

THE INFLUENCE OF CALCIUM NITRATE ON THE PLASTICIZING EFFECT OF CEMENT PASTE

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Abstract. The rheological properties of the cement pastes have been determined using a rotational viscometer under a special testing routine by increasing and retaining a steady rotation speed of the viscometer cylinder up to certain values and for a certain time. These tests, using rotational viscometer, have been conducted by making the assessment of cement paste flow curves, using Bingham rheological model. Rheological properties of cement pastes were tested at different times after pastes mixing: 0 min, 30 min, 60 min, 90 min, 120 min and 180 min. Cement pastes with same W/C ratio equal to 0.35 were tested. The content of admixture was used 0%, 0.5%, 1%, 2%, 3% and 5%. The admixture of $\text{Ca}(\text{NO}_3)_2$ can be used in cement pastes and concretes without a reduction in consistency during 120 min in the dosage of up to 2%. Dosages of $\text{Ca}(\text{NO}_3)_2$ from 3% are not recommended for cement pastes and concrete, they significantly increase the viscosity of mixtures immediately after mixing. The admixture of calcium nitrate changes thixotropy of cement pastes. In this case, a bigger dosage of calcium nitrate increases the thixotropy of pastes.

Keywords: cement pastes, calcium nitrate, admixture, shear stress, viscosity, thixotropy.

Introduction

As it is widely known, antifreeze admixtures for concrete are chemicals which are added to the mixing water of concrete in order to lower the freezing point of the aqueous solution (Karagöl *et al.* 2013). That is, however, not the only effect these admixtures might have on the final product.

Antifreeze admixtures can proceed in two different ways: admixtures that lower the temperature of water freezing in the concrete (e.g. NaNO_2 , NaCl and other) and admixtures that not just lower the freezing temperature but also act as a multifunctional admixture in a relatively high temperature (i.e. 20 ± 2 °C) (e.g. potash, $\text{Ca}(\text{NO}_2)_2$ and $\text{Ca}(\text{NO}_3)_2$). The calcium nitrate can be used for concrete production not only like an antifreeze admixture. It can also be used as an accelerating admixture. The use of accelerators in concrete provides a shortening of setting time and/or an increase in early strength development in the ordinary temperature (Karagöl *et al.* 2013, 2011; Rixom, Mailvaganam 1999).

Mostly, calcium nitrate is used as a concrete admixture performing various functions, e.g. 1) acceleration; 2) neutralization of plasticizer impact with the same rheological features preserved; 3) retaining of long-term strength; 4) anti-frost; 5) corrosion inhibition. In the first two cases, low quantities are used (dry additive content

makes up for 0.2–1% of total cement quantity), in the third and fourth cases, the average quantities are used (1–3%), and in the fourth case, higher quantities (3–4%) (Popovics 1992; Justnes 2007).

Since 1992, technical $\text{Ca}(\text{NO}_3)_2$ has been used as a concrete admixture in some countries. It is a complex agent of calcium and ammonium nitrate containing crystal water (Justnes 1993, 2007). It has been found that $\text{Ca}(\text{NO}_3)_2$ acted as an accelerator of setting in cement paste at low concentrations (Ramachandra 1972). Because the setting is concerned with the development of rigidity in an initially fluid material, it is assumed to be a part of rheology, the study of flow. Calcium nitrate as a set accelerator, and side effects of that have been well proven practically over many years, while the influence of calcium nitrate on the rheological properties of cement paste was only discovered in later years.

The rheology of the cement paste, mortar and concrete mixture may be influenced by various parameters (Esteves *et al.* 2010). Rheological features of mixtures are selected according to construction technologies and the equipment used. It is particularly relevant when modern construction technologies are used, e.g. when mixtures are fed by pumps, self-thickening mixtures are produced, etc.

Cement paste, mortar and concrete mixture are a structural system distinguished by certain rheological features. Cement paste may be considered a flocculant system due to Van der Waals attraction and hydrodynamic impacts (Yahia 2011). Cement pastes are denoted to Bingham bodies (systems), the flow of which is described by two rheological parameters – yield stress τ_0 (below which the suspension displays solid-like behaviour) and plastic viscosity η (Skripkiūnas *et al.* 2005; Skripkiūnas, Daukšys 2004; Tang *et al.* 2001). Plastic viscosity is related to the number and size of the flocs, while the yield stress is a measure of the strength and the number of inter-floc interrelations that are broken down when shear is applied (Puertas *et al.* 2014; Banfill 2003). The microstructure most commonly responsible for a high yield stress is the three-dimensional network that often forms due to flocculation. The yield stress reflects the extent of this flocculation and the strength of the attractive inter-particle forces responsible for the flocculation (Struble, Wei-Guo 1995).

Cement paste is a concentrated suspension of small and heavy particles so rheological measurements are suffering to the disruption of cement operation (Janavičius *et al.* 2013; Miranda *et al.* 2010). The rheological properties of the cement paste gradually change with time (Macijauskas, Gailius 2014; Eisinas, Baltakys 2009). Calcium chloride, calcium nitrate and calcium nitrite, which have the same cations as C_3S and C_2S , accelerate the hydration processes by the nucleating action of such ions and simultaneously make hydrate crystallization processes more intensive (Karagöl *et al.* 2013; Ramachandran 1995). Clearly, the early cement hydration reactions and their associated microstructural changes (i.e. setting) play a major role in controlling the flow behaviour of cement paste. Hydration is known to increase both yield stress and plastic viscosity (Tattersall, Banfill 1983). It presumably increases plastic viscosity only insofar as it increases the volume fraction of the solid material. It is yield stress that is expected to be particularly sensitive to hydration reactions. To the extent that the early hydration products cause the cement particles to be bonded more strongly together or increase the number of inter-particle bonds, hydration is expected to increase the yield stress. Setting corresponds to the development of a substantial yield stress.

Practically, rheological features are useful, whereas they enable forecasting of technological features of a cement paste, mortar, concrete mixture containing corresponding cements with admixtures added or without them, – i.e., the consistency, the possibility to flow, and the possibility to be supplied by pumping.

Numerous methodologies can be used to determine rheological properties of a cement paste. For this purpose, various viscometers could be used, which vary according to the mode of operation (Macijauskas, Gailius 2014; Saak *et al.* 2001; Ferraris, Gaidis 1992; Yahia, Tanimura 2015; Ng, Justnes 2015). One of the most widespread

cement paste rheological characterization techniques is based on the use of a rotational viscometer with coaxial cylinders (Lapasin *et al.* 1983; Banfill 2003; Nedhi, Rahman 2004).

The purpose of the work is to determine the impact of the dosage of the calcium nitrate (from 0.5 to 5%) on rheological properties of the cement pastes (marginal shear tension and viscosity) of a different curing time after mixing from 0 to 150 min.

1. Raw materials and research methods

Cement pastes were prepared by mixing in a mixer in accordance with EN 196-1 (2005). Prior to paste mixing, the admixture was dissolved in water. Water with admixture was added to the cement. Mixing was continuing for 120 s at high speed.

The rheological properties of cement pastes were tested using rotational viscometer with coaxial cylinders Rheotest RN4.1 with a gap of 1.48 mm between cylinders. The cylinder measuring system consists of the measuring cup (assembled) (1) with a coupling (3) and cylinder rotor (2). The dimensions of testing cylinders are presented in Figure 1.

The cement paste is poured into a measuring cup (1), which is fixed to the equipment stand and immobilised. Inside the measuring cup, the cylinder rotor (2) can rotate. Because of the intrinsic friction of the layers of the cement paste (4) appearing between the measuring cup (1) and the rotating cylinder rotor (2) positioned in the measuring cup and connected to a measuring scale; the cylinder rotor (2) makes a turn and the data displayed on the measuring scale changes.

The shear rates used in the tests are presented in Figure 2.

Rheological properties of cement pastes were tested at different times after paste mixing: 0 min, 30 min, 60 min, 90 min, 120 min and 180 min. Cement pastes with same W/C ratio equal to 0.35 with different

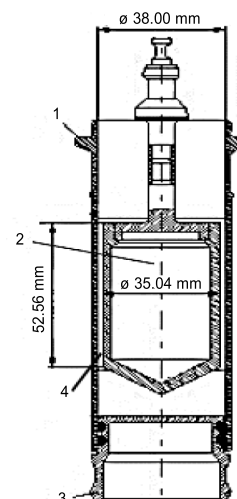


Fig. 1. Principle scheme of testing cylinders

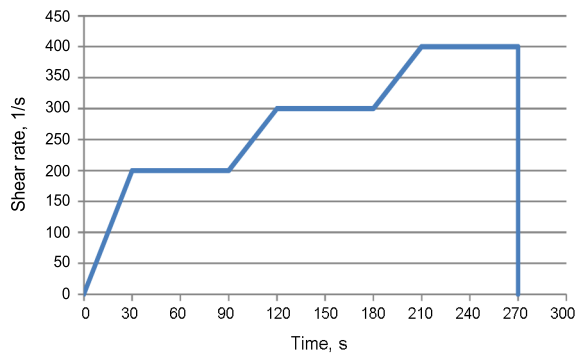


Fig. 2. Shear rates during testing vs testing time

cements were tested. The content of admixture was used 0%, 0.5%, 1%, 2%, 3% and 5%. The tests of rheology were carried out at the temperature of 20 ± 2 °C.

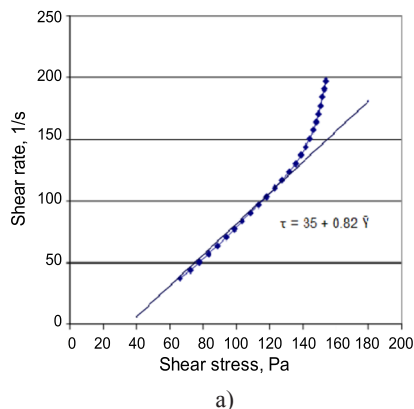
The yield stress and viscosity of cement pastes were calculated from flow curves (τ and \bar{Y} curves) obtained after the test with a rotational viscometer. The flow curves of cement pastes were analysed adopting the Bingham rheological model using the linear approximation of experimental results up to $100\text{--}50$ s^{-1} shear rate when the flow curve of cement paste is of linear character. The EXCEL computer application was used for this purpose (Fig. 3a). The yield stress of cement pastes (τ_0) and viscosity (η) values were obtained from the linear equation (Bingham rheological model):

$$\tau = \tau_0 + \eta \cdot \bar{Y}, \quad (1)$$

where: τ – shear stress, Pa; τ_0 – yield stress of cement paste, Pa; η – viscosity of cement paste, Pa·s; \bar{Y} – shear rate, 1/s.

The thixotropy of cement pastes was calculated from flow curves of cement pastes obtained after testing at different shear rates (Fig. 3b). The thixotropy value was calculated by the reduction of shear stress during the constant shear rate equal 200 1/s by the equation:

$$T = (\tau_1 - \tau_2) / \tau_1, \quad (2)$$



where: τ_1 is the shear stress at 200 1/s before retention during testing, Pa; and τ_2 is the shear stress at 200 1/s after retention during testing, Pa.

For investigations, the following cements (produced in PLLC *Akmenės cementas AB*) were used: Portland cement CEM I 42,5 R (CEM I R); limestone Portland cement CEM II/A-LL 42,5 N (CEM II N); limestone Portland cement CEM II/A-LL 42,5 R (CEM II R) (Tables 1, 2). Portland cement properties are given in Tables 1 and 2 (data from PLLC *Akmenės cementas AB*).

Table 1. Properties of cements

Cement type	Fineness by blaine, cm^2/g	Water content for normal consistency, %	Compressive strength after 28 days, MPa
CEM I 42,5 R	3560	25.4	54.6
CEM II/A-LL 42,5R	4400	26.0	51.1
CEM II/A-LL 42,5 N	4080	25.0	51.2

Table 2. Mineral composition of cements

Cement type	C_3S , %	C_2S , %	C_3A , %	C_4AF , %
CEM I 42,5 R	64.6	7.8	6.4	12.8
CEM II/A-LL 42,5R	58.7	12.9	6.3	10.79
CEM II/A-LL 42,5 N	61.8	8.57	7.1	11.9

2. Experimental results

2.1. Flow behaviour of cement pastes

Cement pastes with different cement type and different $\text{Ca}(\text{NO}_3)_2$ admixture content flow curves (shear stress in Pa vs shear rate in 1/s) are presented in the in Figures 4–9.

Shear stresses of cement pastes with CEM II R cement (the specific surface area of 4400 cm^2/g) is higher than that of cement pastes with CEM I R cement (the specific surface area of 3560 cm^2/g) and cement pastes with CEM II N cement (the specific surface area of

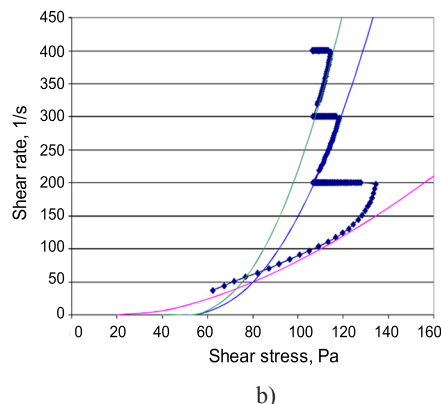


Fig. 3. Calculation of yield stress, viscosity and thixotropy of cement pastes from flow curves

4080 cm²/g). In the range of shear rate from 0 to 200 1/s, shear stresses are 70–130 Pa, 65–135 Pa and 90–300 Pa for the cements CEM I R, CEM II N and CEM II R, accordingly. The surface area of different types of cements has significant effects on the flowability and rheology of the cement paste. Claisse *et al.* (2001) have found from their measurement of cement paste rheology and subsequent data analysis that the yield stress of a cement paste would increase with the specific surface area of the cement used and, therefore, having a larger specific surface area would impair the flowability of the cement paste. The fineness of cement has great influence on cement paste shear stresses, i.e. a bigger specific surface of cement gives a bigger shear stress (i.e. the yield point) for cement paste at defined shear rate.

The changes of cement paste shear stress after 120 or 150 min (Figs 5, 7 and 9) are affected by the Ca(NO₃)₂ admixture. It seems that the admixture accelerated the cement hydration. However, the degree of acceleration varied with the dosage of the admixture. In the pastes without admixture with all types of cements, we don't have changes after 120 or 150 min. Ca(NO₃)₂ admixture content of up to 1% does not increase the shear stresses after 120 or 150 min and 0.5% of admixture usually reduces shear stresses after 120 or 150 min. A bigger content of admixture (from 2% to 3%) significantly increases shear stresses after 120 or 150 min.

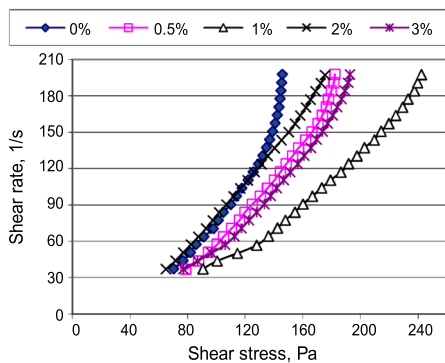


Fig. 4. Cement paste with CEM I R cement shear stress vs shear rate after mixing

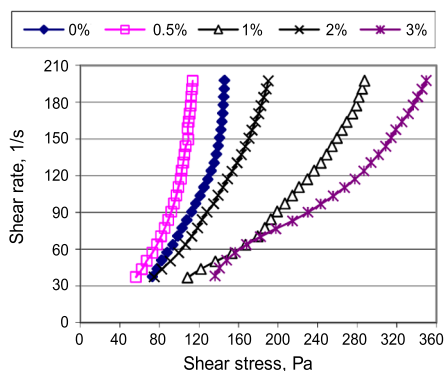


Fig. 5. Cement paste with CEM I R cement shear stress vs shear rate after 120 min

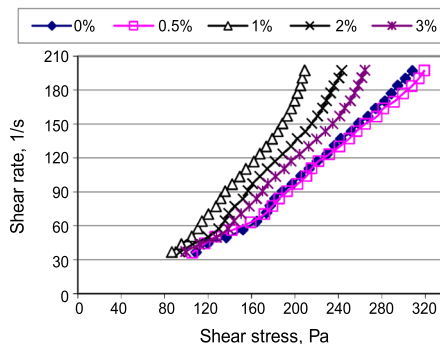


Fig. 6. Cement paste with CEM II R cement shear stress vs shear rate after mixing

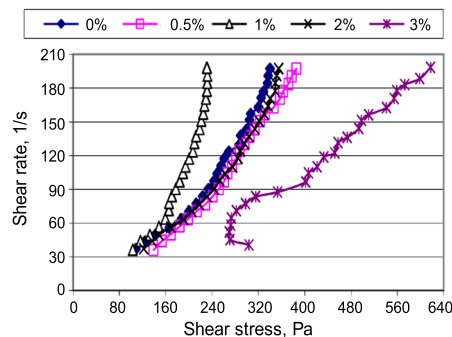


Fig. 7. Cement paste with CEM II R cement shear stress vs shear rate after 150 min

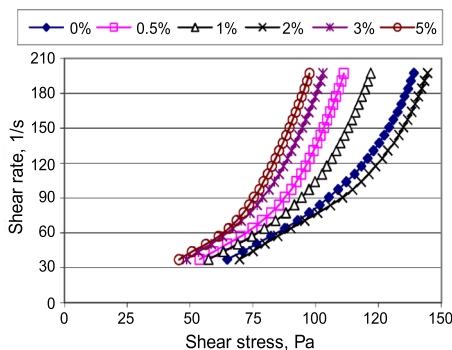


Fig. 8. Cement paste with CEM II N cement shear stress vs shear rate after mixing

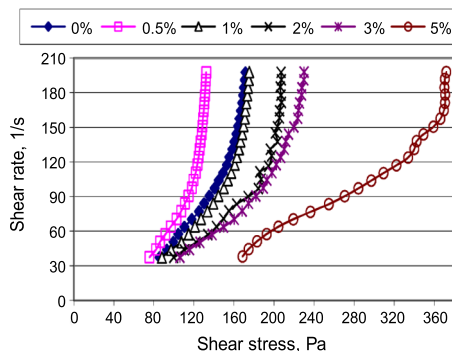


Fig. 9. Cement paste with CEM II N cement shear stress vs shear rate after 150 min

2.2. Yield stresses and viscosities of cement pastes

The yield stress and viscosity of cement paste were calculated adopting the Bingham rheological model. The yield stress and viscosity results vs time after mixing of cement pastes with a different type of cement are presented in Figures 10–15.

The yield stresses of cement pastes are stable during 120 or 150 min after mixing in cement pastes without $\text{Ca}(\text{NO}_3)_2$ admixture. Only 3% of this admixture has an influence on the yield stress of the cement paste. The significant increase of yield point of cement paste with 3% of this admixture starts after 90 min for the cement CEM I R, 60 min for the cement CEM II R and immediately after mixing with 5% of admixture for the cement CEM II N. Dosages of $\text{Ca}(\text{NO}_3)_2$ admixture have no influence on the yield point of the cement paste up to 150 min.

The viscosity of the cement paste after mixing is not higher with $\text{Ca}(\text{NO}_3)_2$ admixture content of up to 3% for the cements CEM I R and CEM II N. For the cement CEM II R (the specific surface of $4400 \text{ cm}^2/\text{g}$), this admixture has the plasticizing effect. A dosage of admixture from 1% to 3% reduces the viscosity of the cement paste immediately after mixing. The admixture of $\text{Ca}(\text{NO}_3)_2$ increase the viscosity of the cement paste with the time in dosage more than 2%.

The increment of the viscosity of cement paste with that content of admixture starts after 60 min in pastes with all cements. A 3% dosage of this admixture significantly increases the viscosity of the cement paste after 30 min for the cements CEM I R and CEM II N and immediately after mixing for the cement CEM II R. A 5% dosage of this admixture starts to significantly increase the viscosity of cement paste immediately after mixing.

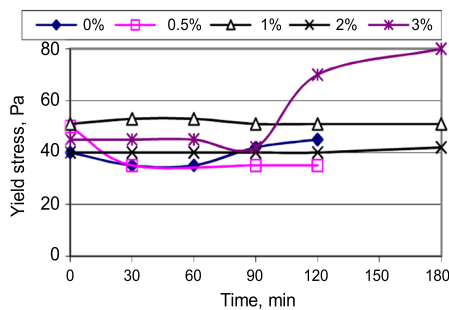


Fig. 10. Cement paste with CEM I R cement yield point

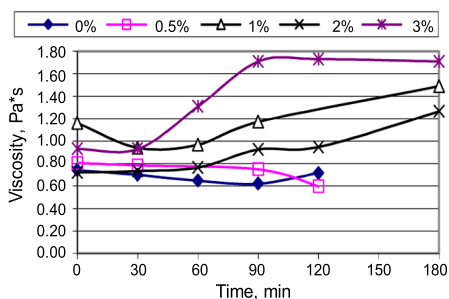


Fig. 11. Cement paste with CEM I R viscosity at shear rate 100 1/s (b) vs time after mixing

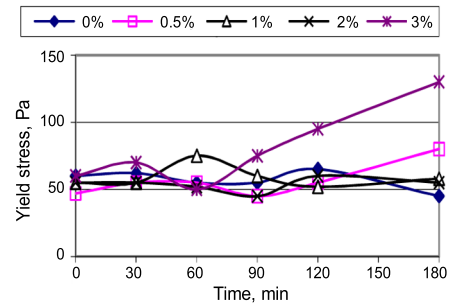


Fig. 12. Cement paste with CEM II R cement yield point

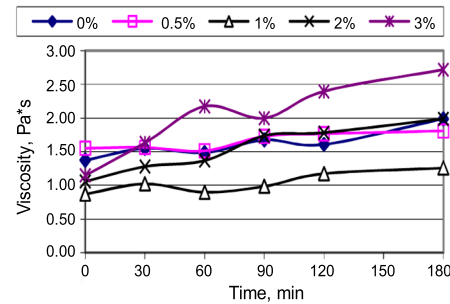


Fig. 13. Cement paste with CEM II R viscosity at shear rate 100 1/s (b) vs time after mixing

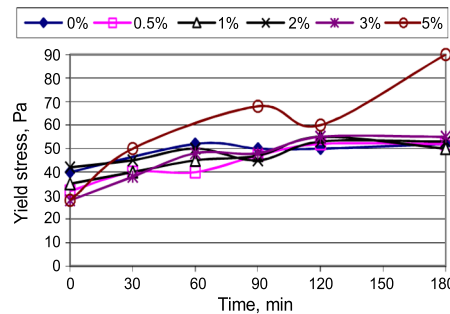


Fig. 14. Cement paste with CEM II N cement yield point

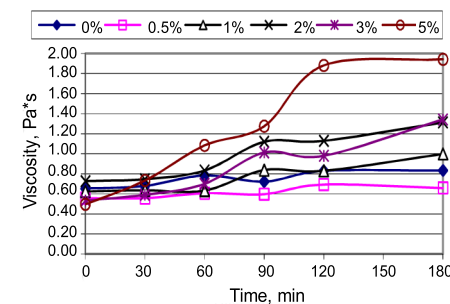


Fig. 15. Cement paste with CEM II N viscosity at shear rate 100 1/s (b) vs time after mixing

Admixture of $\text{Ca}(\text{NO}_3)_2$ can be used in cement pastes and concretes without reduction in consistency during 120 min in the dosage of up to 2%. Dosages from 2% to 3% are suitable for mixtures up to 60 min in time. Dosages of $\text{Ca}(\text{NO}_3)_2$ from 3% are not recommended for cement pastes and concrete, they significantly increase viscosity of mixtures immediately after mixing.

2.3. Thixotropy of cement pastes

Thixotropy is a reversible phenomenon, related to flocculation and deflocculation of the colloidal system under the shear impact load (Roussel *et al.* 2012). Hence, in case of paste, when sufficient shear is present, under which the paste is capable of returning to its initial state, it is not important whether this process takes place due to flocculation of the colloidal system or due to formed C-S-H links between particles. On the microscopic level, an irreversible reaction takes place during hydration, when links between particles are formed and may be quite weak and broken under shear impact (Jiang *et al.* 1995; Nonat *et al.* 1997). During the rest period, additional links form and, if initial links are broken, new links between C-S-H particles appear until there is sufficient place for the formation of new chemical products in the cement system. Formation of chemical linkages in regard to macroscopy may be considered a reversible process, i.e. hydration may be reversible as long as mixing power is sufficient seeking to break C-S-H links between cement particles.

Hydration is the main factor which influences vitality of the mixture, i.e. workability and other technological properties (Flatt, Houst 2001). If the mixture is insufficiently mixed and its internal structure is not fully broken, the mixture has worse workability, other technological properties are also worse (without referring to chemical reactions which take place between chemical admixtures and cement hydration products).

Scientists have examined that the impact of colloidal flocculation of a cement paste on thixotropic properties of the paste is insignificant, and have concluded that formation of C-S-H phases causes the highest impact on thixotropy (Roussel *et al.* 2012).

This explains why one of the most effective methods for production of highly-thixotropic ready-mixed concrete is the use of admixtures which accelerate the hydration process in the mixture (e.g.: $\text{Ca}(\text{NO}_3)_2$ and which are classified as cement accelerators) (Roussel, Cussigh 2008). By mixing the mix using a stirrer of suitable power and by breaking new C-S-H links formed in the paste during the technological process thixotropic properties of the mixture may be improved without significant reduction of its technological properties (slump of concrete mixture), i.e. preserving the viability of the mixture during the whole technological process.

Thixotropic properties of paste are determined to refer to the change in shear stress within 60 s, when constant speed gradient is 200 1/s.

In the case of a cement paste, it means how long shear is sufficient and the substance returns to its initial state. Even if the chemical reaction is irreversible, when bonds between particles are formed, they may be weak and may be terminated under the shearing impact or new bonds may be spontaneously formed during the rest state as long as the chemical impact reserve is sufficient.

Outcomes of tests of paste's thixotropic properties are presented in the Figures 16–18.

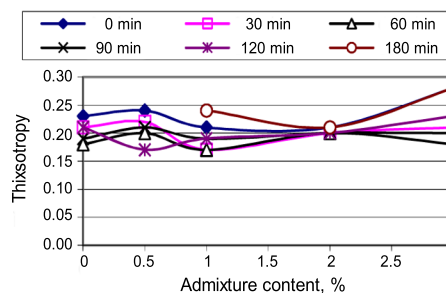


Fig. 16. Thixotropy of cement paste with CEM I R cement

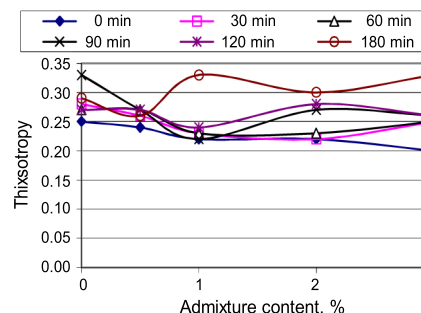


Fig. 17. Thixotropy of cement paste with CEM II R cement

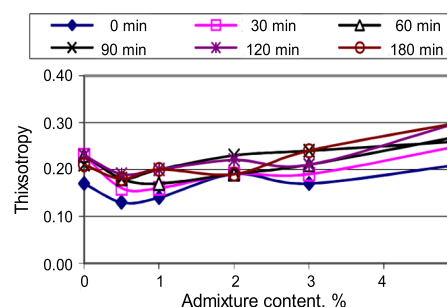


Fig. 18. Thixotropy of cement paste with CEM II N cement

The fineness of cement has a great influence on the cement paste thixotropy. Cement CEM II R with the specific surface of $4400 \text{ cm}^2/\text{g}$ has a higher thixotropy (0.25–0.33) than cements CEM I R and CEM II N with the specific surfaces $3560 \text{ cm}^2/\text{g}$ and $4080 \text{ cm}^2/\text{g}$ (0.15–0.20).

The admixture $\text{Ca}(\text{NO}_3)_2$ does not have the influence on thixotropy of the cement paste up to 120 min after mixing for cements CEM I R and CEM II R. The dosage of admixture $\text{Ca}(\text{NO}_3)_2$ of more than 1.0% increases the thixotropy of the cement paste with cement CEM II N.

Conclusions

The admixture of calcium nitrate can be used in cement pastes and concretes without a reduction in the consistency during 120 min in the dosage of up to 2%. Dosages from 2% to 3% are suitable for mixtures of up to 60 min in time. Dosages of calcium nitrate from 3% are not recommended for cement pastes and concrete as they significantly increase the viscosity of mixtures immediately after mixing.

The admixture of calcium nitrate changes the thixotropy of cement pastes. In this case, the bigger dosage of calcium nitrate increases the thixotropy of pastes.

The fineness of cement has a great influence on shear stresses and viscosity of cement pastes. The bigger specific surface of cement gives the bigger shear stress (the yield point) and viscosity for cement paste at a defined shear rate. After a longer time after mixing, calcium nitrate reduces the fluidity of the pastes; however, the vibration of these pastes restores the fluidity to the same value as without any admixture of calcium nitrate.

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