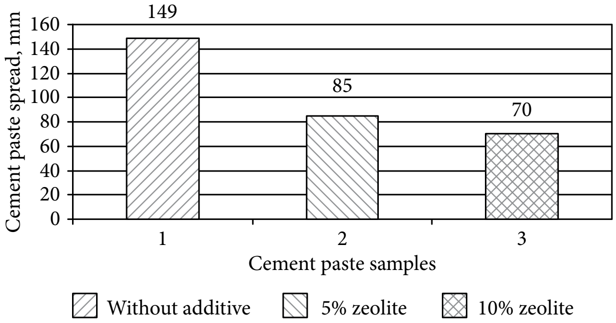

Fig. 2. The results of the spread of cement paste

and containing 10 wt% of zeolite – 70 mm. Thus, the spread of cement paste reduces along with an increase in the content of zeolite.

Figure 3 illustrates the results of viscosity test.

The graph of cement paste without adding zeolite shows an increase in the viscosity of cement paste by 81.11 mPa s during 30 minutes.

With an addition of 5 wt% to synthetic zeolite, the viscosity of the paste increased up to 746.5 mPas in 30 minutes. It should be noted that for 10 minutes the viscosity of cement paste containing 5 wt% of synthetic zeolite remains similar to the viscosity of the paste with no added zeolite (viscosity increases about 16 mPa s).

When 10 wt% of synthetic zeolite is added to cement paste, the viscosity of the paste increases up to 78 mPas in one minute and after 10 minutes continues increasing steadily similarly to the paste containing 5 wt% of zeolite. When 10 wt% of synthetic zeolite is added, the viscosity of cement paste increases up to 827.1 mPas in 30 minutes.

The vibro viscometer also recorded the following changes in temperature: in cement paste with no syn-

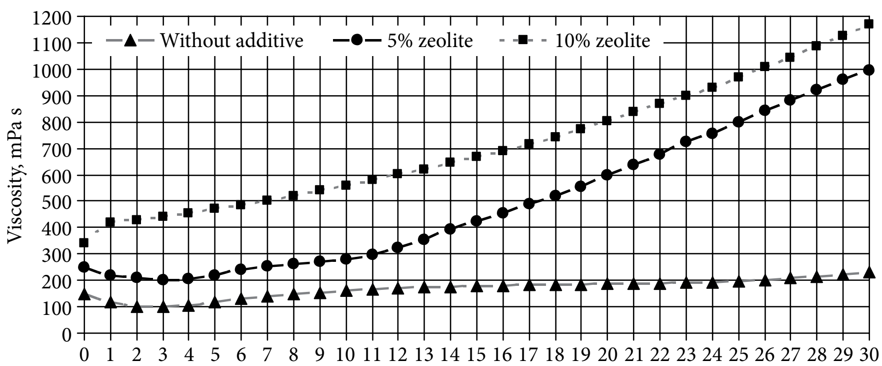

Fig. 3. The relationship between the viscosity of cement paste and time

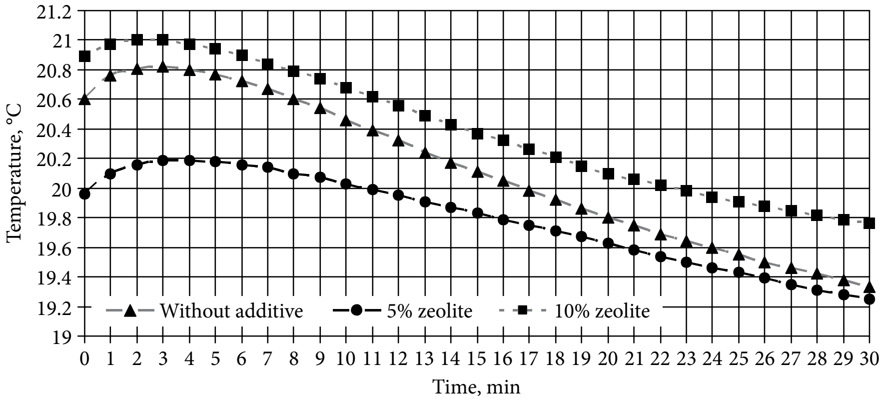

Fig. 4. Temperature changes in cement paste, °C

thetic zeolite temperature dropped up to 1.27 °C, in cement paste containing 5 wt% of zeolyte up to 0.71°C and in cement paste containing 10 wt% of zeolyte up to 1.13 °C.

Figure 4 illustrates time-temperature curves.

5. Statistical Analysis of Data

The obtained data on the viscosity of cement paste were statistically processed employing software *Statistica*. Time-viscosity diagrams presented in Figures 5 – 7 illustrate changes in cement paste containing 0%, 5% ir 10 wt% of synthetic zeolite. The values showing the strength of the relationship between variables, which are the most important measures of the adequacy of experimental data, namely the coefficients of correlation (R) and coefficients of determination (R^2), were established (Sakalauskas 2003).

Regression analysis produced dependent variable equations where x designates time in minutes (Equations 2–4). The coefficient of correlation in cement paste with no synthetic zeolite was 0.938 and the coefficient of determination was 0.880. A strong relationship between the viscosity of cement paste and time can be observed. An equation for the dependent variable can be used for calculating the viscosity of cement paste when the factor of time is known:

$$y = 116{,}602 \cdot \exp\,(0{,}023 \cdot x). \tag{2}$$

Figure 6 illustrates the relationship between viscosity and time in cement paste containing 5 wt% of synthetic zeolite. In this case, the coefficient of correlation was 0.967 and coefficient of determination was 0.935. The viscosity of cement paste can be calculated from Equation (3):

$$y = 176{,}483 \cdot \exp\,(0{,}059 \cdot x). \tag{3}$$

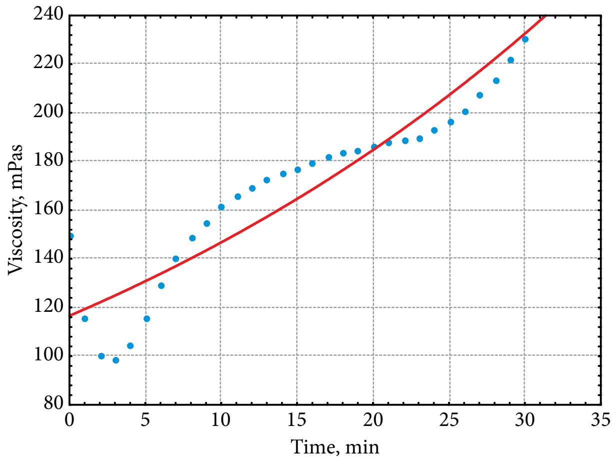

Fig. 5. The relationship between viscosity and time in cement paste with no synthetic zeolite

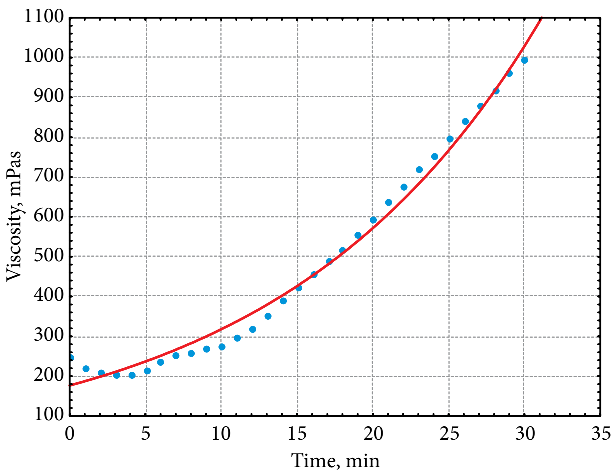

Fig. 6. The relationship between viscosity and time in cement paste containing 5 wt% of synthetic zeolite

Figure 7 illustrates a very strong relationship between viscosity and time in cement paste containing 10 wt% of synthetic zeolite: R = 0.988, R^2 = 0.976.

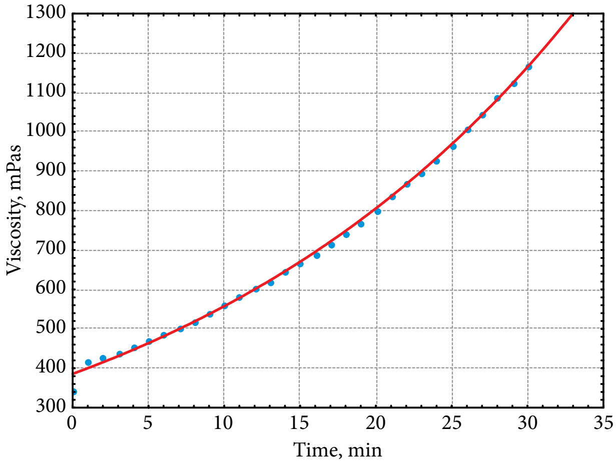

Fig. 7. The relationship of viscosity and time in cement paste containing 10 wt% of synthetic zeolite

When time is known the viscosity of cement paste can be calculated from Equation (4):

$$y = 385{,}802 \cdot \exp\,(0{,}037 \cdot x). \qquad (4)$$

6. Conclusions

1. The test on the consistence of cement paste has showed that a spread in cement paste containing no synthetic zeolite was 149 mm, containing 5 wt% of synthetic zeolite – 85 mm and containing 10 wt% of synthetic zeolite – 70 mm.
2. The test on viscosity has diclosed that within 30 minutes viscosity in cement paste containing no synthetic zeolite has changed into 81.11 mPa s, containing 5% of synthetic zeolite into 746.5 mPa s and containing 10 wt% of zeolyte in 827.1 mPa s.
3. Empirical equations (2–4) and coefficients of correlation and determination have been established with reference to regression analysis.

References

Feng, N. Q.; Yang, H. M.; Zu, L. H. 1988. The strength of mineral admixture on cement concrete, *Cement and Concrete Research* 18: 464–472.

Georgiev, D.; Bogdanov, B.; Angelova, K.; Markovska, I.; Yancho, H. 2009. Synthetic zeolites – structure, classification, current trends in zeolite synthesis review. Stara Zagora, Bulgaria, in *International Science conference Economics and Society Development on the Base of Knowledge*, 4–5 June 2009, Stara Zagora, Bulgaria.

Houssin Christophe, J. Y. 2003. *Nanoparticles in zeolite synthesis*. Eindhoven: Technische Universiteit Eindhoven. 105 p.

LST EN 197-1. 2001. Cementas. 1 dalis. Įprastinių cementų sudėtys, techniniai reikalavimai ir atitikties kriterijai.

LST EN 1008. 2002. Vanduo betonui. Techniniai reikalavimai ėminių ėmimo, bandymo ir tinkamumo reikalavimai, įskaitant grąžinamą iš gamybos betono pramonėje vandenį, pakartotinai naudojamą betono mišiniui ruošti.

Nan, S.; Hung-Yan, F.; Zong, H. C.; Fu-Shung, L. 2000. Reuse of wasre catalyst from petrochemical industries for cement subtitution, *Cement and Concrete Research* 30: 1773–1783. http://dx.doi.org/10.1016/S0008-8846(00)00401-4

Naujokaitis, A. 2007. *Statybinės medžiagos. Betonai*. Vilnius: Technika. 356 p. (in Lithuanian).

Sahmaran, M.; Ozkan, N.; keskin, S. B.; Uzal, B.; Yaman, I. O.; Erdem, T. K. 2008. Evaluation of natural zeolite as a viscosity-modifying agent for cement-based grouts, *Cement and Concrete Research* 38(7): 930–937. http://dx.doi.org/10.1016/j.cemconres.2008.03.007

Sakalauskas, V. 2003. *Duomenų analizė ir STATISTICA*. Vilnius: Margi raštai. 236 p.

Skripkiūnas, G. 2007. *Statybinių konglomeratų struktūra ir savybės*. Kaunas: Vitae Litera. 334 p. (in Lithuanian).

SINTETINIO CEOLITO PRIEDO ĮTAKA CEMENTO TEŠLOS KLAMPUMUI

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Santrauka. Šio darbo tikslas – nustatyti, kokią įtaką cemento tešlos klampumui turi sintetinio ceolito priedas. Naudotos šios medžiagos: portlandcementis CEM I 42,5 R, sintetinis ceolitas, gautas laboratorijoje atliekant žematemperatūrę sintezę, superplastiklis Muraplast FK 63.30 multikarboksilato esterio pagrindu ir vandentiekio vanduo. Buvo maišomos trijų sudėčių cemento tešlos dedant 0 %, 5 %, 10 % sintetinio ceolito priedo nuo cemento masės. Cemento tešlos konsistencijai nustatyti buvo naudojamas Sutardo viskozimetras, klampumui – vibroviskozimetras SV-10. Atlikus cemento tešlos konsistencijos bandymą nustatyta, kad be sintetinio ceolito priedo cemento tešlos pasklidimas – 149 mm, su 5 % sintetinio ceolito priedu – 85 mm, 10 % – 70 mm. Nustačius cemento tešlos klampumą gauta, kad be sintetinio ceolito priedo cemento tešlos klampumas per 30 minučių kinta 81,11 mPas, su 5 % priedo – 746,5 mPas, 10 % – 827,1 mPas. Cemento tešlos klampumo tyrimo duomenys buvo statistiškai apdorojami kompiuterine programa „Statistica“. Šiame darbe pateikiamos sklaidos diagramos, kuriose pavaizduota, kaip kinta cemento tešlos klampumas laiko atžvilgiu naudojant sintetinio ceolito priedą nuo 0 iki 10 %. Nustatyti korealiacijos koeficinetai: 0 % – 0,9380, 5 % – 0,9669, 10 % – 0,9881, bei determinacijos koeficientai: 0 % – 0,8798, 5 % – 0,9348, 10 % – 0,9763. Atlikus regresinę analizę buvo gautos priklausomybių lygtys.

Reikšminiai žodžiai: sintetinis ceolitas, cemento tešla, klampumas, portlandcementis, rišiklis, superplastiklis.

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