

TREATMENT OF COAL ASH LANDFILL LEACHATE USING ZEOLITIC MATERIALS FROM COAL COMBUSTION BY-PRODUCTS

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Article History:	Abstract:
Received 05 March 2018	Three different zeolitic materials derived from coal combustion by-products (CCBs) were used to treat local coal ash landfill leachate at the same power plant. The zeolitic materials properties were characterized in terms of mineralogical composition (XRD), chemical composition (XRF), total carbon content, morphological analysis (SEM), specific surface area (BET method), cation exchange capacity (CEC), loss of ignition, pH, conductivity and bulk density. XRD indicated that sodalite zeolite was formed in all the samples after hydrothermal activation of CCBs. One zeolitic product from baghouse fly ash (ZFB) presented the lowest SiO ₂ /Al ₂ O ₃ ratio, highest CEC and specific surface area, thus having a high capacity in removal of heavy metal pollutants. The zeolitic products were efficient to reduce arsenic concentration to a value below the legislation. All zeolitic materials also showed a significant removal of Ni, Cd, Zn and Co except of Cr at a dose of 10 g L ⁻¹ . Thus, this work provides a sustainable strategy to resolution of solid waste from power plants.
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1. Introduction

Power generation from coal is the most widely used form of electric power production in the world. According to the report from the International Energy Agency [1], coal share in the global energy matrix is the largest and accounts for about 41% of total global electricity generated. The largest consumer of coal is China, which consumes 51% of world coal demand. The United States, India and Japan are also major global consumers [1]. In Brazil, coal accounts for 3.3% of the country's energy matrix, but it has big reserves and the coal use is expected to increase for the coming years [2].

Apart from the emission of large volume of greenhouse gases, another major environmental problem relating to coal burning is the generation of

various types of solid wastes, Coal Combustion By-products (CCBs). Some researchers have shown that CCBs have toxic elements such as As, Cd, Mo, Pb, Ti, U, Zn and Hg in their chemical composition, therefore they can cause environmental problems if not properly disposed [2-3]. Some alternatives have been proposed for disposal and/or reutilization of this waste, such as immobilization of its toxic elements using immobilizing agents [5], and synthesis of adsorbent materials [6-10] for removal of pollutants [11-14].

One of the CCBs disposal ways is landfills. The ash landfills using layers of limestone and clay, for example, are used as a low-cost practice by a coal-fired power plant in southern Brazil. This type of landfill has a drainage system which directs the

landfill leachate to a small stream. However, it is required that the effluent containing the toxic elements must be below the limits allowed by environmental legislation [15].

Recycling of CCBs can prevent soil and water contamination, however, recycling usually cannot use all these wastes and the large amount remaining wastes still require an environmental-benign disposal to avoid site contamination.

The present work is to evaluate the removal of toxic elements presented in the wastewater from an ash landfill leachate located in southern Brazil using zeolitic materials synthesized from different types of CCBs at the same site. This work can contribute to the sustainability of the power plants since the application of modified CCBs to treat the effluent drained from the landfill can use the local ashes in an economic way.

2. Material and methods

2.1. Materials

All the reagents used for this study were of analytical grade. The coal fly ash samples from baghouse filter, cyclone filter and coal bottom ash were collected at the Figueira coal-fired power plant located in Paraná State, Brazil.

These ash samples present different particle sizes and variable physical and chemical characteristics, although they are from the combustion of the same batch coal.

The wastewater used in this study was landfill leachate (LL) from the coal ashes disposal site of the same Figueira coal-fired power plant. The schematic diagram of the ash landfill is shown in Figure 1.

2.2. Methods

2.2.1. Synthesis of zeolitic materials from coal ashes

The zeolitic materials were prepared by hydrothermal activation of 20 g of coal ash at 100 °C in 160 mL of 3.5 mol L⁻¹ NaOH solution for 24 h. The obtained zeolitic material was repeatedly washed with deionized water to remove excess sodium hydroxide until the washed water had pH ~ 10, then it was dried at 80 °C for 12 h. The zeolitic products

obtained from bottom ash, fly ash from cyclone filter and fly ash from baghouse filter were labeled as ZBA, ZFC and ZFB, respectively.

2.2.2. Characterization of materials

The mineralogical compositions were determined by X-ray diffraction analyses (XRD) with an automated Rigaku Mutiflex diffractometer with Cu K- α radiation at 40 kV and 20 mA over a range (2θ) of 5–90° with a scan time of 0.02°/s.

The chemical composition was determined by X-ray fluorescence (XRF) in Bruker S8 Tiger equipment. Total carbon content was determined by a LECO analyzer (model CS-400) with a radio frequency furnace (model HF-400A). The particle morphologies were observed by a JEOL JSM-6701F High Resolution Scanning Electron Microscope (Field Emission).

The pH and conductivity of the suspensions of zeolitic materials were measured as follows: samples (0.25 g) were placed in 25 mL of deionized water and the mixture was stirred for 24 h in a shaker at 120 rpm (Ética — Mod 430). After filtration, the pH of the solution was measured with a pH meter (MSTecnopon — Mod MPA 210) and the conductivity was measured with a conductivimeter (BEL Engineering - Mod W12D).

Bulk density of zeolitic materials was calculated from the weight of each sample divided by the volume of 35 mL of the sample in a beaker. The specific surface area of zeolitic materials was determined by a BET Micromeritics Adsorption Analyzer (ASAP 2010). Prior to determination of the specific surface area, samples were heated at 120°C for 8 h to degas under vacuum conditions. The BET surface areas were obtained by applying the BET equation to the nitrogen adsorption data. In Cation Exchange Capacity measurements (CEC), the samples were saturated with sodium acetate solution (1 mol L⁻¹), washed with distilled water (1 L) and then mixed with ammonium acetate solution (1 mol L⁻¹). The sodium ion concentration of the resulting solution was determined by optical emission spectrometry with inductively coupled plasma – ICP-OES (Spectro – Arcos).

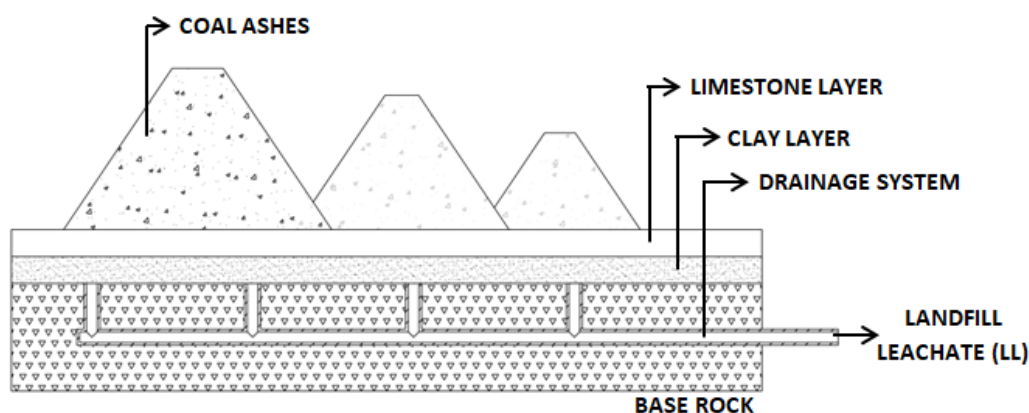


Figure 1. Configuration of coal ashes disposal site

2.2.3. Treatment of coal ash landfill leachate

Batch experiments were carried out at room temperature, with continuous stirring at 120 rpm in 250 mL conical flasks, using 50 mL aliquots of leachate sample mixed with an amount of zeolitic material of 0.5 g and 1 g, corresponding to the adsorbent dosage of 10 and 20 g L⁻¹, respectively. After 1 h of stirring, the mixtures were filtered and the filtrate sample was acidified to pH < 2 by using concentrated nitric acid for metal analysis by optical emission spectrometry with inductively coupled plasma – ICP-OES (Spectro – Arcos) [16, 17]. The arsenic concentration of the landfill leachate was determined by graphite furnace atomic absorption spectrophotometry (GF-AAS), while the concentration of all other elements was determined by the ICP-OES.

3. Results and Discussion

3.1 Characterization of zeolitic materials

3.1.1 Mineralogical composition

The X-ray diffraction patterns of ZBA, ZFC and ZFB samples are shown in Figure 2. It can be observed that sodalite zeolite is present in all the samples. The formation of this material shows that CCBs can be used successfully in the synthesis of zeolites, which have a great potential in removal of pollutants. All the samples also presented a quantity of quartz from the precursor materials. These results agree with the observations presented by other investigators who used the same hydrothermal alkaline activation methodology for coal ashes [18-

21]. It can be noticed that ZBA also presented a crystalline sodium aluminosilicate phase besides sodalite, due to low bulk amount of silicon and aluminium in this material, which was insufficient for the complete formation of zeolite (Table 1 – chemical composition). In addition, ZBA sample was synthesized from the bottom ash of the power plant, which has a shorter exposure time to the flue gases from the burning of coal, and therefore presented different characteristics from the other fly ashes used in this study.

3.1.2 Chemical composition

The chemical compositions and total carbon content of three zeolitic samples (by weight %) are given in Tables 1 and 2, respectively. According to Table 1, the major constituents for all zeolitic materials are: SiO₂, Al₂O₃, Fe₂O₃ and Na₂O. Other components of CaO, SO₃ and TiO₂ were found in less quantity between 1.08 and 4.57%. The SiO₂/Al₂O₃ ratio, related to the cation exchange capacity, followed an increasing order: ZFB (1.46) < ZBA (1.70) < ZFC (1.85). As shown in other studies, a lower SiO₂/Al₂O₃ ratio results in materials with a greater cation exchange capacity [10, 11], which was confirmed in the CEC results shown later.

The total carbon content (Table 2) followed an order: ZBA > ZFC > ZFB, which is similar to the variation in the loss of ignition determined for the samples (Table 1). ZBA was synthesized from the bottom ash, which was exposed to the flue gases for a shorter time and therefore was not completely burnt, containing more unburnt carbon. On the other

hand, the precursor ash of ZFB was exposed to high temperatures in more time, and therefore, lower carbon content was presented both in the raw material and the product. Overall, ZBA is poor in inorganic compounds and rich in unburnt carbon (Table 2 and Table 3 after).

3.1.3 Chemical and physical characteristics

Other physical and chemical characteristics of the three samples of zeolitic materials are given in Table 3. The pH values of zeolitic materials suspension were very close and ranged from 9.1 to 9.5. The pH values are less than 7 due to the hydrothermal activation with NaOH solution. The conductivity values ranged from 213.5 to 296.0 $\mu\text{S cm}^{-1}$ and are consistent with other studies

of zeolitic materials samples synthesized from the ash in the same power plant [10]. ZFB sample presented higher values of pH and conductivity compared to other samples. This result is due to the smaller particle size of this sample, which contributes to the solubilization of a higher quantity of chemical species and is responsible for basicity and higher conductivity of its suspension solution.

The bulk density values followed an order: ZFB > ZFC > ZBA. These results confirm that ZFB sample has a smaller particle size. The specific surface area followed the same order as the bulk density, ranging from 69.5 to 35.2 $\text{m}^2 \text{g}^{-1}$. These results show that the sample with smaller particle size and higher density also presented a higher specific surface area (ZFB).

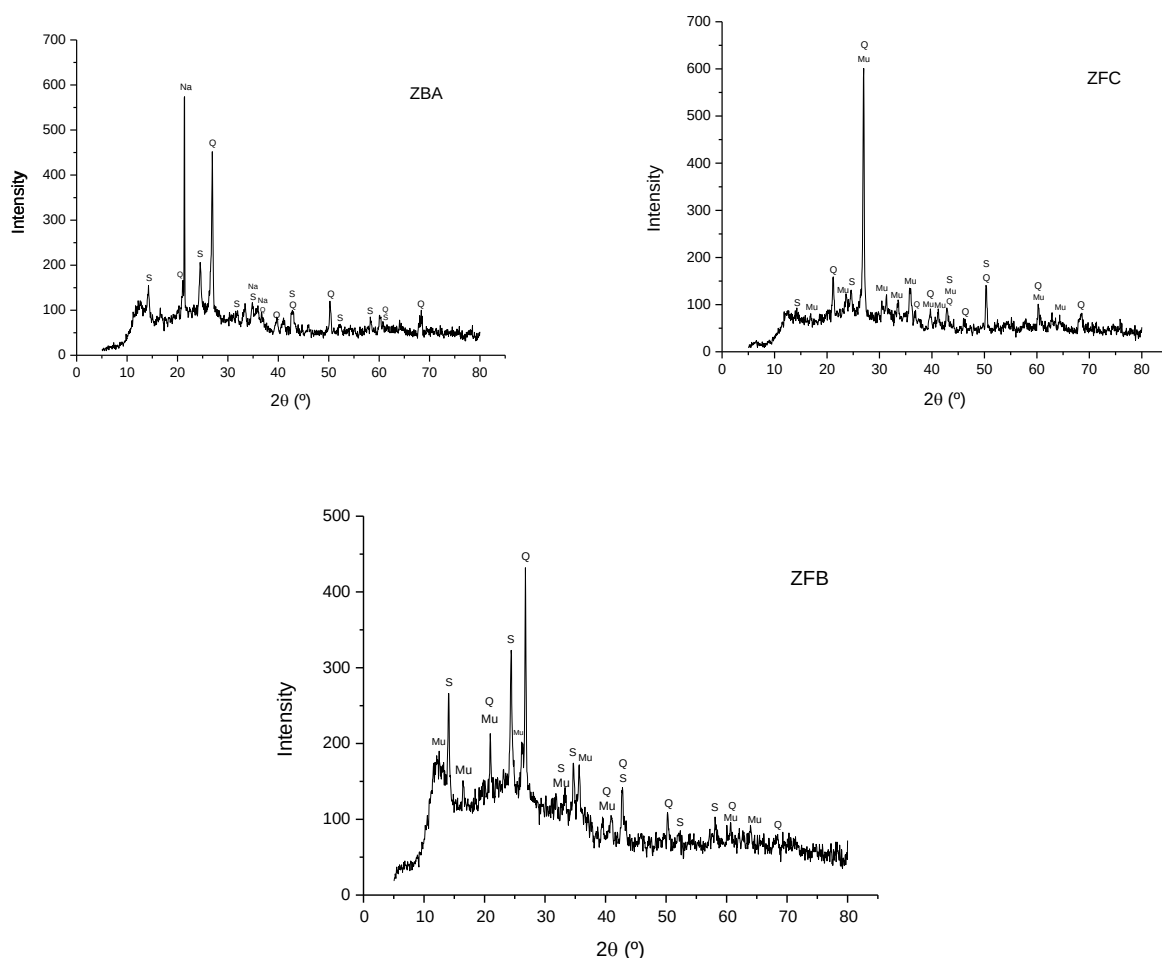


Figure 2. XRD Patterns of zeolitic materials synthesized by hydrothermal alkaline activation of coal ashes (Na = sodium aluminosilicate, Q = Quartz, S = Sodalite and M = Mullite).

Table 1. Compositions (wt%) of zeolitic materials obtained from different coal ashes

Components	ZBA	ZFC	ZFB
SiO ₂	27.3	33.3	33.5
Al ₂ O ₃	16.1	18.0	23.0
Fe ₂ O ₃	13.1	15.2	11.9
Na ₂ O	6.71	5.78	10.7
CaO	3.33	4.57	2.12
SO ₃	3.19	1.08	1.74
TiO ₂	1.25	1.09	1.43
K ₂ O	0.443	0.399	0.383
MgO	0.747	0.909	1.06
ZnO	0.185	0.22	0.65
MnO	0.106	0.124	0.067
Cr ₂ O ₃	<0.001	0.018	0.024
PbO	0.015	0.023	0.07
ZrO ₂	0.103	0.085	0.123
SrO	0.036	0.03	0.034
NiO	0.033	0.015	<0.001
CuO	<0.001	0.015	0.018
Y ₂ O ₃	0.026	0.019	0.037
Cl	0.03	<0.001	<0.001
BaO	0.039	0.035	0.018
Nb ₂ O ₅	<0.001	<0.001	0.003
Ce ₂ O ₂	<0.001	0.037	0.04
LOI*	27.3	19.0	13.1
SiO ₂ /Al ₂ O ₃	1.70	1.85	1.46

*Loss of ignition

The CEC values of zeolitic materials given in Table 3 followed an order: ZFB > ZBA > ZFC. As previously stated, the lower SiO₂/Al₂O₃ ratio is related to the higher cation exchange capacity of these materials. Similar specific surface area and CEC have been reported from other studies on zeolitic material samples synthesized from coal ash in the same origin [10].

3.1.4 Morphology of synthesized products

The scanning electron micrographs (SEM) of the three zeolitic materials are shown in Figure 3.

It can be observed that the crystalline materials (particles with regular morphology) are present in all samples. The same morphology has already been observed in other studies and showed that the

zeolite crystals are deposited on the surface of the ash particles during the hydrothermal activation and therefore have a rough surface [10, 22].

Table 2. Total carbon content (wt%) of zeolitic material samples

Samples	C (%)
ZBA	15.62
ZFC	9.74
ZFB	4.14

Table 3. Chemical and physical characteristics of ZBA, ZFC and ZFB samples

Sample	pH	Cdv ¹	B. D. ²	S _{BET} ³	CEC ⁴
ZBA	9.1	217.0	0.47	35.2	1.60
ZFC	9.2	213.5	0.58	68.0	1.37
ZFB	9.5	296.0	0.63	69.5	2.36

¹Conductivity (μS cm⁻¹); ²Bulk Density (g cm⁻³);
³Surface area (m² g⁻¹); ⁴CEC (meq g⁻¹)

3.2 Treatment of effluents using zeolitic materials

The concentrations of metal elements present in the LL wastewater sample before and after treatment with zeolitic materials (ZFB, ZFC and ZBA), pH and conductivity values are shown in Tables 4, 5 and 6, respectively. The limiting values of metal concentrations and pH set by the current Brazilian legislation [15] for wastewater discharge are also presented in the tables. According to Table 4, the coal ash landfill leachate (LL) presents Cr and As concentrations above the limit, therefore, it needs to be treated prior to discharge. A significant amount of Ca element in the landfill leachate is due to the leaching of the limestone used as a neutralizing layer at the coal ash disposal site (Figure 1).

Table 4 shows that ZFB sample successfully removed Ni, Cd, Zn and Co elements regardless of the ZFB dose, while Mg, Ca, Fe and As removals were higher at the higher zeolitic material dose. ZFB demonstrated high potential removal for arsenic, while it was not efficient for chromium removal at any dosage of zeolitic material. Regarding to Mn, the material is also not efficient for the removal, but the concentrations are below the allowed limit.

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As shown in Table 4, the pH values for the supernatant solutions after the treatment increased with increasing dose of zeolitic material. Thus, it is important to determine if the elements removal was caused by the ion exchange process and/or by precipitation of the elements in the alkaline solution (Moreno et al., 2001). It can be noted that the supernatant solution pH after the treatment at the dose of 20 g L⁻¹ is above the legislation limit, and thereby the dose of 10 g L⁻¹ is good enough for arsenic removal from this effluent.

ZFC sample was found to successfully remove Ni, Cd, Zn, Mg, Ca, Fe and Co elements from landfill

leachate (Table 5). Similar to ZFB, ZFC sample was not efficient for chromium removal at any dose of zeolitic material, however it was satisfactory for arsenic removal at the dose of 10 g L⁻¹. This sample presented the same behavior of ZFB related to pH and conductivity values.

According to Table 6, ZBA sample presented the same behavior for Ni, Cd, Zn, Mg, Ca and Co removals but no removal for Cr and Mn as the other two samples. Moreover, this sample was not suitable for the LL wastewater treatment in consideration of arsenic and iron removals from the solution.

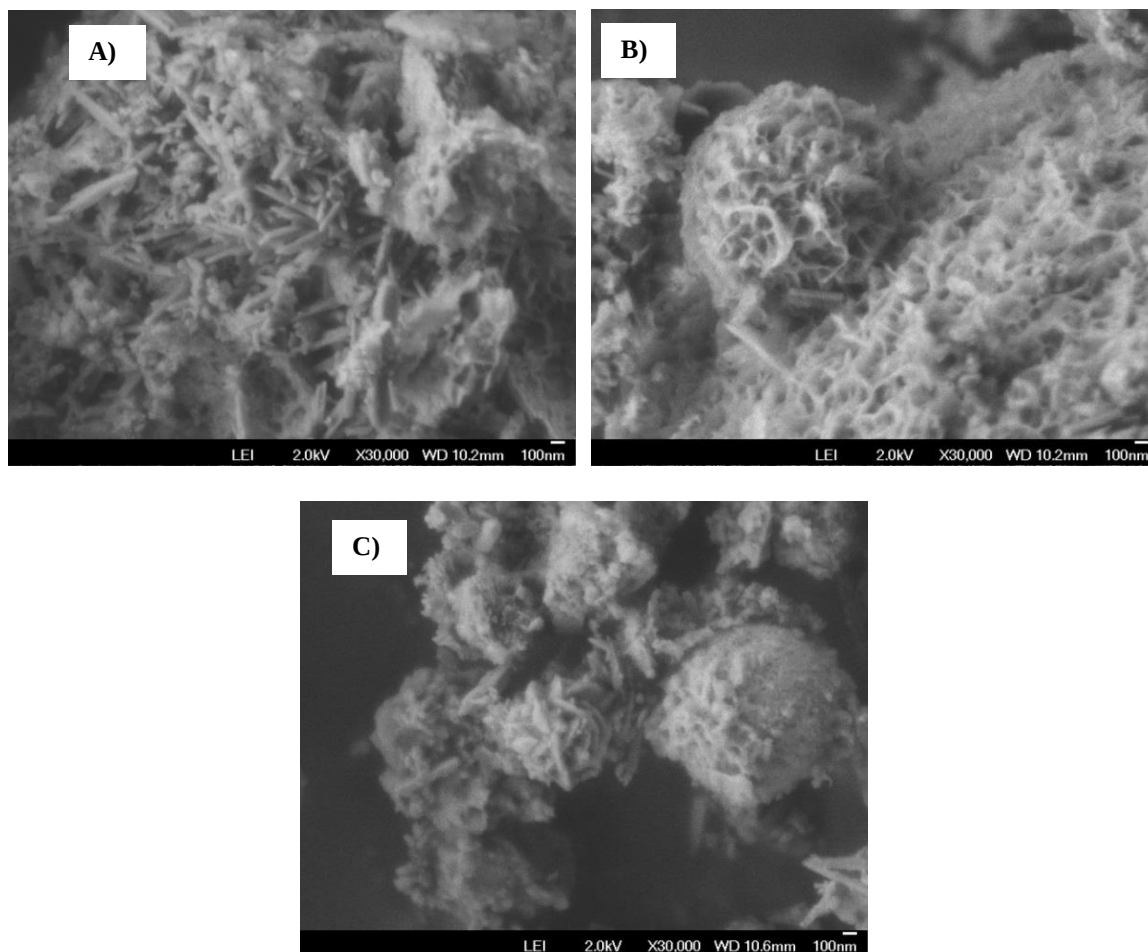


Figure 3. SEM micrographs of zeolitic materials synthesized from different coal ashes by alkaline hidrothermal activation. a) ZBA; b) ZFC; c) ZFB

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Table 4. Analysis of coal ash landfill leachate (LL) before and after treatment with ZFB at different dosage and the allowed limit values

Component	LL	ZFB (mg L ⁻¹)		Limit ¹ (mg L ⁻¹)
	(mg L ⁻¹)	(Dose 10 g L ⁻¹)	(Dose 20 g L ⁻¹)	
Cr	0.15	0.155	0.157	0.1
Mn	0.133	< 0.1	< 0.1	1.0
Ni	1.11	0.012	0.013	2.0
Cd	0.0430	< 0.01	< 0.01	0.2
Zn	0.112	< 0.01	< 0.01	5.0
Mg	5.5	3.23	0.134	-
Ca	1052	193	41.6	-
Fe	0.49	0.243	0.133	15.0
Co	2.0	<0.01	< 0.01	-
As	1.09	0.275	<0.01	0.5
pH	6.43	8.19	11.08	5 a 9
Conductivity ²	106.2	10.85	14.3	-

(1) Established by RESOLUTION No. 430 (May 13th, 2011) of CONAMA [15]; (2) mS/cm

Table 5 Analysis of coal ash landfill leachate (LL) before and after treatment with ZFC at different dosage and the allowed limit values

Component	LL	ZFC (mg L ⁻¹)		Limit ¹ (mg L ⁻¹)
	(mg L ⁻¹)	(Dose 10 g L ⁻¹)	(Dose 20 g L ⁻¹)	
Cr	0.15	0.154	0.157	0.1
Mn	0.133	< 0.1	< 0.1	1.0
Ni	1.11	<0.01	< 0.01	2.0
Cd	0.0430	< 0.01	< 0.01	0.2
Zn	0.112	< 0.01	0.032	5.0
Mg	5.5	0.46	0.292	-
Ca	1052	71	30	-
Fe	0.49	<0.1	0.268	15.0
Co	2.0	<0.01	< 0.01	-
As	1.09	0.128	0.446	0.5
pH	6.43	8.09	11.34	5 a 9
Conductivity ²	106.2	9.9	13.05	-

(1) Established by RESOLUTION No. 430 (May 13th, 2011) of CONAMA [15]; (2) mS/cm

In general, ZFB sample was more effective than the other zeolitic material samples for arsenic removal using different doses. The superiority of ZFB can be related to its higher specific surface

area (Table 3), which should enable a favorable diffusion of arsenate anions into the inner structure of the formed zeolites.

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Table 6 Analysis of coal ash landfill leachate (LL) before and after treatment with ZBA at different dosage and the allowed limit values

Component	LL (mg L ⁻¹)	ZBA (mg L ⁻¹)		Limit ¹ (mg L ⁻¹)
		(Dose 10 g L ⁻¹)	(Dose 20 g L ⁻¹)	
Cr	0.15	0.15	0.15	0.1
Mn	0.133	< 0.1	< 0.1	1.0
Ni	1.11	0.035	< 0.01	2.0
Cd	0.0430	< 0.01	< 0.01	0.2
Zn	0.112	< 0.01	< 0.01	5.0
Mg	5.5	3.46	0.464	-
Ca	1052	186	17	-
Fe	0.49	2.78	0.243	15.0
Co	2.0	0.029	< 0.01	-
As	1.09	0.63	1.043	0.5
pH	6.43	7.65	11.0	5 a 9
Conductivity ²	106.2	8.6	5.01	-

(1) Established by RESOLUTION No. 430 (May 13th, 2011) of CONAMA [15]; (2) mS/cm

The removal of Cr(VI) and As(V) from water by an adsorbent is highly dependent on the pH of the solution, which subsequently affects the surface charge of the adsorbent, the degree of ionization and the speciation of the adsorbate species. The active surface functional groups on the zeolite surface have been well recognized as the silanol (Si-OH) and aluminol (Al-OH) sites. The pH values in all the leachate samples after the treatment with zeolitic materials were about 8 and 11 at adsorbent dosage of 10 g L⁻¹ and 20 g L⁻¹, respectively.

The prevalent forms of arsenate ions are HAsO₄⁻ at pH 8.0, HAsO₄⁻ and AsO₄³⁻ at pH 11.0. It has been confirmed that zeolite terminal aluminol groups are the reactive sites for arsenate anions, not silanol surface groups. Thus, As(V) adsorption will depend on the predominant protonation state of the surface aluminol group and the arsenate oxyanion [23,24].

Usually, there is a decrease in As(V) adsorption at high pH because the adsorption of strong acid anions by metal oxides and hydroxides typically decreases with increasing pH [25]. Furthermore, the surface of the adsorbent becomes more negative with increasing pH, which is not favorable for the adsorption of arsenate anions.

In water, Cr(VI) anion is not a simple monovalent anion but rather a series of chromate

anions depending upon the pH and concentration of the solution. Above pH 6, the dominant species is CrO₄²⁻. In addition, at pH values greater than 6, the presence of OH⁻ ions forms the hydroxyl complexes of chromium [26]. Surface complexation models have been successfully used to describe Cr(VI) ion adsorption onto some solid surfaces [27].

The surface sites for a hydrated zeolitic material can be represented as protonated, neutral and deprotonated surface hydroxyl functional groups, which are S-OH₂⁺, S-OH and S-O⁻, respectively, where S represents both Si- and Al- surfaces [28]. At high pH, the neutral surface hydroxyl functional group, S-OH, is the sole active site providing for CrO₄²⁻ adsorption and the Cr(VI) removal is mainly attributed to the formation of a complex S-CrO₄⁻.

At pH ≥ 8, not only less S-OH functional groups are deprotonated but also more number of OH⁻ competes with the CrO₄²⁻ ions for the active surface sites. Consequently, it is difficult for these species to form complexes. Moreover, as already explained, in the alkaline solutions, surface of the adsorbent became more negatively charged and did not favor the adsorption of anionic Cr(VI) species.

4. Conclusions

Sodalite zeolite was obtained from CCBs in hydrothermal activation. The $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratios of zeolitic materials showed a direct relation with cation exchange capacity (CEC) for all samples. ZFB zeolitic material presented the lowest $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio and the highest CEC and loss of ignition values due to its high content of both inorganic compounds and unburnt carbon. The surface areas and bulk densities of the samples followed the order: ZFB > ZFC > ZBA.

All zeolitic materials showed a significant removal for Ni, Cd, Zn and Co. The concentration of As was below the legislated value after the treatments using different samples. ZFB demonstrated higher

potential removal for arsenic than the other two samples due to its both higher CEC and specific surface area. Chromium was not removed by any of the zeolitic materials and at any of the doses used, therefore further studies are required to understand and promote the removal of this element in this type of effluent. In general, this investigation can indicate a simple approach for self-sustainable control and use of solid waste from power plants.

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