BURNT KAOLIN SAND AS POZZOLANIC MATERIAL FOR CEMENT HYDRATION

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Original kaolin sand from deposit Vyšný Petrovec Slovakia, after temperature transformation provides burnt kaolin sand that contains metakaolin as a pozzolan. In order to verify potential efficiency of pozzolanic effect of its blend with ordinary Portland cement, the research oriented on the related cement-kaolin sand specimens was commenced. The relation between microstructural composition and compressive strength was studied. For this aim two types of kaolin sand with fractions 63-36 µm and below 36 µm were used. The fraction 90-63 µm is not suitable for using in cement-burnt kaolin sand specimens. The optimal replacement of cement by burnt kaolin sand of 20, 30 and 40 wt. % was determined. The Portland cement-burnt kaolin sand blends contain between 4.10 and 14.48 wt.% of pure metakaolin. The strength of cement-burnt kaolin sand specimens depend slightly on the hydration products formed during hydration process of cement. The capability of pure metakaolin in burnt kaolin sand to combine with the most part of $Ca(OH)_2$ was confirmed by the X-ray diffraction analysis. The $Ca(OH)_2$ content is reduced due to pozzolanic reaction.

INTRODUCTION

The reaction of metakaolin (MK) in blended cements has been reported for several years, mainly with the aim to describe the enhanced durability [1-4] and refined pore structure [5]. Other studies were oriented on the reaction of MK with calcium hydroxide (CH) [6, 7]. The common conclusion was that the overall hydration products and the developed pore structure are not the same as those formed with Portland cement (PC) only. During the hydration of PC, CH is typically formed in the range of 16-28 wt.% [8] depending on the mineral composition of the cement. Partial replacement of PC by MK leads to a proportionally lower free lime production resulting in the reduced CH content in the cement paste, whereby new cementitious compounds are arisen. When MK is added to PC, crystalline products of calciumaluminate hydrates and aluminosilicate hydrates such as C_2ASH_3, C_4AH_{13}, C_3AH_6 and CSH gel are formed. Final composition of the hydrate phase depends mainly on MK/CH ratio and the reaction temperature, respectively. The pozzolanic activity of commercial metakaolins varies between 610 and 1150 mg CaO/g, thereby considerably exceeding the pozzolanic activity of silica fume that is around 400-450 mg CaO/g [9]. In summary, the presence of MK in the cement paste and concrete reduces the CH content. Although total porosity may be increasing somewhat by MK addition [10, 11]; metakaolin strongly influences the pore structure of concrete by pore refinement leading to significant delays in mobility of harmful substances [12]. This leads to a denser microstructure that gives the MK–cement system with a larger stability in an aggressive medium. The effects of MK on durability of cement paste and concrete depend on factors such as cement and metakaolin composition, fineness as well as pozzolanic activity of MK and blending percentages [13,14]. The above findings demonstrate that the PC replacement by MK can increase the durability of the concrete by CH reduction and the improvement of microstructure of concrete by refining the pore structure.

Metakaolin is a reactive aluminosilicate pozzolan formed by purified kaolinite clays at a specific temperature range and by grinding it to a high fineness. Chemically, MK encompasses as main constituents SiO_2 and Al_2O_3, and in smaller quantities Fe_2O_3, CaO, MgO, SO_3, Na_2O and K_2O. The efficiency of MK as a pozzolan in cement and concrete is mainly governed by high content of SiO_2 and Al_2O_3. The high pozzolanic activity is also due to the large portion of small particles of metakaolin. Commercial MK is a powder of which the fineness is around 12 000-15 000 m^2/kg. The major part of MK is in 5-10 µm range [15,16].

Metakaolin - blended cement paste with up to 20 wt.% of MK has compressive strength at ages 28 days and later similar to that of the plain cement paste [16]. Only at 180 days of age, the blended paste with 20 wt.% of MK has a lower compressive strength than that of the plain one. Other investigations reveal that the compressive strength of blended cement pastes in the range of 5-15 wt.% of MK are slightly improved when compared to the reference cement paste [17, 18]. The favourable effects of blending with MK can be due to the characteristics of MK (chemical, mineralogical composition, fineness) as well as the thermal treatment in the production stage that affect both pozzolanic activity of MK and strengths of the cement pastes [19-22]. Durability studies on mortars prove that the ones with 20 wt.% substitution of cement by MK exhibit the lowest capillary porosity and highest chloride penetration resistance. The best results show the mortars additioned with MK obtained at 800°C but those gained at 700°C and 600°C also yield much better results than control mortar [23].

The current knowledge on metakaolin is focused on the pozzolanic behaviour of high-purity commercial MK and its effect on cement hydration and concrete properties. It is not satisfactory knowledge level about poor kaolins except of Greek experience how to exploit poor kaolins in concrete technology [24]. This study reveals the effect of four kaolins with kaolinite content between 38 % and 52 % by weight on the cement hydration when compared to the cement paste with high-purity commercial metakaolin with 90 wt.% of MK. Metakaolin cements containing metakaolin derived from poor Greek kaolins show some similarities in the behaviour with cements made with commercial MK with respect to strength development, setting time and hydration. Although a more thorough investigation is required, their exploitation seems to be promising. The same problem is standing in front of Slovakia having deposit of poor kaolin in Vyšný Petrovec. Calculated supplies of kaolin sand are slightly higher than 20 000 kilotonnes. The study [25] reports that this material is not suitable for ceramic and glass industry as input raw material after laboratory - made treatment. The structures of various cement-based materials as well as the influence of their compositions and blending percentages on the process of cement hydration and strength development are of great interest. Therefore, it is not surprising that many authors have investigated numerous blended cement systems with different origins and also studied their mechanical, microstructural, and many other properties [26-29].

In the present paper we are reporting our data on the hydration and strength of cement systems prepared with PC-burnt kaolin sand blends and containing between 4.10 and 14.48 wt.% of pure metakaolin when replacing of Portland cement by kaolin sand up to 50 wt.%.

EXPERIMENTAL

Materials

Two types of raw material - original kaolin sand (OKS) from the deposit Vyšný Petrovec - were used for the experiment (OKS 1 and OKS 2). Each type of OKS was ground and sieved on three fractions: 90-63 μm (thereinafter 90 μm); 63-36 μm (thereinafter 63 μm); below 36 μm (thereinafter 36 μm). These types and fractions of OKS were transformed using high temperature to the burnt kaolin sands (BKS). The transformation conditions were as follows: the rate of heating - 10°C/min; exposure time at 650°C - 1 hour; the rate of self-acting cooling - 10°C/min. Ordinary Portland cement CEM I 42.5 R (PC) was used for preparation of cement-BKS specimens. These specimens were represented by cubes with edge of 20 mm. The water/cement (w/c) ratio was kept at 0.5. Replacement of PC by BKS has manifested 10, 20, 30, 40 and 50 wt.%. The cement-zeolite specimens (the same size 20 × 20 × 20 mm and w/c = 0.5) with the replacement of PC by 20, 30 and 40 wt. % of natural zeolite as well as cement paste with 100% PC (w/c = 0.5) were prepared as reference specimens. Industrially - made ground natural zeolite in fraction below 63 μm from Nižný Hrabovec (Slovakia) was taken as reference pozzolan.

Test methodologies

Chemical analysis of OKS, BKS and PC was made by standard analytical methods. Quantitative X-ray analysis of OKS was realized by the Rockjock program [30]. The program fits the sum of stored XRD patterns of pure standard minerals (the calculated pattern) to the measured pattern by varying fraction of each mineral standard pattern, using the Solver function Microsoft Excel to minimize a degree of fit parameter between the calculated and measured pattern. Samples for analysis were prepared by adding 0.111 g ZnO (internal standard) to 1.000 g sample. The mixture was ground in a McCrone mill for 5 minutes with 4 ml of methanol than dried and sieved.

Cement-BKS specimens were stored for 90 and 180 days in water and then were tested for compressive strength. Powder X-ray diffraction (XRD) profiles of the powder samples were collected in the 2Θ range from 8° to 36° using automatic data recording system on a Philips diffractometer with CuK_α radiation and Ni-filter.

RESULTS AND DISCUSSION

The chemical analysis of OKS is presented in Table 1. Basically, OKS 1 is richer in SiO_2 content and poorer in Al_2O_3 content in comparison with OKS 2. This is the significant difference between two types of OKS. In addition OKS 1 shows the higher content of CaO and the lower Fe_2O_3 content.

As regards alone OKS 1 - it is clear that with the increase of fineness, the SiO_2 content is decreased and Al_2O_3 content is increased. The similar tendencies are valid for OKS 2 as well. Accordingly the values of ignition loss are increased with the increase of fineness in both (OKS 1 and OKS 2) kaolin sands.

Results of quantitative X-ray analysis (Rockjock program) are evaluated in Table 2. The predominant non clay minerals are represented by quartz and feldspar. By contrast the present clay minerals are represented by kaolinite, illite and muscovite. It is evident that major content of kaolinite contains OKS 2 compared to OKS 1. Moreover with the increase of fineness, the content of kaolinite is increased and content of quartz is decreased. Thus the OKS 2 with fineness 36 µm is characterized by the highest content of kaolinite. On the other hand the lowest content of kaolinite is present in OKS 1 with fineness of 90 µm. This value is only on the level 42.5 wt. % of OKS 2 (36 µm). To this effect, the best theoretical assumptions for pozzolanic reaction (after transformation of OKS to BKS) are anticipated for the OKS 2 sample with fineness 36µm.

The chemical analysis of BKS is reported in Table 3. The basic tendencies valid for OKS are characteristic for BKS as well. The highest content of Al_2O_3 and contrary the lowest of SiO_2 is found in BKS 2 with fineness 36 µm.

The chemical and mineralogical composition of PC together with its basic properties is given in Table 4. The course of 90-day compressive strength of cement-BKS specimens when depending on the replacement of PC by BKS is shown in Figure 1. It is clear that fraction 90 µm of both BKS 1 and BKS 2 is not suitable for using in blended systems. These coarse-grained fractions of BKS 1 and BKS 2 namely cause considerable

Table 1. Chemical composition of original kaolin sands (OKS).

Type and fineness of OKS	Humidity (%)	Ignition loss (%)	SiO_2 (%)	CaO (%)	MgO (%)	Fe_2O_3 (%)	Al_2O_3 (%)	SO_3 (%)
OKS 1	0.85	3.55	72.70	2.23	0.30	1.09	19.04	0.24
OKS 1/90	0.75	3.41	72.01	3.27	0.62	1.19	18.47	0.27
OKS 1/63	1.04	5.00	63.76	5.56	0.81	1.82	21.67	0.34
OKS 1/36	1.19	5.97	60.19	5.74	1.17	1.56	23.83	0.35
OKS 2	1.63	5.98	60.66	2.74	1.21	3.25	24.18	0.35
OKS 2/90	1.46	6.38	59.97	2.60	1.26	3.27	24.77	0.29
OKS 2/63	1.53	7.50	58.56	2.50	0.40	3.27	26.01	0.23
OKS 2/36	1.65	7.55	57.51	2.75	0.30	3.03	27.01	0.20

Table 2. Mineralogical composition of original kaolin sands (OKS).

Type and fineness of OKS	Composition (%)				
	Non clay minerals		Clay minerals		
	Quartz	Feldspar	Kaolinite	Illite	Muscovite
OKS 1	43.5	5.6	20.3	8.6	22.0
OKS 1/90	59.6	4.7	15.4	5.4	14.9
OKS 1/63	45.8	4.6	20.5	5.8	23.3
OKS 1/36	34.3	5.3	26.9	5.0	28.5
OKS 2	25.5	4.1	34.4	7.6	28.4
OKS 2/90	31.0	4.6	31.7	9.7	23.0
OKS 2/63	21.9	4.2	36.0	6.9	31.0
OKS 2/36	20.8	3.6	36.2	7.6	31.8

Table 3. Chemical composition of burnt kaolin sands (BKS).

Type and fineness of BKS	Humidity (%)	Ignition loss (%)	SiO_2 (%)	CaO (%)	MgO (%)	Fe_2O_3 (%)	Al_2O_3 (%)	SO_3 (%)
BKS 1/90	0.13	1.00	79.51	3.32	0.31	1.05	14.49	0.19
BKS 1/63	0.26	1.02	72.00	4.03	0.70	1.60	20.15	0.24
BKS 1/36	0.28	1.38	67.69	4.50	1.01	1.37	23.48	0.29
BKS 2/90	0.21	1.61	67.29	2.49	1.02	2.24	24.94	0.20
BKS 2/63	0.28	1.67	59.84	3.34	0.26	4.93	29.40	0.28
BKS 2/36	0.18	1.55	57.56	3.35	0.38	3.27	33.43	0.28

compressive strength decrease. This fact is evidently observed from the values of 20 wt.% replacement of PC. No drops in compressive strength were found when the fractions 63 µm and 36 µm of BKS 1 and BKS 2 were used. It is therefore concluded that the above BKS fractions are suitable for using in blended systems similarly as natural zeolite [31, 32].

The present results show that the optimal fractions of BKS 1 and BKS 2 are those of 63 and 36 µm, and optimal replacement of PC by BKS is 20, 30 and 40 wt.%, respectively. This was the reason for which the above systems have been chosen for more detailed investigations. The calculated pure metakaolin content in PC-BKS blends is summarized in Table 5. It is seen that individual PC-BKS types contain metakaolin as a pozzolan between 4.10 and 14.48 wt.%.

Table 6 shows dependencies between the content of oxide components (SiO_2, Al_2O_3 and Fe_2O_3) of BKS and related compressive strength of cement-BKS specimens after 90- and 180-day curing in water. Negligible differences of compressive strength in these times were found. Cement-BKS specimens with 20-40 wt.% replacement of PC by BKS 1 and BKS 2 in the fraction of 63 µm have approximately equal compressive strength independently on the SiO_2 and Al_2O_3 amounts. Regarding fraction 36 µm, the compressive strengths slightly differ. Cement-BKS specimens with higher content of Al_2O_3 (BKS 2) have strengths lower of about 1.6-4.6 % than cement-BKS specimens with higher content of SiO_2 (BKS 1). These differences are of negligible importance. The fraction 36 µm of BKS gives in the cement-BKS specimens slightly increased strengths

Table 4. Basic characteristics of Portland cement CEM I 42.5 R (Turňa nad Bodvou).

Chemical composition (wt.%)	Properties
Humidity 1.42	Specific gravity .3 019 kg/m^3
Ignition loss 0.99	Surface area . . . . . .420 m^2/kg
Insoluble residue 1.63	Consistency . . . . .29.5 vol.%
SiO_2 . . . . .19.63	Initial set . . . . . . .3 h 30 min
CaO 63.42	Final set . . . . .4 h 40 min
MgO 0.27	Flexural/Compressive strength
Fe_2O_3 . . . . .3.65	1 day (MPa) 1.4/5.4
Al_2O_3 . . . . 7.50	3 days (MPa) 5.1/19.6
SO_3 . . . . .1.48	7 days (MPa) 7.9/32.9
. . . .	28 days (MPa) 10.2/51.6

Mineralogical composition (%) according to Bogue

C_3S	C_2S	C_3A	C_4AF	$C\bar{S}$
50.46	16.00	13.72	11.11	2.90

Table 5. Content of metakaolin in Portland cement (PC)-burnt kaolin sand (BKS) blends.

Type and fineness of BKS	Replacement of Portland cement (%)	Kaolinite content in OKS (%)	Metakaolin content in PC-BKS blends (%)
BKS 1/36	20	26.9	5.38
BKS 1/36	30	26.9	8.07
BKS 1/36	40	26.9	10.76
BKS 1/63	20	20.5	4.10
BKS 1/63	30	20.5	6.15
BKS 1/63	40	20.5	8.20
BKS 2/36	20	36.2	7.24
BKS 2/36	30	36.2	10.86
BKS 2/36	40	36.2	14.48
BKS 2/63	20	36.0	7.20
BKS 2/63	30	36.0	10.80
BKS 2/63	40	36.0	14.40

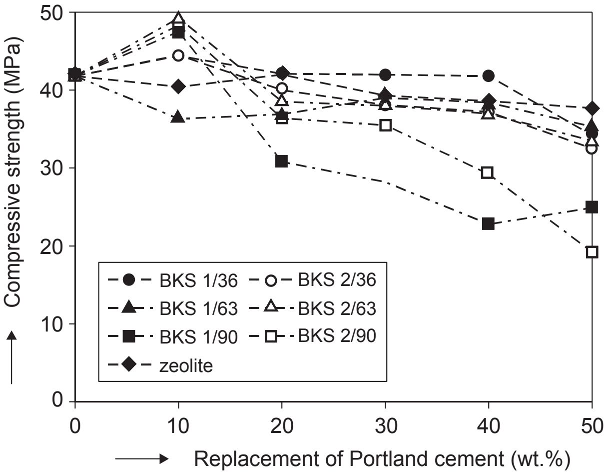

Figure 1. 90-day compressive strength of cement cube specimens with burnt kaolin sand (BKS) in dependence on replacement of Portland cement by BKS.

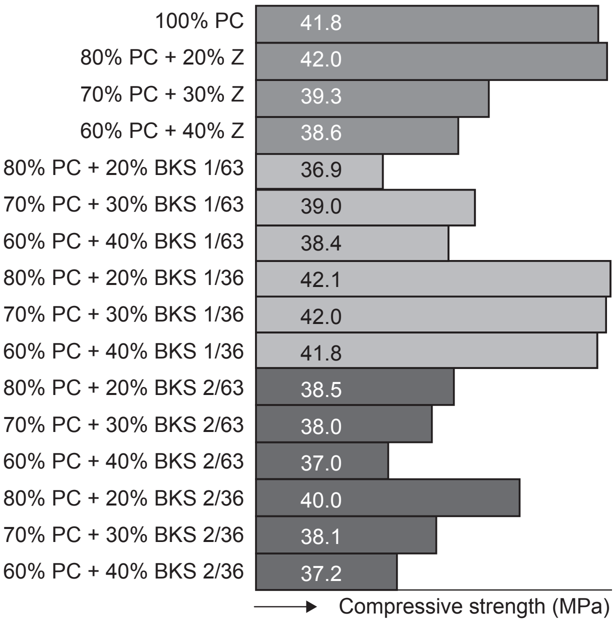

Figure 2. Compressive strength of cement cube specimens with burnt kaolin sand after 90- day curing in water.

than the fraction 63 μm. It was found that BKS 2 (both fractions) besides lower content of SiO_2 and higher content of Al_2O_3 have the higher Fe_2O_3 content related to that in BKS 1. However this fact does not influence the final compressive strength by definitive measure. Thus, compressive strength of cement-BKS specimens does not depend on the BKS type and related chemical composition. Next partial conclusion is that the compressive strength of cement-BKS specimens is either similar or slightly lower than those of reference cement pastes.

The comparison of 90-day compressive strength of cement-BKS specimens each other with that of cement-

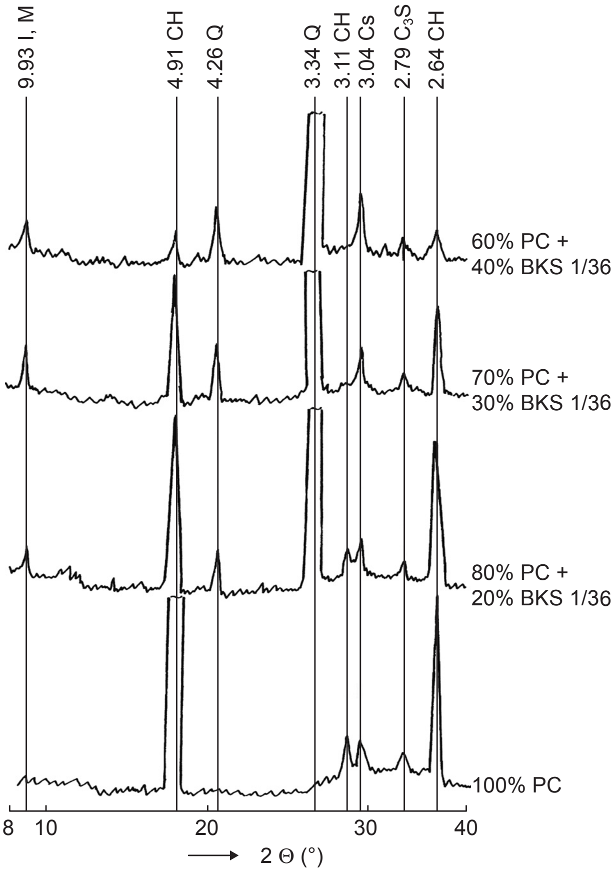

Figure 3. X-ray diffraction patterns of cement cube specimens with burnt kaolin sand - type 1 fineness 36 μm - after 180-day curing in water.

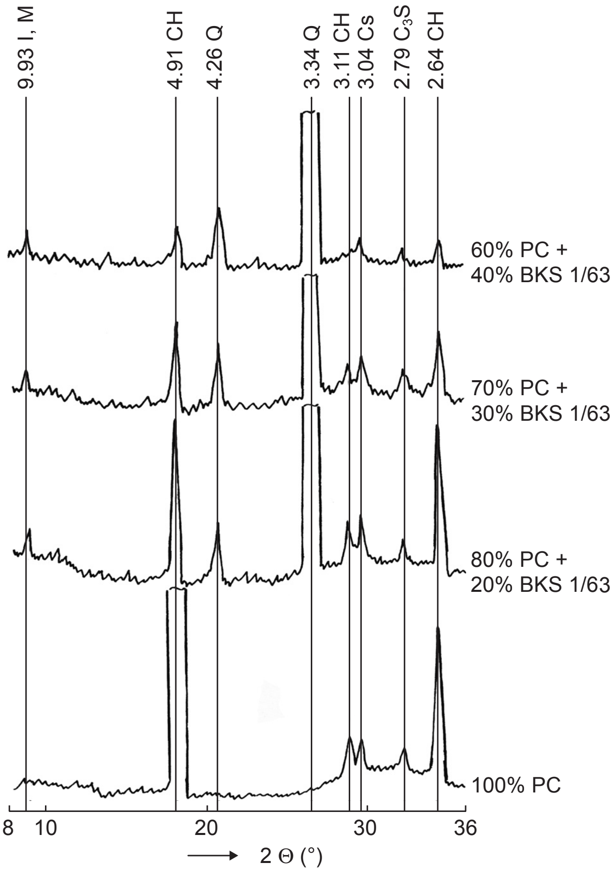

Figure 4. X-ray diffraction patterns of cement cube specimens with burnt kaolin sand - type 1, fineness 63 μm - after 180-day curing in water.

Table 6. Relation between chemical composition of burnt kaolin sands (BKS) and compressive strength of related cement cube specimens after 90- and 180-day curing in water.

Composition of specimen	Content of components (%)			Compressive strength (MPa)	
	SiO_2	Al_2O_3	Fe_2O_3	90 days	180 days
80% PC+20% BKS 1/63	72.00	20.15	1.60	36.9	37.4
70% PC+30% BKS 1/63	72.00	20.15	1.60	39.0	40.1
60% PC+40% BKS 1/63	72.00	20.15	1.60	38.4	40.4
80% PC+20% BKS 1/36	67.69	23.48	1.37	42.1	42.0
70% PC+30% BKS 1/36	67.69	23.48	1.37	42.0	42.1
60% PC+40% BKS 1/36	67.69	23.48	1.37	41.8	42.0
80% PC+20% BKS 2/63	59.84	29.40	4.93	38.5	38.9
70% PC+30% BKS 2/63	59.84	29.40	4.93	38.0	38.9
60% PC+40% BKS 2/63	59.84	29.40	4.93	37.0	38.6
80% PC+20% BKS 2/36	57.56	33.43	3.27	40.0	40.1
70% PC+30% BKS 2/36	57.56	33.43	3.27	38.1	40.5
60% PC+40% BKS 2/36	57.56	33.43	3.27	37.4	39.5

Control specimen - compressive strength: 41.8 MPa (90 days) and 42.3 MPa (180 days) after curing in water zeolite pastes and reference PC paste is illustrated in Figure 2. The above findings indicate that BKS is identically suitable for using in blends with PC than industrially-made and commonly used natural zeolite.

The X-ray diffraction patterns of cement-BKS specimens after 180-day curing in water are illustrated in Figures 3-6. The presence of portlandite $Ca(OH)_2$ (abbreviated as CH) is found in all specimens. The most important facts are significant decreases in intensity of diffraction lines of CH at 0.491 and 0.263 nm with increasing PC substitution by BKS 1 and 2. The higher the BKS (1 and 2) contents, the lower the CH content is found. Pozzolanic reaction of BKS 1 and 2 with hydrating PC is clearly confirmed. Quartz (Q) comes from original kaolin sand. Illite (I), muscovite (M) and calcite (Cc) are found as impurities. The X-ray analysis results show no differences in BKS type. The differences in chemical composition between BKS 1 and BKS 2 have not the influence on the CH reduction due to pozzolanic reaction.

Figure 7 and Figure 8 represent relative content of $Ca(OH)_2$ in cement-BKS specimens after 180-day water curing at significant diffraction lines 0.491 nm and 0.263 nm. Percentage values of diffraction line heights in cement-BKS specimens are related to the height of the same diffraction lines in the reference cement paste that is regarded as 100 %. This relative content of $Ca(OH)_2$ in individual cement-BKS specimens is a measure of relevant pozzolanic activity. The achieved compressive strengths of specimens are inserted in individual columns. The higher content of the BKS and related pure metakaolin contents, the higher the $Ca(OH)_2$ reductions are found. The relative content of $Ca(OH)_2$ indicated by diffraction line at 0.491 nm is between 12 and 18.5 % and that given by diffraction line at 0.263 nm is between 13.5 and 19.5 % at 40 wt.% substitution of PC by BKS. Lower substitution levels give larger data dispersion among individual measurements. This phenomenon was not studied in detail though the reason can lie in the fact that just the less intense pozzolanic effect can be connected with these larger differencies in relative $Ca(OH)_2$ content between 0.491 nm and 0.263 nm due to its preferentially variable consumption.

The above findings enable the last partial conclusion. BKS 1 and BKS 2 though markedly impured have

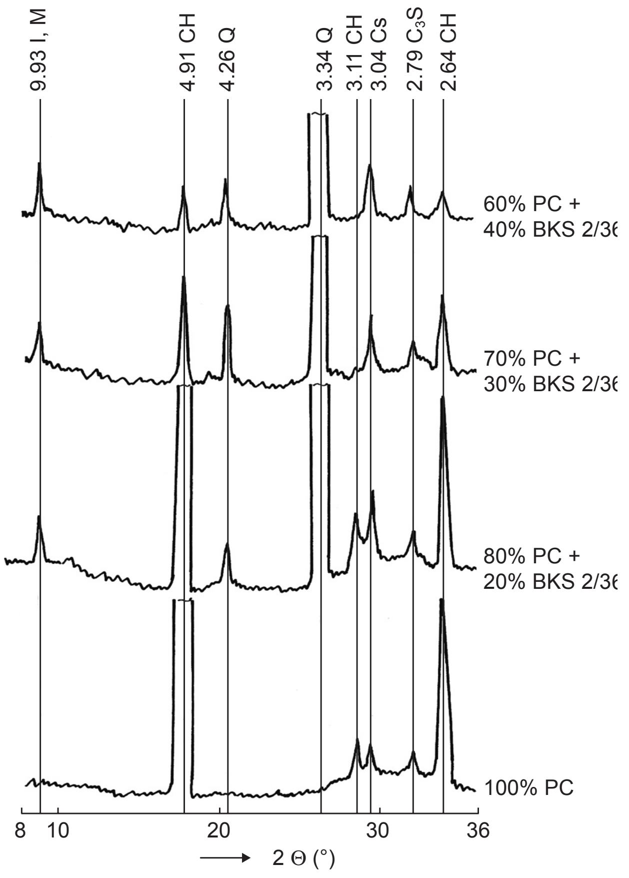

Figure 5. X-ray diffraction patterns of cement cube specimens with burnt kaolin sand - type 2, fineness 36 μm - after 180-day curing in water.

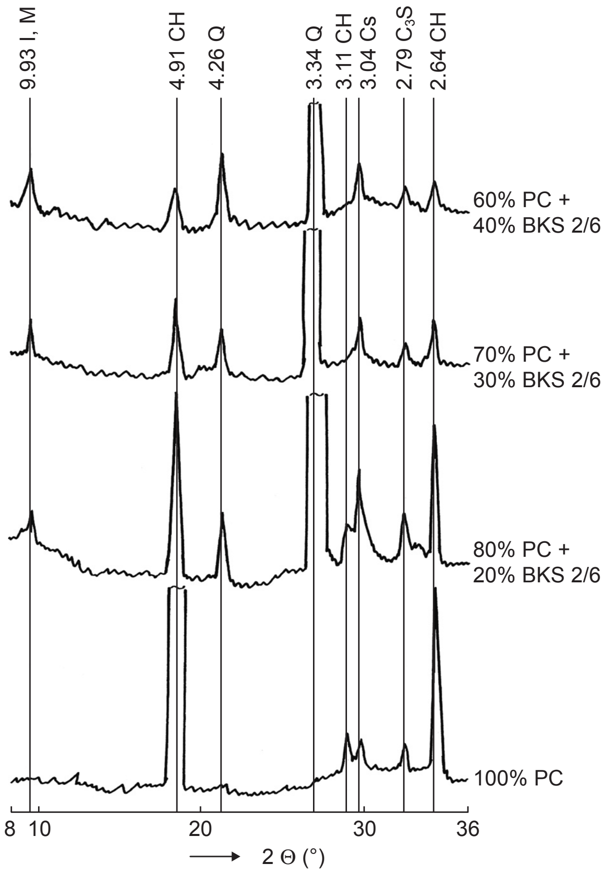

Figure 6. X-ray diffraction patterns of cement cube specimens with burnt kaolin sand - type 2, fineness 63 μm - after 180-day curing in water.

evident pozzolanic effect on hydrating Portland cement. The impurities such as quartz, and mainly burnt illlite and muscovite are intended to be calculated to the amounts of fine sand or aggregate in case of the mortar and concrete. Their influence on workability of mortars and concrete has to be verified in details. The first investigation performed in Slovakia has found BKS 1 and BKS 2 as promising pozzolanic additives to Portland cement systems.

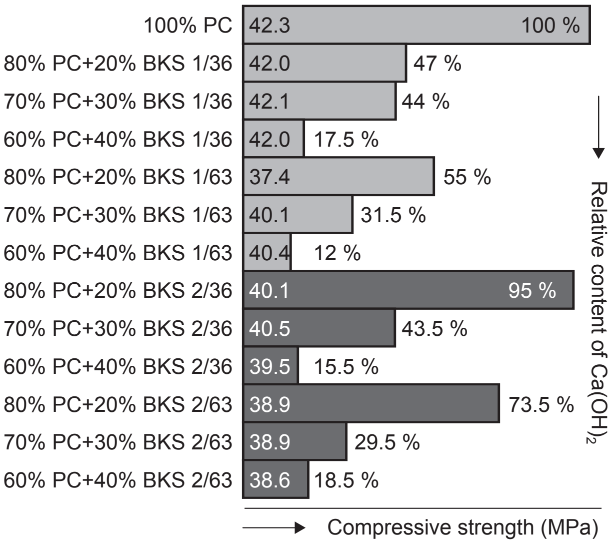

Figure 7. Relative content of $Ca(OH)_2$ in cement cube specimens with burnt kaolin sand after 180-day curing in water - at diffraction line 0.491 nm.

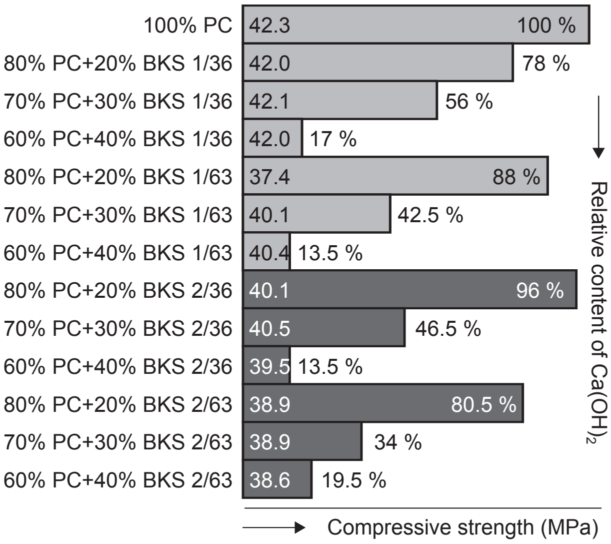

Figure 8. Relative content of $Ca(OH)_2$ in cement cube specimens with burnt kaolin sand after 180-day curing in water - at diffraction line 0.263 nm.

CONCLUSIONS

1. Original kaolin sand (OKS) is completely transformed to burnt kaolin sand (BKS) after heating at 650°C for 1 hour. Types of BKS differ in chemical composition; the BKS 1 has higher SiO_2 and less Al_2O_3 related to BKS 2. Differences in chemical composition have negligible effect on the compressive strengths of Portland cement (PC)-BKS specimens. Both BKS types are therefore changeable.
2. The optimal fractions of BKS 1 and BKS 2 for using in the PC-BKS blends are those of 63-36 μm and below 36 μm at PC replacement by BKS (1 and 2) by 20, 30 and 40 wt. %.
3. The BKS (1 and 2) fraction of 90-63 μm is not suitable for PC-BKS blends.
4. The higher the BKS (1 and 2) contents, the lower the Ca(OH)2 content in PC-BKS blend is found. The related pure metakaolin amounts vary between 4.10 and 14.48 wt. %. Pozzolanic reaction of BKS 1 and 2 with hydrating PC is clearly confirmed. BKS is identically sufficient for using in blends with PC as commonly used natural zeolite.
5. The present investigation indicates that, mainly due to metakaolin presence, BKS can be used as promising pozzolanic additive to Portland cement systems. Quartz and other impurities are necessary to calculate into amounts of fines in the mortars and concrete.

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VYPÁLENÝ KAOLÍNOVÝ PIESOK AKO PUZOLÁNOVÝ MATERIÁL PRE HYDRATÁCIU CEMENTU

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Kaolínový piesok z ložiska Vyšný Petrovec, Slovensko po tepelnej premene poskytuje vypálený kaolínový piesok s metakaolínom ako puzolánom. Pre overenie puzolánovej reakcie v zmesi s Portlandským cementom sa zahájil výskum orientovaný na zistenie vývoja pevností a tvorby mikroštruktúry zmesných systémov na báze Portlandského cementu a vypáleného kaolínového piesku. Tento výskum sa uskutočnil na základe spoločenskej objednávky - priemyselne využiť ložisko Vyšný Petrovec pri súčasnej kvalite materiálu stanovenej predchádzajúcim geologickým prieskumom. Pre tento cieľ sa použili dva typy kaolínového piesku, a to vo frakcii 63-36 µm a pod 36 µm. Frakcia 90-63 µm nie je vhodná pre použitie vo vzorkách cementu s vypáleným kaolínovým pieskom. Určila sa optimálna náhrada cementu vypáleným kaolínovým pieskom, ktorá predstavovala 20, 30 a 40 hmot. %. Zmes vypáleného kaolínového piesku s Portlandským cementom pritom obsahuje 4,10 -14,48 hmot. % čistého metakaolínu. Pevnosť v tlaku vzoriek Portlandského cementu s vypáleným kaolínovým pieskom je minimálne ovplyvnená rozdielnym chemickým zložením kaolínových pieskov. Obsah $Ca(OH)_2$ sa znižuje so zvýšeným obsahom vypáleného kaolínového piesku. V ňom prítomný metakaolín bol preukázaný ako aktívna puzolánová prímes pri hydratácii portlandského cementu.