

Effect of Limestone Particles on Rheological Properties and Hardening Process of Plasticized Cement Pastes

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crossref <http://dx.doi.org/10.5755/j01.ms.21.1.5765>

Received 06 November 2013; accepted 22 February 2014

The rheological properties (flow, fluidity, dynamic viscosity, stiffness rate) and hardening process (hydration, development of structure) of Portland-limestone cement (PLC) and Portland cement (PC) (strength class 42.5 R and N) pastes without and with superplasticizer (SP) based on synthetic polycarboxylate ether (PCE) were investigated. It was found that limestone particles increase W/C and viscosity of cement pastes and reduction of water in plasticized pastes PCL is lower than in pastes PC. On the other hand, the fluidity of plasticized cement pastes PCL is better and its thickening goes slower. The hydration and structure development in pastes PLC are going slower than in pastes PC. The impact of SP (when consistency of cement pastes is the same) on hydration process and hardening structure development are going faster in plasticized cement pastes. The fineness of cements also has an influence on SP effect.

Keywords: Portland cement, Portland-limestone cement, superplasticizer, rheological properties, hardening process

1. INTRODUCTION

It is well known that the production process of Portland cement greatly pollutes the environment by extremely high CO₂ emissions, especially at burning of clinker (up to 0.9 per 1 of clinker). Currently Portland cements CEM II [1], in which clinker is partially replaced by mineral additives (limestone, granulated blastfurnace slag, fly ashes and etc.), are increasingly produced and used. Portland cements CEM II represent a high quality alternative to traditional Portland cement from the point of technology with much less environmental load. Combination of several main components of mixed Portland cements makes it possible to use advantages and disadvantages of individual components. Additions can influence workability of concrete, initial development of strength or durability of concrete [2–4]. Portland-limestone cement CEM II is produced and used at a particularly great pace because of relative cheapness and manufacturability of limestone. The extension of this production was also decided by new advanced technologies of cement products and birth of extremely effective SP. Taking into account specific properties of Portland-limestone cement and selecting proper technological parameters, it is possible to replace by it Portland cement CEM I in many fields of use.

Often SP is indispensable in the use of Portland-limestone cement, especially because all presently used SP practically suit for Portland-limestone cement in contrast to other Portland cements CEM II, e.g. Portland-pozzolana cement [5]. At grinding of limestone together with considerably harder clinker, limestone is crushed very finely and its ultrafine particles in cement are much smaller than those of clinker [6]. Regardless of high fineness, they do not agglomerate in contrast to microsilica, but distribute uniformly in cement and ensure a more even distribution of cementing particles and a more homogenous structure of

hardened cement matrix [7, 8]. However, the mixing of such cement requires greater quantities of water what worsens the properties of cement products, such as freeze-thaw resistance, chemical resistance and etc. The use of SP not only softens or even absolutely eliminates the negative impact of fine limestone particles in cement, but in certain cases also brings out the positive properties of Portland-limestone cement. It is established that limestone added into cement for production of high-performance concretes, where the water and cement ratio (W/C) should be very low (0.25–0.30) and use of SP is indispensable [9], decreases the strength very inconsiderably, meanwhile hardened concrete has smaller, not contacting pores, a lower permeability and a more homogenous structure [10, 11]. Furthermore, at a low W/C, the limestone additive dilutes particles of clinker and by this enables to make a full use of binding properties (full hydration) of cement. According to Taylor [12], to make cement (without additives) to hydrate completely, the W/C of paste should be at least 0.38. When the quantity of water is less, some cement particles do not hydrate and remain as micro aggregates. Not less important is the impact of SP on variation in time of rheological properties of hydrating cement pastes, as well as on hardening. The rate of stiffening of cement paste is influenced by many factors, such as mineral composition of clinker and additives, their content, fineness and W/C. SP changes the process of cement hardening, owing to lower W/C and chemical interaction [13, 14]. The impact of SP on different cements differs and is subject both to properties of cement and SP. The great influence is exerted by mineral composition of clinker, in particular by content of C₃A and C₃S [5, 15]. Limestone presence in the binding system determines the acceleration of initial cement hydration, especially for C₃S; therefore choosing an option of plasticizer in concrete with limestone cement is essential [16].

The SP impact on rheological properties of cement pastes and concretes with limestone powder was investigated rather widely, however so far no unanimous opinion on this issue was arrived at, as the presented results of investigations are not unambiguous, but even

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contradict each other. Tsivilis et al. [17] have found that limestone filler in cement reduces water demand in cement paste of normal consistency. Other authors [18] state, that fine limestone filler increase the viscosity and W/C of cement paste. According Zhang [19] when quantity of ultrafine powder of limestone is smaller or equal to 15 % the viscosity of cement pastes can decrease, when more than 15 % – this decrease is significant. Nehdi [15] has concluded that the impact of limestone additive depends not only on mineral composition of clinker, but also on physical and chemical properties of limestone such as degree of crystallinity, impurities, MgO content.

The aim of this study is to establish the peculiarities of impact of the widely used SP, based on synthetic PCE, on different rheological properties (flow, fluidity, dynamic viscosity, stiffness rate) of Portland-limestone cement and Portland cement pastes, as well as on its hardening process (hydration, development of structure).

2. MATERIALS AND TEST METHODS

Portland cement CEM I (PC) and Portland-limestone cement CEM II/A-LL (PLC) according to EN 197-1 (strength class 42.5 N and R), produced by JSC Akmenės Cementas were used in the investigation. Clinker mineral composition (according Bogue calculation) was: C₃S – 61.0 %, C₂S – 13.5 %, C₃A – 8.5 %, C₄AF – 10.5 %. Lime stone content in cement – 15 %. The codes of used cements and its physical properties are given in Table 1.

Chemical composition of limestone used for production of cement was: CaO – 48.23 %; MgO – 2.98 %; SiO₂ – 2.68 %; Al₂O₃ – 0.78 %; Fe₂O₃ – 0.67 %; SO₃ – 0.48 %; LOI – 41.30 %.

SP Muraplast FK 63.30 based on synthetic (PCE) was used (consistency – liquid, density – 1.06 kg/dm³, max. chloride content < 0.10 %, max. alkali content < 0.5 %).

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

For the investigation of effectiveness of SP for different cements, the same initial flow ($\phi = 120 \text{ mm} \pm 5 \text{ mm}$) of cement pastes was taken as a basic index. It was determined using Suttard cylinder (internal diameter – 40 mm, height – 100 mm) method. A spread diameter of paste was measured after elevation of the cylinder, filled with paste (after 4 min. since the start of cement paste mixing).

The effect of SP on the fluidity of cement pastes was investigated by Marsh cone method [5]. The test consists in measuring the duration of flow of plasticized cement pastes from a bowl of 1 l capacity (for water it is 30 s).

Setting time of cement pastes was determined by Vicat

Table 1. Physical properties of cements (according EN 197-1)

Type of cement	Code	Compressive strength, MPa		Setting time, min		Standard consistence (water content in %)	Fineness	
		7 days	28 days	initial	final		blaine, m ² /kg	> 90 μ m, %
CEM I 42.5 N	PC N	25.5	55.0	120	175	24.6	327	2.7
CEM I 42.5 R	PC R	28.9	54.6	150	200	25.4	356	1.1
CEM II/A-LL 42.5 N	PLC N	23.7	51.1	190	285	25.0	408	4.3
CEM II/A-LL 42.5 R	PLC R	29.9	51.2	160	200	26.0	440	0.5

device. The dynamic viscosity of cement pastes was investigated using the SV-10 vibro-viscometer. This device measures paste viscosity resistance to constant vibration of gauge plates at 30 Hz frequency. It is possible to define the dynamic viscosity of cement pastes up to 12.000 mPa·s with 0.01 mPa·s accuracy. The dynamic viscosity of the freshly prepared cement pastes was measured during 2 h period.

Exothermic reaction (EXO) temperature of cement pastes were measured according to the methodology developed by company Alcoa. The changes of EXO temperature in time and the reached maximum temperature reflect precisely the influence of various factors, such as the fineness of cement, W/C, additives and etc. on hydration process of cement paste. A 1.5 kg specimen of prepared cement paste was placed into the textolite mould (10×10×10) cm. A T-type thermocouple with glass tube was inserted into the specimen during the curing. The mould filled with paste was immediately placed in a metal box insulated with 50 mm of expanded polystyrene. Temperature changes were recorded on the computer.

The investigations of development of hardening structure of cement pastes in early period were made by measuring ultrasonic wave velocity (UWV). The main principle of this method is a change of ultrasonic transition time due to the setting of cement paste and development of its structure. The hydration and hardening processes of cement paste can be conditionally divided into three periods: 1 – induction, (beginning of hydrate formation), till 3 h–4 h. UWV does not change. 2 – structure compaction (precipitation of hydrates with a transition from amorphous to crystal state, quick stiffening), till 24 h. UWV sharply increases. 3 – slow structure compaction period (cement skeleton approaches its final stiffness), follow up 24 h UWV increases slowly and became stable. The Schleibinger Geräte GmbH datalogger with the ultrasonic pulse indicator Pundit 7 was used. Freshly prepared cement paste (following 10 min from start of mixing) was set between two ultrasonic transducers operating at 10 pulses per second and frequency of 54 kHz.

3. RESULTS

The W/C of non-plasticized cement pastes of the same initial flow are provided in Table 2. It may be seen that W/C is higher in pastes PLC than in pastes PC because of the presence of limestone particles of higher fineness [6]. In cement pastes of type R, W/C is also higher.

Table 2. W/C in different cement pastes of the same initial flow ($\phi = 120 \text{ mm} \pm 5 \text{ mm}$)

Type of cement	PC N	PLC N	PC R	PLC R
W/C	0.30	0.32	0.32	0.34

3.1. Cement pastes PC and PLC of type N

Rheological behaviour. The results of fluidity measurements of cement pastes with different SP content by March cone method are provided in Figure 1. SP was added into cement pastes in which W/C was the same as in non-plasticized pastes of the same flow (Table 2). It can be seen that in the course of 60 min, the fluidity (flow time) of cement pastes with 0.75 % and 1.00 % SP, is almost identical while the fluidity of pastes with 0.50 % SP is already worse, especially of pastes PLC N.

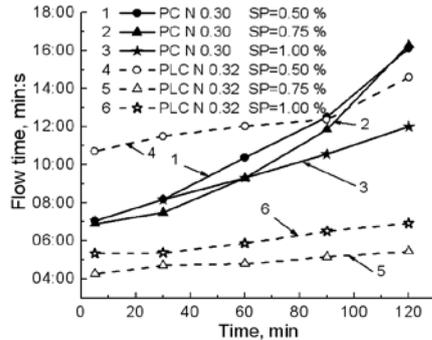


Fig. 1. Flow time of cement pastes (N) with different SP content (0.30 and 0.32 – W/C in cement pastes)

The loss of fluidity of cement pastes is best reflected by difference of flow time from immediately after mixing to a certain established time (in our case after 5 min. and 60 min. respectively, see Table 3). It may be seen that the loss of fluidity of pastes with 0.75 % and 1 % SP is practically the same. Therefore for further investigations the content of SP was selected as 0.75 %.

Table 3. Difference of flow time of cement pastes (N) after 5 min and 60 min

SP content, %	$\tau_{5 \text{ min}} - \tau_{60 \text{ min}}, \text{ min:s}$		
	PC N 0.30	PLC N 0.32	PLC N 0.30
0.50	3:20	1:19	4:05
0.75	2:24	0:32	1:30
1.00	2:17	0:32	1:15

As it is seen, the presence of limestone particles in cement has a positive effect on fluidity properties of plasticized cement paste. The fluidity of paste PLC N is better and its loss is slower, than of pastes PC N. The investigations where W/C in paste PCL N was the same as in paste PC N, also showed that although initial flow time of paste PLC N is longer but it increases slower and this paste stiffens slower than paste PC N (Table 3).

The reduction of water content in plasticized pastes of the same flow is lower in case of PLC (Table 4).

Table 4. Water content in cement pastes (N) of the same flow

Cement type	W/C		Water reduction, %
	without SP	0.75 % SP	
PC N	0.30	0.20	33
PLC N	0.32	0.23	29

The results of dynamic viscosity measurements of non-plasticized and plasticized cement pastes of the same initial flow (W/C according Table 4) are provided in

Figure 2. As it seen, the viscosity of non-plasticized pastes PLC N regardless of higher W/C in it is higher than that of pastes PC N, i.e. the presence of limestone particles increases the viscosity of cement pastes. In all pastes without SP the character of viscosity variation up to 100 min. is similar, i.e. an inconsiderable gradual increase in viscosity is observed. The dynamic viscosity of plasticized pastes PC N and PCL N of the same flow is similar up to 40 min. But afterwards, the viscosity of paste PC N grows faster and the sharp growth of viscosity begins after 90 min. In plasticized pastes PLC N within period of 120 min., no sharp growth of viscosity was observed.

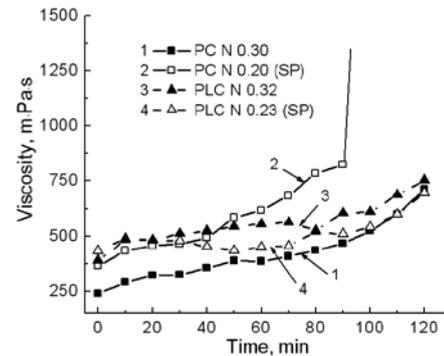


Fig. 2. Dynamic viscosity of cement pastes PLC N and PC N of the same flow (0.20; 0.23; 0.30 and 0.32 – W/C in pastes)

After addition of SP in pastes without any changes in W/C (according Table 2), the dynamic viscosity of pastes decreases greatly and its values in all pastes are close, moreover, in the period of 120 min. they increase very insignificantly.

EXO temperature and structure development in hardening cement pastes. The results of EXO temperature measurements of plasticized and non-plasticized pastes PC and PLC of the same initial flow are provided in Figure 3.

As can be seen, the hydration process in pastes PLC N is slightly slower than in pastes PC N: induction period is longer, the maximum EXO temperature is lower and duration of its reaching is longer. The changes of EXO temperature in both plasticized cement pastes PC N and PLC N versus non-plasticized pastes are similar: the induction period is slightly shorter, the EXO temperature is growing more rapidly and is higher.

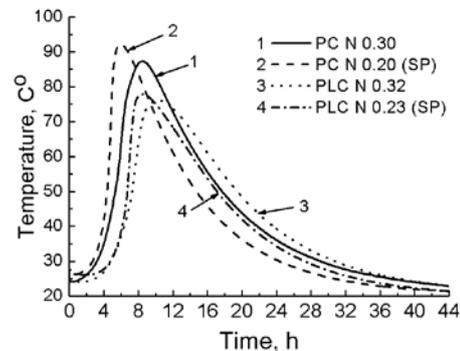


Fig. 3. EXO temperature of pastes PC N and PLC N of the same initial flow (0.20; 0.23; 0.30 and 0.32 – W/C in pastes)

The more rapid hardening of plasticized cement pastes of the same initial flow is also indicated by the results of measurements of setting times (Table 5). The plasticized

cement pastes set more rapidly (especially the final setting time is significantly shortened). The setting of cement paste PLC N is slower than of paste PC N.

Table 5. Setting times of cement pastes PC N and PLC N

Setting time, initial/final, min			
PC N		PCL N	
without SP	0.75 % SP	without SP	0.75 % SP
190/390	165/260	295/485	275/430

UWV investigations also confirmed that hardening structure development in plasticized pastes of the same flow is faster in the period of early hardening. UWV test method enables to observe structure development processes and it is used widely nowadays [22, 23]. The results of UWV measurements are provided in Figure 4.

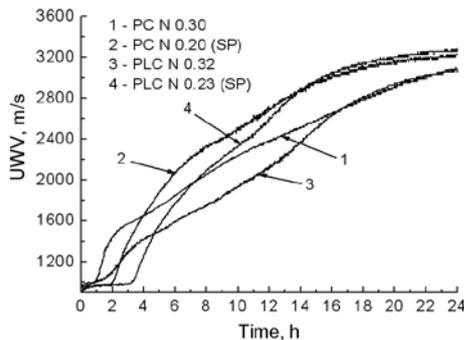


Fig. 4. UWV in the hardening cement pastes (N) of the same flow

As it can be seen, notwithstanding the fact that induction period in plasticized cement pastes is longer, development of structure is going more rapidly. The UWV values of plasticized and non-plasticized cement pastes equalize after about 4 h. After 24 h the microstructure of plasticized pastes is notably more compact. The induction period is also longer in PLC pastes.

3.2. Cement pastes PC and PLC of type R

Rheological behaviour. The results of fluidity investigations are provided in Figure 5 and Table 6. SP was added into cement pastes in which W/C was the same as in non-plasticized pastes of the same flow (Table 2).

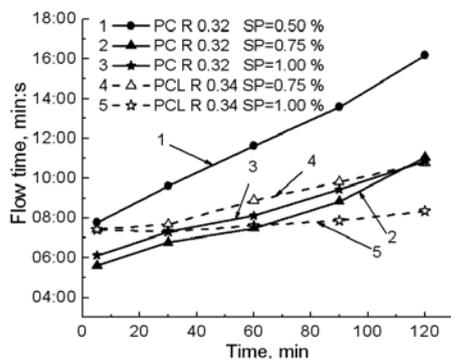


Fig. 5. Flow time of cement pastes (R) with different SP content (0.32 and 0.34 – W/C in cement pastes)

It can be also seen that as in case of cement pastes of type N, the fluidity of cement pastes of type R with 0.75 % and 1 % SP, is similar, and in the course of 60 min. the loss

of fluidity is inconsiderable. The fluidity of cement pastes PLC R also decreases slower comparing with cement pastes PC R. However, the plasticizing effect in cement paste PLC R at a lower content of SP (0.5 %) is considerably worse (initial flow time is 40 min, it thickens after 30 min and does not flow).

The results of investigations, where the W/C in cement pastes PC R and PLC R was the same, i.e. 0.32, showed that although initial flow time of pastes PLC is longer but it increases slower, i.e. they thicken slower than pastes PC (Table 6).

Table 6. Difference in flow time of cement pastes (R) after 5 min and 60 min

SP content, %	$\tau_{5 \text{ min}} - \tau_{60 \text{ min}}, \text{ min:s}$		
	PC R 0.32	PLC R 0.34	PLC R 0.32
0.50	3:52	not flow	not flow
0.75	1:52	1:28	1:35
1.00	1:52	0:12	0:40

In case of PLC the reduction of water content in plasticized pastes of the same flow is also lower (Table 7).

Table 7. Water content in cement pastes (R) of the same flow

Cement type	W/C		Water reduction, %
	without SP	0.75 % SP	
PC R	0.32	0.21	38
PLC R	0.34	0.23	32

The results of dynamic viscosity measurements of plasticized and non-plasticized cement pastes are provided in Figure 6.

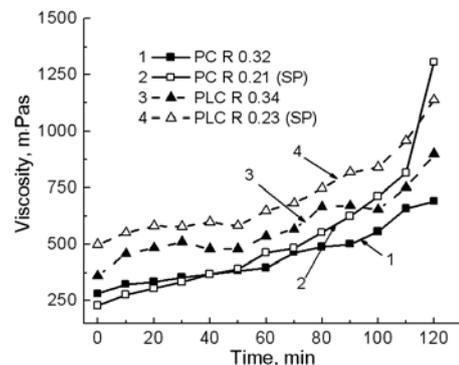


Fig. 6. Dynamic viscosity of PLC R and PC R pastes of the same flow (0.21; 0.23; 0.32 and 0.34 – W/C in pastes)

It may be seen that the effect of limestone particles on dynamic viscosity of cement pastes of type R is little different comparing with pastes of type N. The dynamic viscosity of cement pastes of type R increases a little bit faster and the presence of limestone particles increases the viscosity not only of non-plasticized, but also of plasticized pastes. The sharp growth of viscosity of plasticized PLC R begins at a similar time as of paste PC R.

EXO temperature and structure development in hardening cement pastes. The results of investigations into EXO temperature in plasticized and non-plasticized pastes PC R and PLC R of the same initial flow are provided in Figure 7.

It may be seen that the character of EXO temperatures in plasticized and non-plasticized cement pastes of type R is slightly different comparing with cement pastes of type N. Although as in case of cement pastes of type N, the EXO temperature is growing more rapidly and is higher in plasticized cement pastes. The maximum EXO temperature is lower in PLC pastes, but the difference between maximum EXO temperatures in cement pastes PC and PLC of type R is smaller comparing with cement pastes PC and PLC of type N. Moreover, the duration of its reaching in cement pastes PLC R is almost the same as in cement pastes PC R.

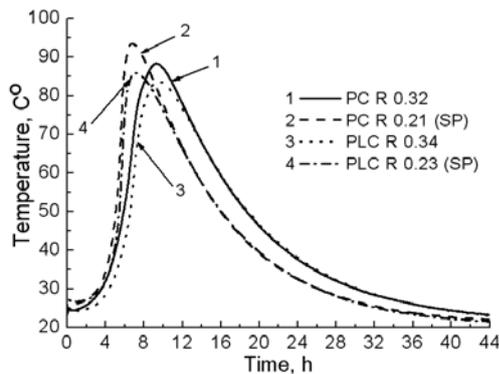


Fig. 7. EXO temperature of pastes PC R and PLC R of the same flow (0.21; 0.23; 0.32 and 0.34 – W/C in pastes)

The results of measurement of setting times of plasticized and non-plasticized cement pastes of type R are given in Table 8. It may be seen that as well as in cement pastes of type N, plasticized pastes set more rapidly and the setting of cement paste PLC R is slower than of PC R. But in case of cement paste of type R the shortening of initial setting time of plasticized cement pastes versus non-plasticized is more significant comparing with cement pastes of type N.

Table 8. Setting times of cement pastes PC R and PLC R

Setting time (initial/final), min			
PC R		PCL R	
without SP	0.75 % SP	without SP	0.75 % SP
235/370	185/355	285/450	230/380

The results of UWV investigations are provided in Figure 8.

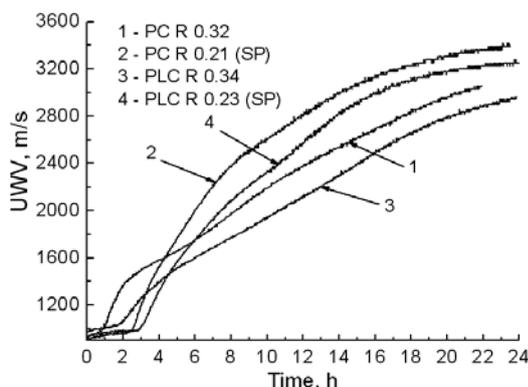


Fig. 8. UWV in the hardening cement pastes (R) of the same flow

One can see that the character of structure development in hardening cement pastes of type R is the same as

in pastes of type N. In plasticized pastes of type R, the induction period is longer comparing with non-plasticized. In pastes PLC R this period is longer comparing with pastes PC R. Further structure development of plasticized cement pastes is going more rapidly and after 24 h the microstructure of plasticized pastes is more compact comparing with non-plasticized.

The same tendency in hardening structure development is observed during further periods of hardening as well: in the course of 3 months the rate of structure development in all samples are of similar character and the UWV values remain higher in plasticized samples.

4. DISCUSSION

The investigations of PC and PCL pastes showed that the presence of limestone particles, also fineness of cement, changes the effect of SP on rheological properties of cement pastes and its effect does not unambiguous. It is obvious, that contrary results were obtained, while comparing the impact of limestone additives on the plasticizing efficiency according to the fluidity of freshly mixed pastes (March cone method) and reduction of water in plasticized cement pastes of the same flow (Suttard cylinder method). When W/C is from 0.30 to 0.34, the presence of limestone particles improves the effect of SP on the fluidity properties of cement pastes (flow time and loss of fluidity). But when W/C is much lower (from 0.20 to 0.23) the presence of limestone particles worsens flow effect of cement pastes. Benefit effect of limestone powder on the fluidity of cement pastes state also authors [5]. Much fine limestone particles has “lubrication” effect on the clinker particles as well as on aggregate particles in mortar and concrete [7, 11], however, as shown by our investigations, this effect does not manifest itself in plasticized cement pastes at low W/C. The measurements of dynamic viscosity of these cement pastes also showed that the presence of limestone particles increased it. Such effect may be partly determined by high MgO content (2.98 %) in limestone [15].

In comparison of cement pastes of type R and N, one can see that the influence of fineness on fluidity of plasticized pastes PC and PLC is different: SP is more effective in finer cement PC R, while in the case of cement PLC the results are contrary. In plasticized cement pastes for reduction of water SP is more effective in cements of higher fineness (of type R).

In plasticized cement pastes of the same initial flow the hydration and hardening process are going faster comparing with non-plasticized. Many authors indicate that PCE slows down the hydration of cement [15, 20, 21]. In our investigations the more intense hydration, setting and development of hardening structure in plasticized pastes was most likely determined by a considerably lower W/C in them.

CONCLUSIONS

These investigations in which the same consistency, i. e. the same flow of cement pastes ($\phi \sim 120$ mm according Suttard’s cylinder) is taken as a basic index leads to the following conclusions:

– presence of limestone particles in cement worsen SP effect on reduction of water to obtain the cement pastes of the same flow. Water reduction for pastes PLC (respectively N and R) was 29 % and 32 %, and for pastes PC – 33 % and 38 %.

– the dynamic viscosity of plasticized and non-plasticized cement pastes PLC is higher than of pastes PC. However pastes PLC thicken more slowly and the period of conditional stability of dynamic viscosity (500–1000 Pa·s) is longer there.

– the fluidity of plasticized cement pastes PLC determined by March cone method is better than of cement pastes PC. The loss of fluidity of plasticized cement pastes PLC also runs slower versus pastes PC.

– the investigations of EXO temperature in hardening cement pastes of the same flow showed that hydration process runs slower in pastes PLC. The nature of impact of SP on EXO temperature of pastes PC and PLC is similar: the maximal EXO temperature is reached more rapidly and it is higher, i.e. hydration process in plasticized cement pastes is more intensive. The development of microstructure in plasticized cement pastes and its setting runs also more rapidly. This is due to lower W/C in it.

Acknowledgments

This work was supported by Research Council of Lithuania (contract No ATE-07/2012). Authors also want to thank JSC Akmenės Cementas for the technical support.

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