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*Original scientific article*

## **COMPARATIVE ASSESSMENT OF ZINC IONS SORPTION AND RETENTION BY PROSPECTIVE UNCONVENTIONAL SOIL ADDITIVES**

Ivana Smičiklas<sup>1</sup>, Marija Egerić<sup>1</sup>, Mihajlo Jović<sup>1</sup>

<sup>1</sup>„VINČA” Institute of Nuclear Sciences - National Institute of the Republic of Serbia, University of Belgrade, Belgrade, Serbia

### **Abstract**

Mining, combustion of coal and waste, and steel processing are the main industrial activities that trigger the emission of large amounts of Zn, which reach the soil and pose a risk to its services, functions, and groundwater quality. A frequently associated process is soil acidification, reducing soil's ability to retain incoming toxic metals. The soil treatments with reactive, low-cost, and locally available materials might be a straightforward approach to decreasing the mobility of toxic metals and alleviating the environmental risks. This study aimed to compare Zn ions sorption and retention by several unconventional soil additives (seashell waste, bone char, and red mud) against the performance of acidic soil. Batch experiments of Zn ions sorption were initially performed using solutions with different Zn concentrations, followed by the determination of sorbed Zn stability in an acidic medium. The waste materials exhibited higher pH values and superior Zn sorption capacities with respect to the soil. The desorption experiment exposed different mechanisms of Zn ions fixation by studied materials. While the substantial amounts of Zn sorbed by seashells, red mud, as well as soil were released in the scope of the ion exchange and carbonate fraction, sensitive to pH decrease, the bone char preserved Zn in more stable fractions. The investigated waste materials can potentially prevent Zn leaching through the soil profile, with the bone char demonstrating the most significant capability for long-term retention performance.

**Keywords:** soil additives, zinc, sorption, retention, soil remediation.

### **Introduction**

The overall mean zinc (Zn) concentration in soil is 50–55 mg/kg and typically does not exceed 300 mg/kg (Noulas et al., 2018). Zinc ions are essential in numerous plant metabolic processes, thus, their deficiency causes reduced growth, tolerance to stress, and chlorophyll synthesis (Sharma et al., 2013). It is also essential for animals' and humans' health, affecting gastrointestinal, central nervous, immune, skeletal, and reproductive systems (Roohani et al., 2013). While the scarcity of available soil Zn represents an important soil quality issue and a cause of malnutrition problems in some parts of the world, Zn concentrations in the soil are rising in many other areas worldwide due to anthropogenic additions.

Characteristic industrial activities that generate and emit the most of Zn are mining, steel processing, and coal and waste combustion (Wuana & Okieimen, 2011). Like other heavy metals, Zn becomes a contaminant in the soil if its total concentrations are elevated compared to natural background levels and its distribution in the soil shifts towards chemical forms with greater mobility and bioavailability. The pollution in croplands results in soil–plant–food transfer of Zn and exposure of numerous living organisms through this chain.

Soil contamination and surface runoff infiltration to the groundwater aquifers result in groundwater contamination with Zn and other toxic metals. Given that groundwater is the most significant source of drinking water in the European Union and the resource for agriculture and industry (EC, 2008), the safety of its quality in part refers to the prevention of metals leaching from the contaminated soil. Numerous techniques for on-site or off-site treatment of the soil contaminated with Zn are developed. Methods based on the physical separation of Zn from soil matrix include chemical leaching using a variety of agents such as carboxyalkylthiosuccinic acid (CETSA), copolymer of maleic and acrylic acid (MA/AA), ethylenediamine tetra acetic acid (EDTA) (Xia et al., 2019), citric acid (Gu et al., 2018; Hu et al., 2021), tartaric acid (Alman-Abad et al., 2020), but also inorganic acids (HCl, H<sub>2</sub>SO<sub>4</sub>, and H<sub>3</sub>PO<sub>4</sub>) (Ko et al., 2006). Phytoremediation (Yan et al., 2020), electro-kinetic remediation (Rezaee et al., 2017) and microbial-induced remediation of Zn pollution (Zhan & Qian, 2016) are also proposed. Furthermore, stabilization/solidification of soil Zn can be achieved using appropriate agents. Recently, cost-effective materials, like limestone calcined clay cement, a low-carbon/cost cementitious material (Reddy et al., 2020), or cement–soda residue (Zha et al., 2021), were found applicable for Zn solidification. The perspective approach is *in situ* stabilization of Zn in soil not by targeting encapsulation but by using amendments (Kumpiene et al., 2008). The role of amendments is to change the chemical forms of Zn in the soil, making it less mobile and less available to plants. Zinc can be chemically stabilized by mixing the soil with liming materials (Martin & Ruby, 2004), phosphate salts (Wuana & Okieimen, 2011), bentonite (Chaves et al., 2017), zeolite, sepiolite, and palygorskite (Ye et al., 2022).

The useful amendment for reducing Zn environmental availability should be effective, inexpensive, available, easy to apply, and non-toxic to the plants. The aim of this study was to evaluate the prospects of several low-cost materials as additives for Zn stabilization in the soil. The sorption and retention of Zn ions by waste-derived materials (seashell waste, bone char, and red mud) were tested in the range of Zn concentrations, and their performance was compared with the Zn sorption/retention ability of a model acidic soil.

### Materials and Methods

Waste-derived materials with different compositions were selected, i.e., calcium carbonate-based seashell powders, calcium phosphate-based animal bones, and Fe-oxide-rich bauxite residue. A composite sample of seashells from the shores of the Aegean Sea in Greece (SW), as well as the samples of *Mytilus galloprovincialis* (MG) shells collected from the mussel farms in the Boka Kotorska bay (Montenegro). The material denoted BC was obtained by the treatment of animal (bovine) bones at 400 °C in an air atmosphere (Dimović et al., 2009). Both the seashells and BC were ground to pass the 0.2 mm sieve. The bauxite residue (red mud, RM) was collected from the Zvornik Alumina Refinery, Republic of Srpska, BiH, and used as a fine powder after rinsing with water and drying at room temperature (Egerić et al., 2019). For comparison, the acidic soil sampled near the mining and metallurgical complex in Bor, Serbia, was used (Egerić et al., 2019).

The sorption of Zn ions was first examined in a series of batch experiments by mixing the investigated solid samples and Zn solutions at a solid-to-solution ratio 1g/10 mL. The solutions with Zn ions concentrations in the range  $10^{-4}$  –  $5 \times 10^{-3}$  mol/L were prepared from Zn(NO<sub>3</sub>)<sub>2</sub> salt in the inert background electrolyte ( $10^{-2}$  mol/L NaNO<sub>3</sub>). The initial pH of all solutions was set to pH 5.0±0.2. The suspensions were agitated at room temperature using an end-over-end shaker (10 rpm) for 48 h. Subsequently, the solid and liquid phases were separated by centrifugation (10 min, 9000 rpm) and filtration. The residual Zn concentrations in the supernatants were measured by Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES, Perkin Elmer Avio 200), and the quantities of Zn sorbed were calculated from the difference between initial and final concentrations. Final pH values

in liquid phases were measured as well, and the concentrations of Ca ions released into the solution following the Zn ions sorption.

For the Zn desorption experiments, residues from sorption experiments were resuspended in 0.11 mol/L acetic acid at the solid-to-solution ratio of 1g/40mL and agitated at room temperature ( $21\pm 2$  °C) for 16 h. These conditions correspond to the first step of the sequential extraction scheme recommended by the European Community Bureau of Reference (BCR) (Ure et al., 1993), which separates the most mobile metal fraction. After centrifugation (10 min, 9000 rpm) and filtration, the extracted Zn concentrations were measured using ICP-OES.

## Results and discussion

Two biological materials were used in the experiments. By their composition, mussel shells are a very rich source of biogenic calcium carbonate (>95%) regardless of the shellfish (Egerić et al., 2018), while (BC) obtained from animal bones is a calcium-phosphate product with the structure of non-stoichiometric hydroxyapatite (Dimović et al., 2009). The RM was composed of oxide compounds and desilication products that result from the Bayer process, with the largest share of hematite (Egerić et al., 2019). The soil was characterized as clay loam, non-carbonate, with an acidic reaction with water (pH 4.93) (Egerić et al., 2019).

As shown in Figure 1a., investigated additives have sorbed Zn ions with very high efficiency viewing the entire range of initial Zn concentrations, specifically SW 98.2-99.7%, MG 96.6-98.7%, B 98.7-99.7%, and RM 99.5-99.9%. Quite the contrary, the soil exhibited a sharp decline in Zn removal efficiency from 93.3% to 62.3%, with a rise in Zn addition.

The experiment outlined the finite capacity of the acidic soil for Zn, which inevitably leads to the leaching of Zn into deeper soil layers or watercourses at higher loads. Amendments, on the other hand, provide sorption sites with a higher affinity for Zn ions with respect to the soil and the pH conditions favorable for limiting the water-soluble fraction of the metal (Figure 1b).

At a given solid-to-solution ratio (1:10), the highest pH values in solution are detected after reaction with RM (10.1-9.1), followed by SW (9.2-7.6), MG (8.6-7.5), BC (7.8-7.4), and finally the soil (5.4-4.8). Processing Bauxite ore via the Bayer process requires the use of a strong base (NaOH), so the residue is strongly alkaline (typically with  $\text{pH} > 12$ ). By washing the red mud with water, the free base was removed but the product still exhibits alkaline properties due to the presence of desilication products (DSP) with high pH and buffering capacity (Gräfe et al., 2011). Seashells and BC as well provide  $\text{H}^+$  neutralization through reaction with bio-carbonate and bio-apatite phases. Since pH is one of the most significant soil quality indicators (Bünemann et al., 2018), adding investigated material could benefit the acidic soil through an increase in pH. However, the materials' mechanisms and capacities for pH regulation differ significantly.

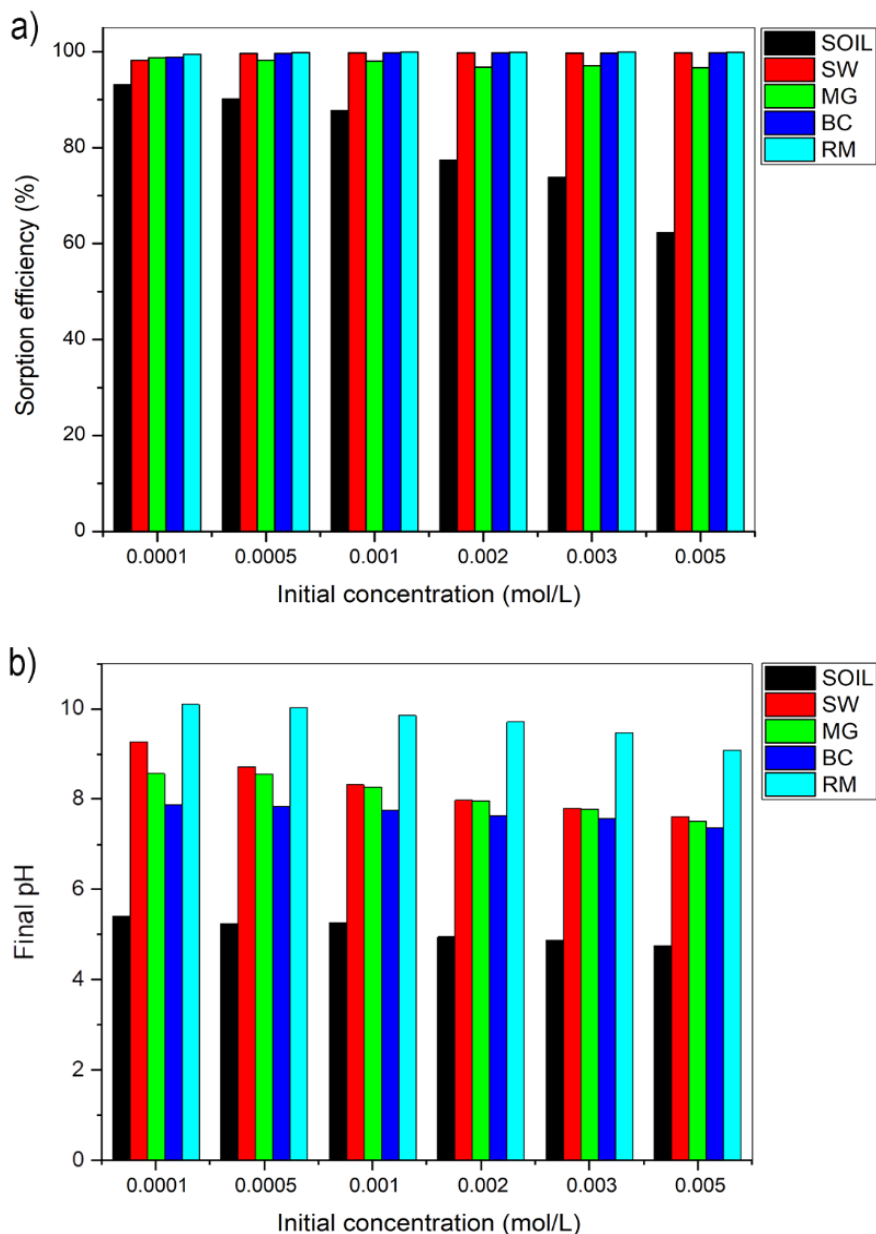


Figure 1. The influence of initial Zn concentration in the solution on the a) Zn sorption efficiency (%) by the investigated unconventional additives and the model soil, and b) final pH values of the solution. Initial pH =5.0, solid-to-solution ratio 1:10, contact time 48 h

After the Zn sorption by all tested materials, the final pH values decreased with the increasing concentration of sorbate (Figure 1b). Over the examined range of Zn concentrations, the final pH values declined by about 1 pH unit using RM and seashell powders, by 0.7 pH units using soil, and by 0.5 pH units using BC. These pH changes may indicate the specific sorption of Zn ions by active protonated surface groups and the release of H<sup>+</sup> ions into the solution.

Relationships between the amounts of Zn ions sorbed and Ca ions released by the additives and the soil are shown in Figure 2. It is noticeable that the application of seashell powders leads to the greatest increase in aqueous Ca concentration (molar ratio Ca:Zn>1:1) due to a partial dissolution of the samples and Zn binding by displacement of Ca in carbonates or precipitation of Zn-carbonate phase. The ratio of sorbed and released ions was linear for the BC and the soil, indicating the ion exchange process and/or dissolution/precipitation mechanism in the case of BC. For the same amount of sorbed

Zn, the RM released the smallest amounts of Ca (Figure 2). RM provides numerous active centers for Zn ion sorption as a composite material with a high hematite content. The addition of different forms of iron, such as hydrous oxides, steel shot, steel sludge, furnace slag, and zero-valent iron, has previously been shown to reduce the leachability and bioaccessibility of soil zinc (Martin & Ruby, 2004). Metal oxides can strongly bind metals through specific sorption and co-precipitation (Derakhshan Nejad et al., 2018).

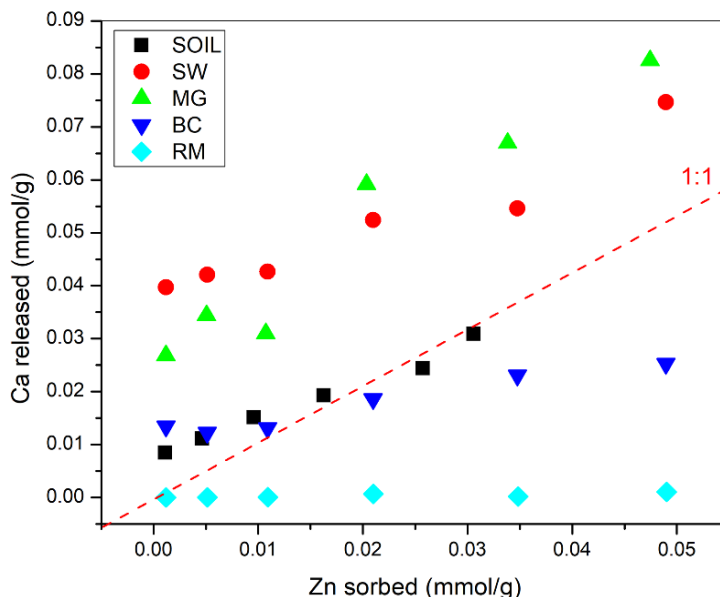


Figure 2. Relationship between the sorbed amounts of Zn and amounts of Ca ions released into the solution by investigated samples

The BCR method proved to be applicable for the assessment of Zn mobility in the soil, showing correlations with the soil constituents and the plant contents (Fernández-Ondoño et al., 2017). The fraction of Zn in the most mobile phase of the BCR extraction protocol was determined to assess the long-term stability of Zn sorbed by different samples. The results of exposure of the residues obtained following Zn sorption to the acetic acid solution are summarized in Figure 3. It can be concluded that Zn extraction in the scope of the ion exchange and carbonate fraction generally increases with the increase in the Zn load, but absolute amounts differ markedly between studied materials.

For soil, maximally, 58% of the sorbed Zn was found in the most mobile fraction. Seashell powders SW and MG accumulated the highest share of Zn in the acid-soluble fraction (up to 86%), whereas the percentage in RM reached 64%, at the highest Zn load. Previously, the positive correlation between Zn concentration extracted within BCR1 soil fraction was established with the content of  $\text{CaCO}_3$  in the soil, as well as with the percentages of  $\text{Fe}_{\text{ox}}$  in some soils (Fernández-Ondoño et al., 2017). In practice, this indicates the potential re-mobilization of Zn ions if the pH of the medium drops sufficiently, which would require periodic repetition of the treatment. On the other hand, with less than 1 % extracted with acetic acid, additive BC proves to accumulate Zn in more stable fractions. Results implied that Zn was immobilized as metal-phosphate precipitates resistant to soil acidification, in accordance with the previous research conducted using various phosphate-based amendments (Kumpiene et al., 2008).



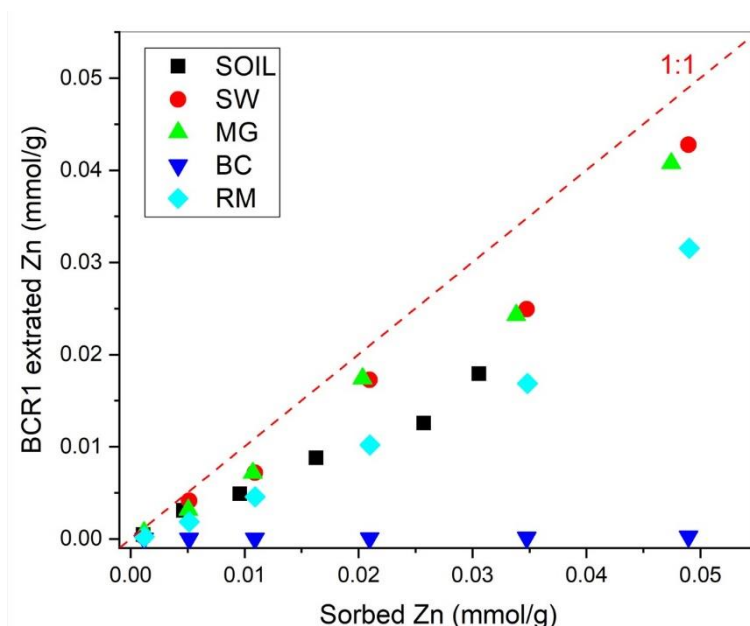


Figure 3. Relationship between the sorbed amounts of Zn and amounts of Zn extracted in the scope of the acid-soluble (BCR1) fraction

Generally, alkaline materials, Fe-oxides, phosphorus materials, and clays were found to be the most efficient amendments for Zn immobilization in the soil (Kumpiene et al., 2008). Identifying and applying waste materials and by-products of various industries, which according to their composition, correspond to one of these classes and do not pose a risk to the living world, is a step toward sustainable soil management. After screening out the efficiency of the potential additives, experiments should be conducted with the specific soil to determine the best soil/additive ratio for the field-fixing treatment.

### Conclusion

The materials studied in this work are available, cheap, easy to handle and apply, and show higher capacity at binding Zn compared to model acidic soil. Consequently, they may be an effective alternative to frequently used soil remediation agents, like limestone, lime, or natural minerals. Application in the soil also has positive effects on reducing the accumulated amounts of these waste materials. Considering their alkaline nature, treatments with selected materials would affect the increase in the soil pH, thus improving soil capacity for natural attenuation of mobility and leaching of toxic metals. Comprehensive research is needed to optimize the addition rates of such unconventional additives, considering not only toxic metals' mobility but other essential properties of the soil and its future use.

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