

Investigation of fresh and hardened properties of Calcium sulfoaluminate (CSA) cement blends

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Abstract. Calcium sulfoaluminate (CSA) is a comparatively new cementitious material that is mainly established in China where it is produced in a large scale. CSA cement is not covered by European standards. However, it provides different beneficial properties such as rapid hardening and high early strength development. Furthermore, the usage of CSA cement can save energy during production process in comparison to established cementitious materials. Therefore it is also more environmental friendly.

Insufficient knowledge of this material behaviour restricts the possibilities and makes further research necessary. The research project applied a laboratory test program to elaborate the characterization of the materials. The obtained knowledge from these tests was then applied to further tests to determine application relevant key properties of CSA based pastes and mortars. The properties of pure CSA cement had been compared with the properties of CSA blends. The additions were PC, HAC, FA and GGBS with quantities of 10, 20 and 30%. The water to cement ratio was varying between 0.4, 0.5 and 0.6. General tests like fineness, XRD and XRF were used to define the present non-standardized material. Investigation of fresh pastes included measurement of setting time and calorimetry. Hardened mortar specimens of different ages were examined for compressive strength.

The results showed that CSA itself hardens very rapidly and gives an early strength development. Possible ways of utilization of CSA based mortars and concretes were also emphasized in the paper.

Keywords: CSA; calcium sulfoaluminate cement; investigation; properties

Introduction

Construction industry is one of the largest industrial sectors in the world. It makes approximately 9 % of the world Gross Domestic Product (GDP) and employs around 7 % of labours all over the world [1]. Construction industry has not only a huge investment volume, but according to some estimates it also emits about 5 % of the anthropogenic carbon dioxide in the world. [2]

The main material used in the construction industry worldwide is concrete. Concrete production exceeds 10 billion tons in 2014 [3]. Gartner [4] specifies in 2004 that in order to produce a cubic meter of concrete, 0.2 tons of CO₂ emissions are produced.

Portland Cement (PC) is one of the most energy-intensive construction materials as it is the most important cementitious binder nowadays [5]. The production process of PC emits approximately the same amount by mass of CO₂ into the atmosphere as the produced mass of PC [6]. PC is a well researched and established cementitious material. Certain chemical additions such as retarders, plasticizers or air entrainments can be added to modify the properties of fresh and hardened concrete. However, these admixtures can be expensive and often have a big impact on the environment. [7]

An alternative to PC can be calcium sulfoaluminate (CSA) cement. The phases of CSA are different to these present in PC. This leads to different properties of the hardened paste, mortar or concrete depending on its usage. This comparatively new material has not been used yet in high amounts in Europe. [8]

While in China, CSA cements are standardized and used in a large scale for approximately 30 years. Glasser [9] specified that the annual production of CSA cements in China is more than 1 million tons in 2014. In most countries as well as in the UK this standardization is missing. It is mainly due to the fact that this material is still poorly researched outside of China. Especially long-term properties of CSA applications are partially unknown.

The aim of this project is to obtain detailed information about the properties of calcium sulfoaluminate cement and to trace back these properties to their origins. Therefore a variety of tests had been carried out. The first set of tests aimed on characterizing the present materials. Tests carried out were namely PSD (particle size distribution), Fineness (via Blaine method, BET and Laser), XRF (X-ray fluorescence) and XRD (X-ray diffraction). The crucial point in this characterisation process was to compare the unknown CSA cement with familiar materials such as PC.

Investigation of fresh cement pastes included setting time tests. Compressive and tensile strength tests had been carried out on mortar specimens.

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Blending the examined materials CSA, PC, GGBS, FA and HAC with each other aimed on taking advantage of the benefits of different materials. Furthermore different w/c ratios were applied throughout the whole testing procedure. The comparatively high w/c ratios that were used compared to the usage of PC is due to the fact that in a related study CSA was found to require more water for the best performance than common cements such as PC. [10]

1. Literature Review. Introduction into CSA cement

In the 1960's Alexander Klein patented a cementitious phase that is called ye'elimite. It means that in comparison to other cementitious materials it is a quite new development. In the cement chemistry ye'elimite is marked as C₄A₃S. In honour to its inventor, it is also called Klein's compound. It has the chemical composition Ca₄Al₆SO₄O₁₂. [8]

Calcium sulfoaluminate cement (CSA cement) contains as main phase ye'elimite (approx. 30–70 %). The actual intention of Klein in using ye'elimite was to use it as a shrinkage compensation additive to normal binders. Therefore it became patented. [8]

CSA cements can be produced in a cement plant in the same way as PC is produced. While the temperature that is required within the clinkering process is only around 1300°C, which is less than the temperature necessary for PC production. The raw materials used for CSA clinker production are bauxite, limestone and calcium sulfate (anhydrite, CaSO₄). The latter is the unhydrated form of gypsum. [8]

Only if gypsum is added to the clinker, hydration process becomes highly accelerated. As long as calcium sulfate is present in the solution, aluminium hydroxide and ettringite are forming instead of aluminium hydroxide and monosulfate. [8]

During the latest studies at the University of Dundee other CSA cements were also examined. Some of these CSA cements did not hydrate at all. The phase composition of CSA cement that did not hydrate, is given in Table 1.

Table 1: Phase composition of a CSA cement examined on University of Dundee

Mineral	Phases	Constituents [%]
Ye'elimite	C ₄ A ₃ S	61,79
Belite	C ₂ S	13,04
Anhydrite	CaSO ₄	-

Zhang et al. [11] also stated that the property of rapid hardening comes from the ye'elimite phase in combination with calcium sulfate. Furthermore, he says that with increasing the amount of calcium sulfate the formation of ettringite replaces the formation of monosulfate.

Due to the special properties of CSA cement, a broad range of possible applications can be listed. First of all, the characteristic shrinkage-resistance is important in order to know for what it actually became invented. Therefore it is often used in the pre-stressed concrete. The China Building Materials Academy (CBMA) used CSA cements to produce self-stress concrete pipes [12]. According to specifications of a Chinese CSA cement supplier, ettringite is able to expand to a volume that is two times greater than the volume of its educts. [13]

Juenger [8] states a variety of applications for the CSA cement such as its usage in concrete pipes, seepage prevention projects, precast concrete, pre-stressed concrete, waterproof layers, glass fibre-reinforced cement products, low temperature construction and shotcrete.

Péra et al. [12] specified the importance of lime in the production of the CSA cement. With no lime ettringite is formed by another reaction and the properties of ettringite are dramatically changing. Now it is no more expansive but achieves a very high early strength that is declared here as 40MPa already after a period of 6 hours.

According to current studies at the University of Dundee CTU (Concrete Technology Unit), the production of foam concrete is reliant on cements that are setting very rapidly. CSA cement is one of the used materials here. [14]

2. Laboratory results and discussion

Portland cement used in this test program was obtained from Hanson who is a member of the Heidelberg Cement Group which is a German cement manufacture. The used material is designated by the supplier as PC 52.5N and is a CEM I Portland cement according to the standard BS EN 197-1: 2000 with a strength of 52.5 MPa and a normal setting velocity [15]. HAC was also obtained from Hanson.

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Hanson HAC was manufactured according to BS915: Part 2: 1972, the standard for High aluminate cements [16]. The used GGBS was also obtained from the supplier Hanson. According to the supplier it is the only type of GGBS produced in the UK [17]. The corresponding standards are BS EN 197-1:2011 and BS EN 15167-1:2006 [18, 19].

Fly ash used in this test program was obtained from Cemex and designated as Cemex 450-S where 'S' stands for special fineness according to BS EN 450-1 [20].

Several CSA cements from various Chinese manufacturers were used in different studies at the University of Dundee. Only one of them was chosen for comparison and characterization within this study. For the CSA cements no European standard can be applied.

2.1. Chemistry

The analysis of the oxide composition was carried out through X-ray fluorescence (XRF). Table 2 shows the oxide compositions of the five compared materials.

For CSA cement the presence of Al_2O_3 and SO_3 is crucial because the formation of ettringite is wanted. SO_3 and CaO can form anhydrite which is able to form with ye'elimite ettringite. Calcium oxide is obviously the main oxide in the examined CSA with an amount of over 45 % by mass. It is important as it is used in ye'elimite and anhydrite formation and therefore required in a higher scale. Al_2O_3 is presented with over 21 % and SO_3 with over 16 %. The oxide composition seems to be suitable for the hydration processes. However, the XRD results will show if the required phases were actually formed by the present oxides.

Table 2. Oxide composition of the examined materials

Oxides	Constituents [%]				
	CSA	PC	HAC	GGBS	FA
CaO	45,11	65,10	40,16	39,91	3,37
Al_2O_3	21,31	4,79	39,37	12,09	20,19
SO_3	16,20	2,64	0,13	1,17	2,03
SiO_2	6,93	19,30	4,40	34,60	43,15
Fe_2O_3	3,02	3,23	13,87	0,55	9,79
MgO	0,79	1,03	0,62	7,02	1,24
TiO_2	0,70	0,45	1,84	0,68	1,06
K_2O	0,30	0,66	0,08	0,58	2,84
Na_2O	0,12	0,39	0,36	0,35	1,99
P_2O_5	0,07	0,23	0,06	0,03	0,41
Cl	0,00	0,10	0,00	0,00	0,00
MnO	0,05	0,05	0,07	0,53	0,08
Total	94,59	97,96	100,95	97,52	86,15

2.2. Mineralogy

By means of X-ray diffraction the mineralogical phases of the tested materials were determined. It is impossible to detect non-crystalline phases. GGBS and FA consist only of 20–30 % by mass of crystalline phases, the rest is non-crystalline. Therefore, GGBS and FA were not investigated quantitatively. The quantitative analysis of the other materials is shown in Table 3 and the qualitative analysis is shown in Table 4.

The amount of calcium oxide was quite high in the investigated CSA cement, so it formed with SiO_2 Larnite (belite), with SO_3 anhydrite and with Al_2O_3 aluminate. However, the important results are the values for ye'elimite and anhydrite which are the highest constituents. It means that the formation of ettringite during hydration process seems to be likely. Belite was also formed in a quite high magnitude. In case belite hydration presumably forms portlandite, the aluminate phase could react with water to form the aluminium hydroxide.

The main phase discovered in Portland cement was alite with 54 % and the second highest value was belite. Both are forming portlandite and CSH while hydrating. CSH is responsible for the strength development during the hydration process and other typical properties of PC.

Table 3. Quantitative XRD analysis of CSA, PC and HAC

Mineral	Phases	Constituents [%]		
		CSA	PC	HAC
Ye'elimite	C ₄ A ₃ S	34,6	-	-
Larnite	C ₂ S	14,6	16,6	8,9
Anhydrite	CaSO ₄	24,0	-	-
Brownmillerite	C ₄ AF	-	9,1	-
Calcium aluminate	CA	-	-	59,5
Aluminate	C ₃ A	4,5	10,8	-
Gehlenite	C ₂ AS	-	-	7,3
Alite	C ₃ S	-	54,1	-
Mayenite	C ₁₂ A ₇	-	-	6,3
Total		77,7	90,6	82,0

Table 4. Qualitative XRD analysis of CSA, HAC, GGBS and FA

CSA	HAC	GGBS	FA
Ye'elimite	Calcium	Feroxyhite	Quartz
Larnite	Gibbsite	Cooperite	Mullite
Anhydrite	Trona	Cassidyite	Magnetite
Lime	Thermonatrite	Imhofite	Maghemite
Gypsum	Calcium aluminium	Cesstibantite	Anhydrite
Calcite	Currundum	Churchidyte	Gypsum
Currundum		Kilchoanite	Currundum
		Currundum	

2.3. Fineness

Testing fineness was determined through three different tests BET, Blaine Test und Laser Test. It cannot be said that one of the three values is the right result because of the fundamentally different testing procedures. However, it is not of interest how big the surface area of a material is, but which material has a higher area than another one. Higher fineness is correlated to a higher surface area which leads to higher water demand in the hydration process.

Table 5 shows the results of the three tests that were carried out. Portland cement has the lowest value in all tests. The fact that the surface area of PC is smaller and there are no rapid hydrating phases which present in the PC tested will presumably lead to a slower setting time.

All tests confirmed that the CSA cement is a comparatively fine material. Thus the fact that the CSA cements are very rapid hardening could be due to its high surface area.

The corresponding standard for testing fineness is BS EN 196-6: 2010 [21].

Table 5. Fineness investigated through three different methods

Method	Fineness [m ² /kg]				
	CSA	PC	HAC	GGBS	FA
Blaine	705	394	713	430	499
BET	1790	971	1120	1171	2260
Laser	450	286	347	419	593

2.4. Setting Time

One of the most important properties is setting time. CSA cement is especially important because of its rapid hardening behaviour. Several special applications depend on rapid hardening.

Previous tests showed that the CSA has a high potential to provide rapid hardening. This presumption was supported by setting time tests. Compared with the familiar PC, CSA cement needed for the whole hardening process only a fraction of time that PC requires to start setting (Table 6).

Table 6 shows that irrespective of the amount of GGBS and FA added setting time is even accelerated in comparison to the plain CSA cement. Only the addition of PC leads to a decrease of setting velocity compared to plain CSA.

In each case, irrespective of which blend is focused on, a higher w/c ratio leads to a decelerated hardening process.

The corresponding standard for testing setting time is BS EN 196-3: 2005 [22].

Table 6. Setting times of pure cement pastes and blended CSA cement pastes

Sample	Initial setting time [hrs:min:sec]	Final setting time [hrs:min:sec]
CSA 100% 0.4	00:04:09	00:06:14
CSA 100% 0.5	00:05:03	00:06:06
CSA 100% 0.6	00:11:18	00:14:27
PC 100% 0.4	03:47:16	05:04:07
PC 100% 0.5	04:22:30	05:45:57
PC 100% 0.6	04:58:53	07:20:35
HAC 100% 0.4	06:52:13	07:44:09
HAC 100% 0.5	08:17:10	09:34:41
HAC 100% 0.6	08:38:36	11:03:48
CSA + 10% PC 0.4	00:05:19	00:06:22
CSA + 10% PC 0.5	00:06:04	00:07:06
CSA + 10% PC 0.6	00:08:20	00:08:20
CSA + 20% PC 0.4	00:05:54	00:06:56
CSA + 20% PC 0.5	00:07:05	00:08:07
CSA + 20% PC 0.6	00:08:08	00:10:14
CSA + 30% PC 0.4	00:07:57	00:07:57
CSA + 30% PC 0.5	00:08:39	00:09:42
CSA + 30% PC 0.6	00:09:35	00:11:40
CSA + 10% PFA 0.4	00:02:28	00:03:31
CSA + 10% PFA 0.5	00:03:26	00:05:31
CSA + 10% PFA 0.6	00:04:49	00:06:54
CSA + 20% PFA 0.4	00:03:22	00:04:24
CSA + 20% PFA 0.5	00:04:18	00:06:23
CSA + 20% PFA 0.6	00:05:39	00:07:44
CSA + 30% PFA 0.4	00:05:43	00:07:49
CSA + 30% PFA 0.5	00:04:57	00:04:57
CSA + 30% PFA 0.6	00:06:48	00:06:48
CSA + 10% GGBS 0.4	00:04:50	00:04:50
CSA + 10% GGBS 0.5	00:05:26	00:06:28
CSA + 10% GGBS 0.6	00:07:21	00:07:21
CSA + 20% GGBS 0.4	00:04:50	00:05:53
CSA + 20% GGBS 0.5	00:06:32	00:06:32
CSA + 20% GGBS 0.6	00:07:31	00:08:33
CSA + 30% GGBS 0.4	00:04:45	00:05:48
CSA + 30% GGBS 0.5	00:06:24	00:07:26
CSA + 30% GGBS 0.6	00:08:27	00:09:30

2.5. Compressive Strength

Strength test results were obtained from a related study at the University of Dundee in 2013 [10].

Within this study the compressive strength of pure CSA cement mortar cubes and blended CSA cement mortar cubes were examined. Mortar specimens were produced with w/c ratios of 0.4, 0.5 and 0.6. The specimens were stored for 1.5 hours, 6 hours, 1 day or 28 days. The results shown in Table 7

are given in the unit MegaPascals [MPa]. In the shown table the three highest and lowest results for a certain storing time are highlighted.

The best performance was observed with the Portland cement mixes. Especially in earlier ages PC cement blended mortars provided good results. It is correlated with the results obtained from calorimetry, which will not be presented in this report. Higher reactivity was observed there when the CSA was mixed with PC, which means that more hydration products were formed. The weakest test results were found in the blends with GGBS.

Focusing on the water content, it can be said that the optimal w/c ratio found in this test program was 0.5. The enlarged water demand is very interesting as it improves the workability of the CSA cement based on concrete or mortar. This is another argument supporting the CSA cement usage in shotcrete or pumped concrete. Furthermore, higher plasticity makes compaction easier.

The determination of the compressive strength was carried out according to the American standard ASTM C 109/C 109M – 02. This was to gain results that are comparable with the ASTM C 1600/C 1600M–08 standard for rapid hardening hydraulic cement.

Table 7. Compressive strength of pure CSA mortar cubes and blended CSA mortar cubes

Sample	Compressive strength [Mpa]			
	1.5 [h]	6 [h]	1 [d]	28 [d]
CSA 100% 0.4	9,0	17,9	17,9	31,4
CSA 100% 0.5	13,8	25,7	39,1	52,6
CSA 100% 0.6	10,2	20,3	30,7	45,6
CSA + 10% PC 0.4	7,9	13,6	25,4	35,2
CSA + 10% PC 0.5	14,8	29,1	40,7	51,1
CSA + 10% PC 0.6	13,8	25,1	31,2	42,4
CSA + 20% PC 0.4	18,1	29,5	37,4	37,1
CSA + 20% PC 0.5	19,3	31,3	40,2	48,5
CSA + 20% PC 0.6	14,3	26,5	33,2	45,9
CSA + 30% PC 0.4	19,5	28,7	38,2	50,2
CSA + 30% PC 0.5	17,1	30,4	36,6	48,9
CSA + 30% PC 0.6	13,1	25,0	31,3	43,9
CSA + 10% PFA 0.4	5,5	11,6	19,8	29,1
CSA + 10% PFA 0.5	12,8	25,3	36,6	55,1
CSA + 10% PFA 0.6	10,3	24,3	30,7	39,5
CSA + 20% PFA 0.4	9,5	16,7	31,3	41,0
CSA + 20% PFA 0.5	10,4	22,2	30,6	43,0
CSA + 20% PFA 0.6	8,7	20,6	25,1	34,2
CSA + 30% PFA 0.4	9,3	14,3	32,8	49,0
CSA + 30% PFA 0.5	9,9	20,5	25,9	36,5
CSA + 30% PFA 0.6	6,8	17,3	20,8	28,5
CSA + 10% GGBS 0.4	5,5	5,5	23,1	33,3
CSA + 10% GGBS 0.5	12,3	24,5	39,1	60,0
CSA + 10% GGBS 0.6	9,8	24,2	29,9	40,3
CSA + 20% GGBS 0.4	3,8	3,8	22,5	36,4
CSA + 20% GGBS 0.5	10,3	10,3	31,9	48,1
CSA + 20% GGBS 0.6	8,4	19,6	22,7	34,8
CSA + 30% GGBS 0.4	4,1	4,1	17,5	19,3
CSA + 30% GGBS 0.5	9,1	9,1	25,6	41,0
CSA + 30% GGBS 0.6	6,8	16,0	19,7	30,5

Conclusions

Within this study, properties of CSA cement and CSA cement blends in fresh and hardened condition were investigated. The obtained knowledge about particle sizes, specific surface areas, oxide compositions, mineralogical phases and morphology was used to link it to the construction industry relevant properties such as setting time and strength. Different w/c ratios and different contents of additions were used to produce the samples. Therefore, a broad range of results was achieved, where all the results are comparable to each other, as the testing procedure and parameters were kept. It enabled back tracking of the developed properties to its origins.

Outcomes of the study

Through the investigation by means of XRF it was observed that the examined CSA cement provided a similar oxide composition as PC but with high differences in the quantitative distribution. XRD tests of the CSA showed that the phase composition was strongly different from what was observed in other cements such as PC and HAC. The exceptional phase ye'elimite in combination with anhydrite achieved very rapid hardening properties.

Furthermore, the pure CSA provided very high early strength. Even greater improvements of the compressive strength could only be achieved by blending the CSA with PC. Calorimetry also showed that reactivity of this cement blend was enlarged. This validates that PC supplies certain components that are absent in CSA which leads to the restriction of hydration reactivity in case of applying pure CSA cement.

Besides, it was discovered that the CSA cement needs higher w/c ratios for the best possible strength performance in comparison with common cements. A w/c ratio of 0.5 was found to be appropriate.

When it comes to heat development, it was discovered that GGBS and FA can enhance the excessive heat development of the CSA cement during the hydration process. Thus the risk of thermal shrinkage induced concrete cracking could be reduced by using GGBS and especially FA as an addition to CSA cements.

Recommendations for further research

The potential of the tested CSA cement is shown in this paper and general behaviour of pure CSA cement and cement blends based on CSA was emphasized. In further research it is important to focus on special ways of application.

The behaviour of the CSA based concretes and mortars in the realistic environment should be investigated. Test programs could be focused on carbonation depth tests related to time, testing of possible correlation with different types of aggregates, water and air penetrability tests or tests to determine the impact of the CSA cement on reinforcement steel.

Long-term effects on structural important properties such as compressive strength or creeping need to be investigated. Long-term tests are obligatory to actuate the formation of standards. Different age accelerating methods can be applied on the specimen to carry out this kind of tests in the laboratory.

Applications in the extraordinary environments such as coastal areas, sewage plants or disposal facilities require knowledge about how far the CSA cement provides resistance against chlorine, sulfate and acid attack.

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Investigation of fresh and hardened properties of Calcium sulfoaluminate (CSA) cement blends

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Key words

CSA; calcium sulfoaluminate cement; investigation; properties

Abstract

Calcium sulfoaluminate (CSA) is a comparatively new cementitious material that is mainly established in China where it is produced in a large scale. CSA cement is not covered by European standards. However, it provides different beneficial properties such as rapid hardening and high early strength development. Furthermore, the usage of CSA cement can save energy during production process in comparison to established cementitious materials. Therefore it is also more environmental friendly.

Insufficient knowledge of this material behaviour restricts the possibilities and makes further research necessary. The research project applied a laboratory test program to elaborate the characterization of the materials. The obtained knowledge from these tests was then applied to further tests to determine application relevant key properties of CSA based pastes and mortars. The properties of pure CSA cement had been compared with the properties of CSA blends. The additions were PC, HAC, FA and GGBS with quantities of 10, 20 and 30%. The water to cement ratio was varying between 0.4, 0.5 and 0.6. General tests like fineness, XRD and XRF were used to define the present non-standardized material. Investigation of fresh pastes included measurement of setting time and calorimetry. Hardened mortar specimens of different ages were examined for compressive strength.

The results showed that CSA itself hardens very rapidly and gives an early strength development. Possible ways of utilization of CSA based mortars and concretes were also emphasized in the paper.

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