University of Belgrade
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# PROCEEDINGS

## XXIII International Conference
## Ecological Truth

Editors
Radoje V. Pantovic
Zoran S. Marković

## EcoIst '15

Hotel "PUTNIK", Kopaonik, SERBIA
17-20 June 2015

UNIVERSITY OF BELGRADE
TECHNICAL FACULTY BOR

**XXIII International Conference**

**"ECOLOGICAL TRUTH"**

# *Eco-Ist'15*

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**Radoje V. PANTOVIC**
**and**
**Zoran S. MARKOVIC**

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# INVESTIGATION OF THE APPLICATION OF THE NATURAL AND WITH LEAD IONS CONTAMINATED ZEOLITE AS AN ADDITION IN PORTLAND CEMENT

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## ABSTRACT

In presented paper results of the removal of lead ions from water solutions by the natural zeolite (clinoptilllite) and application of such contaminated zeolite materials as a supplement for standard additions portland cement are showed. In this investigations, the natural zeolite (clinoptilollite) with particle size 100% -43 µm was used. The experiments of lead ions adsorption showed that the maximum adsorption capacity of the natural zeolite was 66 mg $Pb^{2+}$/g. After adsorption experiments the saturated zeolite with lead ions was used as a supplement to portland cement. Investigations of the mechanical properties of the cement with the addition, natural zeolite and zeolite saturated with lead ions showed that this material can be used as a supplement for portland cement up to 30%.

**Key words:** Natural zeolite, Pb zeolite, Portland cement, Flexural strengths, Compressive strength.

## INTRODUCTION

Natural zeolites are crystalline hydrated aluminosilicate minerals of alkaline and alkaline earth cations, which are very widespread in nature. However, only clinoptilolite, mordenite, filipsite, chabazide, erionite, ferrierite and analcite are in sufficient amount spread in the nature so their exploitation cost effective [1]. Due to the unique properties that they have: high selective and high cation exchange capacity, high degree of crystallinity, uniform channels of molecular dimensions, a high degree of hydration and easy dehydration, low density, high absorption capacity for ions of heavy metals (lead, copper, zinc, etc. ) natural zeolites found wide application:

- catalysts in many reactions,
- such as molecular sieves and cation exchangers [2]

- Adsorption of impurities in the waste water
- adsorbents for radionuclides - 137Cs [3]
- ammonia adsorbents [4,5]
- adsorbents of heavy metals ions - Pb, Cu, Zn [6]
- material for recultivation of degraded lands (ash thermal power plants, flotation tailings)
- material in pharmacy and veterinary medicine
- feed additive in veterinary medicine for the prevention of digestive problems in young animals [7].

In order to improve properties of natural zeolite (clinoptilolite), various methods of modification of the starting minerals were applied [8]. Such modified zeolites can be used as:

- adsorbent of mycotoxins that are found in animal food such as: aflatoxins, zearalenone, ochratoxin A, ergot alkaloids [2]
- tool for removing impurities from the water [9 - 11]; weakly polar organic molecules;inorganic anions - sulfate, phosphate, chromate;
- decontaminant land-radionuclide-uranyl ion [12; 13] cesium etc.

From an environmental point of view, it is possible to use zeolites in certain ecosystems:

1 Air: adsorbent moisture, gases and unpleasant odors in the stables (NH3, CO2)
2 Water: adsorbent cations, anions, heavy metals, low polar organic molecules
3 Land: adsorbent radionuclides; tool for re-cultivation of land
4 Feed: adsorbent of mycotoxins (zearalenone, aflatoxin B1, B2 and G2; vomitoxin, ochratoxin, T-2, DAS -diacetoksiscipirenol), feed additive for the prevention of diarrhea and regulating the pH.

However, storing and keeping of once used zeolite is still not fully resolved, and very low number of studies are engaged in solving this problem. The aim of this paper is to investigate the potential use of natural zeolite (clinoptilolite) contaminated with lead ions in the cement industry as the replacement of standard additions in portland cement.

## EXPERIMENTAL PART

**Material.** In this investigations, natural zeolite originating from Vranjska Banja, Serbia and Portland cement PC 42.5R from Beočin, Serbia were used. Chemical compositions of starting raw materials are given in table 1.

**Metods.** Adsorption isotherm for lead ions removal by the natural zeolite was obtained acording following procedure: 1 g of the natural zeolite was mixed with 50 ml of aqueous solution contaminated with different concentrations of lead ions (350-7200 mg/l). The suspensions were mixed 24 h, at a stirring speed of 350 $min^{-1}$. The initial pH was not adjusted and only was only measured and was in range from 2.8 for the maximum lead ions concentration (7200 mg / l), to 4.3 for the lowest concentration of lead ions (350 mg /l). After reaction time, suspensions were centrifuged for 10 min at 10 000 $min^{-1}$. Then, using the atomic absorption spectrophotometry (AAS) and the instrument "Aanalysis 300" starting and non absorbed concentrations of lead ions were determined. The amounts of adsorbed ions of lead were determined from the difference between the initial and the concentration of ions in the filtrate.

To obtain with lead ions contaminated zeolite which will be used as an addition to Portland cement, concentration of lead ions equal to the maximum adsorption capacity obtained by adsorption isotherm was used . The procedure for obtaining contaminated zeolites was:

Natural zeolite was milled to reach the grain size 100% of -43 μm, and afterwards mixed with $PbNO_3$ under following procedure: 400 g of $Pb(NO_3)_2$ was dissolved in 120 l of distilled water. Then, 3.5 kg of natural zeolite was added in lead ions contaminated water, so the concentration of lead ions in the solution was approximately equal to the maximum adsorption capacity obtained by adsorption isotherm (66 mg/g). Reaction time was 7 days with occasional mixing. After 7 days dewatering, filtration, drying and disintegration of sample was done. Finally, dry zeolite contaminated with lead ions (PbZ) was obtained. Determined adsorbed amount of lead ions was ~60 mg$Pb^{2+}$/g which is very close to the maximum adsorption capacity of natural zeolite obtained from adsorption isotherm (66 mg$Pb^{2+}$/g).

Afterwards, mixing of Portland cement with zeolite: 10% (PC10Z), 30% (PC30Z), 50% (PC50Z), and 10% of Pb-zeolite (PC10PbZ); 30% of Pb-zeolite (PC30PbZ) and 50% of Pb-zeolite (PC50PbZ) in laboratory mill without balls was performed in duration of 10 minutes. On such obtained samples of cement with addition of zeolite and on starting sample (cement PC42.5R) standard physico-mechanical testing have been performed according to European Normative EN197:

- Determination of the specific surface according to Blaine (EN 196-6) (Blaine apparatus was used).
- Determination of standard consistency (EN196-3) (Vicat apparatus was used).
- Determination of the setting time of the cement dough (EN 196-3) (Vicat apparatus was used with the rod replaced by a needle)
- Determination of the compressive strength (EN 196-1).

Thusly, the following cements with additions are obtained:

PC42.5R
PC+10%-PZ = PC10Z
PC+30%-PZ = PC30Z
PC+50%-PZ = PC50Z
PC+10%PbZ = PC10PbZ
PC+30%PbZ = PC30PbZ
PC+50%PbZ = PC50PbZ

All stated cements had mass of 4000 g.

## RESULTS AND DISCUSSION

### Results of adsorption of lead ions from water by using natural zeolite

To determine the maximum adsorption capacity of natural zeolite for lead ions, at first adsorption isotherm was determined. This part of experiments was important, in order to define the conditions for obtaining the contaminated zeolite which will be used as an addition to portland cement.

Results of adsorption of lead ions on the natural zeolite, with particle size -43 µm are given in Figure 1. Figure 1a showed the adsorption isotherm, which is obtained as a dependence of the amount of adsorbed lead ions in mg/g vs. equilibrium concentration of pollutant in the solution after adsorption on zeolite in mg/l. Figure 1b showed dependence of the adsorption percent of lead ions vs. its initial concentration. From Figure 1a, it can be seen that, under applied experimental conditions, with increasing initial concentration of pollutants increases the concentration of adsorbed ions of lead.

The results showed in Figure 1a are fitted using the Langmuir and the Freundlich adsorption model. Mathematical forms of this two isotherms are given in equations 1 and 2.

Langmuir isotherm: $$c_{ads} = \frac{abc_e}{1+bc_e} \quad (1)$$

Freundlich isotherm: $$c_{ads} = bc_e^{\beta}, \quad (2)$$

where $c_{ads}$ is the concentration of the adsorbed lead ions (mg/g), $c_e$ is the equilibrium concentration of lead ions in the filtrate (mg / l), *a is* the maximum adsorbed lead ions concentration of adsorbed per gram of adsorbent, *b* is the adsorption equilibrium constant of the surface (l /mg), and the β is factor heterogeneity and determined affinity of the adsorbent according to adsorbate.

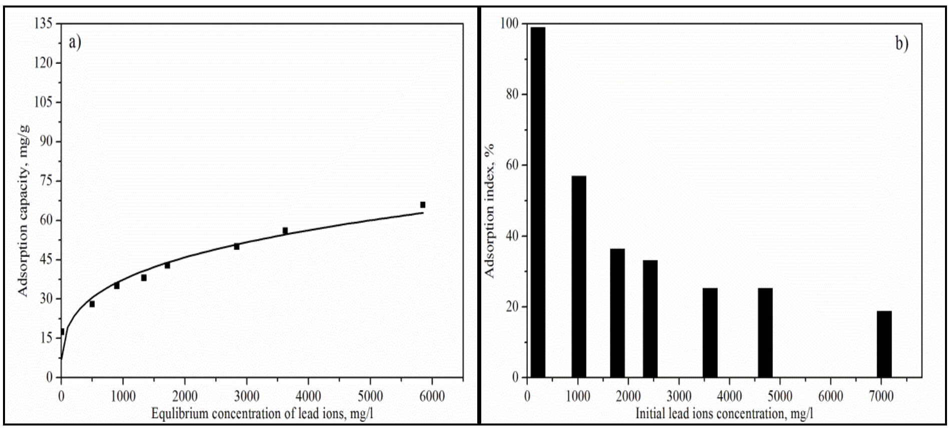


**Figure 1.** a) Adsorption isotherms of lead ions on natural zeolite; b) The dependency index sorption of lead in the function of the starting concentration of lead ions in solution

For results showed in Figure 1 the best fit of experimental data was obtained by the Freundlich adsorption model ($r^2$> 0.93) indicating a complex mechanism of binding ions lead to the natural zeolite. The maximum amount of adsorbed lead ions was 66 mg/g.

Also, we measured the pH of the suspension after the reaction time. For all initial concentrations of lead ions equilibrium pH were higher than initial, but, both, initial and equilibrium pH, decreased with increasing initial concentration of lead ions. Measuring of the equilibrium pH showed that final pH, for all initial concentrations of lead ions was lower than pH 6.0 what according to [14] mining that lead was in cationic, and dominantly $Pb^{2+}$ form, i.e. precipitation of lead in form of hydroxide did not occur. These investigations showed that the natural zeolite (clinoptilolite) from the Vranjska banja, Serbia has a good adsorption capacity for binding lead ions from water and aqueous solutions. However, because storage of waste material (zeolite contaminated with lead ions) is still not solved, we did the second part of experiments, and investigated opportunity of using that material as an additive in Portland cement production instead of the existing standard additions.

### Results of the application of the natural zeolite contaminated with lead ions as an additive in portland cement

The influence of zeolite and saturated zeolite on Portland cement quality was monitored by determining of the chemical composition, physical and mechanical characteristics.

The results of the chemical analysis of all portland cement mixes are given in the Table 1, while in the Table 2 are presented the results of physical properties of PC42.5R and PC with addition of zeolite and contaminated in various mass ratios.

As can be seen (Table 1) the content of PbO in the PbZ was 2.22. When such zeolite is added into PC in mass ratio 10 %, the 0.29 PbO is obtained (PC10PbZ). By increasing of the PbZ content in the PC, the content of PbO also increased. Thus, with 50 % content of Pb zeolite and 50 % PC 42.5R, there was 1.08 % of PbO in the PC50PbZ sample. In our previous paper [15] it was found that lead ions are connected via strong forces with natural zeolite and there is no desorption of its ions. Because of that, the Pb leaching test has not been performed in this investigation.

**Table 1.** Chemical composition of zeolites and portland cements

| Oxide, % | $SiO_2$ | $Al_2O_3$ | CaO | MgO | $Fe_2O_3$ | $K_2O$ | $Na_2O$ | $TiO_2$ | $SO_3$ | MnO | PbO | LoI |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Zeolite samples | | | | | | | | | | | | |
| PbZ | 65.12 | 13.50 | 3.47 | 0.39 | 2.25 | 1.15 | 1.07 | - | - | - | 2.22 | 10.83 |
| Cement samples | | | | | | | | | | | | |
| PC 42.5R | 24.02 | 6.03 | 59.60 | 2.03 | 1.48 | 0.76 | 0.34 | 0.346 | 3.4 | 0.087 | 0.0065 | 1.89 |
| PC10Z | 29.40 | 6.46 | 48.87 | 1.84 | 2.25 | 0.68 | 0.32 | 0.17 | 3.13 | 0.080 | 0.0043 | 6.75 |
| PC30Z | 33.11 | 7.89 | 43.73 | 1.64 | 2.23 | 0.81 | 0.45 | 0.17 | 2.70 | 0.066 | 0.0038 | 7.20 |
| PC50Z | 40.60 | 9.34 | 34.41 | 1.40 | 2.23 | 0.89 | 0.36 | 0.17 | 2.28 | 0.053 | 0.0033 | 7.92 |
| PC10PbZ | 28.10 | 6.71 | 53.87 | 2.00 | 1.58 | 0.82 | 0.55 | 0.646 | 2.80 | 0.084 | 0.29 | 2.75 |
| PC30PbZ | 34.83 | 8.02 | 43.66 | 1.91 | 1.57 | 1.04 | 0.71 | 0.346 | 2.68 | 0.071 | 0.72 | 4.44 |
| PC50PbZ | 40.75 | 9.54 | 34.98 | 1.80 | 1.53 | 1.01 | 0.88 | 0.329 | 1.88 | 0.054 | 1.08 | 6.16 |

Since the initial zeolite sample had grain size -43 µm, when this sample is mixed with Portland cement the specific surface area significantly increased what can be seen in Table 2. Standard consistency (SK, %) of all investigated cements is higher than SK of PC (29.0%), and was in the range from 31.6 % for PC10PbZ to 44.0 % for PC50FeZ.

**Table 2.** Results of physico-mechanical properties of the investigated cements

| Sample | Fineness, % | Sp, $cm^2/g$ | SK, % | Setting time, min | | |
|---|---|---|---|---|---|---|
| | | | | Start of the setting | | End of the setting |
| *PC42.5R* | *0.6* | *3940* | *29.0* | *220* | | *270* |
| PC10Z | | | | | | |
| PC30Z | | | | | | |
| PC50Z | | | | | | |
| PC10PbZ | 0.05 | 4880 | 31.6 | 190 | | 250 |
| PC30PbZ | 0.06 | 6960 | 37.0 | 270 | | 340 |
| PC50PbZ | 0.1 | 9840 | 36.8 | 190 | | 250 |
| Sample | Flexural strength, MPa | | | Compressive strength, MPa | | |
| | 2 days | 7 days | 28 days | 2 days | 7 days | 28 days |
| *PC42.5R* | *6.4±0,8* | *8.4±0,6* | *9.6±0,8* | *33.0±0,7* | *49.0±0,8* | *61.1±1.4* |
| PC10Z | 1.8±0,1 | 5.4±0,2 | 10.3±0,4 | 5.3±0,1 | 22.5±1,8 | 58.9±0.6 |
| PC30Z | 2.1±0,1 | 6.1±0,1 | 9.7±0,5 | 7.5±0,2 | 29.8±0,3 | 57.4±1.5 |
| PC50Z | 2.0±0,1 | 5.8±1,1 | 7.8±0,5 | 6.7±0,3 | 26.9±1,1 | 50.5±1.9 |
| PC42.5R | 6.4±0.8 | 8.4±0.6 | 9.6±0.8 | 33.0±0,7 | 49.0±0.8 | 61.1±1.4 |
| PC10PbZ | 6.2±0.4 | 7.5±0.7 | 9.2±0.2 | 25.2±0,8 | 41.2±0.9 | 63.3±2.1 |
| PC30PbZ | 4.3±0.1 | 5.9±0.2 | 8.5±0.5 | 17.0±0.4 | 31.3±0.7 | 57.3±0.6 |
| PC50PbZ | 2.4±0.1 | 4.2±0.6 | 6.9±0.3 | 10.0±0.6 | 23.4±0.1 | 44.9±1.5 |

According to the Point 17 of Regulation [15] which concerns cement PC 42.R and similar cements (according to the SRPS EN 196-3), initiation of setting time for cement PC 42.5 R must be ≥ 60 min. All investigated cements are fulfilling this condition. However, the shortest setting time (initiation and ending) is noticed in the case of PC30Z (80/100 min) and PC50Z (80/130 min). The longest setting time is obtained in the case of PC30PbZ (270/340 min), which is longer time period than PC setting time for 50 min (initiation), i.e. 70 min – end of setting.

Table 2 show values of flexural strength and compressive strength of Portland cement and cement with addition natural and saturated zeolite for 2, 7, 28 days of hydration. Based on these results it can be seen that values of flexural strength and compressive strength depend on the mass content of addition used in Portland cement. Strengths of investigated cements are decreasing with the increasing of saturated zeolite content in the PC for all investigation periods (2, 7 and 28 days). However, the decrease of the strength was after 2 days of hydration, lower for 7 days, and the lowest for 28 days.

According to the EN 197 Standard, cement marked as CAM I should have compressive strength 42.5 MPa. Compressive strengths of newly-formed cements with addition of Pb ion saturated zeolite after 28 days of hydration are mostly above these values. It can be said that all used zeolites can be applied as additives for Portland cement in the quantity up to 30 %. Namely, values of compressive strength (after 28 days of solidification) for all cements with 30 % of additive are high (PC30PbZ – 57.3 MPa and PC30Z – 57.4 MPa) in comparison with requested minimum - 42.5 MPa.

## CONCLUSION

Based on the results obtained from adsorption isotherms, it can be seen that increasing of the concentration of the lead ions in the water solutions the concentration of adsorbed lead ions increased. The maximum amount of lead ions adsorbed on zeolite under certain experimental conditions was 66 mg $Pb^{2+}$/g.

Results of using zeolite saturated with lead ions as the addition for portland cement, instead of the more common additives, showed that this material can be used as Supplementary Cementitious Materials (SCM). Since the flexural strengths (after 28 says of solidification) for the samples obtained by addition of 50 wt. % zeolite into Portland cement are in the range 7 - 8 MPa, they can be considered as low values. The optimal mass content of zeolite or with lead ions contaminated zeolites in Portland cement should not exceed 30 %. Since values of compressive strength (after 28 days of solidification) for all cements with 30 % of additive are higher (PC30PbZ – 57.3 MPa and PC30Z – 57.4 MPa) in comparison with requested minimum - 42.5 MPa, it may be cncluded that used zeolite (lead ions saturated) can be applied as additives for Portland cement in the quantity up to 30 wt. %.

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