

INFLUENCE OF PORTLAND CEMENT ADDITION TO SULPHOALUMINATE - BELITE CEMENT ON HYDRATION AND MECHANICAL PROPERTIES OF HARDENED MORTARS

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Submitted January 24, 2002; accepted July 5, 2002

Keywords: Calcium sulphoaluminate-belite cement, Microstructure, Mortar

Ordinary Portland cement (PC), calcium sulphoaluminate - belite (SAB) cement and a 85 wt. % SAB cement/15 wt.% PC blend were used for mortar manufacture (cement to sand weight ratio 1 : 3, w/c ratio 0.5) of 40×40×160 mm specimens kept in wet air cure (20°C, 100 % R.H. for 90 days), and used for the tests. The results reveal the influence of cement type on mortar properties. The mortars, made from tested SAB cement have the worst mechanical properties. The replacement of 15 wt. % of SAB cement by PC contributes to the increase in dynamic modulus of elasticity (DME), flexural strength of the mortar by 15 % and compressive strength almost by 40 % compared to the SAB cement mortar. Such mortar behaves mechanically similar to PC mortar. Differences in mechanical properties of mortars are caused by differences in chemical composition of cements (in SAB/PC mortar content of CaO is increased by 6.4 %, contents of Al₂O₃ and Fe₂O₃ are decreased by 8.2 % and 7.0 %, respectively compared to SAB cement mortar; SO₃ content is 2.6× higher than that in the PC mortar), and mineralogical composition and hydration kinetics of cements. Differences in mineralogical composition, mainly ettringite formation, and consequently hydration kinetics are the substantial reason of higher demands on batch water, rapid initial and final set of SAB cement and lower DME and strength of SAB cement mortar relative to PC and PC mortar, respectively. The 85 wt. % SAB cement/15 wt. % PC blend gains on hydration activity, DME and strength growth by the C₃S activator from PC supporting less-active SAB cement hydration. Utility properties of SAB/PC mortar are more similar to PC mortar than SAB cement mortar.

INTRODUCTION

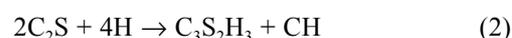
The key component of calcium sulphoaluminate-belite (SAB) cements is calcium sulphoaluminate (C₄A₃S̄) due to its ability of generating ettringite (C₆A₃S̄₃H₃₂) when combined with lime, calcium sulphate dihydrate and water [1-4]. Depending on the composition and proportion of raw materials used for their manufacture, SAB cements also contain other components e.g. β - C₂S and C₄AF [5-9]. The compounds C₂S and C₄AF behave similarly in SAB cements as they do in Portland cements [4]. C₂S generates C-S-H gel and therefore contributes to the strength of cement at medium and longer ages, and it also contributes to the durability of hydrated cements. C₄AF generates hydration products which give a little contribution to the mechanical strength [10].

In order to understand the chemistry of SAB cement hydration, processes of all its individual clinker minerals are necessary to consider. C₄A₃S̄ hydrates according to the following equation:



In this rapid hardening cements the role played by C₄A₃S̄ is similar to that of C₃S in ordinary Portland cements. For this reason SAB cements are also termed as non-alkalic cements [11] because of lower pH value at hydration. Due to the fact that C₄A₃S̄ needs synthesis temperatures 200-300°C lower than that required for the formation of C₃S, cements incorporating it are also called low-energy cements [12]. C₄A₃S̄ often known as Klein's compound is readily synthesized along with belite (β - C₂S) and ferrite (C₄AF) at about 1300°C [13-15]. The theoretical heat requirement for SAB cement is 1337 kJ/kg compared with 1756 kJ/kg for ordinary Portland cement. Enthalpy of formation of C₃S is 1848 kJ/kg clinker, of β - C₂S 1336 kJ/kg clinker, and of C₄A₃S̄ only around 800 kJ/kg clinker. The amount of carbon dioxide release during firing from C₃S is 0.578 kg/kg clinker, β - C₂S 0.511 kg/kg clinker, and C₄A₃S̄ is only 0.216 kg/kg clinker [16].

Hydration reaction of C₂S and C₄AF are represented by following chemical equations:



The products formed are a calcium silicate hydrate known as C-S-H gel and calcium hydroxide. The formula given for C-S-H is only a rough approximation because also more than one variety of C-S-H is formed during the hydration reaction. C_4AF is a solid solution of C_2A - C_2F system whose hydration process is very similar to that of C_3A . The hydration rate, however, is slower [17] and in the products, iron replaces some of aluminium. The C_4AF (ferrite phase) reacts with water by this way:



When sulphates are plentiful ettringite is formed and when sulphates are insufficient monosulphates are formed. Conversion from one product to other is possible [18]. Reaction of C_4AF with $C\bar{S}$ proceeds as seen [19]:



In general, SAB cements harden at a notably slower rate than Portland cement. They do indeed attain the required strength, usually lower than that of Portland cement [20]. In our previous papers of this series [21-24] we studied laboratory samples (3-50 kg) of SAB cements prepared in Slovakia and their blends with ordinary Portland cement with the aim of upgrading SAB cement systems by blending with Portland cement.

In the present paper we are reporting our data on the hydration and properties of foreign SAB cement and mixed SAB/PC. The tested SAB cement was manufactured under industrial conditions in Romania (2000 t) in Cimius Campulung cement plant during the solution of COPERNICUS project (Contract ERB CIPA CT 94 01105) with the participation of the UK, Romania and Slovakia.

EXPERIMENTAL

Ordinary Portland cement CEM I 42.5 (PC), calcium sulphoaluminate - belite (SAB) cement and the blend consisting 85 wt.% of SAB cement and 15 wt.% of PC were used in the tests. Specific weight, specific surface area, chemical composition, initial and final set and mineralogical composition of the cements are listed in tables 1, 2, 3 and 4.

Mixing and curing

Cement mortars (cement to sand ratio 1:3 by weight and water to cement ratio of 0.5) using a standard sand as specified by STN 72 1208 [25] were prepared as prismatic specimens $40 \times 40 \times 160$ mm in steel moulds on a vibration table (50 Hz, 0.35 mm) with vibration time of 30 seconds. The specimens were stored 24 hours in a climate chamber at 20°C, and 100 % R.H., and subsequently at 20°C/100 % R.H. - wet cure for the next 89 days.

Table 1. Specific weight and specific surface area (Blaine) of cements.

property	type of cement		
	PC	SAB/PC	SAB
specific weight (kg/m ³)	3 313	3 310	3 008
specific surface (m ² /kg)	398	396	395

Table 2. Chemical composition of tested cements.

component (wt.%)	type of cement		
	PC	SAB/PC	SAB
humidity	0.15	1.08	1.17
ignition loss	1.38	1.06	1.01
insoluble residue	0.84	0.89	0.90
SiO ₂	20.82	18.42	18.00
CaO	62.30	54.85	53.53
Al ₂ O ₃	6.00	7.82	8.14
Fe ₂ O ₃	4.39	4.86	4.94
MgO	1.84	1.42	1.35
SO ₃	2.18	9.48	10.77
free CaO	0.10	-	0.18

Table 3. Initial and final set of the cements.

type of cement	normal consistency (%)	initial (min)	final (min)
PC	29	190	305
SAB/PC	31	30	37
SAB	34	20	28

Table 4. Mineralogical composition of cements employed.

constituent (wt.%)	type of cement		
	PC	SAB/PC	SAB
C ₃ S	44.5	6.7	-
C ₂ S	25.8	47.6	51.5
C ₃ A	8.5	1.3	-
C ₄ AF	13.4	14.7	15.0
C ₄ A ₃ \bar{S}	-	8.5	10.0
C \bar{S}	4.7	14.4	16.1

Table 5. Dynamic modulus of elasticity, flexural and compressive strength of the mortars.

type of mortar	curing (days)	DME (GPa)	strength (MPa)	
			flexural	compressive
100 % PC	28	41.7	8.7	42.8
	90	43.0	10.3	48.3
85 wt.% SAB +15 wt.% PC	28	31.9	6.9	24.6
	90	36.2	8.4	41.3
100 % SAB	28	26.4	5.6	17.4
	90	31.4	7.3	30.1

Test methods

Dynamic modulus of elasticity, flexural and compressive strength of the mortars were tested. Ultrasonic pulse velocities were measured on ultrasonic apparatus UNIPAN type 543. The dynamic modulus of elasticity (DME) values were calculated by the formula:

$$E_{bu} = \rho_{vb} \cdot \gamma_L^2 \cdot 10^6 \quad (5)$$

where E_{bu} - DME (MPa), ρ_{vb} - volume density (kg/m^3), γ_L - impulse speed of longitudinal ultrasonic waves (m/s^{-1}). Strength characteristics were estimated according to EN 196 - 1 Standard [26].

To study the cement hydration X-ray diffraction patterns (Philips, $\text{CuK}\alpha$ - radiation, Ni - filter) and thermal curves were made. Simultaneous thermogravimetry (TG) and differential thermal analysis (DTA) were conducted from 20°C to 1000 °C using a T.A.I. SDT 2960 Instrument (sample mass 10-20 mg, heating rate 10°C/min, in flowing air). The chemical composition of

the mortars was estimated by standard analytical methods. The oxide content in the soluble portion related to the cement binder in wt.% was calculated.

The pH values of mortar extracts were determined by a pH meter OP 113 (Radelkis, Hungary). Potentiodynamic curves of steel bars were obtained using a Potentiostat OH 405 (Radelkis) at a polarisation rate of 30 mV/min. Steel bars with diameter of 6 mm were immersed in mortar extracts to determine the corrosion state.

RESULTS AND DISCUSSION

Dynamic modulus of elasticity (DME), flexural and compressive strength values of mortars are considerably influenced by the type of cement (table 5). With increasing portion of SAB cement, DME, flexural and compressive strength of mortars decrease. The rate in DME and strength growth in time is slower in SAB cement-compared to Portland cement mortar. However a signi-

Table 6. Chemical analysis of mortar specimens.

type of cement mortar	a. ignition loss b. soluble portion c. insoluble portion	a.composition of soluble portion b.oxide content related to cement binder					
		SiO _{2,5}	CaO	Fe ₂ O ₃	Al ₂ O ₃	MgO	SO ₃
MPC	a. 6.90						
	b. 21.28	a. 4.33	13.28	0.96	1.37	0.53	0.81
	c. 71.82	b. 20.35	62.41	4.51	6.44	2.49	3.80
MB	a. 5.90						
	b. 21.08	a. 4.09	11.60	1.03	1.87	0.39	2.10
	c. 73.02	b. 19.40	55.03	4.89	8.87	1.85	9.96
MSAB	a. 3.72						
	b. 21.11	a. 4.06	11.13	1.11	2.04	0.36	2.41
	c. 75.17	b. 19.23	51.72	5.26	9.66	1.71	11.42

Table 7. Results of thermal analysis of the mortars.

type of cement mortar	curing time (days)	content of bound water in hydration products (%)	CaO bound in		total ignition loss (%)
			Ca(OH) ₂ (%)	CaCO ₃ (%)	
100 % PC	28	2.98	0.87	2.12	6.35
	90	2.80	1.90	2.10	7.40
85 wt.% SAB + 15 wt.% PC	28	3.99	0.67	0.83	5.83
	90	2.60	1.70	1.70	6.40
100 % SAB	28	4.12	0.44	0.88	5.57
	90	2.20	1.00	0.80	4.20

Table 8. Corrosion characteristics of steel in mortar extracts after 90 - day wet cure.

type of cement mortar	stationary potential (mV)	current density of passivation (A/m ²)	potential of breakdown (mV)	state of steel	pH of extract
MPC	- 330	0.033	585	passive	12.48
MB	- 330	0.033	585	passive	12.18
MSAB	- 330	0.037	580	passive	12.04

ficant improvements of DME and strength characteristics in the 85 wt.% SAB/15 wt.% PC blend are observed compared to the pure SAB cement mortar. After 90 - day wet cure this increase is 15.3 % for DME, 15.1 % for flexural strength, and 37.2 % for compressive strength values. This supports our previous concept to make possible the improvement in SAB cement properties by blending with more active Portland cement.

Differences in chemical composition of 90 - day mortars kept in 20°C/100 % R.H. - wet air are evident from table 6. The highest CaO content available for C_3S and C_2S hydration reactions, and the lowest Al_2O_3 and Fe_2O_3 contents disposable for aluminoferrite hydration reactions in PC mortar compared to those made from SAB cement and SAB/PC blend clearly confirm the highest DME and strength values of the PC mortar, as seen in table 5. The replacement of 15 wt.% of SAB cement by PC increases the CaO content by 6.4 % and decreases Al_2O_3 and Fe_2O_3 contents by 8.2 % and 7.0 %, respectively compared to values found in the SAB cement mortar. This affects markedly DME and strength growth in SAB/PC mortar compared to SAB cement mortar. The SO_3 content is 2.6 and 3.0 times higher in SAB/PC and SAB cement mortar, respectively compared to PC mortar and this is the main reason of ettringite formation during mortar hydration, as seen in XRD results.

Mineralogical composition of mortars is illustrated in figure 1 (28 - day wet cure) and figure 2 (90 - day wet cure), respectively. High portlandite [$Ca(OH)_2$ or (CH)] formation characterizes diffractogram of the PC mortar (MPC) in figure 1. Traces of ettringite ($C_6A\bar{S}_3H_32$) are found (E). Doublet at 2.79-2.74 Å showing unreacted clinker minerals C_3S and $\beta - C_2S$ is of minor significance. This shows a high degree of clinker minerals conversion to gel - like hydrate phase of C-S-H type. The presence of unreacted ferrite phase (C_4AF) proves diffraction line at 7.35 Å (FP), and that of calcite, a by-product of cement hydration, mainly of $Ca(OH)_2$ diffraction pattern at 3.03 Å (Cc). Quartz (Q) comes from the sand. Main hydration product of SAB cement mortar is ettringite (MSAB, figure 1). The portlandite occurrence is minor. The diffraction line intensity of 2.79-2.74 Å doublet is higher compared to that in PC mortar. This is due to the absence of C_3S and $\beta - C_2S$ hydrates in a slower rate. Gypsum ($C\bar{S}$) originates from SAB cement, calcite is a carbonation product and quartz comes from sand. The replacement of 15 wt.% of SAB cement by PC in SAB/PC mortar (MB, figure 1) increases $Ca(OH)_2$ portion, decreases ettringite formation and accelerates high - strength gel - like C-S-H phase development in the SAB/PC mortar opposite to the SAB cement mortar. This is caused by 6.7 % content

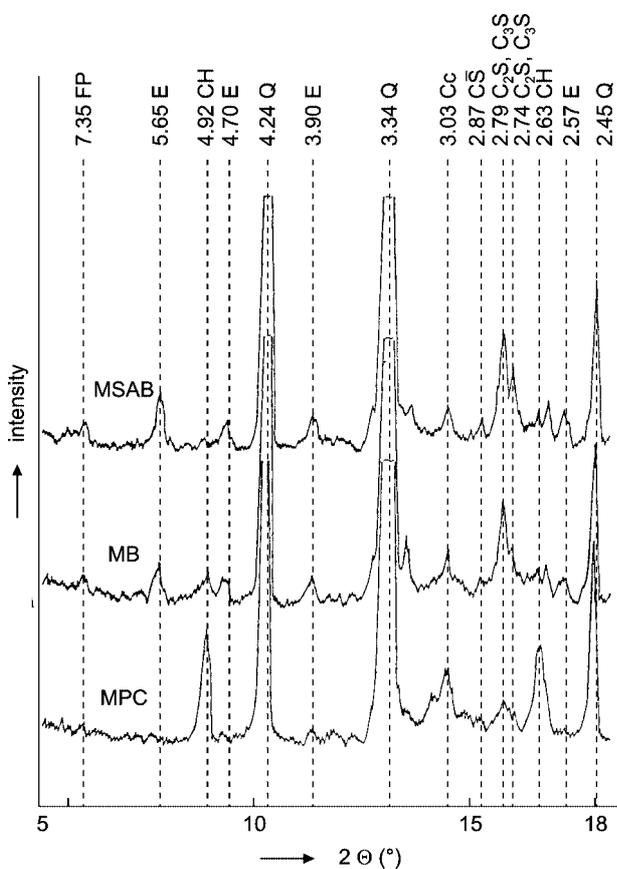


Figure 1. XRD patterns of 28 - day mortars in wet air cure.

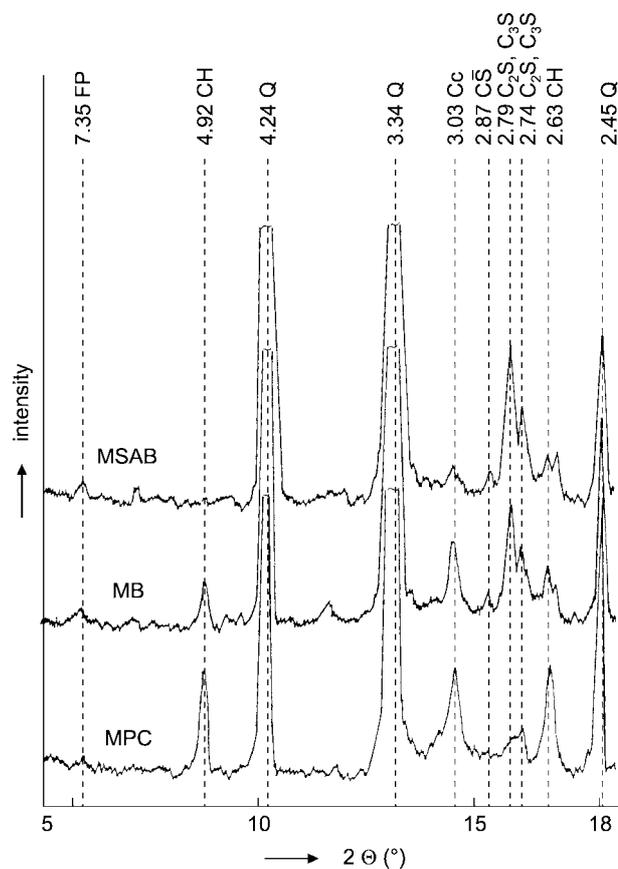
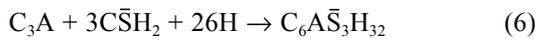


Figure 2. XRD patterns of 90 - day mortars in wet air cure.

of C_3S (table 4) in the mortar made from 85 wt.% SAB/15 wt.% PC blend. The reason of different DME and strength values among tested mortars lies in differences between chemical and mineralogical composition of the cements. The replacement of SAB cement by more active PC is advantageous for cement hydration and consequently on strength growth (table 5). Mineralogical composition of 90 - day mortars is very similar to those of 28 - day mortars. The only significant difference is ettringite decomposition in SAB and SAB/PC mortars (figure 2). Different mechanism of hydration process between PC and SAB cement systems is illustrated on ettringite formation. One molecule of ettringite ($C_6A\bar{S}_3H_{32}$) is formed by the reaction of C_3A with three molecules of gypsum ($C\bar{S}H_2$) as seen in equation (6):



On the contrary, one molecule of calcium sulphoaluminate ($C_4A_3\bar{S}$) needs for three molecules of ettringite formation, 8 molecules of gypsum, 6 molecules of portlandite, and almost three times more water than C_3A , as shows equation (7):

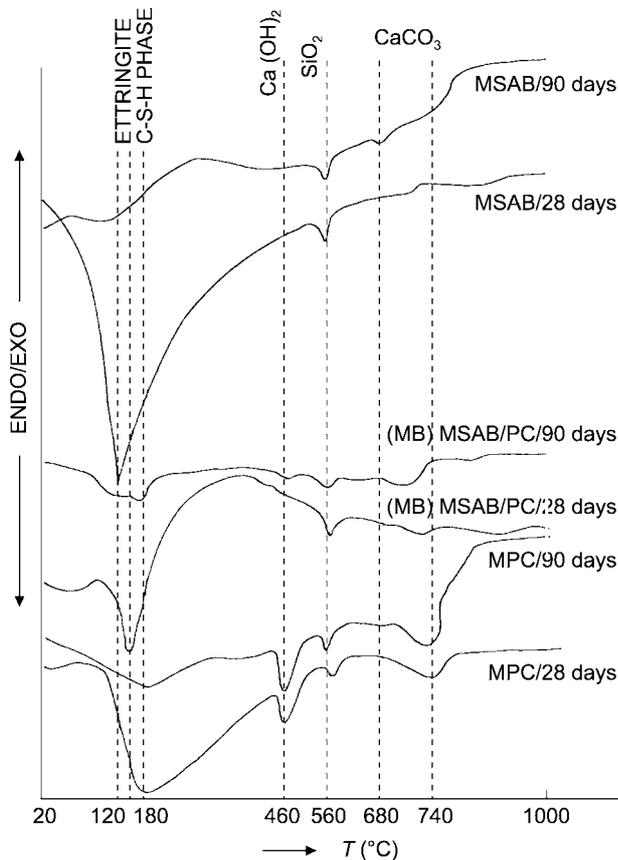
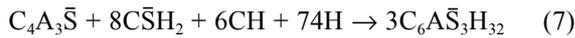


Figure 3. DTA thermograms of mortars of 28 - day and 90 - day age.

Differences in ettringite formation are the substantial reason of:

- a) higher demands on batch water of SAB cements towards PC (table 3 - normal consistencies),
- b) rapid initial and final set of SAB cements towards PC (table 3 - set characteristics),
- c) lower DME and strength values of SAB cement mortars towards PC mortars (table 5 - DME and strength characteristics and figures 1,2 - differences in mineralogical composition).

It is clear that blending of low-energy and low-active SAB cement with high-energy and high-active PC cement is unavoidable process of preparing blended cement system of higher quality when an improvement of original SAB cement quality is of an interest.

The increase in hydration activity of SAB/PC mortar relative to that of SAB cement, and its comparison with PC mortar is reported by thermal analysis results (table 7, figure 3). Percentage portion of individual hydration phases, $CaCO_3$ content and total ignition loss of mortars are given in table 7. Significant differences are evident in bound water contents. The PC mortar indicates an initial growth (28 - day wet cure) in gel - like hydration products of C-S-H type. Contrary, high amounts of bound water in SAB/PC and SAB cement mortar are contributed to ettringite. Ettringite undergoes decomposition in time, and therefore bound water contents of 90 - day SAB/PC and SAB cement mortar are pronouncedly lower than those of 28 - day cement mortars. Ettringite decomposes in water with separation of alumina gel, gypsum with non - binding properties and $Ca(OH)_2$ with binding potential, the final concentration are 35 mg CaO/l , 215 mg Al_2O_3/l and 43 mg SO_3/l [27]. The ettringite decomposition results in a drop of the total ignition loss of SAB cement mortar. This phenomenon together with lower formed $Ca(OH)_2$ content is the main reason of markedly decreased compressive strength of the SAB cement mortar opposite to the PC mortar (table 5). Development in the bound water con-

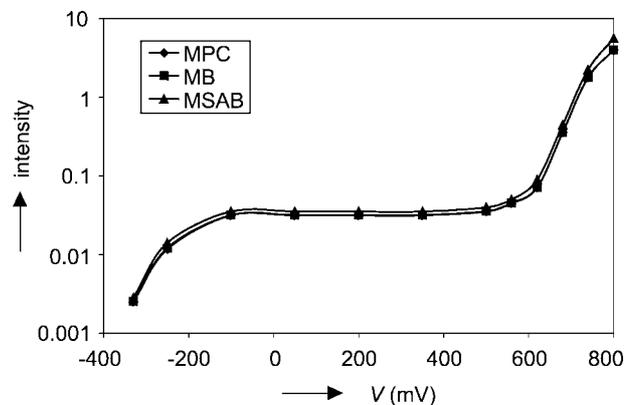


Figure 4. Potentiodynamic curves of steel in mortar extracts of 90 - day wet - cured mortars.

tent of SAB/PC mortar copies that of SAB cement mortar. Contrary, $\text{Ca}(\text{OH})_2$ and CaCO_3 , as well as total ignition loss values of SAB/PC mortar copy the development of PC cement mortar. This is confirmed by DTA curves of cement mortars (figure 3). PC mortar is characterized at 28 - day and 90 - day cure by an endothermic peak maximum at 180°C indicating prevailing C-S-H phase decomposition. The endothermic peaks maximized at 460°C and 740°C correspond to $\text{Ca}(\text{OH})_2$ dehydroxylation and CaCO_3 dissociation, respectively. The fourth endothermic peak maximized at 560°C corresponds to SiO_2 modification conversion. On the other hand DTA plots of SAB cement mortar at 28 - day cure shows a large, sharp endotherm at 120°C contributed to ettringite decomposition. The endotherms at 560°C and 680°C are due to quartz (SiO_2) modification and CaCO_3 dissociation, respectively. No typical endotherm characterizing ettringite decomposition was observed for 90 - day SAB cement mortar. A small wide endotherm with the peak maximized at 180°C belongs to C-S-H phase decomposition. The C-S-H phase formation is the result of β - C_2S hydration. DTA plot of 28 - days SAB/PC mortar is characterized by a sharp endotherm at 140°C belonging to ettringite decomposition and by the endotherms at 560°C and 720°C corresponding to SiO_2 modification and CaCO_3 dissociation, respectively. On the contrary, DTA plot of 90 - days SAB/PC mortar exhibits a wide endotherm at 140°C , a very small endotherm at 460°C corresponding C-S-H gel - like phase and $\text{Ca}(\text{OH})_2$ decomposition, respectively. The endotherms at 560°C and 720°C are due to SiO_2 modification and CaCO_3 dissociation, respectively. The SAB/PC mortar with 15 wt.% replacement of SAB cement by PC behaves partially as like as SAB cement mortar, and partially as like as PC mortar. The 15 wt.% of PC contributes markedly to the formation of gel - like hydration products and $\text{Ca}(\text{OH})_2$, and thus enhances the strength to the values similar to PC mortar.

Corrosion characteristics of steel in extracts of the mortars indicate negligible differences among individual parameters (table 8). Slightly decreased *pH* values of SAB/PC and SAB cement mortar extracts exhibits less alkaline character of SAB cement. Related potentiodynamic curves of steel bars (figure 4) are of the same course. It means that steel is sufficiently passivated in each mortar system.

CONCLUSIONS

The following conclusions are applicable to the tested cements and related mortar specimens:

1. The SAB cement is characterized by higher demand of batch water, quicker setting, lower CaO, and higher Al_2O_3 , Fe_2O_3 and SO_3 content as compared to Portland cement. This is the consequence of quite different mineralogical composition between both cement systems. The SAB cement is composed from low - energy clinker minerals such as $\text{C}_4\text{A}_3\text{S}$, C_2S and C_4AF which gives low - strength mortar compare to high-energy Portland cement mortar containing mainly C_3S which gives high-strength mortar.
2. The 85 wt.% SAB cement/15 wt.% PC blend gains the C_3S support for a C-S-H phase and $\text{Ca}(\text{OH})_2$ formation. Cement properties are markedly influenced by the PC addition, and are more similar to those of Portland cement. The 15 wt.% replacement of SAB cement by more active Portland cement evokes the 37.2 % compressive strength increase of 90 - day SAB/PC mortar compare to the SAB cement mortar. The decrease of 90 - day compressive strength of SAB cement mortar compare to the PC mortar represents 37.7 %, whereas the strength reduction of the SAB/PC mortar is only 14.5 %. This confirms the positive influence of Portland cement addition to upgrading the origin SAB cement properties and in the final effect of increasing the strength of mortar.

Acknowledgement

The authors are thankful to the Slovak Grant Agency (project 2/7035/21) for the financial support of this work.

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VPLYV PRÍDAVKU PORTLANDSKÉHO CEMENTU
K SULFOALUMINÁT - BELITOVÉMU CEMENTU
NA HYDRATÁCIU A MECHANICKÉ VLASTNOSTI
ZATVRDNUTÝCH MÁLT

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Prešetroval sa Portlandský cement (PC) CEM I 42,5 (SR), kalcium sulfoaluminát-belitový (SAB) cement (Rumunsko) a zmesný cement (85 hmot.% SAB cement/15 hmot.% PC) na maltových vzorkách veľkosti 40×40×160 mm (cement : piesok = 1 : 3 hmot., w/c = 0,5) ošetrovaných 90 dní vo vlhkom vzduchu (20°C, 100 % rel. vlhkosť). Hlavné slinkové minerály SAB cementu s teplotou výpalu 1 250°C sú C₄A₃S a C₂S a PC s teplotou výpalu vyššou o 200°C C₃S a C₂S. SAB cement patrí do kategórie nízko-energetických cementov.

Pevnostné (pevnosť v ťahu za ohybu a v tlaku) a pružné charakteristiky (dynamický modul pružnosti, DMP) malty zo SAB cementu sú najnižšie. Náhrada 15 hmot.% SAB cementu za PC zvyšuje 90 - dňovú pevnosť v ťahu za ohybu a DMP malty o 15 % a pevnosť v tlaku skoro o 40 % v porovnaní s maltou zhotovenou zo SAB cementu. Mechanické vlastnosti malty zo zmesného SAB/PC sú blízke malte vyrobenej z portlandského cementu.

Rozdielnosti v mechanických vlastnostiach mált sú zapríčinené odlišnosťami v chemickom a mineralogickom zložení cementov. To v konečnom efekte vedie k rozdielnostiam v kinetike hydratačného procesu podľa hydratačnej aktivity rozhodujúcich slinkových minerálov každého cementu. V hydratovanom PC je dominantný hydratačný produkt C-S-H gél, ktorého vznik doprevádza intenzívna tvorba Ca(OH)₂. V SAB cimente dominuje časovo nestabilný etringit s minoritným podielom Ca(OH)₂. Rozdielna kvalita a kvantita vytvorených hydratačných produktov a Ca(OH)₂ je hlavnou príčinou zvýšenej normálnej hustoty, rýchleho počiatku a doby tuhnutia SAB cementu a nižšieho DMP a pevnosti malty zo SAB cementu v porovnaní s Portlandským cementom resp. maltou zhotovenou z PC. Zmesný cement (85 hmot.% SAB cement/15 hmot.% PC) získava na hydratačnej aktivite a tým aj na DMP a pevnostiach C₃S aktívatorom z PC podporujúcim hydratačný proces menej aktívneho SAB cementu. Všetky zistené skutočnosti svedčia o rozhodujúcom význame hydratačnej aktivácie SAB cementu portlandským cementom, pričom na zvýšenie úžitkových vlastností SAB cementu postačuje 15 hmot.% PC.