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## Variability in cement properties - influence on bleeding of cement paste

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# Variability in cement properties - influence on bleeding of cement paste

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**Abstract.** The aim of this study is to find relation between cement properties and tendency of cement paste (concrete) to bleeding. We tested 19 cement samples collected from different batches. On cements were tested granulometry, specific surface, strengths, alkali content, content of C<sub>3</sub>A modifications, setting times and bleeding. Tendency to bleeding of all 19 samples was tested in 3 types of cement paste mixtures. One reference mix and two mixtures with superplasticizers from different producers. Results show, that we can see partly relation between specific surface area and tendency to bleeding. With lower specific surface area, the tendency to bleeding is higher. While the content of alkali seems to have small influence. Where the higher content of alkali means higher tendency to bleeding. On the other hand, these results are not conclusive and it is required further testing on different parameters such as content of sulphur and type of gypsum added to clinker during grinding.

## 1. Introduction

Cement is one of the most worldwide used binder in building materials. In European Union is cement under control of standard EN 197 and tested according to EN 196. This standard controls strength, chemical composition, setting times, fineness, heat of hydration and amount of water soluble chromium. If all these properties are in allowed range, cement should have constant behavior in concrete mixture. Nevertheless, sometimes there are differences in concrete behavior on situ. Even if the composition of concrete mixtures is constant, workability is different and we can see signs of concrete bleeding in various intensities.

Bleeding is a phenomenon where water in a freshly mixed cement-based composite is drained to the surface when solid component of the mixture consolidate in a form [1]. Bleeding influences the quality of fresh concrete and durability after the concrete is hardened [2]. There were several bleed channels and pores on the surface of freshly placed concrete, which bled too much [3]. It is known that concretes which exhibit less bleeding, have a stronger surface [4].

During the investigations, it was observed that concrete mixtures containing high cement contents reduce bleeding [5]. When cements having high fineness were used, the quality of bleeding and the rate of bleeding were decreased. It was also found that cements having a high C<sub>3</sub>A content reduces bleeding [6].

Topic for our research came from a concrete producer. They have a problem with instability of concrete behavior on situ. Even if they use the same composition of mixture, concretes with cements from different batches have different behavior - sometimes there were signs of bleeding. In



cooperation with cement producer we took 19 samples of cements from consecutive batches in cement plant. Samples were determined on granulometry, alkali content, C<sub>3</sub>A content and its modifications, setting time, strengths and tendency to bleeding.

## 2. Materials and Methods

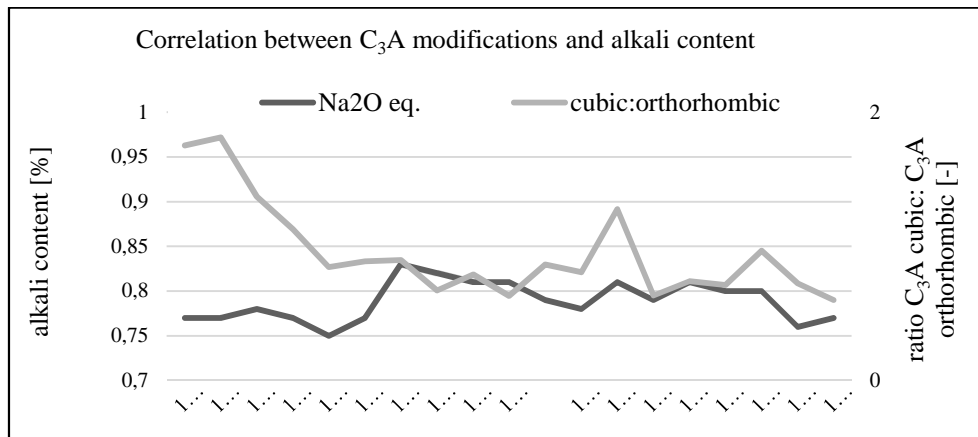
Tendency to bleeding was tested on 3 types of cement paste mixtures. In these mixtures were used all 19 samples of cement. All tests were done in laboratory room with temperature around 22 °C. First mixture - REFERENCE - plain cement paste, second mixture - with superplasticizer Dynamon SX 14, third mixture - with superplasticizer Stachement 2489

### 2.1. Tested cements

19 samples of cement CEM I 42.5 R were taken from consecutive batches in the time span from September to November 2017. C<sub>3</sub>A and alkali content in cement is show in table 1.

**Table 1.** C<sub>3</sub>A and alkali content.

Sample no.	C3A			alkali		
	cubic	ortho-rhombic	total	K <sub>2</sub> O	Na <sub>2</sub> O	Na <sub>2</sub> O eq.
<b>1505</b>	3.75	2.14	5.89	0.96	0.14	0.77
<b>1535</b>	3.86	2.13	5.99	0.96	0.14	0.77
<b>1553</b>	3.37	2.46	5.83	0.97	0.14	0.78
<b>1580</b>	3.17	2.81	5.98	0.94	0.15	0.77
<b>1601</b>	2.86	3.38	6.24	0.91	0.15	0.75
<b>1630</b>	3.02	3.40	6.42	0.94	0.15	0.77
<b>1651</b>	2.48	2.76	5.24	1.00	0.17	0.83
<b>1675</b>	2.32	3.46	5.78	0.99	0.17	0.82
<b>1692</b>	2.51	3.18	5.69	1.00	0.15	0.81
<b>1721</b>	2.23	3.54	5.77	0.97	0.17	0.81
<b>1738</b>	2.54	2.94	5.48	0.95	0.16	0.79
<b>1767</b>	2.52	3.12	5.64	0.96	0.15	0.78
<b>1785</b>	2.81	2.20	5.01	0.95	0.18	0.81
<b>1810</b>	2.36	3.73	6.09	0.95	0.16	0.79
<b>1827</b>	2.34	3.16	5.50	0.99	0.16	0.81
<b>1850</b>	2.71	3.81	6.52	0.97	0.16	0.80
<b>1867</b>	2.92	3.02	5.94	0.97	0.16	0.80
<b>1889</b>	2.52	3.48	6.00	0.93	0.15	0.76
<b>1905</b>	2.35	3.92	6.27	0.93	0.16	0.77



**Figure 1.** Correlation between content of C<sub>3</sub>A modifications and content of alkali.

Total content of C<sub>3</sub>A ranges between 5.0 and 6.5 % and the ratio between cubic and orthorhombic modification is changing from 0.6 to 1.8. As it can be seen on Figure 1 with higher content of alkali is lower ratio C<sub>3</sub>A cubic / C<sub>3</sub>A orthorhombic.

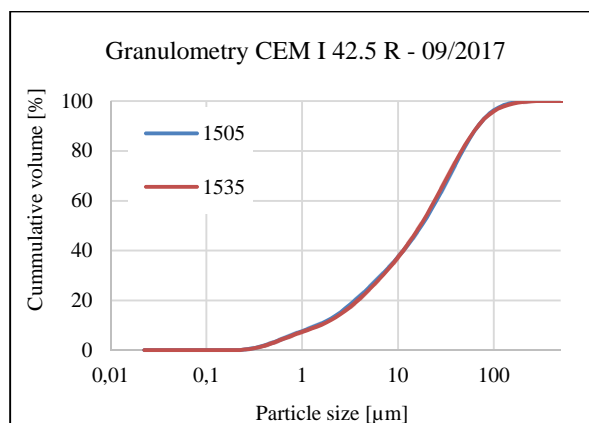
### 2.2. Superplasticizer

For preparation testing cement pastes were used two types of superplasticizers. Dynamon SX 14 from MAPEI producer which is a totally formaldehyde-free water solution of acrylic polymers that can effectively disperse cement granules with secondary components that significantly improve the cohesion and the pumpability of concrete. Second admixture was Stachement 2489 from Stachema producer. Stachement 2489 is polycarboxylate based admixture with high plasticizing effect especially used for ready-mix concrete.

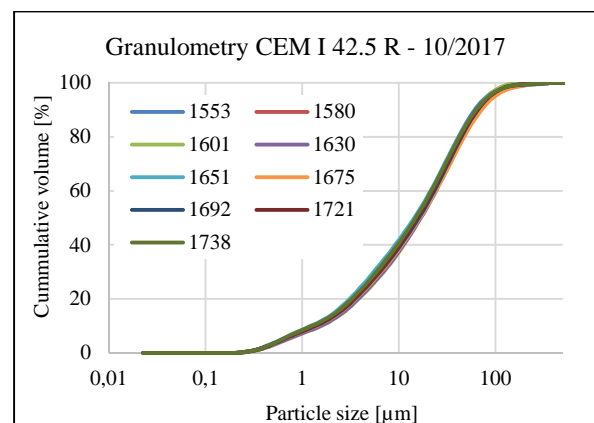
## 3. Results and discussion

### 3.1. Granulometry and specific surface area

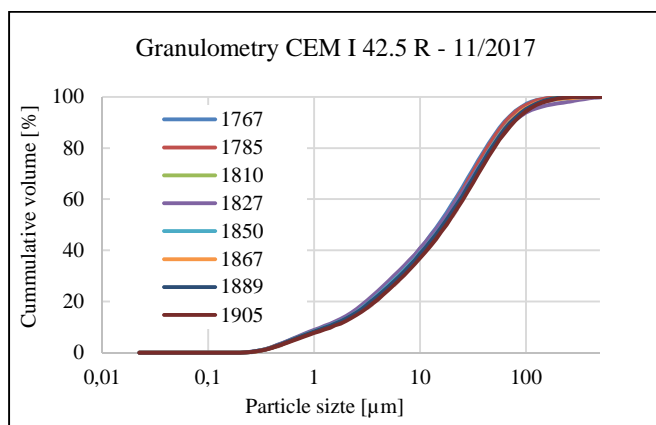
Cement granulometry was determined by laser particle size analyser Mastersizer 2000. Results of the analysis are shown in Figure 2 - 4. As the second parameter of particle size distribution was determined the middle particle size - Figure 5-6. Specific surface area was determined by automatic analyser according to CSN EN 196-6 - Permeable method (Blaine).



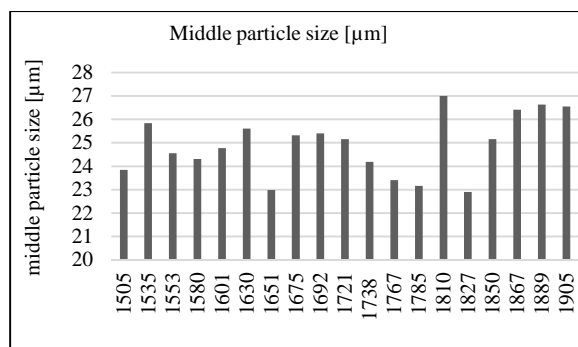
**Figure 2.** Granulometry CEM I 42.5 R- September.



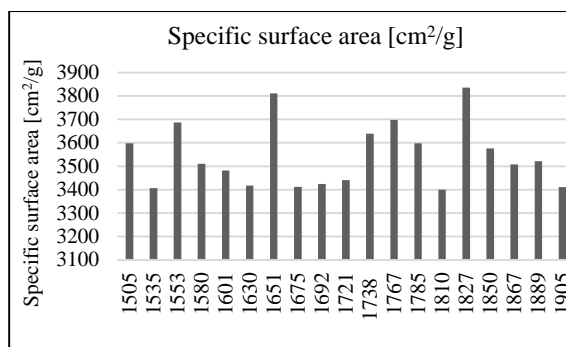
**Figure 3.** Granulometry CEM I 42.5 R- October.



**Figure 4.** Granulometry CEM I 42.5 R – November.



**Figure 5.** Middle particle size - CEM I 42.5 R.



**Figure 6.** Specific surface area - CEM I 42.5 R.

As it can be seen in Figure 2 - Figure 4 granulometry of cements is quite stable. Even if granulometry of cement seems to be constant, differences are in middle particle size as can be seen in Figure. Value of middle particle size is in range 23 - 27 µm. Specific surface area is variable too and is in the range of 3400 - 3820 cm<sup>2</sup>/g.

**3.2. Setting time and strengths**

Setting time was determined according to CSN EN 196-3. Tensile strength and compressive strength were determined according to CSN EN 196-1. Results are shown in table 2.

Sample no.	Setting time [min]	Tensile strength [N/mm <sup>2</sup> ]		Compressive strength [N/mm <sup>2</sup> ]		Sample no.	Setting time [min]	Tensile strength [N/mm <sup>2</sup> ]		Compressive strength [N/mm <sup>2</sup> ]	
		2 days	28 days	2 days	28 days			2 days	28 days	2 days	28 days
<b>1505</b>	196	6.0	9.3	34.0	60.7	1738	227	5.6	9.0	32.5	58.7
<b>1535</b>	216	5.6	8.8	30.8	57.2	1767	196	5.9	8.8	32.8	59.7
<b>1553</b>	197	5.8	8.7	33.5	59.8	1785	220	5.4	8.7	31.0	57.8
<b>1580</b>	219	5.5	8.6	29.6	59.0	1810	246	5.4	9.2	29.7	57.4
<b>1601</b>	229	5.6	8.8	30.5	59.4	1827	211	6.2	9.1	35.3	59.8
<b>1630</b>	235	5.6	9.1	30.7	58.4	1850	215	5.8	8.6	32.2	58.6
<b>1651</b>	191	5.9	9.4	34.3	59.9	1867	241	5.7	8.6	32.4	58.7
<b>1675</b>	225	5.4	8.7	30.7	56.6	1889	246	5.7	8.8	32.9	59.1

<b>1692</b>	231	5.4	9.2	29.4	56.8	1905	236	5.7	8.8	30.4	56.6
<b>1721</b>	251	5.3	8.9	30.6	56.8						

Whereas the cement is supposed to have compressive strength in 28 days 42.5 N/mm<sup>2</sup>, the range of 56.6 - 60.7 N/mm<sup>2</sup> is enough. Setting time is in the range of 191 - 251 minutes. This variability is wide but still in allowed limits.

**Table 2.** Setting time, tensile strength and compressive strength.

Sample no.	Setting time [min]	Tensile strength [N/mm <sup>2</sup> ]		Compressive strength [N/mm <sup>2</sup> ]		Sample no.	Setting time [min]	Tensile strength [N/mm <sup>2</sup> ]		Compressive strength [N/mm <sup>2</sup> ]	
		2 days	28 days	2 days	28 days			2 days	28 days	2 days	28 days
<b>1505</b>	196	6.0	9.3	34.0	60.7	1738	227	5.6	9.0	32.5	58.7
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<b>1630</b>	235	5.6	9.1	30.7	58.4	1850	215	5.8	8.6	32.2	58.6
<b>1651</b>	191	5.9	9.4	34.3	59.9	1867	241	5.7	8.6	32.4	58.7
<b>1675</b>	225	5.4	8.7	30.7	56.6	1889	246	5.7	8.8	32.9	59.1
<b>1692</b>	231	5.4	9.2	29.4	56.8	1905	236	5.7	8.8	30.4	56.6
<b>1721</b>	251	5.3	8.9	30.6	56.8						

3.3. Bleeding

Tendency to bleeding was determined on cement pastes. Compositions of mixtures are in

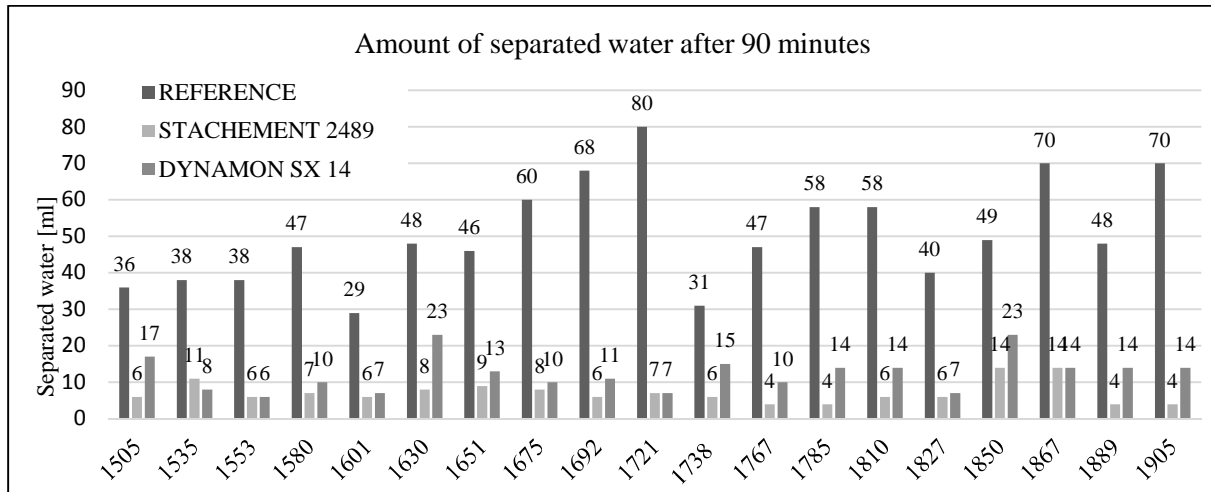
	name of mixture		
	Reference	DYNAMON SX 14	STACHEMENT 2489
cement CEM I 42,5 R	450.0 g	500.0 g	500.0 g
Water	292.5 g	175.0 g	175.0 g
superplasticizer Dynamon SX 14	-	2.5 g	-
superplasticizer Stachement 2489	-	-	2.5 g

Cement was put into water and measuring of time started. Cement paste was mixed for 1 minute by plastic spatula. Mixed paste was put into 500 ml glass volume cylinder and the high of level was noted. After 15 and 90 minutes was determined level of separated water. After 90 minutes water from the top pipetted out with small amount of cement. After drying we got real amount of separated water - intensity of bleeding. Separated water was recounted according to real amount of cement paste. Amount of separated water is indicator of cement paste tendency to bleeding.

**Table 3.** Composition of cement paste mixtures for bleeding determination.

	name of mixture		
	Reference	DYNAMON SX 14	STACHEMENT 2489
cement CEM I 42,5 R	450.0 g	500.0 g	500.0 g

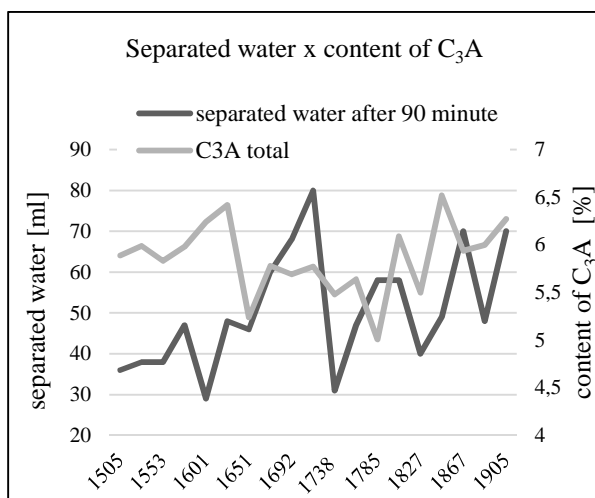
Water	292.5 g	175.0 g	175.0 g
superplasticizer Dynamon SX 14	-	2.5 g	-
superplasticizer Stachement 2489	-	-	2.5 g



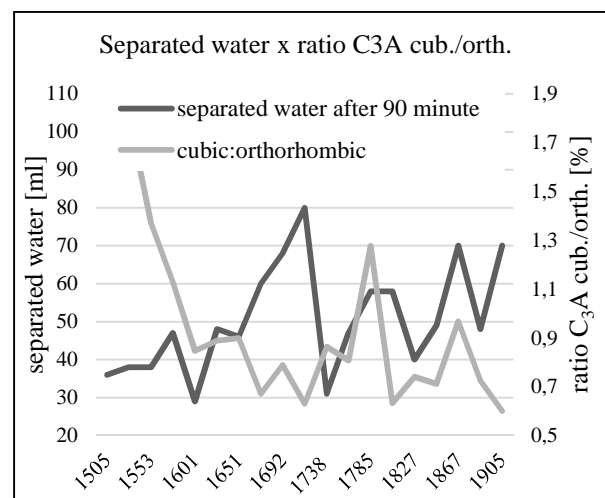
**Figure 7.** Amount of separated water after 90 minutes.

In 15 minutes, the amount of separated water is around 7 ml without superplasticizer and around 3 ml with admixture. Difference between with/without admixture is more visible after 90 minutes. Whereas the mixtures without superplasticizer have up to 80 ml of separated water, the mixtures with plasticizer have less than 23 ml. It is apparent, that the variability in amount of separated water is quite wide. With admixture adding, the variation is smaller. Of course, this fact is caused by reducing water-cement ratio. Nevertheless, this can be the manner how to reduce variability in the concrete tendency to bleeding.

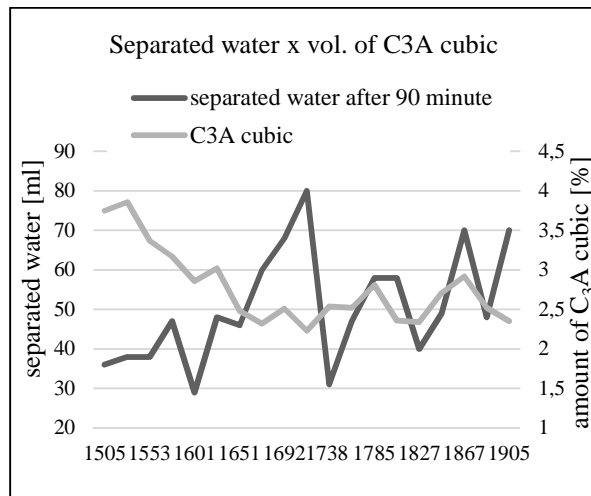
The comparisons of cement properties and the amount of separated water from reference mixture after 90 minutes are shown on the following Figures.



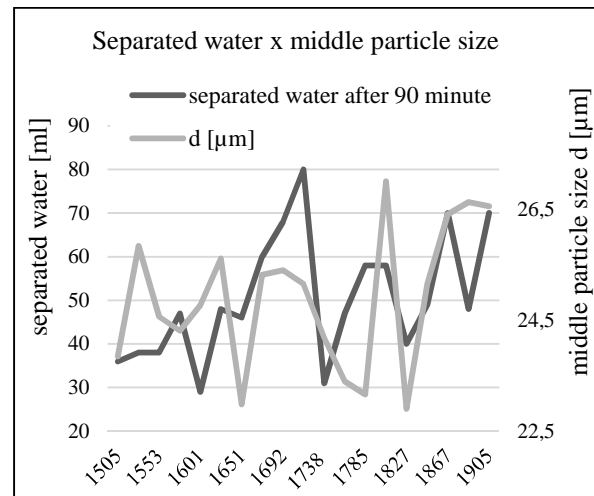
**Figure 8.** Relation between content of C<sub>3</sub>A and amount of separated water.



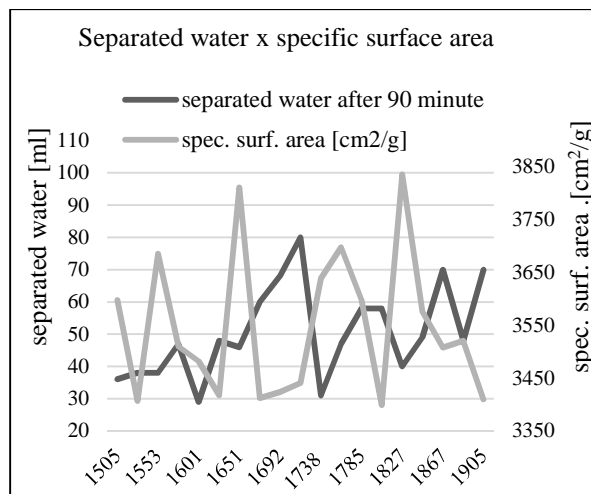
**Figure 9.** Relation between ratio of C<sub>3</sub>A modification and amount of separated water.



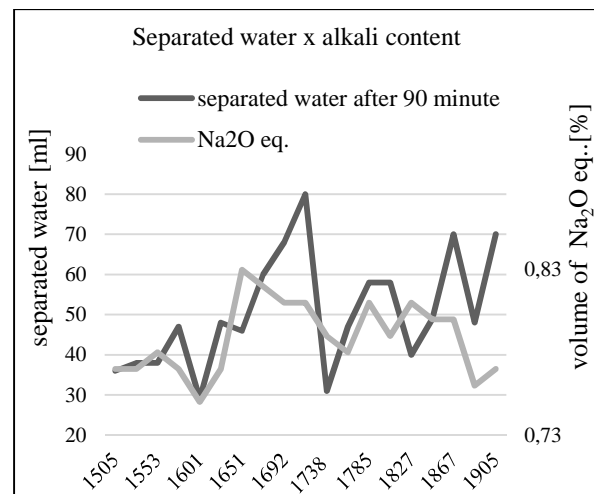
**Figure 10.** Relation between content of C<sub>3</sub>A cubic and amount of separated water.



**Figure 11.** Relation between middle particle size and amount of separated water.



**Figure 12.** Relation between specific surface area of cement and amount of separated water.



**Figure 13.** Relation between content of alkali and amount of separated water.

As it can be seen on Figure 8, the total content of C<sub>3</sub>A is not valid parameter to describe tendency of cement paste to bleeding. Same situation is visible on Figure 10 where is compared the content of C<sub>3</sub>A in cubic modification with the amount of separated water. On samples 1785 - 1905 (cements produced in November) can be seen the correlation between content of C<sub>3</sub>A cubic and amount of separated water. With increasing amount of these C<sub>3</sub>A modifications is visible increasing amount of separated water. On Figure 9 is amount of separated water compared with the ratio of cubic and orthorhombic modification of C<sub>3</sub>A. Higher ration leads to higher tendency to bleeding. These results do not match the assumption, that higher amount of orthorhombic modification of C<sub>3</sub>A increases tendency to bleeding. Comparison on figure 12 is between specific surface area of cements and amount of separated water. Here we can see demonstrable the dependence of tendency to bleeding on specific surface. With higher specific surface area of cement the tendency to bleeding is lower. On Figure 13 where is compared the amount of separated water with content of alkali is visible the dependence of these two parameters. Tendency to bleeding is higher with higher amount of alkali.



#### 4. Conclusion

As mentioned in previous chapter, major influence on tendency to bleeding looks to have specific surface area or middle particle size. With higher specific surface tendency to bleeding decrease. Partial influence has ratio between content of C<sub>3</sub>A cubic and orthorhombic. Higher amount of orthorhombic C<sub>3</sub>A leads to higher tendency to bleeding. Addition of superplasticizer helps decrease tendency to bleeding, because of lower water/cement ratio. Whereas the results are not final another testing is requested. As another parameter, the content of sulphur and type of gypsum added to clinker during the grinding will be taken into consideration.

#### Acknowledgment

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