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Research paper

Properties of mortars with Calcium Sulfoaluminate cements with the addition of Portland cement and limestone

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Abstract: Calcium Sulfoaluminate cements (CSA) may be an alternative to Portland cements due to their very high early strength and more environmentally friendly production technology, however they are characterized by a short setting time and high cost. A possible solution to these problems is to mix CSA cement with other binders or additives. In order to test this possibility, CSA cement was mixed with Portland cement and limestone in the amount of 10, 20 and 30 wt. %. A hydration heat test was carried out in the first 72 hours after the components were mixed, measured were compressive and flexural strength after 1, 2, 7 and 28 days, and rheological properties, including early shrinkage. A negative interaction between CSA and CEM I 42.5R was observed, leading to deterioration of mechanical properties of the mortars. The study did not indicate a similar negative interaction between CSA cement and limestone.

Keywords: Calcium Sulfoaluminate cements, CSA cements, mortar properties, rheological properties, limestone

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1. Introduction

The need to decrease the carbon footprint has become one of the main concerns of the cement industry, due to the high carbon dioxide production in the process of Portland clinker burning, which amounts to between 0,6 - 0,8 t of CO_2 per 1 t of Portland clinker [1–4]. This situation creates the demand for less widely popularized clinkers and cements that contain no Portland clinker, such as alkali- activated binders [5] or Calcium Sulphoaluminate cements [6].

Calcium Sulfoaluminate (CSA) cement is a mineral hydraulic binder made with clinker obtained by burning limestone, bauxite and gypsum [7]. Main mineral phases of the clinker are ye'elemite ($C_4A_3\hat{S}$), belite (C_2S), and calcium sulphate ($C\hat{S}$). Hydration process of CSA clinker is drastically different from the hydration of ordinary Portland cement (OPC), as the main product of the reaction is ettringite ($C_6A\hat{S}_3H_{32}$), stratlingite $C_2A\hat{S}H_8$, monosulphate, and low amounts of CSH [8,9]. Due to its different hydration process, CSA cement is characterized by short setting time, high early strength and low shrinkage [10,11]. It is also characterized by a lower CO_2 emissions in comparison to OPC clinker [12,13]. However, CSA cement is not commonly used, due to high cost of bauxite required in the clinker production. Therefore research of CSA cements often focuses on mixing CSA cement with other binders or additives such as limestone [14].

It must be noted, however, that the topic of properties of CSA cements with an addition of OPC (in amount lower than 50% of cement mass) is not well developed in the available literature, as the most prominent direction of the research is connected to addition of 10-30% of CSA to OPC [15]. Research by Huang et al. [16] had shown that the rheological properties of mortars were dependant on the ratio between CSA and OPC; with the increase of CSA content in relation to OPC, the yield stress and viscosity increased. The results presented by Le Saoût et al. [17] and Chaunsali and Mondal [18] indicated that mixing OPC with 10-15% of CSA caused a significant expansion of cement paste. The research into the compressive strength of mortars with CSA-OPC mixes had shown that the addition of 20-50% of OPC to CSA may have negative effect on the strength [19]. Moreover, the research of Huang et al. [16] had shown that addition of OPC to CSA cements in amount of 10-50% increased the early heat flow. In similar vein, there is very little information concerning the properties of the CSA cements with limestone. Available studies conducted by Martin at al. [14], and Pelletier-Chaignat et al. [20] show the possibility of a beneficial effect of limestone on the hydration of CSA cements, due to stabilization of ettringite.

The aim of presented research was to investigate the properties of fresh and hardened mortars with CSA cement mixed with ordinary Portland cement CEM I 42.5R (OPC) or limestone in amount of

10, 20 and 30% of cement mass. Following properties were tested: heat of hydration in the first 72 h of hydration, early shrinkage, rheological properties of fresh mortar, as well as compressive and flexural strength of mortars.

2. Materials and testing methods

The composition of the CSA cement, ordinary Portland cement CEM I 42.5R and limestone which were used in the research are presented in Table 1. The phase composition of the cements is shown in Table 2. The cements were prepared by homogenizing CSA cement with 10, 20 and 30% of OPC or limestone, measured by cement mass. The compressive strength after 28 days of CSA cement used in the research was 66.4 MPa, and of CEM I 42.5R - 52.8 MPa.

Table 1: Chemical composition of materials used in the research

Cement type	Constituent [%]									
	LOI	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Na_2O	K_2O	Na_2O_{eq}
CSA	0.46	9.2	28.1	1.52	39.2	3.5	11.4	0.08	0.35	-
CEM I 42,5R	2.8	20.5	4.67	2.8	64.35	1.18	2.79	0.18	0.43	0.46
Limestone (LL)	42.7	1.4	0.4	0.5	53.2	1.5	0.02	-	-	-

Table 2: Phase composition of cements used in the research

Cement type	Constituent [%]						
	C_3S	C_2S	C_3A	C_4AF	$C_4A_3\hat{S}$	$C\hat{S}$	$3C_2S$* $3C\hat{S}$*CaF_2
CSA	-	10.4	-	1.2	64.9	5	9.4
CEM I 42,5R	62.4	12.2	7.6	8.5	-	~2	-

The mortars that were used for the tests were prepared on the basis of the standard mortar according to EN-196-1 [21], meaning 450 g of cement, 1350 g of standard sand, and 225 g of water. The standard w/c ratio of 0.5 was changed in case of rheological parameters to 0.6 due to the technical issues. Only in case of the heat of hydration, the tests have been conducted on cement paste, with w/c ratio of 0.5. Flexural and compressive strengths were tested according to the procedure described in standard EN-196-1 [21], after 1, 2, 3, 7 and 28 days of curing. Three prismatic specimen were used to determine flexural strength, and six half-prismatic specimen – for compressive strength tests. Early shrinkage in the first 24 h was determined by laser measurement system Shrinkage Cone deltaEL, that allows continuous measurement of changes in height of a fresh mortar cone. Due to its shape, linear changes in cone height correspond to a volumetric change in size, and therefore cone height can be used as a way to measure shrinkage of fresh mortar. During the measurement, the apparatus with the sample were kept in a climatic chamber in a temperature 20^{o}C and humidity 60%.

Rheological properties, namely yield stress and plastic viscosity, were measured in a rheometer 5 min from mixing and calculated using simplified Bingham model. The Heat of hydration was tested for 72 h in an isothermal calorimeter TAMair, according to the standard EN 196-11 [22].

3. Results and discussion

2.1. Heat of hydration

The results of test of heat of hydration are presented in Fig. 1. As it can be seen, CSA cement hydration is rapid, with basically no induction phase, as the lowest heat flow during first 20 h of hydration, is still higher than the maximum heat flow of OPC hydration. The heat flow of CSA cement hydration peaked after 4 h, and the cement was mostly hydrated after 20 h mark.

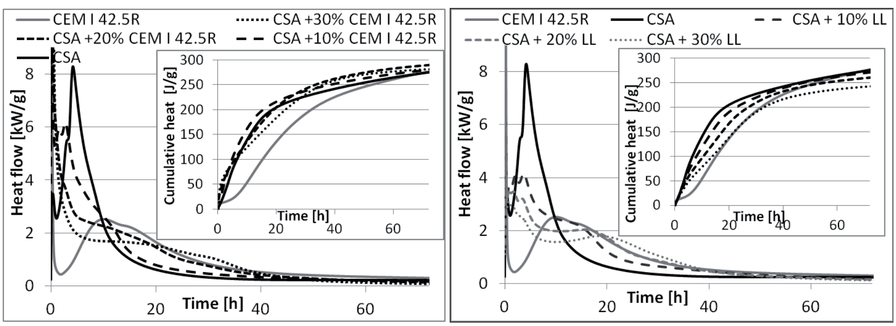

Fig. 1. Heat flow and cumulative heat of CSA cement with OPC(left) and limestone (right)

In case of OPC, the induction phase lasted for around 3 h, and the peak of heat flow occurred around 10 h mark. It should be noted, that the cumulative heat of CSA cement and OPC chosen for the research is similar. Adding 10-30% of OPC to CSA cement had drastically changed the hydration process. It can be noticed, that addition of 10% OPC accelerates the initial hydration, moving the peak heat flow to the 3^{rd} hour after mixing binder with water. This may be attributed to the gypsum and free lime from OPC reacting with ye'elimite and water to create additional ettringite, as was proposed by Li et al. in [23] and [24]. At the same time, hydration of OPC in case of its 20 and 30% addition to the binder is visibly slowed down, what is noticeable both in case of heat flow and cumulative heat. As it can be seen, in case of CSA cement with 30% OPC addition, the heat flow peaks connected to C_3S and C_3A reaction which for OPC occur after 10 and 14 h respectively, are not readily visible, and the reaction seems to be prolonged up to 40 h of hydration. This could be connected to the rapid reaction of ye'elimite, as the products of this reaction may create a barrier between unreacted clinker particles and available water. As the speed of hydration is considered to be dependent on the diffusion rate, this may lead to a visibly slower reaction [25,26].

The addition of limestone to CSA cement had significantly reduced the rate of heat emission, however visibly accelerated the early hydration. This effect, which was observed also by Pelletier-Chaignat et al. [20], may be connected to the filler effect, which prevents the agglomeration of the clinker particles and therefore allows for better hydration rate. At the same time, limestone particles act as a nucleation seeds, what can speed up the hydration process [27]. As indicated by research by Hargis et al. [28] and Pelletier-Chaignat et al. [20], limestone has also a chemical effect on CSA hydration, stabilizing ettringite by enhancing the formation of hemicarbonate and monocarbonate at the cost of monosulphate.

2.2. Shrinkage

CSA cement was characterized by almost four times lower early shrinkage than in case of OPC used in the research (Fig. 2), and the shorter time in which the shrinkage stabilized, as the shrinkage did not substantially change for CSA cement after 1 hour, while in case of OPC cement, this time was 5 h.

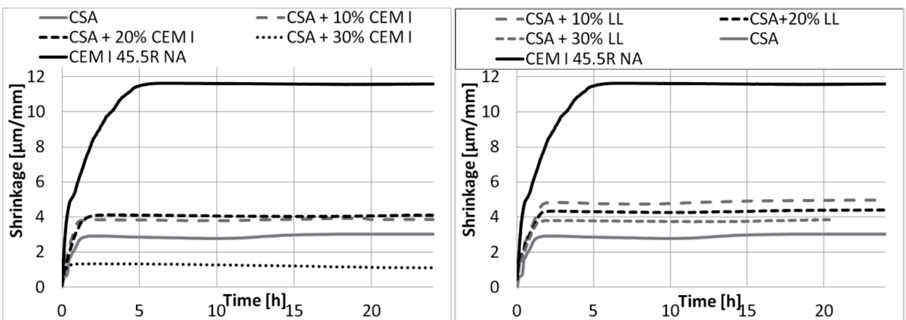

Fig. 2. Early shrinkage of CSA cement with OPC (left) and limestone (right)

CSA cements with an addition of 10% and 20% of OPC were characterized by increase in shrinkage, while the addition of 30% of OPC to CSA cement caused a decrease in the shrinkage. The low shrinkage of CSA cement is connected to high volume of non-expansive ettringite, which is the main product of basic ye'elimite hydration [29]. It should be noted that 30% addition of OPC to CSA led to the lowest early shrinkage, indicating the appearance of more expansive phases. This may be due to the different ye'elemite hydration process in the presence of alumina phases from Portland clinker, which leads to the production of expansive ettringite [30].

CSA cements with limestone addition in amount of 10-30 wt. % are characterized by a higher early shrinkage than in case of CSA cement, but also lower than in case of OPC . Increased shrinkage may be linked to the fact, that limestone particles reduce the capillary pore diameter due to the filler effect. This can lead to increased shrinkage in binders, as it is directly proportional to the capillary forces which increase as capillary pores decrease in size [31].

2.3. Rheology

The results of rheological tests are presented in Fig. 3. In case of CSA cements with 20% and 30% addition of OPC, it was impossible to conduct the measurement in the given timeframe (4 minutes of mixing procedure + 4 minutes rheological measurement), due to the rapid setting and loss of consistency that occurred during the measurement. This effect was present also in case of 10% CEM I addition, however the effect was less pronounced, and therefore led just to a significant, almost five-fold, increase in yield stress of CSA cement. This effect may be connected to the possible increase in ye'elimite hydration speed, that was apparent in the test of the heat flow (Fig. 3). Plastic viscosity of CSA-OPC blends was low, what was connected to a high w/c ratio [32].

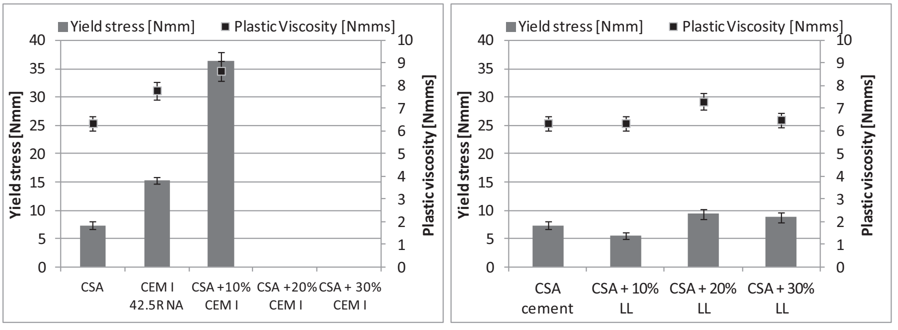

Fig. 3. Rheological parameters of CSA cements with OPC cement (left) and limestone (right).

Similarly negative effect on rheological properties was not observed in case of CSA cement with limestone addition. The yield stress and plastic viscosity did not change significantly with limestone addition, and remained consistently low (Fig.3). The lack of change in rheological properties of cement with increasing limestone addition is consistent with previous tests performed on mortars with OPC [33,34].

2.4. Compressive and flexural strength

The results of compressive strength tests are shown in Fig. 4. It can be noticed, that in case of substitution of CSA with both OPC and limestone, the compressive strength of mortars was lower than in case of mortars with only CSA cement. Substitution of 10-30 wt. % of CSA cement with OPC led to a substantial decrease in the compressive strength after 1 day, amounting to 60% drop in the case of the addition of 30% of OPC. Similar effect was observed by Biolzi et al. [35]. The substantial decrease in strength may be linked to the rapid early hydration of CSA which can negatively impact hydration rate by restricting the access to water for cement grains, what is supported by the results of heat of hydration tests described in chapter. 2.1.

It should be noted, however, that after 2 and 3 days of curing, the compressive strength of mortars with CSA and OPC is comparable to mortars with only CSA. After 28 days, the decrease of compressive strength is much lower, amounting to less than 15%. This drop in compressive strength may be due to the fact that the compressive strength of OPC after 28 days is lower than in case of CSA cement by 22%, so replacing CSA cement with OPC should result in a reduced strength.

Limestone addition to CSA cement does not show similarly negative influence on early compressive strength of mortars. Even though limestone is considered to be mostly inert [36], replacing 30% of CSA with limestone led to a decrease of compressive strength after 1 day by only 25%. This may be attributed to a beneficial effect of limestone on ye'elimite hydration, proved by Hargis at al. [37], as well as its filler effect, which allows for a higher degree of hydration of cementitious materials [20]. It should be noted, that the positive effect of limestone on hydration of CSA cement does not extend to strength characteristics after 3 days. Then, the compressive strength is substantially lower in case of CSA-limestone binders, up to 50% in case of 30% limestone content. This decrease is comparable to the effect of adding 10-30% of limestone to ordinary Portland cements, as was presented in [31], therefore this result can be attributed to dilution effect. In case of flexural strength, in can be noticed, that there is a tendency for a drop in strength after 2^{nd} or 3^{rd} day of curing. While there are not many sources available in this topic, similar effect of decrease in flexural strength have been observed by Martin et al. [14]. By the timing alone, the decrease may be linked to the start of stratlingite production in CSA hydration process, which occurs around 40-72 h of hydration [38]. However, this effect requires further research.

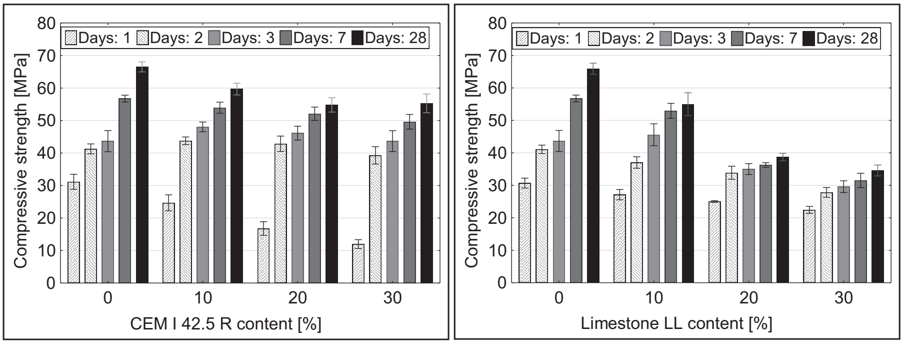

Fig. 4. Compressive strength of CSA cements with OPC cement (left) and limestone (right).

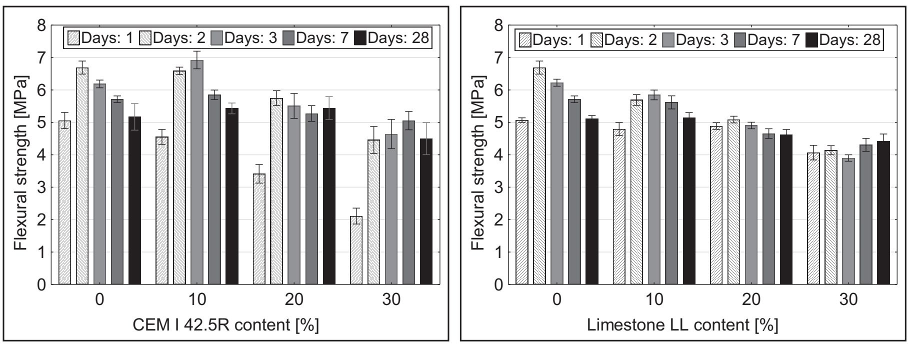

Fig. 5. Flexural strength of CSA cements with OPC cement (left) and limestone (right).

4. Conclusions

Presented results lead to following conclusions:

- Addition of 10-30% of OPC to CSA cement had significant effect on the tested mortar properties. With the increase in OPC content, yield stress and plastic viscosity increased, in the case of 20% and 30% addition to the point that made the measurement impossible. The compressive strength od CSA-OPC blends decreased with the increase in OPC content, and the effect was most visible after 1 day of curing. Those effects can be linked to the rapid reaction of CSA in presence of OPC, what can negatively impact hydration rate by restricting the access to water for cement grains.
- Shrinkage of mortars with CSA-OPC binder was significantly lower than shrinkage of OPC mortars. In case of 30% OPC content, the shrinkage was lower than the shrinkage of CSA cement mortar. This effect may be connected to the expansive ettringite produced in the CSA-OPC binder hydration.

- Limestone addition to CSA cement had affected negatively both early and 28-day strength of mortars , what can be linked to the dilution effect. Mortars with CSA-limestone were characterized by a higher shrinkage than in case of CSA cement, however it was ~3 times lower than in case of OPC. No significant influence of limestone addition to cement on yield stress and plastic viscosity of tested mortars was observed.

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Właściwości zapraw z cementów wapniowo siarczanoglinianowych z dodatkiem cementu portlandzkiego i wapienia

Słowa kluczowe: cement wapniowo siarczanoglinianowy, cementy CSA, właściwości zapraw, właściwości reologiczne, **wapień**

Streszczenie:

Potrzeba zmniejszenia śladu węglowego stała się jednym z głównych problemów przemysłu cementowego ze względu na wysoką produkcję dwutlenku węgla w procesie spalania klinkieru portlandzkiego, która wynosi od 0,6 do 0,8 tony CO_2 na 1 tonę klinkieru portlandzkiego Sytuacja ta stwarza zapotrzebowanie na mniej popularne klinkiery, takie jak cement CSA (cement wapniowo siarczanoglinianowy). Cement CSA charakteryzuje się krótkim czasem wiązania, wysoką wytrzymałością początkową i niskim skurczem, a podczas jego produkcji emitowane jest mniej CO_2 w porównaniu z klinkierem OPC. Jednakże wysoki koszt cementu CSA zachęca do mieszania CSA z innymi, tańszymi spoiwami lub nieklinkierowymi składnikami głównymi cementu.

Celem prezentowanych badań było zatem zbadanie właściwości zapraw z użyciem cementu CSA zmieszanego ze zwykłym cementem portlandzkim CEM I 42,5R (OPC) lub wapieniem w ilości 10, 20 i 30% masy cementowej. Badano następujące właściwości: ciepło hydratacji w pierwszych 72 h od połączenia cementu z wodą, wczesny skurcz zaprawy w pierwszych 24 h, właściwości reologiczne, czyli granicę plastyczności i lepkość plastyczną świeżej zaprawy, a także wytrzymałość na ściskanie i zginanie po 1,2,3,7 i 28 dniach. Zaprawy przygotowano na bazie normowej zaprawy wg EN 196-1, zawierającej 450g cementu, 1350g piasku normowego i o stosunku w/c wynoszącym 0,5. Ze względów technicznych stosunek w/c został zmieniony dla pomiarów reologicznych na 0,6.

Hydratacja cementu CSA jest szybka, a najniższy przepływ ciepła podczas pierwszych 20 h nawadniania jest nadal wyższy niż maksymalny przepływ ciepła w przypadku nawadniania OPC. Dodanie OPC zwiększyło początkową szybkość reakcji ye'elimitu, jednakże późniejsza reakcja została znacznie spowolniona, co może być związane z konglomeracją produktów hydratacji na ziarnach cementu. Dodatek wapienia do cementu CSA znacznie zmniejszył szybkość emisji ciepła, jednak wyraźnie przyspieszył wczesną hydratację. Efekt ten można powiązać z efektem wypełnienia, który zapobiega konglomeracji cząstek klinkieru, a tym samym pozwala na lepsze uwodnienie.

Skurcz zapraw ze spoiwem CSA-OPC i CSA-wapień był znacznie mniejszy niż skurcz zapraw OPC. W przypadku 30% zawartości OPC skurcz był mniejszy niż skurcz zaprawy z CSA. Efekt ten może być związany z ekspansywnym ettryngitem wytwarzanym w procesie uwodnienia spoiwa CSA-OPC.

Dodanie 10-30% OPC do cementu CSA znacząco wpłynęło na wczesną wytrzymałość zapraw, zmniejszając po 1 dniu wytrzymałość na ściskanie nawet o 60%, co może być związane z powolną reakcją cementów CSA-OPC. Po 28 dniach różnica pomiędzy cementem CSA a cementami CSA-OPC była znacznie mniejsza, I prawdopodobnie związana z różnicą wytrzymałości cementów OPC i CSA zastosowanych w badaniach. Dodatek wapienia do cementu CSA wpłynął negatywnie zarówno na wczesną, jak i 28-dniową wytrzymałość zapraw, co można wiązać z efektem rozcieńczenia.

Zaprawy z dodatkiem 10-30% OPC dodawane do cementu CSA charakteryzują się szybkim usztywnieniem, co przekłada się na wysoką granicę plastyczności zapraw z 10% dodatkiem OPC oraz brakiem możliwości pomiaru parametrów reologicznych dla zapraw z 20% i 30% OPC. Nie stwierdzono istotnego wpływu dodatku wapienia do cementu na granicę plastyczności i lepkość plastyczną badanych zapraw.

Badania wskazują na możliwość dodawania 10% wapienia lub 10% OPC do cementów CSA.

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