HYDRATION AND LEACHING OF THE CEMENT – ZEOLITE COMPOSITE

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This study has examined the effect of zinc-saturated zeolite on hydration processes and its stabilization in cement composites. Measurements were made on cement pastes with defined water/solid ratios, W/S, different contents (10-50 %) of zinc-saturated zeolite and the constant addition of 0.3wt.% of plasticizer admixture. The hydration process was studied by determining the heat effects of reactions using differential microcalorimetry and by measuring specific electric conductivity. Stabilization after solidification was evaluated by leaching test and UV/Vis spectrophotometrical analysis of zinc in elutes. The results show that higher zeolite addition brings lower hydration heat and relative hydration degree. Changes in specific conductivity and appearance of maxima on conductograms indicate that when the content of saturated zeolite in cement pastes increases, specific conductivity decreases, and the recorded maxima appear at later hydration and solidification times. Zinc concentration in elutes after the leaching depends on the duration of solidification, quantity of saturated zeolite and duration of rinsing after cement sample solidification. Low zinc concentration in elutes indicates that zinc-saturated zeolites can be successfully stabilized in Portland cement composites.

INTRODUCTION

Solidification processes in Portland cement composite are based on cement hydration. It is a very complex process during which the basic components, clinker materials, such as C_3S, C_2S, C_3A and C_4A, together with gypsum, $CaSO_4\ 2H_2O$, as the regulator of binding, and water, form insoluble Ca-hydrate compounds. With time, this cement-water reaction system solidifies, develops, and forms the final structure of the hydrated cement gel, resulting in final properties of formed and hardened composite [1-3].

When saturated zeolite is introduced as substitute addition to cement in the cement-water reaction system, the system becomes even more complex. Hydration conditions change, interactions take place between the introduced zeolite and cement and various reaction products are formed. The products formed affect the dynamics and kinetics of cement hydration and solidification processes, changing both the composition and the properties of the cement gel. As the zeolite addition contains zinc as a component harmful to the environment, zinc is thereby introduced into the newly formed cement composite. Because of possible environmental hazards, such wastes containing heavy metals: Pb, Cd, Cr, Zn, Cu and others, must be stabilized. Stabilization must prevent uncontrolled releasing of bound harmful components into the environment even under conditions of long exposure to the action of possible agents (atmospheric conditions and other aqueous electrolytes). The quantities of harmful components released into the environment through the rinsing processes must not exceed the quantities allowed by standards and rules on taking care of harmful wastes. Studies so far indicate that such wastes can be successfully stabilized in processes of solidification in cement composites [4, 5, 6].

Stabilization by solidification represents the physical and chemical binding of the harmful component into the composition and structure of the cement composite. The success of stabilization is based on the above mentioned cement hydration processes, in which the cement binder physically and chemically transforms with time, passing from the state of paste to the state of hydrated cement gel with the properties of the solid state. The zeolite waste containing zinc, is distributed in the cement gel, included in its composition and structure and thus bound, immobilized and stabilized [4-9]. Stabilization of such wastes in the cement-water reaction system is possible after a certain hydration time also due to the additional effect caused by the pozzolanic reaction [1, 10, 11].

This study has examined the effect of such wastes, as substitute additions to cement, on the hydration and stabilization processes using various methods, i.e. differential microcalorimetry, conductometry, and UV/Vis spectrophotometrical analysis. The study had the additional goal to determine the conditions for preparing and methods for predicting the properties of newly formed composite materials.

EXPERIMENTAL

Materials

Industrial Portland cement, PC-45B is a commercial product of Dalmacijacement-RMC Group, Kaštel Sućurac, Croatia, and is classified according to EN-197 as CEM I. It is a highly alite cement with increased early strengths. Table 1 shows its average chemical composition and properties.

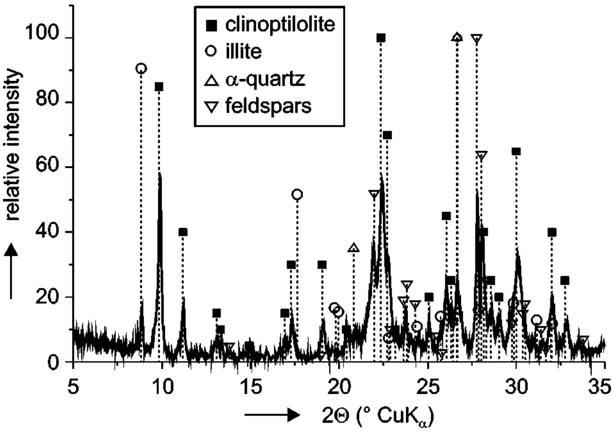

Figure 1. The XRD patterns of natural zeolite samples - deposit of Donje Jesenje, Croatia.

Saturated zeolite is the natural zeolite from the deposit of Donje Jesenje, Croatia, saturated by Zn^{2+} - ions. The zeolite applied contains 40-50 wt.% the clinoptilolite and has the ion exchange capacity CEC = = 1.12 mmol/g determined according to the Khjeldal method. The impurities include illite, montmorillonite, feldspars, calcite and quartz (figure 1). The chemical compositions of the zeolite samples tested was (wt.%): SiO_2 - 64.93; Al_2O_3 - 13.66; Fe_2O_3 - 2.03; Na_2O - 3.66; K_2O - 1.88; MgO - 1.10; CaO - 2.99; loss of ignition - 9.84 [12]. The zeolite particle size at saturation ranged from 0.04-0.10 mm. After saturation by Zn^{2+} - ions the zeolite was dried at 105°C, ground and sieved through the standard 4900 mesh/cm^2 sieve, so that its particle had the same or similar size as the Portland cement used.

Polymeric admixture-plasticizer based on lignosulfonates was added to the reaction system to reduce the water sorption observed in substitute additions to cement, such as zeolites and other pozzolanically active substances, in preparation of cement composite pastes of specific consistency. The admixture was added in the content 0.3 % to the mass of the solid reaction mixture of cement and saturated zeolite.

Water used in this study as the reactant in the processes of hydration and solidification of cement composites was distilled water of standard properties.

Sample preparation for examination and measurements

Cement pastes for microcalorimetrical measurements have been prepared by mixing cement and saturated zeolite. The part of saturated zeolite in test samples was 10%, 20%, 30%, and 50% relative to the mass of the solid mixture (cement + saturated zeolite). The total mass of the solid sample in each microcalorimetrical measurement was constant, 4 g. The plasticizer was

Table 1. Physical, chemical and mechanical properties of the PC-45B cement.

Component	Component content (%)	Physical property	Value
SiO_2	20.85	Specific surface according to Blaine (cm^2/g)	3300
Al_2O_3	4.81	Standard consistency (%)	26
Fe_2O_3	2.79	Setting time - start (min)	85
CaO	65.23	Setting time - end (min)	150
MgO	1.61	Average bending strength (MPa)	
Na_2O	0.18	after 3 days	6.26
K_2O	0.75	after 28 days	8.44
		Average compressive strength (MPa)	
		after 3 days	33.5
Total	99.31	after 28 days	50.7

added to the reaction system with the water for hydration. The W/S ratio was constant, 0.5. Hydration and solidification of prepared cement pastes took place in a differential microcalorimeter at 20°C for 48 hours.

Cement pastes for conductometrical measurements were prepared in the same way as the pastes for microcalorimetrical measurements, except that the W/S ratio corresponded to pastes of normal consistency, and the total mass of the solid sample was 150 g for each measurement.

The specific electrical conductivity of prepared cement pastes was determined by conductometric measuremet at 20°C.

Cement pastes for leaching and UV/Vis spectrophotometrical analysis were prepared in the similar way as those for microcalorimetrical measurements. The content of the zeolite waste in the samples was 15% or 30% relative to the mass of the solid. The mass of the solid was also constant, 150 g. The *W*/*C* ratio of the prepared cement pastes was 0.5. The hydration and solidification process took place on pastes formed in the shape of cylinders of constant height, h = 45 mm, and diameter, d = 34 mm. The pastes hydrated in air for the first 24 hours, were then immersed into the thermostat at the temperature of 20 C (isolated contact of cement paste and water) and hydrated for the defined solidification time: 1, 3, or 7 days. Cement pastes, thus solidified, were rinsed with 400 cm^3 of 0.5 mol/dm^3 acetic acid, HAc, according to USEPA TCLP test [13]. Rinsing took place in the automatic shaker for the defined period of 1 to 24 hours. After rinsing, the concentration of zinc in elutes was determined by means of UV/Vis spectrophotometry.

Equipment and methods of examination

Microcalorimetrical measurements were carried out by means of a differential microcalorimeter of the conduction-isoperibolic type [14]. To record heat effects of hydration processes in the microcalorimeter, the Data Logger, ALMEMO 2290-8, was used, measuring the change in voltage $\mathrm{d}U = \mathrm{f}\ (t)$. The data obtained were analyzed by a computer program on a PC against the given properties of the measuring system. In this way the time dependences of hydration heat Q (J/g), relative hydration degree, α (%), and hydration rate $\mathrm{d}Q/\mathrm{d}t$ (J/gh) were obtained. Measurements were carried out at 20°C, and the duration of hydration and solidification was 48 hours.

Conductometrical measurements were carried out at 20°C by means of a microprocessor conductometer ISKRA MA 5964. The conductometric cell electrode, made out of stainless steel, and showing the constant value of CC = 0.5673 cm^{-1}, was used to determine the specific conductivity of cement pastes which were used to trace the electrolytic properties of the prepared cement systems.

UV/Vis spectrophotometrical analysis was carried out with the Lambda EZ201 UV/Vis spectrophotometer PERKIN ELMER, when determining Zn^{2+} concentration in elutes after rinsing and leaching of solidified cement composites.

RESULTS AND DISCUSSION

Figures 2-4 summarize the results of determination of effects of saturated zeolite as a substitute addition on the hydration processes, obtained by microcalorimetrical measurements and analyzed by a computer program. The results for the cement paste sample without the addition of saturated zeolite shown in figure 2 indicate that the thermovoltaic curve $\mathrm{d}U = \mathrm{f}\ (t)$ shows two maxima. The first occurs immediately after the contact of the cement binder with water, and the second one after the specific hydration duration. After the second maximum, the thermovoltaic values decrease. The hydration heat curve, recorded as the function $Q = \mathrm{f}\ (t)$, indicates that heat increases continuously during hydration and solidification. The hydration degree curve, shown as the function $\alpha = \mathrm{f}\ (t)$, shows great similarity with the hydration heat curve.

The kinetic curve $\mathrm{d}Q/\mathrm{d}t = \mathrm{f}\ (t)$, as the hydration rate curve, is very similar to the thermovoltaic curve $\mathrm{d}U = \mathrm{f}\ (t)$. It also shows changes with time in the form of maxima and minima. Appearance of a maximum, its shape, size and position on the time axis indicate individual hydration interval that take place during hydration. Each interval is characterized by hydration time in which one of possible controlling processes becomes dominant and governs the hydration and solidification kinetics.

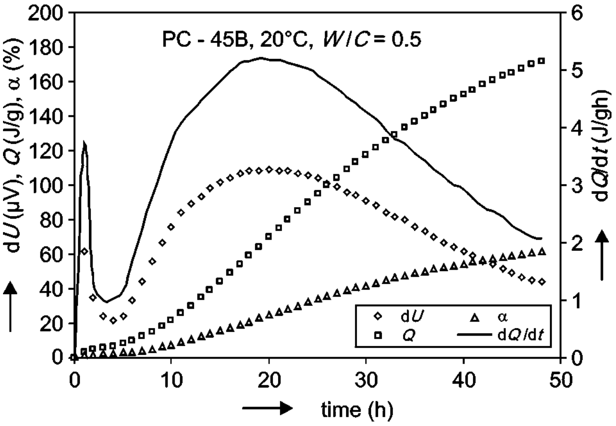

Figure 2. Thermovoltaic value $\mathrm{d}U$ (μV), hydration heat Q (J/g) hydration degree α (%), and hydration rate $\mathrm{d}Q/\mathrm{d}t$ (J/gh) of the cement paste PC-45B vs. hydration and solidification time.

Figure 3 presents the hydration heat during solidification and the hydration degree in cement pastes with or without saturated zeolites. As can be seen, the increase in the content of saturated zeolite brings the increase in the final hydration heat, whereas the hydration degree after 48 hours of hydration decreases relative to the sample without addition. The basic shapes of the recorded curves Q = f (t) and α = f (t), relative to time and content of the zeolite addition in the cement pastes, are the same as or very similar to the curves for the sample without addition. This indicates that the saturated zeolite as the addition to the reaction system does not affect the basic processes and does not change the hydration reaction mechanisms of solidification. The cement pastes without the zeolite addition show the highest values for both hydration heat (Q =171.7 J/g) and hydration degree (α = 59.3 %) after 48 hours of hydration. Cement composites with the maximum content of 50 % of saturated zeolite show the lowest values for hydration heat (Q = 104.7 J/g) and hydration degree (α = 37.4 %) for 48 hours of hydration.

The dynamics of heat release with time indicates that the heat release values until about 15 hours of hydration are somewhat higher in samples containing saturated zeolite as addition than in samples without addition. After 15 hours of hydration, with increasing content of saturated zeolite, the heat released gradually decreases in comparison with the heat released in the sample without addition and at 48 hours of hydration shows a much lower value. The results obtained are the expected ones, as the content of cement being the active component releasing heat during hydration decreases. and the content of the saturated zeolite in the composite increases.

The dynamics of change of the hydration degree with time, especially in the early stadium of hydration, depends on the content of saturated zeolite in the reaction system and the actual water - cement ratio. The W/C - ratio increases with the decrease in the content of cement in the composite, which leads to the increase in the relative degree of cement hydration. Besides diluting the system on cement, the W/C ratio is affected by the properties of saturated zeolite as addition, which has the increased water sorption affinity owing to its characteristic zeolite structure. This property of increased water sorption is found in all natural pozzolanas and similar substances such as zeolites. This is one of the reasons why plasticizer-type admixture is introduced into such composite systems. It is due to this phenomenon and the decreased content of cement in the composites with higher zeolite contents that in the early hydration stage up to 15 hours the values of the relative hydration degree are higher and found above the curve for the cement paste without additions, similar to examinations of hydration heat. The results obtained are due to the shift in equilibrium to the product side in the given hydration and solidification conditions [5,6,7].

The analysis of results for the hydration rate, dQ/dt = f (t), shown by kinetic curves in figure 4 for cement pastes with varying contents of saturated zeolite indicates that the kinetic curves are very similar to that for the hydration rate for the sample without zeolite addition. At early stadium of the reaction process, immediately after the contact with water, short-lasting maxima are recorded. The magnitude of these maxima depends on the content of saturated zeolite in the sample. When the content increases from 0-50 %, their values increase from 3.7 to 5.7 J/gh. When the first maxi-

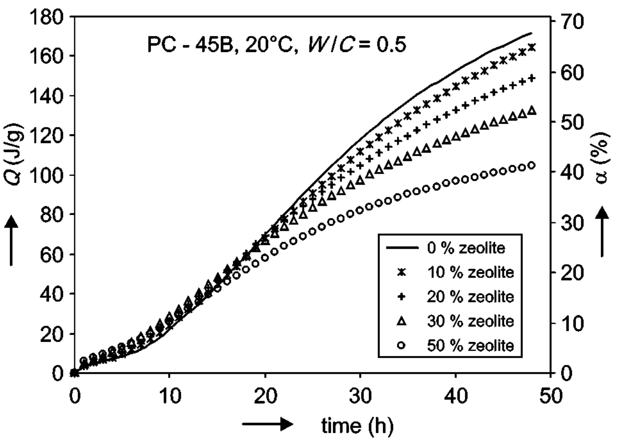

Figure 3. Hydration heat Q (J/g) and hydration degree α (%) vs. hydration time and varying contents of saturated zeolite in Portland cement pastes.

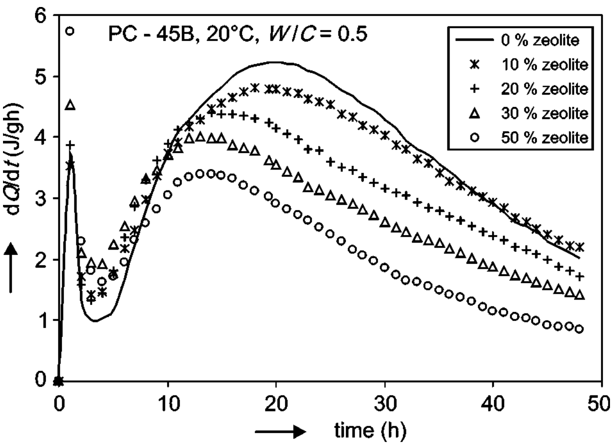

Figure 4. Hydration rate, dQ/dt (J/gh) vs. hydration time and varying contents of saturated zeolite in Portland cement pastes.

mum is finished, the heat release rate abruptly changes and declines, and minimum is formed on kinetic curves. This initial time period, lasting to the end of minimum on the kinetic curves, represents the induction period. After the induction period, the kinetic curve again shows the increase in the hydration rate. On the $dQ/dt = f(t)$ curve, the second maximum appears eventually. The value of this maximum and its position on the temporal axis is in direct correlation to the amount of zeolite added to the reaction system.

When the saturated zeolite content in cement pastes increases, the magnitude of this maximum decreases from 5.2 to 3.4 J/gh, and the position is shifted to shorter time. The recorded maximum for cement pastes without addition appears at about 20 hours, and for cement pastes with 50% of zeolite at about 13 hours.

During the hydration of silicate components C_3S and C_2S and alkali, the reaction system shows a high *pH* value and the content of $Ca(OH)_2$. Under such conditions the zinc present in the system is exposed to possible ion exchange and sedimentation processes accompanied by formation of surface layers in the form of $CaZn_2(OH)_6\ 2H_2O$. The formed zincate is sorbed on the cement particle surface and decreases the cement hydration rate. The retarding effect is especially observed in hydration of C_3S, which is the main component of the cement examined [8, 15, 16].

The earlier occurrence of maxima on the kinetic curve is due to dilution of the system by the zeolite addition. This hydration period with the maxima described represents the accelerated hydration period. Hydration reactions take place; reaction products as well as the structure and microstructure of cement binder are formed. The reaction system binds and starts to harden. Further progress of hydration after the period of accelerated hydration shows a continuous decrease in the hydration rate and represents the retarded hydration period. Processes and reactions started earlier continue in this period. The reaction system keeps hardening, and hydration and solidification kinetics is governed by diffusion as the slowest process [1-3,11,17-19].

The results shown in figure 5 indicate the linearity of the function $y = f(t_{Qmax})$, which can be expressed by the equation $y = -7.12x + 138.43$. The independent variable x represents the time at which the maximum hydration rate occurs. This form of dependence makes possible to predict the necessary content of saturated zeolite, for which the maximum hydration rate will be realized in a given hydration time. This linearity also indicates the fact that the saturated zeolite did not affect the basic processes as well as the controlling mechanism of the hydration.

Figure 6 presents the results of conductometrical measurements determining specific conductivity of cement pastes whose consistency corresponds to the normal one. At the initial period the curves show the abrupt initial increase of measured specific conductivity. Then the specific conductivity decreases and a minimum occurs on the curve. This initial very early hydration period is characterized by the initial reactions of dissolving and hydrolysis, in which the aqueous phase of the cement paste gradually fills with ionic species. After that the dissolution processes and transfer of ionic species into the solution slow down. This corresponds with characteristic minimum on the curve. After that, there is a marked increase in specific conductivity that lasts for some time, eventually reaching the maximum [20]. The shape of the $\kappa = f(t)$ curves, the position of occurrence of main maximum, and measured values for specific conductivity depend on the content of saturated

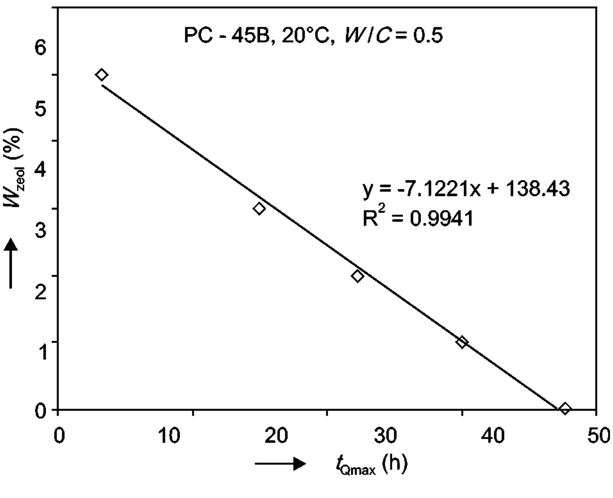

Figure 5. Content of saturated zeolite in cement pastes vs. time of occurrence of the maximum hydration rate.

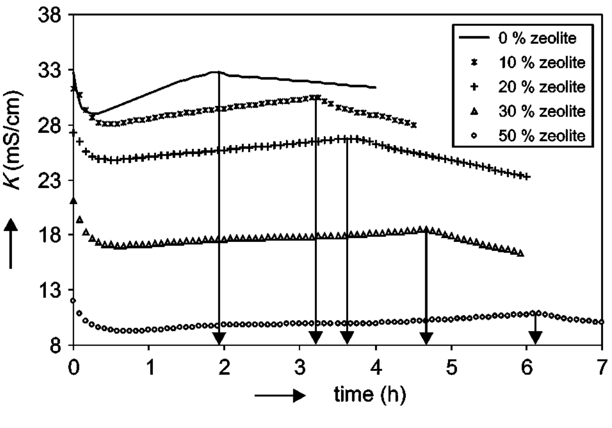

Figure 6. Specific conductivity, $\kappa = f(t)$, vs. contents of saturated zeolite in cement pastes of normal consistency.

zeolite in cement pastes. When the content of saturated zeolite increases, the position of maximum shifts towards later times and the values of specific conductivity become lower.

Figure 7 shows the dependence of the position of the conductivity maximum on the content of saturated zeolite. This dependence is linear and can be expressed by the equation W_{zeolite} (%) = 11.96 X - 24.96, where X represents the time at which the maximum conductivity of cement paste occurs on the temporal axis. This linearity indicates that the addition of saturated zeolite does not affect the hydration and solidification process mechanisms, but affect the kinetic parameters.

Figures 8 and 9 present the results of spectrophotometrical analysis of elutes after the leaching test. The results shown in the figures indicate that zinc concentration in elutes decreases with the solidification time. After one day of solidification and 24 hours of elution, the sample with 15 % of saturated zeolite has the zinc concentration of 2.181 mg/dm^3, and the sample with 30 % has the concentration of 3.137 mg/dm^3. After 3 days' solidification the 15 % sample has the zinc concentration of 1.742 mg/dm^3, and the 30% sample has the concentration of 2.816 mg/dm^3. Samples after 7 days' solidification and 4 hours' elution show even lower values. Zinc concentration in elutes of the sample with 15% of saturated zeolite was 1.371 mg/dm^3, for the sample with 30 % it was 2.682 mg/dm^3. The values obtained for all solidification times are lower than the limit values allowed by regulations of harmful waste management. The reason is the stabilization and binding of zinc in the structure of the cement composite, partially aided by a possible pozzolanic reaction of the part of silicate and alumosilicate component from the zeolite structure composition, and especially from amorphous part of the zeolite rock. This additional binding of the zeolite addition by the pozzolanic reaction is very probable, especially when hydration and solidification last for a longer period (3 days and more), thus realizing the conditions for this reaction taking place. The reaction system shows a high pH value and a high concentration of OH-ions, which may lead to de-polymerization of silicate and alumosilicate groups from the zeolite basis. This might lead to formation of hydrate phases C-S-H and AFm, which thereby substitute a part of $Ca(OH)_2$. These more stable hydrate phases are included in the already formed structure and composition of the cement gel, whereby the hydrated and solidified product becomes even denser and more stable with time, at the same time immobilizing zinc added through zeolite as waste [5-8,10,11, 21-23]. In this way the possibility of migration of the harmful zinc admixture from the zeolite waste to the surface of the cement composite is reduced and made more difficult, decreasing the possibility of its eluting from the surface of the composite. The results obtained agree with the results of studies of applicability of harmful waste stabilization processes by solidification in cement composites [1-8].

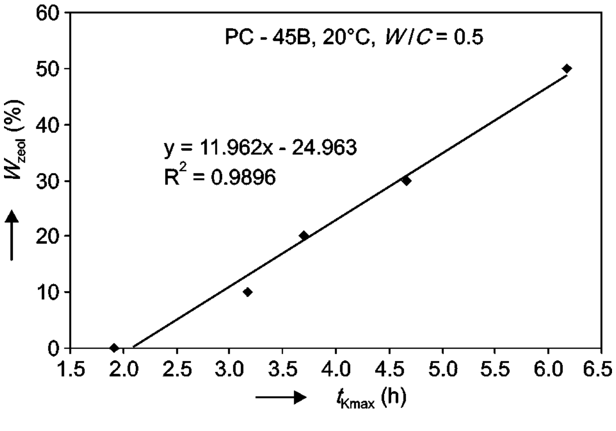

Figure 7. Content of saturated zeolite vs. time of occurrence of the maximum conductivity in the curve, κ = f (t), for cement pastes of normal consistency.

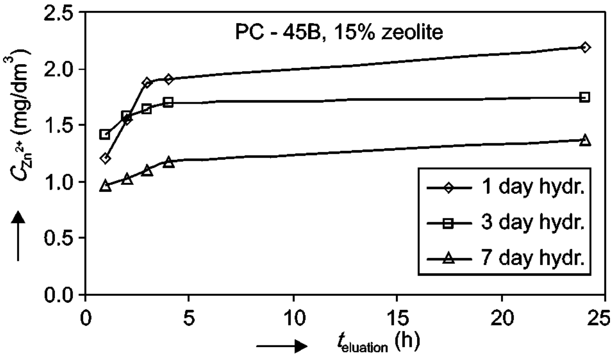

Figure 8. Zinc concentration in elutes of cement composites with 15% of saturated zeolite vs. solidification time and time of eluting with 0.5 M HAc.

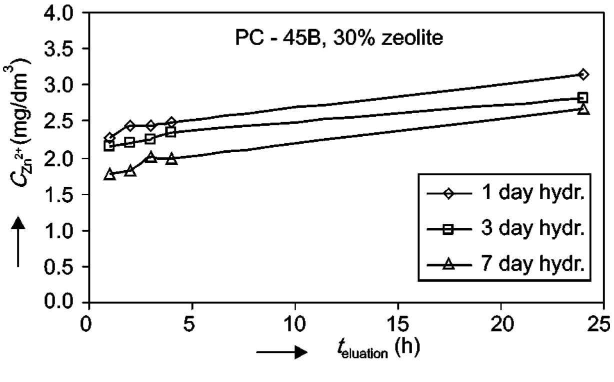

Figure 9. Zinc concentration in elutes of cement composites with 30 % of saturated zeolite vs. solidification time and time of eluting with 0.5 M HAc.

CONCLUSION

The examinations carried out indicate that the content of saturated zeolite containing Zn^{2+} ions affects the hydration and solidification processes, but does not affect the mechanism of hydration. Saturated zeolite modifies the cement hydration dynamics.

The action of saturated zeolite and interactions in the reaction system result in changes in intensity of effects caused and their occurrence on the temporal axis representing the duration of hydration and solidification. When the content of saturated zeolite in the reaction system increases, the hydration heat and the hydration degree have lower values. The dependence between the occurrence of the maximum hydration rate and the content of the zeolite on time is linear. When the saturated zeolite content in cement pastes increases, maximum hydration rates occur earlier. The higher is the content of zeolite containing harmful zinc admixture, the lower is the maximum magnitude.

Specific electrical conductivity is lower when the content of saturated zeolite in the cement paste increases, and maximum on the $\kappa = f(t)$ curve occurs later. The linear dependence of the occurrence of the specific conductivity maximum on the content of saturated zeolite also confirms that the zeolite waste does not affect the reaction mechanisms of hydration and solidification.

Leaching tests on cement composites with saturated zeolites show lower values for zinc concentration with longer times of hydration and solidification. Low zinc concentrations in elutes confirm that hydration and solidification of cement composites can be successfully used to manage wastes hazardous to the environment.

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HYDRATACE A LOUŽENÍ KOMPOZITU CEMENT – ZEOLIT

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Tato studie zkoumala účinek zeolitu se sorbovaným zinkem na hydratační proces a jeho stabilizaci v cementových kompozitech. Měření byla prováděna na cementových pastách s definovaným poměrem voda/cement, s obsahem zeolitu 10-50 % a konstantním přídavkem 0,3% plastifikátoru. Hydratační proces byl studován stanovením tepelného účinku reakcí diferenční mikrokalorimetrií a měřením specifické elektrické vodivosti. Stabilizace zeolitu po ztuhnutí cementové matrice byla hodnocena loužícími testy a UV/VIS spektrometrickou analýzou zinku ve výluzích. Výsledky ukazují, že rostoucí přídavek zeolitu snižuje hydratační teplo a relativní stupeň hydratace. Změny specifické elektrické vodivosti a existence maxim v konduktogramech dokládají, že rostoucí přídavek zeolitu snižuje specifickou vodivost a poloha maxim se na konduktometrických křivkách posouvá k delším hydratačním časům. Nízká koncentrace zinku ve výluzích ukazuje, že zeolity se sorbovaným zinkem mohou být úspěšně stabilizovány v kompozitech s portlandských cementem.