NATURAL AND CALCINED CLAYEY DIATOMITE AS CEMENT REPLACEMENT MATERIALS: MICROSTRUCTURE AND PORE STRUCTURE STUDY

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Abstract

This manuscript introduces clayey diatomite from Jelšovec deposit in Slovakia intended for use in cement and concrete industry. Jelšovec deposit contains 3350 kilotons of clayey diatomite to be easily mined. Natural clayey diatomite (CD) and calcined clayey diatomite (CCD) were used to substitute 5 to 15 % by wt. of CEM I according to EN 197-1 Standard. CCD was prepared from CD by calcination at 900 °C for 1 hour. Frattini test was applied for the estimations of pozzolanic activity. Pozzolanic activity of CCD is found to be comparable to that of traditional natural and industrial pozzolans. The pozzolanic activity of the CD is only slightly lower opposite to that of CCD. Both clayey diatomites are behaving as high-efficient pozzolans when partially substituting the Portland cement. Cement composites of Water/Solid ratio of 0.5 with a substitution rate of CEM I between 5 and 15 wt. % CD and CCD were used for the tests. The specimens were studied by X-ray diffraction and thermal analysis as well as mercury intrusion porosimetry after 28- and 365-days test on compressive strength. The composites consumed a larger amount of $Ca(OH)_2$. They had a higher volume of gel-like hydrate phase and finer pore structure relative to the reference cement paste. The present findings indicate that Slovak diatomite from Jelšovec can be considered as a suitable pozzolan for use in cement-based materials. The results show that the microstructural properties of the respective materials are comparable to those of the traditional natural and industrial cement modifiers. Moreover, the substitution of CEM I by CD and CCD save energy and protect the environment by reducing the CO_2 emissions.

Keywords: *Cement, Diatomite, Pozzolanic Activity, Hydration, Pore Structure*

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1. INTRODUCTION

Siliceous and aluminium-bearing silicate rocks such as diatomite and expanded clays are quite important raw materials, and have currently found many applications by several industries (chemicals, construction materials, environment protection, etc.). Diatomite is a highly porous silica-rich material mainly composed of diatom shells. Even though the shells are porous themselves, the remarkably high porosity of diatomite is due to the open spaces present between the diatom shells composing the rock. The porous structure of diatomite makes it a poor conductor of heat and a good insulator.

The principal use of diatomite as insulator is as industrial fireproofing, at operating temperatures of about 750 °C in the semi-refractory range; the diatomaceous material bonded with suitable binders and combined with other ingredients such as bentonite clays can produce these materials, which can be manufactured in the shape of bricks, blocks, pipe coverings and others [1, 2, 3].

The effect of diatomaceous rocks from Hungary and Greece on the cement properties were studied [4]. Hungarian diatomite samples were extracted from active diatomite deposit in the Tokaj Mountains and that of Greek from pozzolanic tuff quarry located on the island of Milos. Laboratory tests and measurements showed that the diatomite samples exhibited good pozzolanic properties and could replace the currently used natural pozzolans. The highest compressive strength values obtained at later ages were obtained by use of the diatomaceous earth with the highest amorphous silica content. The thread-like shape of the diatom shells predominant in this sample could be one of the parameters contributing to the high strength of the cement produced with this material [4]. The water demand of all cements blended with diatomite from Samos Island and in the Elassona, Greece was higher than that of the Portland cement [5]. The late compressive strength of most diatomite cements was improved compared to that of the reference cement [5]. Standard cement test results indicate the water demand and the setting times of the pastes were related to diatomite ratio [6]. The physical, chemical, mineralogical, micro-structural and mechanical tests of the mortars, prepared by mixing Portland cement clinkers with 5 to 20 wt. % raw and calcined diatomite produced comparable strength values with respect to the reference Portland cement. The increased additions resulted in a decrease in the strength due to the higher water demand related to the porosity while Blaine values and reactive SiO_2 content were increased.

Calcination changed the porous structure of diatomite and rendered the grindability easier providing it to be used as a cement addition and suggested its use with substitution rate higher than 20 wt. % in this work [7].

Cement mortars and pastes with 10 to 35 wt. % substitution of cement with calcareous diatomaceous rocks of Zakynthos Island, Ionian Sea, Greece were examined [8]. Blended cements, having up to 10 wt. % diatomite content developed the same compressive strength as the Portland cement (PC), while the presence of diatomite leads to an increase of the paste water demand. The pozzolanic nature of the diatomite results in the formation of higher amounts of products, specifically at the age of 28 days [8].

Diatomite is characterized as natural pozzolan, as it satisfies the requirements of EN 197-1 [9] concerning the active silica content. The usability of diatomite, a natural amorphous and porous raw material, as an additional blend in an effort to improve the performances of natural pozzolan-blended cements was studied [10]. To this aim, varying proportions of pozzolan, diatomite and pozzolan-diatomite-blended cements were prepared and chemical, physical, mechanical, mineralogical and microstructural tests were applied to the resulting cements. The test results indicated that diatomite blend may be effective in improving pozzolanic properties, reducing setting time, reducing alkali content and increasing early-ultimate strengths of pozzolan-blended cements. Diatomite, used as an additive can eliminate excess water, improve water tightness and strength by promoting a more uniform distribution of voids in the mixture, and yields an improved finished surface. Diatomite can combine with free lime and the formed gel-like reaction products could improve the hardened qualities [10].

Diatomite was used in mortars at 5 to 15 wt. % substitution for cement, sand and water quantities were kept constant. The compressive and flexural strength decreased with increasing diatomite content. Calcined diatomite substitution generally increased the compressive strength of the cement mortar after 25 freezing and thawing cycles. Water absorption of the mortars decreased with the increase of diatomite content except the mortar containing of 15 wt. % diatomite. The expansion of the cement mortar prisms immersed in 5 wt. % sodium sulphate solution decreased with increasing diatomite content and generally the sulphate resistance of the mortars was higher than that of the reference mortar [11].

The concrete specimens containing 10 % diatomite substitution by weight for cement had the best compressive and flexural strength [12]. The substitution of cement with diatomite using a superplasticizing admixture could be utilized to improve the mechanical properties of the conventional concrete mixtures [12].

Diatomite aggregates in the production of lightweight concretes can be also satisfactorily used [13].

The investigation carried out in this study consists of the following:

a) the preparation of binary cement systems of PC + clayey diatomite (CD) and PC + calcined clayey diatomite (CCD) by mixing and intergrinding cement with the selected CD and CCD at different percentages,
b) the determination of the physical and chemical characteristics of the used cement, CD and CCD,
c) the preparation of cement composites using the prepared binary cement systems and testing the 28-day and one-year strength of the samples,
d) the investigation of the effects of CD and CCD on the hydration characteristics of binary cement composites,
e) the determination of pozzolanic activity of CD and CCD and its comparison with the other traditional pozzolans.

2. EXPERIMENTAL

2.1 Materials

The following materials were used in the study: ordinary Portland cement (PC) {Holcim, a.s. (Slovakia), Rohožník, in accordance with STN EN 197-1 [9]}, and clayey diatomite mined at Jelšovec deposit, Slovakia: one as received (CD) and the second calcined at 900 °C for 1 hour (CCD). Both diatomite samples were prepared with fineness below 0.063 mm. The properties of the used cement are summarized in Table 1. The chemical composition of PC (STN EN 196-2 [14]), CD and CCD (STN 72 0101[15]) are reported in Table 2 and 3. The mineralogical composition of CD and CCD is presented in Table 4. Quantitative XRD analysis of diatomite (CD and CCD) samples was performed applying the RockJock program [16].

2.2 Preparation of Specimens, Casting and Curing

The PC was substituted by 5, 10 and 15 wt. % of CD and CCD, respectively to prepare binary composites (PC + CD and PC + CCD). The binary cement composites were prepared with Water/Solids ratio of 0.5. The reference cement paste was prepared without CD and CCD.

The fresh specimens were compacted on a vibration table (40 Hz) for 30 seconds prior to casting. They were cast into 20 × 20 × 20 mm steel moulds. The moulds were stored in the atmosphere with ~ 95 % relative humidity (R.H.) at (20 ± 1) °C for the first 24 hours and after demoulding the specimens were cured underwater at (20 ± 1) °C until the testing.

2.3 Testing Methods

The SEM examination on the as received diatomite (CD) was performed on SEM instrument JEOL JXA-840A. Small pieces of the composites were appropriately prepared and observed in the mode of secondary electrons. SEM images of the samples are depicted in Figures 1 to 4.

Thermal studies were conducted simultaneously from 20 to 1000 °C on Thermogravimetric analyzer TGA-DSC1 with sensor TG-DTA, Metler Toledo, Zürich, Switzerland. Samples weighing 200 mg were heated in flowing air at a heating rate 20 °C/minute. Powder X-ray diffraction (XRD) patterns were recorded in the 16° to 41° of 2Θ range on Philips diffractometer coupled with an automatic data recording system, using CuK_α radiation and Ni-filter.

The pore structure was studied by mercury intrusion porosimetry (MIP) on Porosimeter Poremaster GT-60, Quantochrome, Florida, USA.

The pozzolanicity of the CD and CCD was determined by soaking fine samples passing 0.063 mm (63 μm) in a saturated lime solution at (40 ± 1) °C for 1, 7 and 28 days. The CaO concentration of the solution was analyzed at the specified test time according to STN EN 196-2 [14]. The amount of lime fixed (mM/litre) was calculated as the difference between the CaO concentration in the original saturated lime solution and the CaO content remaining in the solution. The pozzolanic reactivity obtained for CD and CCD was compared to the values of other pozzolans such as commercial metakaolin, silica fume, fly ash (Spanish materials) zeolite and metakaolin sand from Vyšný Petrovec deposit (Slovakia) [17].

Table -1: The characteristics of Portland cement CEM I 42.5 R

Chemical composition (wt. %)		Properties	
Humidity Ignition loss Insoluble residue CaO SiO_2 Al_2O_3 Fe_2O_3 MgO SO_3	0.02 3.56 0.16 61.64 19.70 5.00 2.70 3.01 2.65	Specific gravity Surface area Consistency Initial set Final set	3 104 kg/m^3 452 m^2/kg 34.2 vol. % 2 h 40 min 4 h 35 min
		Flexural/Compressive strength (MPa)	
		1 day 3 days 14 days 28 days	4.5/16.7 6.6/35.6 7.0/46.3 9.0/52.9
Mineralogical composition according to Bogue (%)			
C_3S	C_2S	C_3A	C_4AF
56.18	8.42	8.68	8.22

Table -2: The chemical composition and physical properties of clayey diatomite (CD) and its calcined form (CCD)

Chemical composition (wt. %)	Sample	
	CD	CCD
Humidity	4.65	1.14
Ignition loss	8.90	2.39
Insoluble residue + SiO_2	56.68	63.70
Al_2O_3	16.93	19.83
Fe_2O_3	9.93	9.33
CaO	0.84	1.13
MgO	0.68	0.86
SO_3	0.06	0.11
Physical properties	CD	CCD
Specific gravity (kg/m^3)	2576	2738
Surface area (m^2/kg)	1936	1796

Table -3: The mineralogical composition of clayey diatomite (CD) and calcined clayey diatomite (CCD) identified by RockJock program

Mineralogical composition (%)		Sample	
		CD	CCD
Clay minerals	Ka	15	1
	Ill + Mu	7	10
	Bi + Chl	1	4
	Sm	23	-
Amorphous matter (diatoms)		41	69
Non clay minerals	Q	5	7
	F	1	1
	Px + Am	-	2
	Ilm	-	1
	An + Rt	-	1
	He + Gt + Fer	7	4

Abbreviations:
Ill-illite; Mu-muscovite; Q-quartz; F-feldspar; Ka-kaolinite; Bi-biotite; Chl-chlorite; Sm-smectite; Px-pyroxene; Am-amphibole; Ilm-ilmenite; An-anatase; Rt-rutile; He-hematite; Gt-goethite; Fer-ferrihydrate (details on the minerals appear at www.webmineral.com)

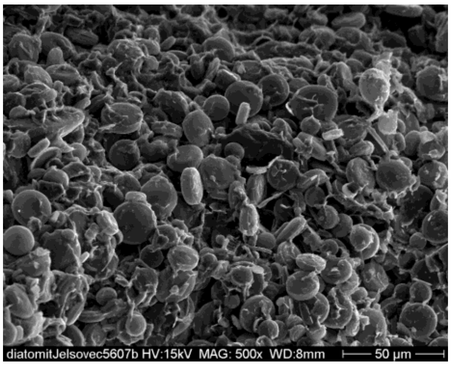

Fig -1: SEM image of clayey diatomite (CD)

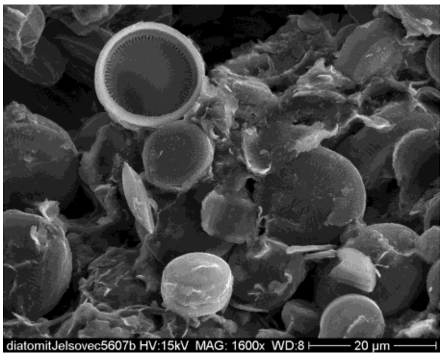

Fig -2: Couple of clayey diatomite shells (CD)

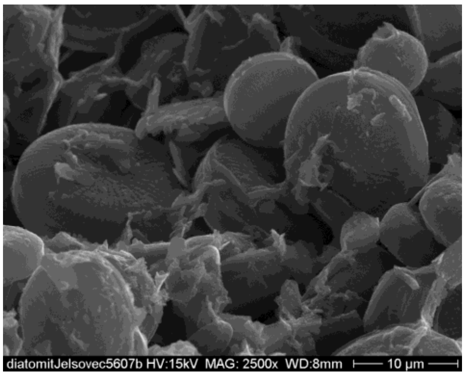

Fig -3: Cluster of clayey diatomite particles (CD)

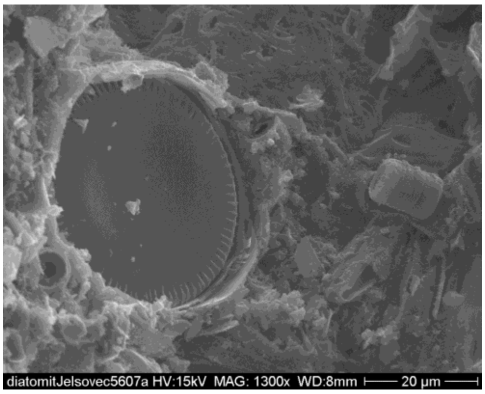

Fig -4: Detailed view on diatomite shell (CD)

3. RESULTS AND DISCUSSION

The 28-day and one-year strengths of the cement pastes (20 × 20 × 20 mm) are presented in Table 4. The one-year values are seen to increase compared to the reference, except in the 5CD system. Similar tendency is observed for 28-day strength. Higher substitution of PC by 10 and 15 wt. % CD or CCD is associated with higher strength values. No significant differences in the one-year strength are found between PC + CD and PC + CCD systems.

Table -4: The compressive strength of binary composites with clayey diatomite (CD) and calcined clayey diatomite (CCD) after 28- and 365-day water cure at (20 ± 1) °C

Sample	Compressive strength (MPa)	
	28 days	365 days
PC	39.3	51.5
5CD	34.5	50.2
10CD	39.0	53.3
15CD	43.8	56.8
5CCD	46.6	54.5
10CCD	47.1	56.1
15CCD	47.9	57.8

The diffraction line at 4.93 Å (0.493 nm) and 2.63 Å (0.263 nm) clearly prove the presence of $Ca(OH)_2$ (CH) in the cement composites (Figures 5 and 6); that at 3.34 Å (0.334 nm) belongs to quartz SiO_2 from the used diatomite. The small peak at 3.03 Å (0.303 nm) is associated to the carbonation product $CaCO_3$ accomplished with the cement hydration. The presence of non reacted cement clinker minerals C_3S and C_2S is proved by diffraction line at 2.78 Å (0.278 nm). The (5, 10 and 15 wt. %) CD (Figure 5) and CCD (Figure 6) cement composites differ from the reference specimen by higher CH consumptions. The content of $Ca(OH)_2$ gradually decreases with percentage substitution of PC by CD or CCD. The higher $Ca(OH)_2$ consumption is detected in pastes with higher diatomite (CD and CCD) contents. The comparison of CH diffraction line intensities at 4.93 Å (0.493 nm) and 2.63 Å (0.263 nm) shows a higher CH consumption by CCD compared to that of CD at 15 wt. % of diatomite.

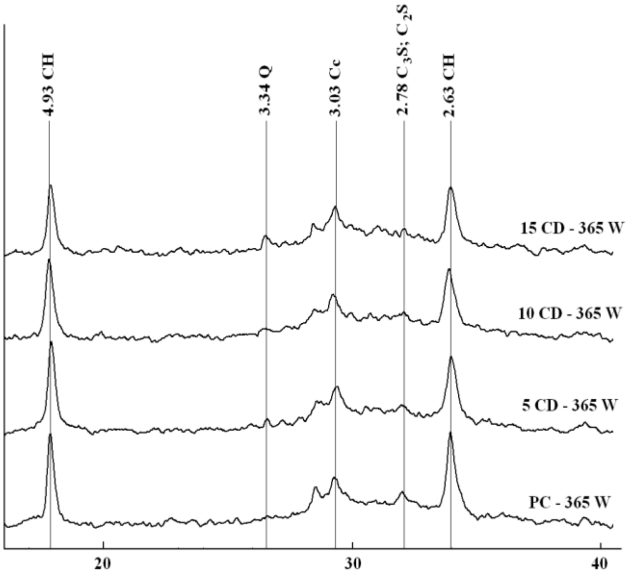

Fig -5: X-ray diffraction patterns of cement binary composites with clayey diatomite (CD) after 365-day curing in water [CH – portlandite $Ca(OH)_2$; Q – quartz SiO_2; Cc – calcite $CaCO_3$]; C_3S, C_2S – still non reacted clinker minerals of the cement

The greater consumption of $Ca(OH)_2$ in the composites with CD and CCD relative to that in the reference PC paste is also distinguished by TG - DTG analysis. (Figures 7 and 8). The results of TG analysis show trends towards releasing bound water from the hydration products and $Ca(OH)_2$ (Table 5): the composites made with CD and CCD contain slightly higher percentage portion of gel-like hydration products towards markedly lower content of $Ca(OH)_2$ crystals relative to the reference PC paste. The content of calcium hydroxide in CD and CCD - bearing cement composites gradually decreases with the percentage of PC substitutions. Total ignition loss of the composites with CD and CCD remain unchanged relative to that of PC paste. This finding is mainly contributed to the reduced content of PC in binary cement composite. DTG plots related to CH dehydroxylation at around 454 - 464 °C indicate more intense CH consumption in the CD and CCD - bearing cement composites compared to the PC paste.

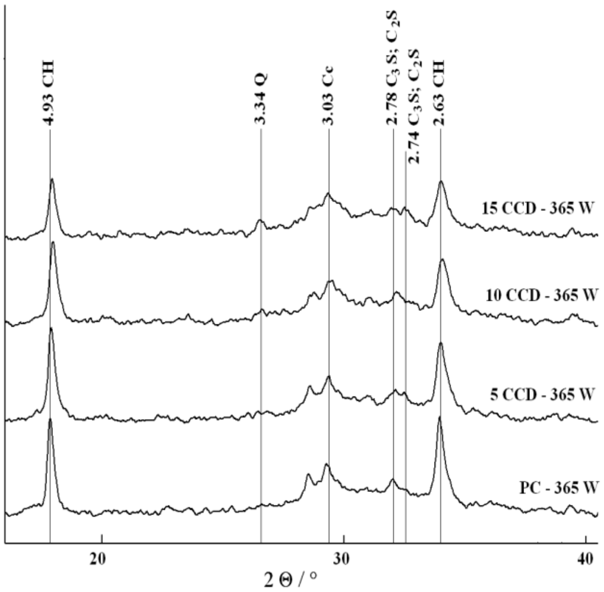

Fig -6: X-ray diffraction patterns of cement binary composites with calcined clayey diatomite (CCD) after 365-day curing in water [CH – portlandite $Ca(OH)_2$; Q – quartz SiO_2; Cc – calcite $CaCO_3$]

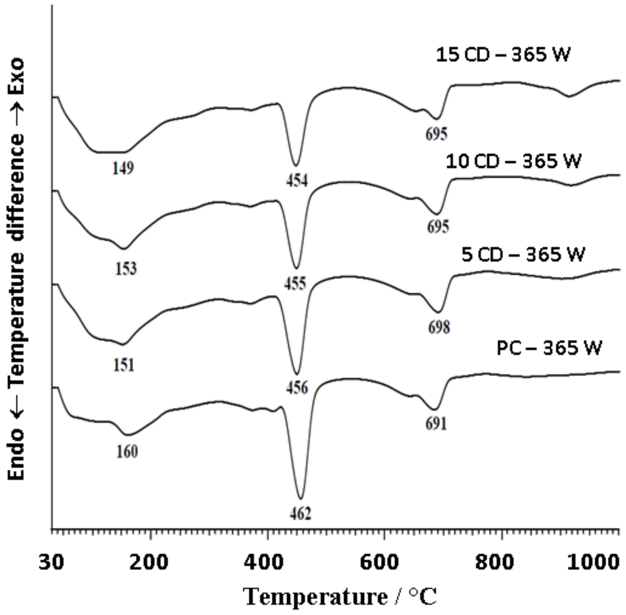

Fig -7: DTG curves of cement binary composites with clayey diatomite (CD) after 365-day curing in water [(149 - 160) °C: C-S-H and C_2ASH_8 (gehlenite); (454 - 462) °C: CH (portlandite); (691 - 698) °C: Cc (fine grained calcite)]

The pore structure parameters of cement composites stored one year in water at (20 ± 1) °C are listed in Table 6.

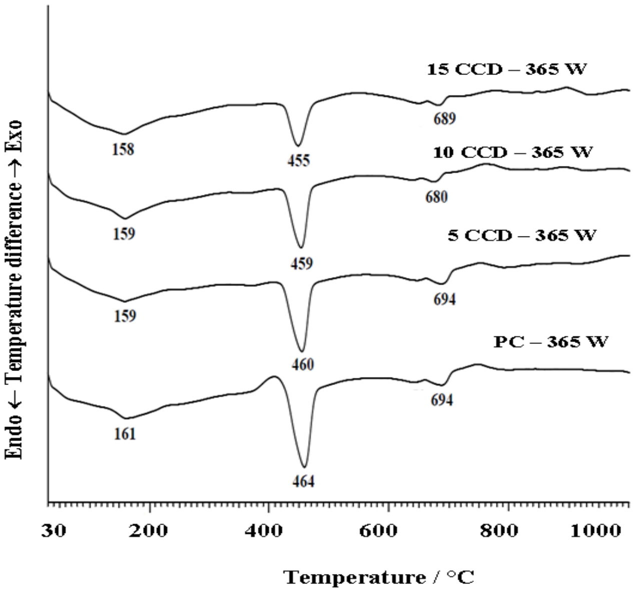

Fig -8: DTG curves of cement binary composites with calcined clayey diatomite (CCD) after 365-day curing in water [(158 - 161) °C: C-S-H and C_2ASH_8 (gehlenite); (455-464) °C: CH (portlandite); (680 - 694) °C: Cc (fine grained calcite)]

Table -5: TG analysis evaluation after one-year water cure

Sample	Surface bound water (%)	Water bound in C-S-H, C-A-H (%)	$Ca(OH)_2$ (%)	$CaCO_3$ (%)	Total ignition loss (%)
PC	2.0	10.3	20.0	6.4	21.3
5CD	2.1	11.1	15.4	7.5	22.1
10CD	2.3	10.8	13.7	7.2	20.8
15CD	2.3	10.7	12.5	7.4	21.0
5CCD	2.0	10.7	15.8	6.3	21.1
10CCD	2.2	10.8	13.6	5.3	20.3
15CCD	2.1	10.9	10.5	5.1	20.0

Table -6: One-year pore structure parameters of tested cement composites stored in (20 ± 1) °C water

Sample	SSA (m^2/g)	V_{TP} (cc/g)	M_{TP} (nm)	M_{MP} (nm)	TP (%)	K (m/s)
PC	35.98	0.177	21.2	17.4	24.3	0.3×10^{-11}
5CD	40.89	0.175	18.8	16.8	26.9	0.3×10^{-11}
10CD	48.72	0.193	16.1	14.1	26.1	0.2×10^{-11}
15CD	52.56	0.186	14.8	13.1	27.9	0.2×10^{-11}
5CCD	30.64	0.163	21.1	19.4	22.8	0.3×10^{-11}
10CCD	35.70	0.174	19.6	17.5	23.8	0.3×10^{-11}
15CCD	91.78	0.218	10.8	8.7	29.0	0.1×10^{-11}

The abbreviations related to the pore structure parameters given in Table 6 are listed as follows: SSA - specific surface area of the pores; V_{TP} - volume of total open pores (1.82 nm - 0.534 mm); M_{TP} - total pore median radius (1.82 nm - 0.534 mm); M_{MP} - micropore median radius (1.82 – 5250 nm); TP - total open porosity (1.82 nm - 0.534 mm); K - permeability coefficient (calculated from the measured pore structure data).

The composites with PC substitution of 5, 10 and 15 wt. % CD and CCD are characterized by a pore structure similar to that of the reference. Both structures are characterized by a homogenous dense skeleton of the hydration products with comparable strength values. The significant difference is found in the higher CH consumption in the cement composites made with CD and CCD, mainly at 15 wt. % substitution of PC by CD and CCD. This indicates a pozzolanic reactivity of CD and CCD occurring in the binary cement systems used.

The pore structure results of the cement composites reveal that the micropore (M_{MP}), total pore (M_{TP}) median radii decrease and the permeability coefficients (K) remain unchanged or decrease slightly for all CD and CCD - bearing cement composites compared to those of PC paste. The gradual increase in the very fine pores volume confirmed by SSA, V_{TP} and TP is caused by the incorporation CD and CCD into the cement composite. Higher levels of the pore structure refinement were found for the cement composites with 15 wt. % substitution of PC by CD and CCD. The effect of CCD on the pore structure refining is stronger than that of CD when comparing the pore structure parameters listed in Table 6. The pore structure results indicate that CD and CCD percentage substitution levels mainly between 10 and 15 % by weight mean effective additions to Portland cement causing in the final effect pore structure refinement. This refinement results in the increased compressive strength values of the CD and CCD - bearing cement composites compared to the reference (as proved in Table 4). Comparison of pozzolanic activities of various pozzolans is presented in Table 7.

Table -7: Pozzolanic activity of various pozzolans

Pozzolan	The fixed CaO (mM/litre)/1g pozolan in the supersaturated solution of $Ca(OH)_2$; percentage pozzolanic activity in every time is shown in the brackets		
	1 day	7 days	28 days
CD	13.73 (62)	19.22 (86)	20.66 (93)
CCD	16.49 (74)	20.97 (94)	21.03 (95)
Zeolite	19.24 (86)	21.23 (95)	21.66 (97)
MK	12.85 (58)	17.59 (79)	18.95 (85)
Fly ash	5.63 (25)	12.70 (57)	19.47 (87)
Blast furnace slag	7.54 (34)	10.42 (47)	12.80 (57)
Cupola foundry slag	5.31 (24)	7.63 (34)	7.89 (35)

Both diatomites: clayey raw diatomite in Jelšovec deposit (Slovakia) – CD and its calcined form – CCD are effective pozzolans fully comparable with those of zeolite, metakaolin and blast furnace slag tested previously on the pozzolanity by Frattini test.

The evident difference between CCD and CD is recognized in the 1-day pozzolanity values; those of CD are lower relative to those of CCD. The final values after 28 days are almost the same (CD = 93 % and CCD = 95 %).

Figure 8 shows that the higher pozzolanic reactivity has been observed for natural zeolite when compared to clayey diatomite (CD) and calcined clayey diatomite (CCD). The uptake of pozzolanic activity of CCD opposite to CD is observed at one-day pozzolanic activity only. Smaller and negligible, differences between the activities of CD and CCD were found with the advanced time (7 and 28 days).

Natural CD occurred in Jelšovec deposit and its calcined form CCD are both confirmed as high-efficient pozzolans, which are suitable for use with the cement, substituting the presently applied pozzolans of natural and industrial origin.

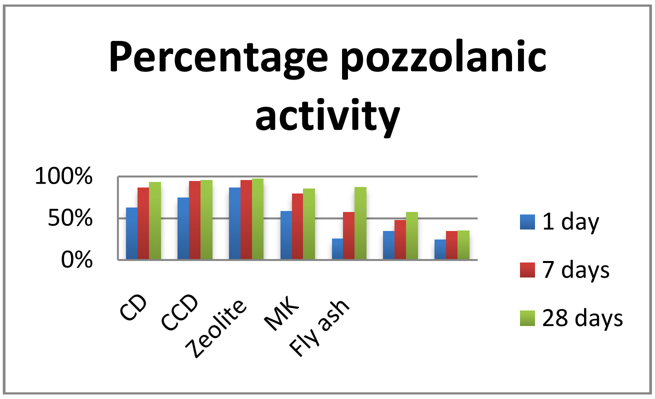

Fig -8: Percentage comparison of pozzolanic activities

4. CONCLUSIONS

1.) Frattini test results show that 28-day pozzolanic activity of natural clayey diatomite occurred in Jelšovec deposit (Slovakia) – CD and its calcined modification – CCD fired at 900 °C/1 hour are represented by 93 % and 95 % binding efficiency of CaO by the pozzolan. The pozzolanic reactivity is slightly increased relative to the other commonly used pozzolans: fly ash – 87 %, metakaolin sand (MK) – 85 %, markedly increased than that of blast furnace slag – 57 % and cupola foundry slag – 35 %.

2.) The calcination of CD is not necessarily needed because of the almost equivalent pozzolanic activity in 28-day values. This results in energy saving and subsequently in the possibly broader utilization of Jelšovec diatomite in cement and concrete practice.

3.) Diatomite is regarded as a suitable pozzolanic addition fully comparable by the pozzolanic properties with the presently applied conventional pozzolans, and also meets the requirements given on pozzolanic additions by EN 197-1 Standard.

4.) One-year compressive strength of CD and CCD - bearing cement composites substituted by 10 to 15 wt. % of CD and CCD is characterized by strength gain opposite to the reference PC paste.
5.) Strength gains are mainly caused by the higher $Ca(OH)_2$ consumption, the higher portion of gel-like hydration products and the refined pore structure of binary cement composites composed of PC + (10, 15) wt. % CD and PC + (10, 15) wt. % CCD opposite to the reference PC paste.
6.) Differences among strength, microstructure and pore structure parameters of PC + (5, 10, 15) wt. % CD and PC + (5, 10, 15) wt. % CCD binary cement composites are of negligible importance.
7.) This fundamental research of Slovak Jelšovec diatomite exhibits the full justification of consequent applied research made on the concretes according to the relevant STN and STN EN Standards.

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