THERMOPHYSICAL PROPERTIES OF BLENDS FROM PORTLAND AND SULFOALUMINATE-BELITE CEMENTS $^{\rm 1}$

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The behavior of mortars with blends consisting of sulfoaluminate-belite (SAB) cements and ordinary Portland cement (OPC) made with cement to sand ratio of 1:3 by weight and w/c =0.5 maintained for 90 days at 20°C either at 60% relative humidity (RH)-dry air or 100% RH-wet air. The results show insufficient character of hydraulic activity of SAB cements. Their quality has been improved. The replacement of 15 wt % of SAB cement by OPC influences strength positively and elasticity modulus values as well as hydrated phases and pore structure development of SAB/OPC blends relative to pure SAB cement systems. The above statements confirm the possible making technologies, when improvements in SAB cements quality will be achieved. One would then anticipate the competition in usages between SAB/OPC and blast furnace-slag Portland cement (BFSPC) systems in the practice. It is important to consider because SAB cements are of great advantage from the viewpoint of energy savings and quantity of CO₂ released during their production. Thermal characteristics of the samples were studied by thermogravimetry (TG) and differential thermal analysis (DTA) from room temperature to 1000°C in air atmosphere. Generally, four significant temperature regions on TG curves with the respective DTA peak temperature for all types of samples are observed.

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1 Introduction

Blended cements have attracted intensive attention. There have been many successful practices of blended cements in the concrete industry in the past. There is still a necessity to describe important roles in advancing the use of blended components: 1) incorporating those components

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	Cements employed					
	OPC	BFSPC	SAB 1	SAB1/OPC	SAB 2	SAB2/OPC
Humidity	0.02	0.24	0.35	0.02	0.04	0.03
Igmition loss	1.40	1.02	0.60	0.46	0.13	0.40
Ins. residue	1.66	1.51	0.16	0.57	4.60	3.94
SiO_2	19.51	24.88	23.46	22.62	20.87	21.09
CaO	63.60	55.18	55.06	58.50	54.59	55.41
MgO	1.49	4.70	1.78	0.46	1.38	1.78
Fe_2O_3	3.48	4.70	6.25	5.45	6.46	5.69
Al_2O_3	5.67	4.27	7.35	7.27	7.03	7.06
SO_3	3.01	3.22	4.99	4.65	4.90	4.60
CaO free	0.10	0	0.02	0.02	0.05	0.04
Bulk density						
$(kg.m^{-3})$	3 168	3 047	3 2 3 6	3 152	3 2 3 4	3 166
Surface area						
$(m^2.kg^{-1})$	443	342	440	419	371	390
Initial set						
(minutes)	125	160	15	15	10	10
Final set						
(minutes)	205	245	20	20	15	20

Tab. 1. Chemical composition and basic properties of the cements

into more products and processes, 2) modifying products to provide their compatibility with engineering properties, and 3) optimizing cement and concrete systems by optimizing resistance to chemical attack, freezing and thawing and repressing alkali-aggregate reaction [1-3]. In recent times, much attention has been given to the development of some modified special cement clinkers, leading to energy saving [4]. One of such cements containing the main phases C_2S , $C_4A_3\overline{S}$, C_4AF and $C\overline{S}$ was developed and reported by many researches [5–10]. The notations used herein are explained at the end of this paper. This special cement contains C2S, C4AF and two sulfate phases $C_4A_3\overline{S}$ and $C\overline{S}$ instead of high-temperature C_3S and C_3A . Raw mixes for $C_4A_3\overline{S}$ clinkers differ from those for OPC due to the significant amounts of sulfates; therefore reactions and products are quite different than those normally found in OPC production [11-14]. Calcium sulfoaluminate-based cement compositions containing more than 50% $C_4A_3\overline{S}$ show expansion, cracking and loss of strength at later ages. Compositions with more than 20% $C_4A_3\overline{S}$ gave progressively lower mechanical strength, when hydrated with the fixed water/reactants ($C_4A_3\overline{S}$ + \overline{CS}) ratio of 0.86. The best behavior was obtained with cements containing 30–40% $C_4A_3\overline{S}$ at water to cement ratio about 0.35 to 0.50 [15]. Cements containing $C_4A_3\overline{S}$, β - C_2S and $C\overline{S}$ at 1.5:1:1 weight ratio hydrate rapidly to ettringite and develop early strength. All these compositions have very good dimensional stabilities, similar to OPC and good resistance to atmospheric carbonation, although the ettringite component in hydrated cement tends to carbonate [16-18]. Preliminary results show the resistance of sulfoaluminate-based cements to hydrochloric acid, sodium sulfate, and sodium chloride solutions is very similar to that of OPC [19], but consider-

Type of		Portion of clinker minerals (%)						
the cement	C_3S	C_2S	C_3A	C_4AF	$C_4A_3\overline{S}$	$C\overline{S}$		
OPC	67.6	5.1	9.2	10.6	no	5.9		
BFSPC		no calculation was done						
SAB 1	no	67.1	no	19.0	6.7	7.0		
SAB 1/OPC	10.1	57.8	1.4	17.7	5.7	6.8		
SAB 2	no	59.7	no	19.6	5.8	7.1		
SAB 2/OPC	10.1	51.5	1.4	18.3	4.9	6.9		

Tab. 2. Mineralogical composition of test cements.

Note: Mineralogical composition of Portland cement (PC) is calculated by the Bogue method and those of SAB cements and SAB/PC blends by the method developed at the Faculty of chemical engineering Slovak Technical University, Department of ceramics, glass and cement, Bratislava using data of chemical compositions of the cements.

ably lower to freezing and thawing [20]. MDF cements based on sulfoaluminate-belite clinkers were synthesized and showed sufficient moisture resistance [21–23]. An experimental study on the upgrade of sulfoaluminate-belite cement systems by blending with OPC indicates increased strength and dynamic modulus of elasticity as well as the decreased water absorption capacity of the blends compare to SAB cements.

The present paper is concerned with the influence of sulfoaluminate-belite cements and their blends on mechanical properties of mortar specimens, hydrated phases and pore structure development. The results obtained with pure sulfoaluminate-belite cements and their blends with OPC are confronted with those of OPC and blast furnace-slag Portland cement.

2 Experimental

2.1 Materials

Ordinary Portland cement CEM I 42.5, blast furnace-slag Portland cement (BFSPC) with 30 wt % of blast furnace-slag and two types of sulfoaluminate-belite (SAB 1 and SAB 2) cements were used in this investigation. The SAB cements were mixed with OPC in the weight ratio of 85% to 15%. The characteristics of the cements are listed in Tab. 1 and 2. Four cements (OPC, BFSPC, SAB 1 and SAB 2) and two mixed cements were used to prepare mortar specimens, 40x40x160 mm in dimension, with cement to sand ratio of 1:3 and water/cement ratio of 0.5.

2.2 Test methodologies

The specimens were stored at 20°C and 100% RH for 7 days and subsequently at 20°C/100% RH and at 20°C/60% RH, respectively for 83 days.

Mortar specimens were tested for their compressive strength, water absorption capacity, specific gravity, volume density and total porosity. The water absorption capacity was determined from the decrease in weight of the specimens saturated by water and then dried at 105°C to

Property of	Тур	Type of cement mortar cured in dry (D) and wet (W) air						
the mortar	MPC	MBFSPC	MSAB 1	MB (1)	MSAB 2	MB (2)		
Strength (MPa) (D)	52.1	35.1	5.8	20.6	6.4	18.7		
Strength (MPa) (W)	66.2	40.2	12.8	32.0	12.8	26.9		
Absorption capacity								
(wt %) (W)	7.1	8.0	10.7	8.4	10.9	9.4		
Specific gravity								
$(kg.m^{-3})$ (W)	2 6 3 6	2 678	2 796	2 714	2 808	2 771		
Bulk density								
$(kg.m^{-3})$ (W)	2 286	2 274	2 159	2 253	2 148	2 2 3 2		
Total porosity								
(vol. %) (W)	13.3	15.1	22.8	17.0	23.5	19.5		

Tab. 3. Physico-mechanical properties and total porosity of mortars after 90-day curing Note: Compressive strength was tested either in $20^{\circ}C/60\%$ RH-dry cure (abbreviated as D) or in $20^{\circ}C/100\%$ RH-wet cure (abbreviated as W).

constant weight. The results are expressed in weight percent (wt %). The bulk density was determined on the specimens of regular shape by weighing at calculated volume of the prisms. The specific gravity was measured by the pycnometric method as the weight of the volume unit of solid constituents of the mortar specimen in powdered stage.

The total porosity was calculated from the bulk density and specific gravity values using the formula:

$$TP = \left(1 - \frac{\rho_{\rm VD}}{\rho_{\rm SG}}\right) \times 100 \tag{1}$$

where TP is total porosity as the content of pores and voids in the specimens %, $\rho_{\rm VD}$ is volume density (kg.m⁻³) and $\rho_{\rm SG}$ is specific gravity (kg.m⁻³). Ultrasonic pulse velocities were measured on an ultrasonic apparatus (UNIPAN type 543, Poland) calculating the dynamic modulus of elasticity ($E_{\rm bu}$) by the formula

$$E_{\rm bu} = \rho_{\rm VD} \gamma_{\rm L}^2 \times 10^{-6} \tag{2}$$

where $E_{\rm bu}$ is dynamic modulus of elasticity (MPa), $\rho_{\rm VD}$ is volume density (kg.m⁻³) and $\gamma_{\rm L}$ is impulse speed of longitudinal ultrasonic waves (m.s⁻¹).

The changes in length of prisms were measured by a portable mechanical strain gauge apparatus (in a glass tube to minimize extraneous effects). The gauge length was 100 mm, with a measuring accuracy of 0.001 mm. Changes in length of mortar specimens are expressed in per mille (‰). The relationship of per mille to microstrain is 1 μ s = 0.001‰. The pH values of mortar extracts were determined by a pX meter OP 113 (Radelkis, Hungary).

Powder X-ray diffraction patterns were recorded on a Philips X-ray diffractometer coupled with an automatic data recording system. CuK_{α} radiation and Ni-filter were used. Chemical compositions of the mortars determined by analytical methods. Simultaneous thermogravime-try (TG) and differential thermal analysis (DTA) were conducted from 20°C to 1000°C using a



Fig. 1. Changes in dynamic modulus of elasticity $(E_{\rm bu})$ of test mortars kept in 20°C/60% RH air



Fig. 3. Development in length changes of the mortars cured in 20°C/60% RH—dry air



Fig. 2. Changes in dynamic modulus of elasticity $(E_{\rm bu})$ of test mortars kept in 20°C/100% RH air



Fig. 4. Development in length changes of the mortars cured in 20°C/100% RH—wet air

T.A.I. SDT 2960 instrument (sample mass 10–20 mg, heating rate 10°C/min, in flowing air). The pore structure was measured by the mercury porosimetry test using the high-pressure porosimeter model 2000 and macroporosimetry unit 120 (both Carlo Erba, Italy). The following pore structure characteristics were determined: volume of micropores (to 7 500 nm); micropore and pore median radius and porosity ranged between pore radius 3.75 nm and 0.2 mm.

3 Results and discussion

3.1 Mechanical properties

Six types of mortar specimens—the mortar with 100 wt% of OPC (signed in the text as MPC), 100 wt % of BFSPC (MBFSPC), 100 wt % of SAB 1 cement (MSAB 1), 100 wt % of SAB 2 cement (MSAB 2) as well as the mortar with blend 85 wt % of SAB 1 cement/15 wt % of OPC [signed in the text as MB (1)] and the mortar with blend 85 wt % of SAB 2 cement /15 wt% of OPC [MB (2)]—were used in the experiment. It is evident from the experimental results that

Mortar	Chemical composition of mortars (wt. %)								
	Ignition loss	5.18	Compo	sition of	soluble p	ortion			
MPC	Ins. portion	70.60	SiO_2	CaO	Fe_2O_3	Al_2O_3	MgO	SO_3	
	Sol. portion	22.67	4.96	14.42	0.73	1.66	0.20	0.70	
	Total oxide co	ontent in	Oxide	Oxide content related to cement binder					
	sol. portion	100.00	21.88	63.61	3.22	7.32	0.88	3.09	
	Ignition loss	Ignition loss 4.55 Composition of soluble portion							
	Ins. portion	72.82	SiO_2	CaO	Fe_2O_3	Al_2O_3	MgO	SO_3	
MBFSPC	Sol. portion	21.34	5.24	11.14	1.58	1.97	0.78	0.63	
	Total oxide co	ontent in	Oxide	content r	elated to a	cement bi	nder		
	sol. portion	100.00	24.56	52.20	7.40	9.23	3.66	2.95	
	Ignition loss	2.18	Compo	sition of	soluble p	ortion			
	Ins. portion	74.35	SiO_2	CaO	Fe_2O_3	Al_2O_3	MgO	SO_3	
MSAB 1	Sol. portion	22.57	4.80	12.80	1.65	1.92	0.28	1.12	
	Total oxide content in		Oxide content related to cement binder						
	sol. portion	100.00	21.27	56.71	7.31	8.51	1.24	4.96	
	Ignition loss 4.54 Composition of soluble portion								
	Ins. portion	71.18	SiO_2	CaO	Fe_2O_3	Al_2O_3	MgO	SO_3	
MB (1)	Sol. portion	22.61	4.77	13.28	1.39	1.97	0.15	1.05	
	Total oxide content in		Oxide content related to cement binder						
	sol. portion	100.00	21.10	58.74	6.15	8.71	0.66	4.64	
	Ignition loss	2.48	Composition of soluble portion						
	Ins. portion	74.86	SiO_2	CaO	Fe_2O_3	Al_2O_3	MgO	SO_3	
MSAB 2	Sol. portion	21.72	4.63	12.35	1.59	1.97	0.10	1.08	
	Total oxide co	ontent in	Oxide content related to cement binder						
	sol. portion	100.00	21.32	56.86	7.32	9.07	0.46	4.97	
	Ignition loss	3.03	Compo	sition of	soluble p	ortion			
MB (2)	Ins. portion	74.56	SiO_2	CaO	Fe_2O_3	Al_2O_3	MgO	SO_3	
	Sol. portion	20.99	4.40	12.39	1.05	2.15	0.10	0.90	
	Total oxide co	ontent in	Oxide	content r	elated to a	cement bi	nder		
	sol. portion	100.00	20.96	59.03	5.00	10.24	0.48	4.29	

Tab. 4. Chemical composition of mortars cured 90 days in 20°C/100% RH-wet air

properties such as compressive strength, water absorption capacity and total porosity of mortars are influenced considerably by the type of cement and curing conditions. Experimental data also indicate that the blending of SAB cement with OPC is a possible way to improve SAB cement properties (Tab. 3, Figs. 1 and 2). The shrinkage compensation properties of SAB cements over 90 days are evident from Fig. 3. The difference in 90 day values of length changes between specimens with SAB cements and other samples is approximately 0.60‰. This is in good agreement with English and Chinese experience of calcium sulfoaluminate cements in practice [24–27]. The OPC mortar MPC, and MB (1) as well as MSAB 2 mortar show a slight shrinkage under moist air curing (Fig. 4). The MBFSPC, MB(2) and MSAB 1 show a tendency to expand slightly.

Туре	Micropore volume	Micropore	Pore	Porosity
of mortar	up to 7 500 nm	median ra	ndius	(3.75 nm – 0.2 mm)
	(%)	(nm)	(nm)	(%)
MPC	95.4	52.0	53.7	11.3
MBFSPC	89.2	55.9	62.1	13.9
MSAB 1	97.5	255.6	263.7	22.0
MB (1)	90.5	55.8	56.9	15.9
MSAB 2	91.5	276.5	304.4	23.0
MB (2)	94.1	45.3	47.2	17.4

Tab. 5. Pore structure of mortar specimens cured 90 days in 100% RH-wet air

Type of	Pores portion (%)						
mortar	3.75 - 50	50 - 100	100 - 500	500 - 1000	1000 - 7500	over 7500	
	nm	nm	nm	nm	nm	nm	
MPC	40.4	44.3	5.9	3.0	3.4	3.0	
MBFSPC	37.0	30.6	10.5	3.2	7.3	11.4	
MSAB 1	17.2	7.8	57.0	11.0	4.9	2.1	
MB (1)	34.5	48.3	2.0	2.1	4.1	9.0	
MSAB 2	16.5	11.7	52.6	6.1	4.7	8.4	
MB (2)	48.0	29.3	11.8	0.7	5.0	5.2	

Tab. 6. Pore size distribution of mortars after 90-day wet air cure

Differences in 90-day length changes of mortars cured in moist air are only ca 0.20‰. The results given in Tab. 3 and Figs. 1, 2, 3 and 4 provide a true picture of the relationship between the cement mortar composition and important engineering properties.

Importantly, increased strength and elasticity modulus on the one hand and decreased water absorption capacity and total porosity on the other hand are indicted by the results of mortars made with the above blends in comparison with pure SAB cement mortars. SAB cement mortars also show reduced shrinkage than the OPC mortar.

The disadvantage of SAB cements may be overcome by the selection of a more appropriate raw material composition for their manufacture. Significant improvements in utility properties of SAB/OPC blends can be then expected.

3.2 Cement hydration and pore structure

It is well known that mechanical properties of mortars are largely dependent on the microstructural development through changes induced by the processes of hydration of the cement paste under a given curing condition. Properties including strength, elastic modulus, total porosity and shrinkage/expansion are influenced mainly by the hydrated phases and development of the pore structure.

Sample	TG	DT	A
MPC	20-400 (CSH, CSH ₂ , AFt, Afm)	71, 148	Endo
	400-550 (CH)	433	Endo
	550-800(\overline{C})	570	Endo
	Above 800 (recrystallization)	904	Exo
MBFSPC	20-400 (CSH, CSH ₂ , AFt, Afm)	31, 73	Endo
	400-550 (CH)	435	Endo
	550-800(\overline{C})	571	Endo
	Above 800 (recrystallization)	899	Exo
MSAB1	20-350 (CSH, CSH ₂ , AFt, Afm)	68, 229	Endo
	350-550 (CH)	416	Endo
	550-800(\overline{C})	574	Endo
	Above 800 (recrystallization)	897	Exo
MB (1)	20-250 (CSH, CSH ₂ , AFt, Afm)	71	Endo
	250-525 (CH)	430	Endo
	$525-800(C\overline{C})$	571	Endo
	Above 800 (recrystallization)	897	Exo
MSAB2	20-450 (CSH, CSH ₂ , AFt, Afm)	66, 197	Endo
	450-580 (CH)	409	Endo
	$580-785(\overline{C})$	574	Endo
	Above 785 (recrystallization)	898	Exo
MB (2)	20-340 (CSH, CSH ₂ , AFt, Afm)	66, 146	Endo
	340-500 (CH)	367	Endo
	$500-80(\overline{C})$	571	Endo
	Above 800 (recrystallization)	902	Exo

Tab. 7. Summary of TG intervals (°C) and DTA peak temperatures (°C) of the samples

The chemical compositions of the test mortars cured for 90 days in moist air are given in Tab. 4. The soluble portions of SAB and SAB/OPC cement mortars have less CaO and more Fe_2O_3 , Al_2O_3 and SO_3 , in comparison with OPC mortar. Results of X-ray diffraction analysis of the mortars are shown in Fig. 5. The presence of Ca(OH)₂ (d-values of 4.92 Å and 2.64 Å) is clearly seen in OPC and BFSPC mortars, and also in MB (1) and MB (2) mortars due to the 15 wt % addition of OPC in them. Conversion of unreacted belite or alite is characterized by the doublet in the region of 2.78 Å and 2.74 Å, which generally corresponds to this two clinker minerals. Both SAB mortars exhibit a considerably reduced conversion of unreacted belite to hydration products. Its conversion in MB (1) and MB (2) mortars is evident; and markedly accelerated in BFSPC and OPC mortars.

Thermal characteristics of the samples, studied by TG and DTA are summarized in Tab. 7. Generally, four significant temperature regions for all types of samples are observed:

 Up to 400°C — main temperature region of decomposition of the cement hydration products (CSH, CSH2, AFt and AFm phases). The interpretation of the thermogravimetric curves in this region is difficult because the decomposition temperatures of CSH, ettringite, gypsum and Afm phases lie close together [28–31].



Fig. 5. Diffractograms of test mortars after 90-day wet air cure

- 2. 400–550°C temperature region of Ca(OH)₂ decomposition [28–30].
- 3. 550–800°C temperature region of decomposition of carbonate, hydrated aluminate and the final stage of CSH phases [28–31].
- 4. Above 800°C temperature region of recrystallization of new non binding phases arising from hydrated cement minerals under recombustion [32].

However, a little bit differences of the temperature ranges for different samples were observed which are exactly presented in Tab. 7.

The observed loss of bound water in MB (1) and MB (2) mortars and mainly OPC mortar; and the dehydroxylation of $Ca(OH)_2$ in OPC, BFSPC and partially in MB (1) mortar, correlate

well with the results of X-ray diffraction analysis. It is evident from the degree of hydration of the OPC and BFSPC mortars contributing to their strength and elasticity characteristics, that they are considerably more reactive than both the SAB cements. The 15 wt % addition of OPC to SAB significantly improves the formation of hydrated phases in comparison with the pure SAB cement, but not to such an extent to be comparable with hydrated phases development in OPC and BFSPC mortars. This aspect of SAB cements must be improved before they can be expected to compete effectively with other blended cements like BFSPC.

The reduced volume of the hydrated phases in SAB cement mortars induces a micro-pore and pore median radius, and porosity is increased (Tab. 5) compared to OPC and BFSPC mortar. Nevertheless, the pore structure characteristics of mortars with mixed cements [MB (1) and MB (2)] show distinct similarities with those made from OPC and BFSPC. The positive role of OPC in the blends with SAB cement is evident. Data given in Tabs. 5 and 6 unequivocally confirm the phenomenon of pore structure "coarsening" due to the SAB cement in the mortar. The increase in porosity is due to a higher proportion of micro-pores with a radius of 100–500 nm within the SAB cements mortar structure in comparison with the OPC and BFSPC mortars with the decisive percentage of micro-pores between a radius of 3.75 and 100 nm. The pore size distribution of MB (1) and MB (2) mortars is similar to those of OPC and BFSPC mortars. The 15 wt % addition of OPC in the blends with SAB cements appears to be sufficient to improve the pore structure and pore size distribution of the observed mortars in comparison with pure SAB cements mortar. The pore structure characteristics of MB (1) and MB (2) mortars are similar to those of OPC and BFSPC mortars. This reiterates the positive influence of OPC portion in blends with the SAB cements.

Strength and elasticity characteristics and deformational behavior ability of the mortars show variation depending on the type of cement or cement blends used in the mortar. Different types of hydration products and of degree of hydration in the studied mortars generate pore structures with diverse and markedly characteristic pore size distribution. The utility properties of SAB cement mortars are considerably improved by 15 wt % replacement of SAB cements by OPC. The reasons for this fact are the increase in volume of the hydrated phases and Ca(OH)₂; and the formation of finer pore structure in the mortar with a mixed cement.

4 Concluding remarks

The following conclusions are applicable to the particular mortars and test conditions employed:

- 1. The replacement of 15 wt % SAB cement by OPC offers the blend suitable for the mortar preparation with increased strength and elasticity modulus as well as decreased water absorption capacity and total porosity compared to the pure SAB cement mortars.
- 2. The reason for this fact are different in the crystalline structure of developed hydrated phase, pore structure and percentage portion of individual pores of distinct pore radii.
- 3. The improvement in SAB cement quality is unavoidably required to be competitive with at least BFSPC in mortar and concrete-making technological procedures in the future.
- 4. Thermal decomposition of the samples was studied by the TG and DTA. There are four significant temperature regions on TG curves with the corresponding DTA peak temperature for all types of cement and mortar samples.

5 Notations used

The following notations are applied to distinguish main cement minerals:

Special abbreviations for clinker minerals of cement are used in the silicate chemistry worldwide due to simplification of chemical formulae. Four main clinker minerals of Portland cement are C_3S with the meaning of 3 CaO. SiO₂ (solid solution of 3 molecules of CaO and 1 molecule of SiO₂, the completely accurate formula does not exist), C_2S (2 CaO. SiO₂), C_3A (3 CaO. Al₂O₃) and C_4AF (4 CaO. Al₂O₃. Fe₂O₃). Sulfoaluminate-belite cement is characterized by C_2S , C_4AF , $C_4A_3\overline{S}$ (4 CaO. 3 Al₂O₃. SO₃) and $C\overline{S}$ (CaO. SO₃, what means CaSO₄).

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