Research Article

Ceramic matrices for immobilization of heavy metals adsorbed on rice husk

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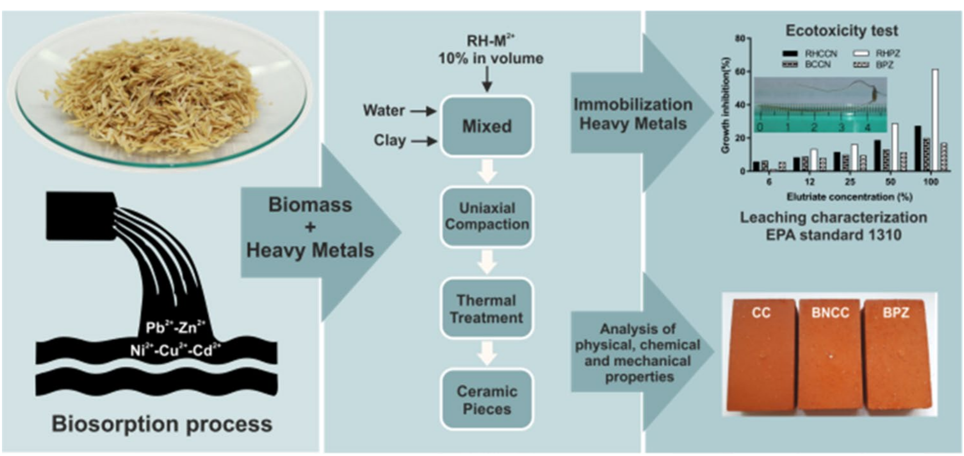
Abstract

Numerous studies have been conducted about the removal of heavy metal ions from aqueous solutions using lignocellulosic materials, which are capable of adsorbing various contaminants present even at low concentrations. Rice husk constitutes biomass waste that has found multiple applications in different fields, such as the removal of heavy metals by adsorption. After this process, a biomass-contaminant waste is generated, which currently does not have viable and low-cost alternatives for its final disposal. Thus, the objective of this work is to evaluate the possibility of incorporating rice husk contaminated with Cu–Ni–Cd and Pb–Zn into ceramic matrices, to immobilize these hazardous metals. Ceramic pieces from mixtures of commercial clay and rice husk with adsorbed metal, added by 10% in volume were obtained. The raw materials were characterized by different techniques, such as SEM-EDS, XRD, DTA-TGA, particle size distribution, ecotoxicity, etc., while in ceramic products were analysed porosity, permanent volumetric variation, weight loss of ignition, ecotoxicity, among others. From the overall results, it is possible to conclude that the obtained ceramic pieces have immobilized within their structure, the heavy metals present on rice husk. The bricks have excellent physical and mechanical properties that make possible their use in civil construction.

Graphic abstract



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Keywords Heavy metals · Rice husk · Ceramic matrices · Clay bricks

1 Introduction

The continuous population growth and industrial development have led to the demand for new products and services whose processes generate liquid effluents with high concentrations of bioaccumulative, non-biodegradable and carcinogenic toxic substances. These effluents must be adequately treated to eliminate their toxicity or reduce it to adequate levels so they can be discarded to watercourses without damaging ecosystems.

Heavy metals such as lead, copper, cadmium, zinc and nickel, are considered priority environmental pollutants, due to the toxicity that metal ions present in living organisms. After their release from various sources, such as industrial and agricultural activities, heavy metals are not degraded and they persist in the environment [1].

Numerous technologies are currently used to remove these contaminants from liquid solutions: chemical precipitation, ion exchange, advanced oxidation processes, membrane separation and adsorption can be mentioned [2–6]. These treatments are expensive, produce secondary waste and, in many cases, do not meet environmental requirements. Consequently, there is great interest in alternative removal systems, such as biosorption processes since they have proved to be an effective, economical, versatile and simple technique [7, 8].

On the other hand, agroindustry generates significant amounts of biomass waste without a specific use. Nowadays, there are a large number of investigations that study these type of wastes as heavy metal biosorbents [9–15]. However, there is no scientific information about the treatment, reuse or final destination of the new biomass-contaminant waste.

Rice is one of the greatest consumed cereals in the whole world and it is cultivated in more than 100 countries around the world. According to FAO, the annual global rice production is more significant than 740 million tons. In Argentina, it is approximately 1,400,000 tons. Rice seed is mainly formed by endosperm, husk, bran and germ, in which the endosperm accounts for 70%, husk 20–21%, rice bran 6–8%, and rice germ 1%, respectively, of the total seed weight. During rice production, large quantities of husks are generated, causing wastes and a problem for the production regions of this cereal [16].

In general, the husk is burned or deposited in landfills with the corresponding environmental impacts that this entails. The use of this residual material would improve the sustainability of this agricultural activity and help to reduce its effect. Rice husk (RH) has been used in different applications: as a fuel due to its calorific value [17], as a source of silica and silicon due to the high content of SiO_2 [18–22], for the preparation of activated carbon [23], in construction materials [24, 25], and as a biosorbent in water treatment [26–28], among others. In the latter case, after the retention of the contaminants on the husk, a biomass-contaminant residue is generated, which nowadays does not have viable and low-cost alternatives for final disposal.

In this context, the rice husk used in the first stage of adsorption experiments, under the optimal conditions of the process, was utilized to ensure that it retains a determined amount of contaminants. Thus, this paper is going to evaluate the feasibility of incorporating the rice husk-contaminant residue in clay ceramic matrices, to immobilize the heavy metals lead, zinc, nickel, copper and cadmium contained in these materials.

2 Materials and methods

2.1 Raw materials

The rice husk used was obtained from a cooperative in the province of Entre Ríos, after the rice grain husking.

Since chemical surface modifications of biomaterial can improve its adsorptive capacity, the rice husk was treated with potassium hydroxide (KOH-RH). The natural material was mixed with 1 M potassium hydroxide (ratio 1:10) and boiled for 30 min. After that, this mixture was left overnight. Then, the rice husk was filtered, washed with distilled water and 2.5 M hydrochloric acid to reach pH 5 and finally dried at 70 °C in an oven for 24 h.

To determine the optimal conditions of adsorption process, several factors were analyzed such as pH, contact time, initial M^{2+} concentration and adsorbent mass.

Sorption tests were performed under batch conditions. For adsorption tests, a suitable mass of rice husk was put in contact with a solution of known concentration of lead, nickel, zinc, copper, lead or cadmium at a given pH. The samples were kept on a mechanical stirrer for the necessary time to achieve chemical equilibrium. The experimental conditions were pH = 5–6; adsorbent dosage: 3 g/L for Cd^{2+}, 4 g/L for Cu^{2+}, 4 g/L for Ni^{2+}, 2 g/L for Pb^{2+}, 3 g/L for Zn^{2+}; initial metal concentration: 50 mg/L; contact time: 60 min; temperature: 20 ± 4 °C.

Once the sorption tests were completed, the samples were filtered and the residual metal was quantified in the collected solution. The determination of the remaining metal was performed by atomic absorption spectroscopy using an air-acetylene flame at the characteristic wavelength for each metal under study. The results obtained have showed that metal removal percentages of chemically pretreated rice husk were significantly higher than those of untreated material (data not shown).

The husks loaded with the different contaminants were dried at 70 °C and reserved for later use in compact ceramic bodies.

The commercial clay used comes from a local company.

2.2 Ceramic bricks design and manufacture

The manufacturing process of ceramic bricks has been designed considering the characteristics and properties of the clay used and the thermal behavior of the biomass.

The rice husk loaded with the contaminants was subjected to a grinding and sieving process to obtain a particle size of less than 1 mm. Porous ceramic pieces were obtained from green bodies made with mixtures of 10% in volume of residue in commercial clay. The decision to begin the study adding this percentage of residue is based on previous experiences with other biomass wastes [29].

The pieces have been compacted by uniaxial pressure of 25 MPa, with the addition of 8% in weight of water, into molds of 70 mm x 40 mm, resulting in bricks approximately 15 mm thick. After a drying period, the samples were thermally treated at 1000 °C following heating curves similar to those used in the ceramic industry.

2.3 Characterization of raw materials and compact bodies

The raw materials were characterized using different techniques such as scanning electron microscopy (SEM), energy-dispersive analysis of X-ray (EDS), X-ray diffraction (XRD), thermogravimetric analysis (TGA), differential thermal analysis (DTA), Fourier-transform infrared spectroscopy (FTIR), granulometric distribution, ecotoxicity and leachate.

For SEM–EDS analyses, a FEI Inspect S50 scanning electron microscope with energy-dispersive analyser (EDAX-Phoenix) was used.

The X-ray diffraction spectrum was obtained by using a PANalytical X'Pert PRO diffractometer. Samples were exposed to X-ray CuKα (λ = 1.5406 nm) with the 2θ angle varying between 10° and 80°. The applied voltage and current were 40 kV and 40 mA, respectively.

The thermal behavior of rice husk was obtained with Shimadzu TGA-50 and Shimadzu DTA-50 instruments with TA-50 WSI analyser, using a heating speed of 10 °C/min, in the range of ambient temperature – 1000 °C. In all cases, Pt support and air atmosphere were used.

FTIR spectra were obtained with Perkin Elmer 1605 Fourier-transform Infrared Spectrophotometer (USA), using KBr disks to prepare the rice husk samples. The spectral range varied from 4000 to 650 cm^{-1}.

The compact bodies were characterized by several techniques: porosity, permanent volumetric variation (PVV), weight loss of ignition (LOI) and mechanical properties, among others.

The porosity and water absorption of the samples were determined according to ASTM C20-00.

The module of rupture (MOR) was obtained in a Digimess Model TG100L machine, with a maximum capacity of 500 kg. The speed of the test was 0.5 mm/min.

The PVV and LOI values were obtained by measuring the dimensions (length, width and thickness) and the weight of each sample before and after sintering at 1000 °C.

The ecotoxicity tests were carried out according to IRAM 29114 (Argentina). The KOH-RH charged with adsorbed metals was first tested and then, the ceramic pieces obtained by replacing part of the commercial clay with residue were tested too. Thus, it was sought to determine the fixation of the metals into the ceramic matrix. In this ecotoxicity test, the initial elutriate was obtained by suspending the residue in distilled water in a 1:4 ratio, stirred for 2 h and filtered. The experiments were carried out by triplicate using concentrations of 6, 12, 25, 50 and 100% of the initial elutriate. Twenty seeds of the ryegrass species were placed in 90 mm Petri dishes, on two filter papers (Whatman type 1) and 3.5 mL of the solution to be evaluated was added. The dishes were covered and incubated at 24 °C for 120 h. Reference controls are carried out with distilled water. The evaluation points were the number of germinated seeds and the elongation of the radicle, which is expressed as a percentage of inhibition of the radicle growth, concerning the reference samples with distilled water. The evaluation of the effect on the elongation of the seedling radicle makes it possible to measure the toxic effect of soluble compounds, present in such a low concentration that they are not sufficient to inhibit germination, but which nevertheless can delay or completely inhibit the process of elongation of the radicle.

The leaching characterization procedure of contaminated rice husk and bricks were carried out by adopting EPA standard 1310. An adequate amount of rice husk or brick blocks were placed together with the extraction solvent. The amount of water used was 16 times the weight of the solid. The systems were stirred for 24 h. Finally, they were filtered and in the filtered solutions, the concentration of heavy metals was determined by an atomic absorption spectrophotometer Buck 210 VCG (USA) equipped with an air-acetylene flame at the characteristic wavelength of each metal.

3 Results and discussion

3.1 Characterization of raw materials

The semi-quantitative chemical analysis by EDS of commercial clay and natural rice husk, expressed as a weight percentage of their elements, is shown in Table 1. As can be seen, the significant elements in both materials are carbon, oxygen and silica.

Morphologically, the commercial clay presents a homogeneous structure of fine and large particles, being the latter agglomerates of small particles (Fig. 1a). The natural rice husk is characterized by a globular structure, which shows a cellular pattern of the epicarp or outermost layer, well organized, with a homogeneous structure (Fig. 1b). Regular spherical grains of almost equal sizes appear in parallel rows. Also, elongated acicular structures are observed, which resemble "hairs", typical of the fibrous structure of certain cereals. Other authors [20, 30, 31] also observed this morphology. In Fig. 1c, the effect of the chemical treatment on the rice husk can be seen. The detachment of these "hairs" from the base thereof is observed.

The DTA-TGA profile corresponding to rice husk is shown in Fig. 2 (a). Several exothermic peaks in the DTA curve that have been assigned to the combustion

Table 1 Chemical analysis of the raw materials

	C	O	Na	Mg	Al	Si	K	Ca	Fe
RH [wt %]	29.7	38.4	–	–	–	22.3	1.9	2.1	5.6
Clay [wt %]	15.6	40.7	1.1	1.3	8.7	23.8	2.0	1.3	5.5

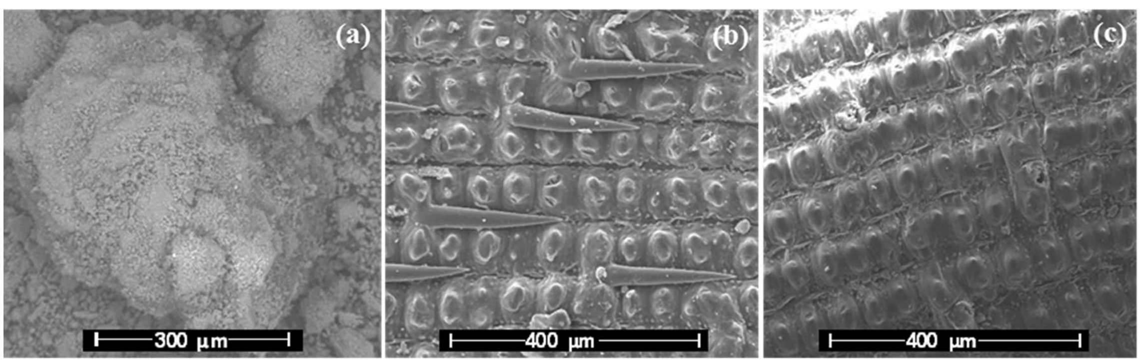

Fig. 1 Scanning electron microscopy images of **a** commercial clay, **b** RH, **c** KOH-RH

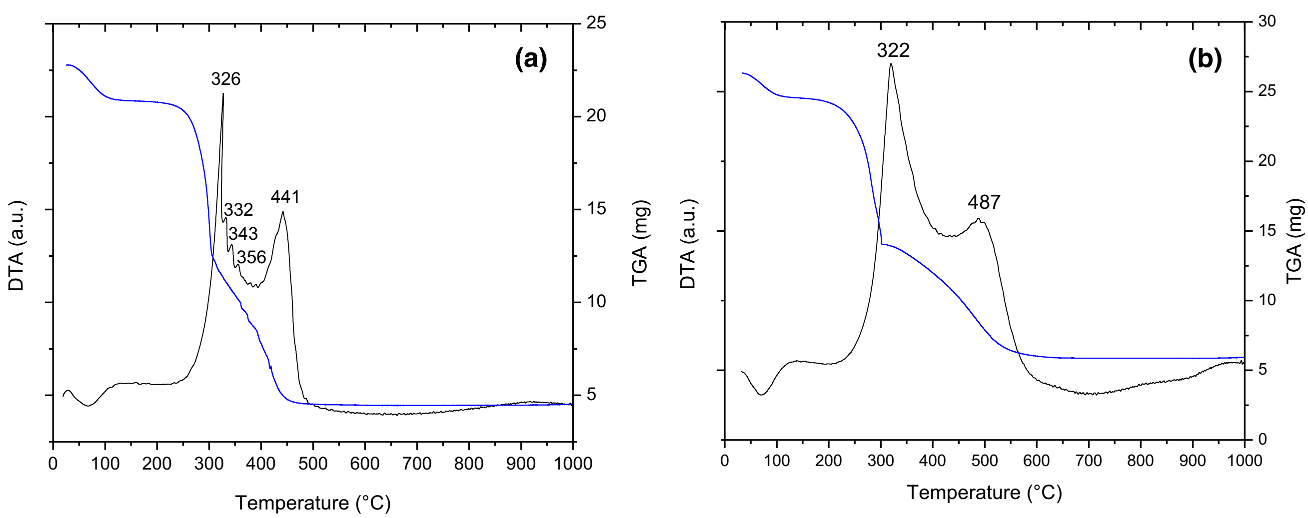

Fig. 2 DTA-TGA curves **a** RH and **b** KOH-RH

decomposition of the biopolymers present in the biomass are observed. The rice husk sample presents a first pronounced peak at 326 °C with loss of weight, followed by other small peaks at 332 °C, 343 °C and 356 °C and finally another pronounced broad peak at 441 °C, with continuous weight loss. This behavior corresponds to the presence of hemicellulose, cellulose and lignin. Since these compounds are intimately related in the whole structure, the thermal decomposition of each one cannot be analyzed individually. However, it should be expected that lignin decomposition occur at the highest temperature, while hemicellulose degradation takes place at the lowest one. Therefore, it should be considered that the first peaks are associated with hemicellulose and cellulose decomposition and the last one with the lignin degradation. In Fig. 2b, the DTA-TGA curve, corresponding to KOH-RH sample is shown. As can be seen, the peak assigned to lignin is displaced toward higher temperatures, suggesting that the alkali treatment performed makes the material structure more resistant.

XRD pattern of the commercial clay used in this work is presented in Fig. 3a. It is observed that the main crystalline phases present are quartz, hematite, potassium feldspar, aluminum oxide and iron.

The diffraction pattern of RH (Fig. 3.b) exhibits a broad peak of high intensity centered at 21.9°, showing the amorphous nature of silica [32–34]. Besides, peaks corresponding to a semi-crystalline cellulose structure were observed (2θ = 16.0°, 21.9°, 34.7° and 46.4°). Similar spectra were obtained for the samples of KOH-RH and correspond to contaminated samples after the adsorption experiences.

Figure 4 shows FTIR spectra of biomass (RH and KOH-RH) and in Table 2 have been assigned the functional groups bands that might participate in the metal adsorption [33, 35, 36].

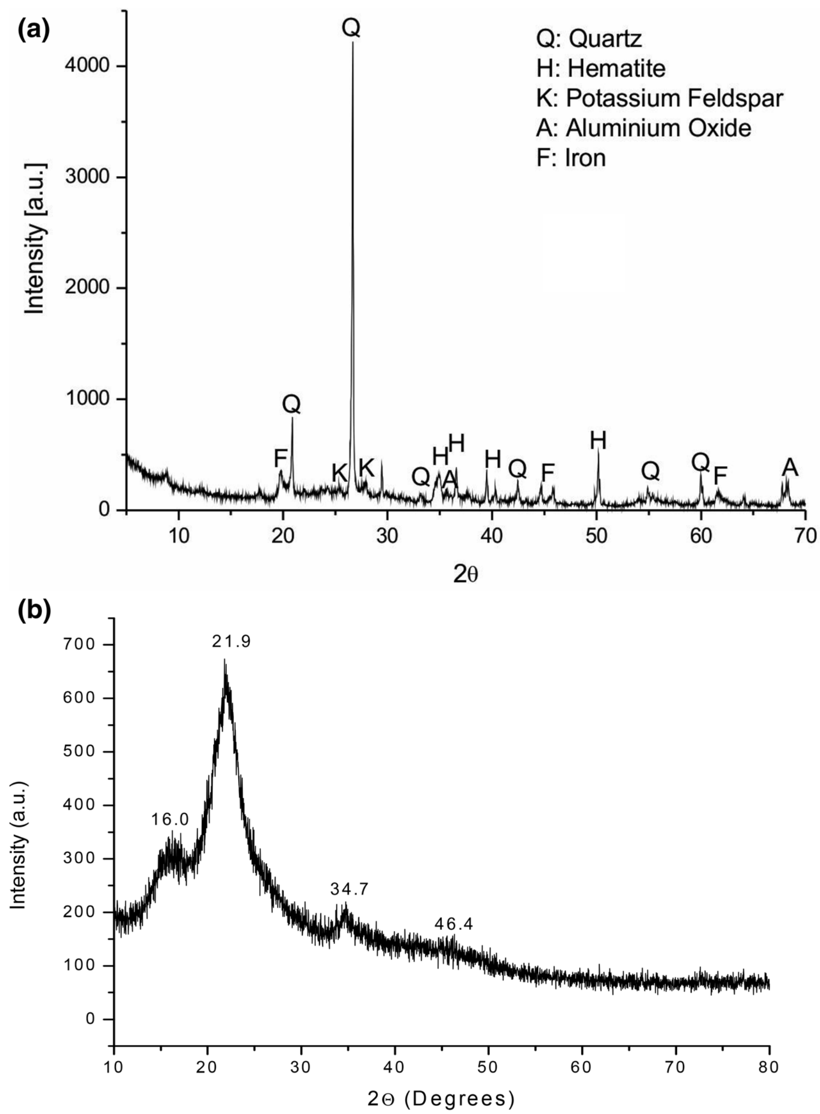

Fig. 3 XRD pattern of **a** commercial clay and **b** RH

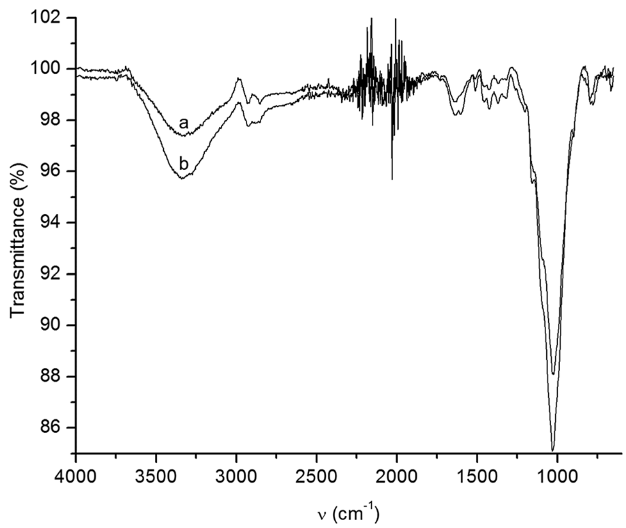

Fig. 4 FTIR spectra of biomass (**a** RH, **b** KOH-RH)

Table 2 Functional groups present in RH and KOH-RH

ν (cm^{-1})	Functional group present in biomass
~3330	–OH stretching due to the –Si–OH group
~2930 and ~2850	Aliphatic C–H stretching of methyl and methylene groups present in lignin
~1730 and ~1630	>C=O stretching frequencies due to ketonic and aldehydic moieties
~1030 and ~798	–Si–O–Si– stretching and bending of siloxane

The infrared spectra of natural and activated rice husk did not differ significantly. FTIR peak analysis of RH and KOH-RH identified the Si–OH band that appears at 3300 cm^{-1}. This group plays an important role in cation exchange. Other functional groups that could participate in ligand biosorption are –Si–O–Si, –OH and carbonyl moieties [37].

3.2 KOH-RH loaded with heavy metals from the adsorption process

Once the adsorption experiences were completed under the optimal process conditions for each divalent metal, the amount of metal retained by KOH-RH was calculated. These results are observed in Table 3.

Since the quantities of each contaminant retained were different, for the preparation of the ceramic pieces it was decided to use on the one hand the waste containing Pb and Zn and on the other hand, the waste loaded with Cu, Cd and Ni because the amount retained in these cases were similar.

Heavy metals adsorbed on KOH-RH after adsorption experiences have not been able to be detected by EDS, possibly due to their low concentration. Thus, to determine the presence of the contaminants in the KOH-RH, a leaching test was carried out on the material loaded with the metal contaminants. The results are presented in Table 4.

Taking into account Tables 2 and 3 is possible to note that the Pb^{2+}, which recorded the highest amount adsorbed per gram of treated biomass, in the leachate tests shows the lowest concentrations. This could be indicating that its retention in biomass is excellent, of the type of chemical adsorption. On the other hand, the Zn ion is the one with the least retention, probably due to a physical adsorption process. With the same criteria, it can be concluded that the Cd and Cu ions exhibit similar behaviors, both in adsorption and in leaching, while the Ni ion, which behaved similarly to these in adsorption, leaches more easily.

Ecotoxicity test determines the toxicity of water-soluble chemicals for seeds of certain species. Evaluation of the effect on the elongation of the seedling radicle makes it possible to determine the toxic effect of soluble compounds, present in such low concentration that they are not sufficient to inhibit germination, but which nevertheless can delay or completely inhibit the processes of radicle elongation. The results of this test conducted on the shells contaminated with Ni, Cu and Cd (RHCCN) on the one hand and contaminated with Pb and Zn (RHPZ) on the other, at different concentrations, will be incorporated for comparative purposes to the figure corresponding to the results of the ecotoxicity test performed on the ceramic bricks obtained (Fig. 6). It is observed that as the concentration of the contaminated biomass elutriate increases, the inhibition in the growth of the radicle increases.

3.3 Characterization of ceramic pieces

Figure 5 shows the macroscopic appearance of the bricks obtained with commercial clay (CC) and with the addition of 10% by volume of KOH-RH with Ni-Cu-Cd (BNCC) on the one hand and Pb–Zn (BPZ), for the other. It can be seen that the products have a uniform coloration and a well-defined structure without shelling.

Table 3 Quantity Adsorbed of each metal in KOH-RH, in milligrams

	Pb^{2+}	Zn^{2+}	Ni^{2+}	Cu^{2+}	Cd^{2+}
q_e [mg M^{2+}/g KOH-RH]	16.09 ± 0.14	9.83 ± 0.39	6.89 ± 0.39	7.81 ± 0.40	7.66 ± 0.06

Table 4 Leachate analysis of KOH-RH loaded with metals after adsorption processes

	Ni	Cu	Cd	Pb	Zn
Concentration [ppm]	38.5	19.8	19.2	1.5	66.1

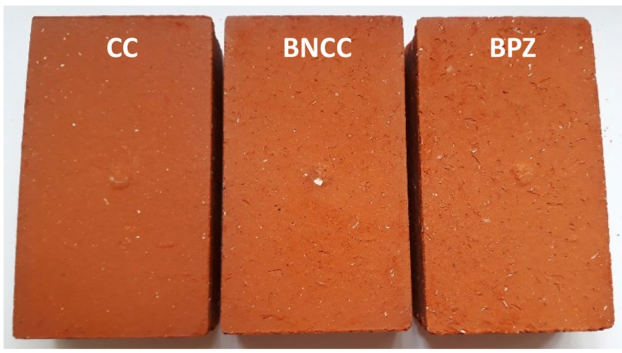

Fig. 5 Macroscopic appearance of the obtained bricks

The properties analyzed in the ceramic products obtained: LOI, PVV, porosity, water absorption and modulus of rupture, are shown in Table 5. Samples with rice husks have higher loss of ignition values (more than 50%) compared to commercial clay samples, with no added waste. In contrast, the permanent volumetric variation of all samples is around the same range, presenting a difference of 10% in the BPZ sample, taking into account the value of commercial clay. This fact can be explained because, during the heat treatment, the rice husk combustion takes place, generating pores in the structure. The increase in the number of pores is reflected in the porosity of the pieces. In the case of BNCC, there is an increase of 20% compared to CC. In contrast, flexural strength is lower in samples with added residue. This behavior is related to the increase in porosity that affects cohesion and reduces the resistance to fracture of the material.

The characteristics of the ceramic pieces obtained are within the values established by the market and those set by ASTM C410-60 for industrial floor bricks.

Figure 6 shows the results of the ecotoxicity tests carried out on ceramic bricks. It was observed that for low concentrations, the percentages of inhibition of root growth do not differ significantly from those obtained in the shells with adsorbed metals. However, from higher levels (50%), the inhibitory effect of the bricks is less compared to that obtained in contaminated rice husk.

On the other hand, the analysis of leached liquids of the ceramic pieces with contaminated shells was performed employing atomic absorption spectroscopy. The concentration values are below the method detection limit, and in consequence, the levels of the pollutants analyzed are below the values established by current regulations.

Table 5 Properties of the ceramic products obtained

	CC	BNCC	BPZ
LOI [%]	5.2	8.7	8.4
PVV [%]	− 10.62	− 10.03	− 9.62
Porosity [%]	29	35	32
Water absorption [%]	16	21	18
MOR [MPa]	7.0	5.2	5.2

The results of the ecotoxicity and leaching tests carried out on the final products suggest the feasibility of incorporating rice husks with adsorbed heavy metals, into ceramic matrices to achieve fixation of these by the structure.

4 Conclusions

In this work, the feasibility of incorporating in ceramic matrices residual rice husks treated with KOH and loaded with heavy metals, in order to immobilize them (lead, zinc, nickel, copper and cadmium) was evaluated. The pollutant loads of rice husks were made by adsorption processes.

From the results, it can be concluded that the ceramic pieces have immobilized within their structure, the heavy metals adsorbed in the added rice husks.

Besides, the ceramic products obtained have a defined structure without shelling, with similar and homogeneous coloration, defined edges, a reasonable degree of sintering and properties suitable for use in service, within the market requirements.

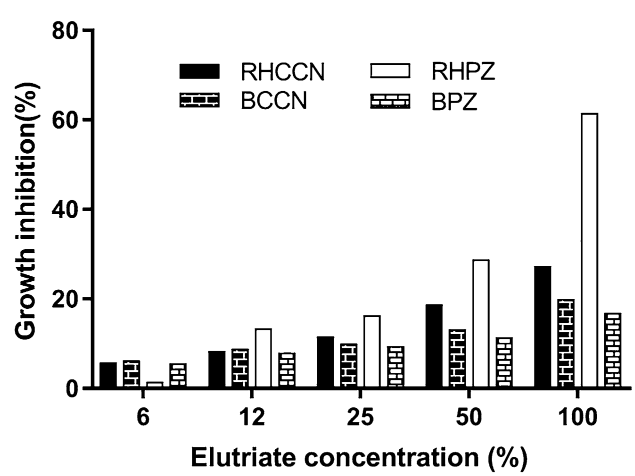

Fig. 6 Ecotoxicity analysis of KOH-RH and the bricks obtained

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Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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