Influence of Plasticizer Amount on Rheological and Hydration Properties of CEM II Type Portland Cements

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Abstract. The article analyzes the effect of plasticizer (based on polycarboxilates) amount (0.3 – 1.2% wt. of cement) on the rheological and hydration properties of two Portland cements pastes: CEM II/A-S 42.5N and CEM II/A-LL 42.5N. Increase of plasticizer amount reduces viscosity of CEM II/A-LL 42.5N cement paste from 3 to 12 times, where viscosity of CEM II/A-S 42.5N cement paste reduces from 5 to 20 times. The optimum plasticizer dose (0.3%) in case of CEM II/A-S 42.5N and (1.2%) in case of CEM II/A-LL 42.5N was established. Calorimetry studies have shown that plasticizer reduces the wetting heat release rate in CEM II/A-LL 42.5N cement twice and in CEM II/A-S 42.5N cement – by 25%. Plasticizer prolongs the maximum heat release rate time by 16 h in CEM II/A-LL 42.5N samples and reduces heat release rate by 19%. In CEM II/A-S 42.5N cement samples plasticizer prolongs maximum heat release rate time by 14.5 h and increases heat release rate by 15%. The goal of this study is to analyze the effect of the dosage of the most widely used plasticizer on solubility characteristics, rheological and hydration properties of two cements CEM II/A-S 42.5N and CEM II/A-LL 42.5N to establish the optimum dose of plasticizer in cements pastes.

1. Introduction
Concrete has been known as a versatile and reliable construction material for many years. Despite this its production and final product’s properties constantly change due to the newly acquired knowledge, development of products and technologies and environmental issues. Seeking to make the production of cements less costly and to address environmental issues, various additives are used, including limestone and slag. It was estimated that production of cements causes (5 – 7)% [3, 6] of the total quantity of CO₂ emitted to the environment. Therefore recently the number of researches of various

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additives which may partly substitute Portland cements has increased. Blended cement is a well-known component of concrete. CEM II blended Portland cements, in comparison with ordinary CEM I Portland cements have ecologic advantages, since the use of admixtures reduces fuel costs and CO₂ emissions into the atmosphere during Portland cement production and somewhat solve waste utilization problems [1, 2, 3, 4]. That's why it is expected that the future world production of CEM II blended Portland cement will be continuously increasing. In general, fillers are incorporated in order to complete the granulometric distribution of cement, to enhance its granular packing factor and to block up capillary pores [4]. Whereas limestone and slag fillers used in composite cements are very fine and they impose an impact on rheological properties of grout, the use of plasticizers is a necessity. Plasticizers are widely used in concrete and have become common components of concrete. Various plasticizers have been widely offered to the users, therefore choosing an option and amount of plasticizer in blended cements is essential [5, 6, 7].

According to their chemical composition plasticizers may be classified into the following main groups: sulphonated naphthalene formaldehydes, sulphonated melamine formaldehydes, modified lignosulphonates, polycarboxylates, polycarboxylate ethers [8, 9]. Researchers have performed several cement and superplasticizer compatibility researches and have analyzed rheology of grouts [10, 11]. Flowability of fresh mixtures of concrete and mortars may be influenced by various factors, related to chemical and mineralogical composition of Portland cements, as well as their fineness, type and quantity of superplasticizers, as well as ambient temperature [12]. Pozzolanic and cementitious materials, when mixed with Portland clinker and water, produce C₃S–H similar to that generated from the hydration of calcium silicate of clinker [13]. This reaction can be significantly influenced by the type and amount of plasticizer. Moreover, plasticizers may affect (impede or promote) hydration of the silicate and alumina phases of clinker grains. And it can lead to a lower strength at early ages. Superplasticizers can interact with cement particles physically and chemically. They can also react with hydration products of the cement. It is reported that some of them can lower the initial hydration rate by reacting with C₃A preferentially, while others delay the hydration reaction of C₃S component [14]. Among the plasticisers lignosulphonate base works like retarders. It prolongs the hydration period of cements.

It is reported that the lignosulphonate base plasticisers greatly modify the initial rheological properties and the final compressive strength of cement base materials [15]. Chandra et al. [16], have suggested that the hydration retardation effects of superplasticisers in cement/cement admixtures is due to the interaction of calcium ions [or Ca(OH)₂ particles] formed during C₃S hydration with sulphonate ions in superplasticizers, or due to superplasticiser micellization of water available for the hydration of the silicates. Results from a study of lignosulphonic acid used, - as a plasticiser [17] shows that a plasticiser has dual role; one mainly of a kinetic inhibitor (poison) in cement hydration mechanism and the other of a dispersant. It was found that the decrease of rheological parameters depends on fine fraction contents in cement. It was determined that lignosulphonates may cause retarded strength development and reduced flexural strength of slag cement concrete [18]. Addition of lignosulphonates and superplasticizer lowers rheological parameters of cement pastes containing blast furnace slag [19]. It is proved, that the slag–cement formulation demonstrates a lower viscous property under the influence of the plasticizer additive, than ordinary Portland cement [19]. Limestone presence in the binding system determines the acceleration of cement initial hydration, especially of tricalcium silicate [2]. As one can see, a considerable number of researches were performed, however, there still remain some uncertainties during selection of a suitable plasticizer for cements of type CEM II. That's why choosing an option of plasticizer for limestone cement and slag cement are essential. The goal
of this study is to analyze the effect of the dosage of the mostly used plasticizer on solubility characteristics, rheological and hydration properties of two cements CEM II/A-S 42.5N and CEM II/A-LL 42.5N to establish the optimum dose of plasticizer in cements pastes.

2. Materials and research methods

Two-types of Portland cements, namely CEM II A-LL 42.5 N (L) and CEM II/A-S 42.5 N (B) – type cement, meeting LST EN 197-1: 2011 requirements, manufactured by Akmenės Cementas (Lithuania) were tested in the research. The main properties of both cements are provided in Tables 1 and 2 below. Fineness (by Blaine) of L cement is 4080 cm$^2$/g, of B cement – 3860 cm$^2$/g.

Table 1. Mineral compositions of cements in %.

| Cement type | $C_2S$ | $C_3S$ | $C_3A$ | $C_4AF$ | MgO | SiO$_2$ | CaCO$_3$ | CaSO$_4$ $\cdot$ 2H$_2$O | $K_2SO_4$ | $Na_2O_{eqv.}$ |
|-------------|--------|--------|--------|--------|-----|-------|---------|-----------------|--------|------------|
| B           | 10.84  | 60.25  | 5.85   | 10.70  | 2.05| 0.51  | 2.75    | 2.94           | 3.34   | 0.77       |
| L           | 7.44   | 55.30  | 6.25   | 10.55  | 3.15| 0.32  | 11.15   | 2.19           | 2.91   | 0.74       |

Polycarboxylate ester based plasticizer (PCE) was used in research. Electric conduction (EC) and pH measurements in PCE solution were tested by MPC 227 device, (pH electrode InLab 410, measuring accuracy 0.01 S/cm, EC electrode InLab 730, measuring range 0 S/cm – 1000 S/cm). PH and EC characteristics of PCE are 4.22 and 2.41 mS/cm. In the study different concentration solutions were used, where PCE amount changes from 0.3 to 1.2% in the distilled water. The PCE amount range was selected according to the manufacturer recommendation. Increasing of the amount of PCE leads to reduction of pH from 3.9 to 3.6 and increases of EC from 82 to 250 mS/cm.

Cement paste was mixed in a planetary mixer according to EN 196-1. The mixing procedure was as follows: cement (1kg) and 3/4 of required water were mixed for 2 min. at low speed, after that the remaining water and plasticizer were added and cement paste was mixed further for 1 min. at high speed.

The influence of PCE on dynamic viscosity of cement pastes was tested using the SV-10 vibro-viscosimeter. With SV-10 vibro-viscosimeter it is possible to define the dynamic viscosity of pastes up to 12,000 mPa·s with 0.01 mPa·s accuracy in a very small amount of paste (35 ml). At the same time, the device records the temperature changes in cement paste with 0.1 °C accuracy with the purpose to monitor the PCE effect on the cement paste temperature. W/C ratio in cement pastes in all cases was constant 0.28. Cement pastes with PCE contents dosed from 0 to 1.2% (depending on cement amount) were prepared for the tests. The dynamic viscosity of the prepared pastes was measured instantly and then after 5, 10, 15, and 20 min. In order to evaluate PCE impact on rheological and hydration characteristics of on L and B cements, 10 cements pastes compositions have been made (table 2). Calorimetric investigations of samples L-0, L-0.6 and B-0, B-0.6 were performed, using Toni CAL III equipment. Pure L-0 and B-0 cements compositions of proportions 0.28 parts of distilled water and 1 part of cements were studied. For compositions with PCE additive preparation was as follows: 0.28 parts of distilled water, 0.06 parts of PCE and 1 part of cements. The heat evolution curves of all pointed compositions were registered up to 48 h.
Table 2. Cement paste compositions by weight (in %).

| Batch | L-0 | L-0.3 | L-0.6 | L-0.9 | L-1.2 | B-0 | B-0.3 | B-0.6 | B-0.9 | B-1.2 |
|-------|-----|-------|-------|-------|-------|-----|-------|-------|-------|-------|
| Cement L | 100 | 99.7  | 99.4  | 99.1  | 98.8  |     |      |      |       |       |
| Cement B | 100 | 99.7  | 99.4  | 99.1  | 98.8  |     |      |      |       |       |
| PCE, g | 0.3 | 0.3   | 0.6   | 0.9   | 1.2   | 0.3 | 0.6  | 0.9  | 1.2   |       |
| W/C ratio | 0.28 | 0.28 | 0.28 | 0.28 | 0.28 | 0.28 | 0.28 | 0.28 | 0.28 | 0.28 |

3. Test results
Cement paste viscosity measurements is very informative, as it is possible to monitor the kinetics of cement pastes variation at selected time interval until the viscosity reaches the limit value of instrument readings. Fluctuations of dynamic viscosity illustrate the binding process of cement pastes (deterioration of rheological properties and loss of workability) during the hydration process and formation of new compounds. The results of PCE impact on L and B cement pastes’ viscosity are presented in figure 1. Monitoring shows, - that viscosity of pure L cement samples paste increased within the testing period (from 610 Pa·s to 1600 Pa·s (figure 1). Effect of PCE additives on viscosity of L cement paste (figure 1a) shows, - that increase of PCE additive amount in the paste (from 0.3 to 1.2%) immediately reduces viscosity from 270 to 43 Pa·s. The last measurement shows, - that viscosity in the pastes increases to 830 and 430 Pa·s. It is observed that the higher amount of PCE additive is there in the paste, the lower the viscosity. It has been noticed that the difference in viscosity using 0.6, 0.9 and 1.2% of PCE additives in the paste is small, thus we can conclude that amount of 0.6% of PCE additive is optimal and it is enough for maintain constant level of viscosity.

Meanwhile, viscosity of B cement (B-0 sample) is lower right after mixing, 390 Pa·s, than in L-0 sample. Noticeable growth of viscosity up to 1420 Pa·s has been observed after 10 – 15 min. The observed differences in viscosity in L and B cements may be conditioned by their mineral compositions. As it is described in [19], the influence of limestone on rheological properties of the paste depends not only on mineral composition of clinker (amount of C₃A) but also on the amount of...
MgO. According to the data presented in table 1, the amount of MgO in L cement is higher than in B cement. Growth of PCE additive in the paste of B cement (from 0.3 to 1.2%) reduces viscosity from 170 Pa·s to 30 Pa·s (figure 1b) and during last measuring viscosity increases up to 315 Pa·s and 80 Pa·s respectively. It is obvious that PCE additive decreases viscosity of B cement more significantly than L cement paste. We can assume that less than 0.6% of PCE additive is enough for decrease of viscosity.

During the viscosity research, measurements of changes in temperature of pastes were performed. Seeking for better presentation of on-going processes, changes in temperature were recorded immediately after the mixing procedure, when the temperature of all samples was the same, i.e. 20 ºC, then it was recorded in 1 min and 5, 10, 15 and 20 min. When the 1-st minute of the research passed (figure 2a), the highest temperature was achieved in sample L-0.3 – 21.7 ºC, meanwhile temperature of other samples was as follows: L-0.6 – 21.5 ºC, L-1.2 – 21.35 ºC, and L-0 – up to 21 ºC. It is obvious that the minimum quantity of PCE causes higher temperature, lower quantity of PCE causes lower temperature, and the temperature of the samples with no additives seems to be the lowest. It means that higher quantity of PCE reduces wetting heat of sample L. During 19 min reduction of temperature of the samples was observed. After 20 min. the temperature of sample L-0.3 was 20.3 ºC, L-0.6 – 20.2 ºC, L-1.2 – 20 ºC, and L-0 – 19.55 ºC.

Different tendencies have been noticed in case of sample B. When 1 st min. of the test passed (figure 2b), the highest temperature was reached in the sample B-1.2 with the highest amount of PCE 25.37 ºC. It is noticed that the temperature of the sample B-0 was higher than samples L-0 (by almost 2 ºC), and it was equal to – 22.98 ºC. If the quantity of PCE is consistently increased, the temperature of the samples rises. It means that clinker minerals of cement B (in total their quantity in cement B exceeds by 8.28% the quantity in cement L) generates more heat than cement L during dissolution and additive PCE interacts with dissolving minerals of the cement and causes growth of the temperature of the sample. Because of cement minerals interaction with PCE additive, temperature of the sample rises even more and the viscosity drops. During the last 19 min. temperature of the samples drops, however, in 20 min. the recorded temperature of sample B-0 is 20.16 ºC, and of B-1.2 – 22.67 ºC. It shows that temperature of the samples from cement B drops faster than in case of samples of cement L.

![Figure 2. Changes of viscosity in L (a) and B (b) cement paste with PCE additive.](image-url)
Some correlation between viscosity and temperature changes can be seen. One of the factors which influence interaction of cement and PCE is mineral composition of the cement. If the cement contains more cement clinker minerals, wetting heat during dissolution and correspondingly temperature of the sample rises. The heat generated during cement hydration activates the impact of PCE. Cement L generates less heat during dissolution process, than cement B, therefore viscosity of the sample is higher than of cement B. Additive PCE a little raises temperature of L cement samples, and this may be the reason why reduction in viscosity in this samples is slower than in case of B cement samples, where additive PCE significantly influences increase of temperature and reduction of viscosity.

For the purpose to further clarify the effect of PCE additive on both cements pastes and to explain the conducted viscosity tests, measurements of the EC were performed within 2 h. It has been explained that EC depends on the amount of ions in solution and well describes the course of the hydration process during cement mineral dissolution [16, 17]. Scientists singled out three main periods of EC changes. During the first period fast increase of EC is observed, which is interpreted as solution of cement minerals and ion transit to the solution. During the second period EC stabilization is observed. The third period consists in hydrate generation and reduction of EC.

EC values in L-0 sample during the first period (~20 min) grow from 17.3 to 19.0 mS/cm (figure 3a). In the course of the second period (next 20 min) EC does not change noticeably and during the third period, EC reduction is visible. Increase of PCE additive (figure 5), in L cement paste from 0.3 to 1.2%, slightly decreases the primary EC values (from 16.4 to 15.6 mS/cm). Less amount of PCE additive in the paste defines a shorter first period and quicker EC growth at the end of the period. Larger amount of PCE defines a longer second period. At the end of the second period the difference among all pastes EC is slight (0.5-0.7 mS/cm) and during the third period a larger amount of PCE defines lower EC values in the paste.

Cements mineral solution in the B-0 cement paste slightly differs from mineral solution of L cement. EC in the B-0 cement paste at the start of the first period (figure 3b) is by almost 2.3 mS/cm less than in the L-0 paste and its value is just 15.0 mS/cm. Perhaps this can be explained by lower amount of C\textsubscript{3}A in B cement (table 1). During the second period, EC values quickly grow and at the end of the third period EC rise to 19.0 mS/cm, like in L-0 paste. Same as in case of L cement, EC changing in B cement paste is minimal during the second period and during the third period EC diminishes. Same as in the case with L cement paste, increase of PCE additive amount in B cement paste a little decreases EC values (from 14.6 to 13.8 mS/cm). The shorter is the first period and the superior is the rate of EC increase at the end of the period, the lower amount of PCE additive is there in the paste. The larger amount of PCE additive extends the second period; EC also decreases during the third period. We can conclude that additive PCE retards transition of cements minerals ions to the solution. It can be stated that despite the type of cement, PCE additive, characterized by its moderate EC and acidic pH, decrease the values of EC in the paste, retards transition of ions to the solution, and decreases viscosity of the paste. Efficacy of PCE additive depending on the type of cement is different: for B cement 0.3 – 0.6% is enough and for L cement larger amount – 0.9 – 1.2% is the most efficient.
Calorimetric test well describes the differences between L and B cements and PCE additive influence on cement hydration process. Investigation shows (figure 4a, 4b), that wetting heat release rate in both cements samples is different: in B-0 sample 12.4 J/g and in L-0 sample 8.6 J/g. May be it is due large quantity of gypsum in B cement and its reaction with C$_3$A.

The highest heat release rate (10 J/gh) in L-0 sample was reached after 12.5 h and in B-0 sample (8.6 J/gh) – after 11.5 h of hydration. Noticed differences may be conditioned by different fineness and mineralogical composition of both cements, containing different amount of C$_3$A, C$_2$S and C$_3$S. It is known that most of plasticizers retard the hydration of the Portland cements [33, 35, 21, 34]. Comparing with rezults of L-0 sample, PCE additive (figure 4a) reduces a wetting heat release rate from 8.6 J/g to 4.4 J/g. These results verify that wetting heat has a direct influence on the development of temperature, decline of viscosity and EC values changes in cement pastes (figures 2, 3). PCE additive increases time required to achieve maximum heat release rate in L cement samples from 12.5 to 17.5 h. In L-0.6 sample heat release rate increases to 8.1 J/gh. It is stated that PCE additive decelerates hydration process more in L than in B cement. Possibly, addition of limestone may reduce the potency of PCE additive [11].

Investigations show that in comparison with B-0 sample (figure 4 b) PCE additive in B cement reduces wetting heat release rate from 12.4 J/g to 9.8 J/g. The maximum heat release rate in the B-0.6 sample is reached after 14.5 h, in B-0 sample – after 11.5 h.

The highest heat release rate in sample B-0.6 is 9.8 J/gh. Probably bigger total amount of C$_3$A in B cement determines quicker action of PCE additive on the hydration process in B cement than in L cement, because in the study significance of C$_3$A amount on cement hydration in presence of plasticizer has been pointed out [22].
Total heat value curves confirmed by data of figure 5a indicate that PCE additive in L cement impedes heat release by almost 6 h in comparison with L-0 sample. PCE additive influences faster heat releases in B cement, than in L cement samples. After one day total heat value in L-0 sample reaches 175 J/g, in L-0.6 sample reduces by one fifth – 133 J/g. After two days of measurements the total heat value in sample L-0 was 240 J/g, while in L-0.6 sample 225 J/g. PCE additive impedes heat release in B cement by almost 2 h (figure 5 b). Heat release starts increasing in a B-0.6 sample after this time is over. After one day we can see that the value of released heat in both samples (B-0, B-0.6) becomes equal and reaches 150 J/g. After two days of measurements total heat value in sample B-0 reaches 225 J/g and little differs from B-0.6 sample. We can conclude that L cement is more sensitive to pH and EC values of plasticizer, than B cement. This fact attests that these two cements need attentive choosing of plasticizer and it is confirmed by researches [24, 25, 26, 27]. It is possible to generalize that the observed temperature, viscosity and EC changes tendencies in pastes basically reflect in calorimetric tests.

**Figure 4.** Heat release rate in L (a) and B (b) cement paste without and with PCE additive.

**Figure 5.** Amount of released heat in L (a) and B (b) cement paste without and with PCE additive.
4. Conclusions
It was found that mineral composition of the cement is one of the important factors affecting the cement and plasticizer interaction. If cement contains more cement clinker minerals, wetting heat and temperature in the sample paste rise. In case of cement CEM II/A-S 42.5N, containing more cement clinker minerals, wetting heat and temperature of the paste are higher than in CEM II/A-LL 42.5N cement. Cement CEM II/A-LL 42.5N generates less heat than cement CEM II/A-S 42.5N, therefore viscosity of the sample is higher than that of cement CEM II/A-S 42.5N. It can be assumed that higher wetting heat in the cement paste more markedly activates PCE additive and as the consequence this leads to viscosity drop.

It has been established that increase of PCE additive (from 0.3 to 1.2%) reduces viscosity of the CEM II/A-LL 42.5N cement paste after 20 min of mixing from 1600 to 430 Pa·s and EC – from 17.7 to 16.4 mS/cm, where viscosity of CEM II/A-S 42.5N cement paste reduces from 1420 to 80 Pa·s and EC – from 16.8 to 15.6 mS/cm. During hydration PCE additive decreases the wetting heat release rate in CEM II/A-LL 42.5N cement from 8.6 to 4.4 J/g and in CEM II/A-S 42.5N cement from 12.4 to 8.1 J/g.

Additive PCE prolongs the time, required for reaching the maximum wetting heat release rate in CEM II/A-LL 42.5N cement samples from 12.5 to 17.5 h, in CEM II/A-S 42.5N cement samples – from 11.5 to 14.5 h. It is determined that in comparison with pure CEM II/A-LL 42.5N sample the heat release rate in CEM II/A-S 42.5N sample with PCE additive is reduced by 19%, and the total heat value – by 6% in the samples after 48 h of hydration. In CEM II/A-S 42.5N cement samples PCE additive increases heat release rate by 15% and 4% total heat value in samples after 48 h of hydration. It is assessed that efficacy of PCE additive depending on the type of cement is different: for CEM II/A-S 42.5N cement 0.3-0.6% is enough and for CEM II/A-LL 42.5N cement larger amount – 0.9-1.2% - is the most efficient.

5. References
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