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ACID ROCK DRAINAGE NEUTRALIZATION BY BAUXITE RED MUD: LABORATORY BATCH TESTING

NEUTRALIZAÇÃO DE DRENAGEM ÁCIDA DE ROCHAS COM LAMA VERMELHA DE BAUXITA: ENSAIOS DE BATELADA EM LABORATÓRIO

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RESUMO - A Drenagem Ácida de Rochas (ARD) é uma das questões ambientais mais desafiadoras do mundo. Correções alcalinas nas barreiras para prevenção de ARD ou tratamento alcalino direto de ARD têm sido usados rotineiramente para o manejo de ARD. Por sua vez, a lama vermelha de bauxita (BRM) é um resíduo alcalino proveniente da indústria de alumina, com produção média anual próxima de 120 milhões de toneladas. Portanto, o uso do BRM para neutralização da DRA parece ser uma boa alternativa e deve ser investigado. O principal objetivo deste experimento é avaliar proporções adequadas de BRM para tratar ARD sob a perspectiva dos procedimentos de gestão de resíduos. Assim, foram realizados testes laboratoriais de equilíbrio de lotes de curto prazo utilizando diferentes proporções volumétricas e de peso. A utilização da abordagem volumétrica teve como objetivo simular a mistura da polpa de BRM com as soluções líquidas de ARD, enquanto a abordagem de massa refere-se às misturas de BRM desidratado e ARD. O pH e a porcentagem de remoção de metal, aqui denominados de extração, foram utilizados respectivamente como parâmetros de neutralização e eficiência do tratamento. Os resultados mostraram que o BRM foi eficiente na neutralização da ARD em proporções volumétricas de BRM superiores a 60% e relação sólido (BRM):líquido (ARD) de 1:1,5. Esta relação resulta em valores de extração superiores a 90% para a maioria dos produtos químicos da ARD.
Palavras-chave: Tratamento de Drenagem Ácida de Rochas. Lama Vermelha de Bauxita. Neutralização. Reuso. Testes em lote. Resíduo de Alumínio.

ABSTRACT - Acid Rock Drainage (ARD) is one of the world's most challenging environmental issues. Alkaline amendments to barriers for ARD prevention or direct ARD alkaline treatment have been routinely used for ARD management. In turn, bauxite red mud (BRM) is an alkaline residue from the alumina industry, with annual average production close to 120 million tons. So the use of BRM for ARD neutralization seems to be a good alternative and must be investigated. The main objective of this experiment is to assess suitable proportions of BRM to treat ARD under the perspective of waste management procedures. So, laboratory short-term batch equilibrium tests were carried out using different volumetric and mass proportions. The use of a volumetric approach was intended to simulate the mixture of BRM pulp with the ARD liquid solutions, while the mass approach refer to the mixtures of dewatered BRM and ARD. The pH and metal removal percentage, called here as extraction, were respectively used as neutralization and treatment efficiency parameters. The results have showed that BRM was efficient for ARD neutralization on BRM volumetric proportions greater than 60% and solid (BRM): liquid(ARD) ratio of 1:1.5. This ratio result on extraction values greater than 90% for most of the ARD chemicals.
Keywords: Acid Rock Drainage Treatment. Bauxite Red Mud. Neutralization. Reuse. Batch Testing. Aluminum Residue.

INTRODUCTION

Acid Rock Drainage (ARD) is generated by exposing sulfide minerals to weathering elements, such as oxygen, water, and acidophilic bacteria. Rock excavations for mining and civil construction can accelerate this phenomenon very much, creating one of the world's most challenging environmental issues (Tuffnell, 2017). ARD solutions carry a great load of metals besides the acidity itself, that can contaminate surface and underground water.

In many ways, engineered barriers such as covers and liners, have proved to be effective on ARD attenuation (INAP, 2009). They are classified as a passive method, preventing or reducing ARD generation in a cost-effectively way, especially for mine post-closure applications (Australia, 2016).

Alkaline amendment can be very useful for these barriers since immediate acid neutralization is provided. Besides neutralization, increasing the alkalinity serves to reduce or to stop: (1) the rate of sulfide oxidation; (2) the Fe^{3+} dissolution and (3) the acidophilic bacterial growth (Duchesne & Doye, 2005). Commercial alkaline materials include hydrated lime [$Ca(OH_2)$], quick lime (CaO), limestone ($CaCO_3$), liquid and solid caustic soda (NaOH) and others (INAP, 2014; Thisani et al., 2021). The possibility of using industrial by-products of alkaline nature to neutralize ARD is quite appealing, since the destination will be addressed for two different hazardous solid and liquid wastes.

Research on the application of alkaline industrial solid wastes as covers and liners include metallurgic slag (Simmons et al., 2002; Hamilton et al., 2007; Salviano & Leite, 2014; Pereira de Almeida et al., 2015 and others) and Bauxite Red Mud – BRM (Duchesne & Doye, 2005; Sutar et al., 2014; World Aluminum, 2015). BRM is an alkaline residue from the bauxite processing (Bayer Process) for alumina (Al_2O_3) production. Typically, 0.3 to 2.5 tonnes of BRM are produced for each ton of alumina (World Aluminum, 2015). The world's annual production is estimated to be 120 million tonnes (Shinzato et al., 2009; World Aluminum, 2015), which makes BRM one of the greatest industrial by-products. Usually, BRM is mainly composed by metallic oxides (Fe, Al and Ti), SiO_2, CaO and Na_2O with pH values usually ranging from 10 to 13 due to the addition of NaOH in the Bayer Process (Schmitz, 2006).

As pointed out by Evans (2016), the solid proportion of BRM has intentionally been increased from around 20% in the past to near 80% nowadays by filter-pressing it. So, BRM disposal methods have changed from pumped slurry disposal on natural water (rivers, lakes, and seas) or constructed impoundments (dams and levees), to much drier stocking, such as landfills and stockpiles. Despite this improvement on the BRM's mechanical behavior, in its natural state BRM is still very alkaline and harmful to the environment, forcing waste managers to neutralize it with acid solutions before disposal, which can be very expensive. So, the option of using ARD to neutralize BRM has both an economic and an environmental appeal.

Doye & Duchesne (2003) used batch experiments (10 to 20 rpm) to investigate the neutralization of sulfidic mine tailings (pH around 2.5) by BRM and other alkaline residues. They found that the volumetric proportions of 2% and 5% of BRM were not sufficient to neutralize the reactive tailings, and the proportion of 10% had initially raised the pH to 8, decaying to 4 after one year of rotation. Paradis et al. (2006) conducted batch experiments with mixtures of RMB with an acidic mine tailings sample with original pH of 3.29. The percentages of 0, 2, 3.5, 5.8, 7.8 (dry weight basis) of BRM have been applied in mixtures with the acid tailings sample. These mixtures and deionized (solid:liquid ratio of 2:1) were rotated at 10 to 20 rpm for 6 months. They found neutralization with BRM proportions above 3.5%. Taneez & Hurel (2019) present a good review on water remediation and metal removal by using BRM. Despite using the same laboratory approach (batch test), these investigations applied different laboratory procedures, sometimes unrealistic considering the waste management practice.

The main objective of this experiment is to assess suitable proportions of BRM to treat ARD under the perspective of waste management procedures. So, laboratory short-term batch equilibrium tests were carried out using different volumetric and mass proportions. The use of a volumetric approach was intended to simulate the mixture of BRM pulp with the ARD liquid solutions, while the mass approach refer to dewatered BRM. The pH and metal removal percentage, called here as extraction, were respectively used as neutralization and treatment efficiency parameters.

MATERIALS AND METHODS

Acid Rock Drainage (ARD): generation and characterization

The ARD sample was generated by percolating distilled-deionized water (DDW) through an acidic soil sample using a leaching column. This soil was collected on a residual soil outcrop derived from phyllite rocks of an abandoned pyrite (FeS_2) open mining pit, in the vicinity of Ouro Preto - MG, Brazil. According to Martins (2005) and Moraes (2010), pyrite had been extracted from fresh rocks of this site between 1930 and 1960 for commercial sulfuric acid production. Nowadays, a huge gully took place at the pit, releasing a very acidic effluent downward to the Carmo River.

In the field, the soil pH was approximately 2.5 as indicated by Merck pH test strips.

Soil collection and preparation was done according to ordinary disturbed sampling procedures for geotechnical testing (ABNT-NBR 6457/86), which mainly consist of hand excavation, identification and packing in plastic bags. In the laboratory, the samples were dried at room temperature to hygroscopic moisture, homogenized and reduced. Table 1 summarizes the acidic soil properties before the leaching procedures.

Table 1 - Acidic soil properties before the leaching procedure.

Property-Method	Value
Grain Size (φ) – ABNT-NBR 7181/84	
Clay (ϕ < 0,002 mm)	11%
Silt (0,002 < ϕ < 0,075 mm)	10%
Fine Sand (0,075 < ϕ < 0,42 mm)	11%
Medium Sand (0,42 < ϕ < 2,0 mm)	28%
Coarse Sand (2,0 < ϕ < 4,75 mm)	21%
Gravel (4,75 < ϕ < 60,0 mm)	29%
Liquid Limit – ABNT-NBR 6459/84	39.3%
Plastic Limit – ABNT-NBR 7180/84	27.6%
Plastic Index	11.7%
Density of Solids (g/cm^3) – ABNT-NBR 6508/84	2.915
Soil pH at 1:2.5 soil:solution	1.9
Saturated Hydraulic Conductivity (cm/s) ABNT-NBR 13292/95	1.0 x 10^{-3}
Porewater Electrical Conductivity at 1:1 soil:solution (mS/cm)	17.0

The chemical composition of this acid soil sample was investigated (acid digestion) by Pereira de Almeida (2016), as presented on Table 2. It can be highlighted the high content sulfur (3.04% - which corresponds to 6.07% of SO_2), iron and aluminum oxides. In terms of mineralogy, quartz, pyrite, muscovite, and kaolinite dominate this sample.

Table 2 - Chemical composition (weight basis) of the acidic soil sample (Source: Pereira de Almeida, 2016).

Oxide	(%)	Oxide	(%)
SiO_2	18.45	Cr_2O_3	0.0603
Fe_2O_3	36.27	MnO	0.06
Al_2O_3	31.01	ZrO_2	0.034
SO_2	6.07	As_2O_3	0.0261
TiO_2	2.25	CuO	0.0234
K_2O	1.24	NiO	0.0184
MgO	1.05	Sc_2O_3	0.0113
Na_2O	0.83	CoO	0.0063
CaO	0.54	Y_2O_3	0.0045
P_2O_5	0.27	ZnO	0.0016
BaO	0.0827	SrO	<LOQ
V_2O_3	0.0696	ThO_2	<LOQ

LOQ: Limit of QuaLOQ: Limit of Quantification.

The leaching column was manufactured using plexiglass and PVC. It basically consisted of a 1-cm thick plexiglass pipe, with 15.2 cm of internal diameter and 104.0 cm in length. Perforated PVC plates were adapted at the top and the bottom of the columns for percolation, along with non-woven geotextiles.

The column itself was fixed by iron rods and screws. A twenty-liter barrel served for DDW storage and feeding device for the leaching procedure.

About 20 liters of ARD were generated during seven days of percolation. Table 3 presents the average chemical composition of this leachate determined by Inductively Coupled Plasma – Optical Emission Spectrometry ICP-OES (Varian 725-ES), as well as some physico-chemical parameters. It can be observed that the maximum concentration level (MCL) was trespassed for many elements according to a wastewater discharge standard, reflecting the very acidic nature of this leachate, as a typical ARD.

Table 3 - ARD chemical composition (mg/L) and physico-chemical parameters.

Al	As	Ca	Cd	Cu	Fe	Mg	Mn	Ni	P	Pb	S	Zn
1005.36	16.34	465.56	4.73	3.78	12322.22	52.9	8.41	18.63	42.44	8.71	35639.67	6.38
Wastewater Discharge Standard (COPAM/CERH-MG, 2008)												
---	0.2	---	0.1	1.0	15.0	---	1.0	1.0	---	0.1	1.0	5.0

Physico-Chemical Parameters		
pH	**Eh (mV)**	**Electrical Conductivity (mS/cm)**
1.80	639	16.84

Bauxite Red Mud (BRM) characterization

The BRM sample came from the alumina industry Hindalco, Ouro Preto, Brazil. One hundred kilograms were randomly collected before the filter-press process, with an initial solid:liquid proportion close to 1:1.5 (39% of solids), according to the manufacturer. For primary characterization, the sample was firstly dried for two days using infrared lamps, to be reduced to 10 kg on a small-scale splitter.

The chemical composition of the BRM was determined by mixing 0.2 g of this dried sample with 3 g of a 2:1 blend of sodium carbonate (99.5%) and sodium tetraborate (99.5%). This mixture was homogenized and melted in a platinum crucible at 1000 °C for 30 min. After melting, the mixture was cooled on a desiccator and then dissolved in a 16% hydrochloric acid solution and the chemicals were analyzed by Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES – Varian 725-ES).

The results are presented on Table 4, which shows Fe, Si, Al and Ca as major elements, as expected.

Table 4 – Initial BRM chemical composition.

Element	Quantity
$FeOOH + Fe_2O_3$ (%)	66.07
SiO_2 (%)	16.29
Al_2O_3 (%)	11.90
CaO (%)	4.10
Na_2O (%)	0.64
Pb (ppm)	2639
Mn (ppm)	1888
Ni (ppm)	332
Cr (ppm)	328
Zn (ppm)	112
As (ppm)	170

The BRM mineralogical composition was investigated by x-Ray Diffraction – XRD (D2 Phaser – Bruker), and Scanning Electron Microscopy (SEM - Tescan – VEGA3 LMH), coupled with Energy Dispersive Spectroscopy (INCA – 51ADD-0007). The x-ray patterns were interpreted using the database standards of the Diffract.EVA software and the Rietveld refinement method (TOPAS 5.0 software).

XRD results are presented on Figure 1 and Table 5, which are similar to other BRM compositions found in the literature (Doye & Duchesne, 2003;

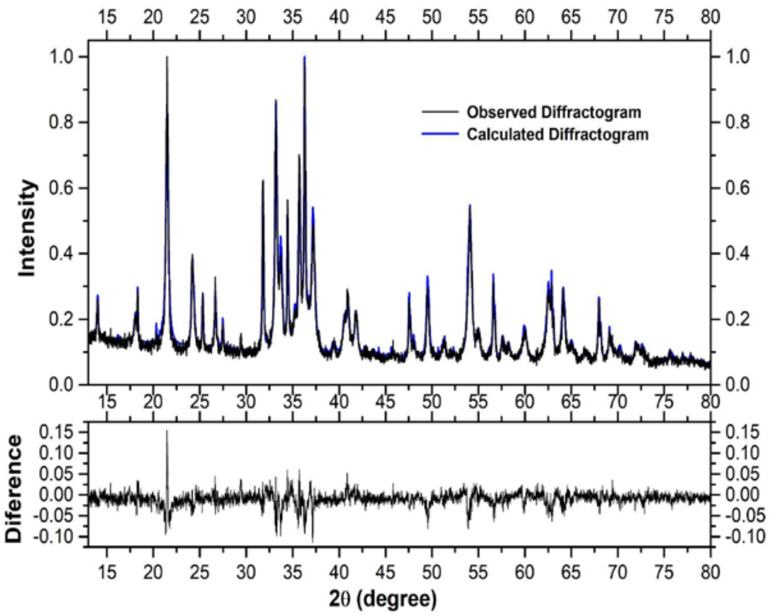

Figure 1 - Red mud X-ray diffraction (XRD).

Table 5 - BRM mineralogy obtained by XRD.

Mineral	Percentage
Goethite [FeOOH]	28.39
Hematite [Fe_2O_3]	24.01
Sodalite [$Na_8Al_6Si_6O_{24}Cl_2$]	2.52
Anatase [TiO_2]	1.89
Gibbsite [$Al(OH)_3$]	1.74
Rutile [TiO_2]	1.41
Quartz [SiO_2]	1.24
Portandite [$Ca(OH_2)$]	0.35
Davyne [$Na_4K_2Ca_2Si_6Al_6O_{24}(SO_4)Cl_2$]	0.33
Lime [CaO]	0.20
Amorphous	37.92

Bertocchi et al., 2006; Klauber et al., 2009; Evans, 2016). As can be seen, it is dominated by iron oxides/hydroxides and by an amorphous phase.

The SEM image of Figure 2 (a,b) shows angular particles of 50 to 500 µm. Some elements identified by EDS (Figure 2b) corroborate the chemical analysis of Table 4 and the XRD patterns (Table 5). An exception is made to the element Ti, which was present on the XRD as anatase and rutile, as well as in the EDS, but it was absent in the BRM chemical analysis. Maybe this absence was related to sample heterogeneities.

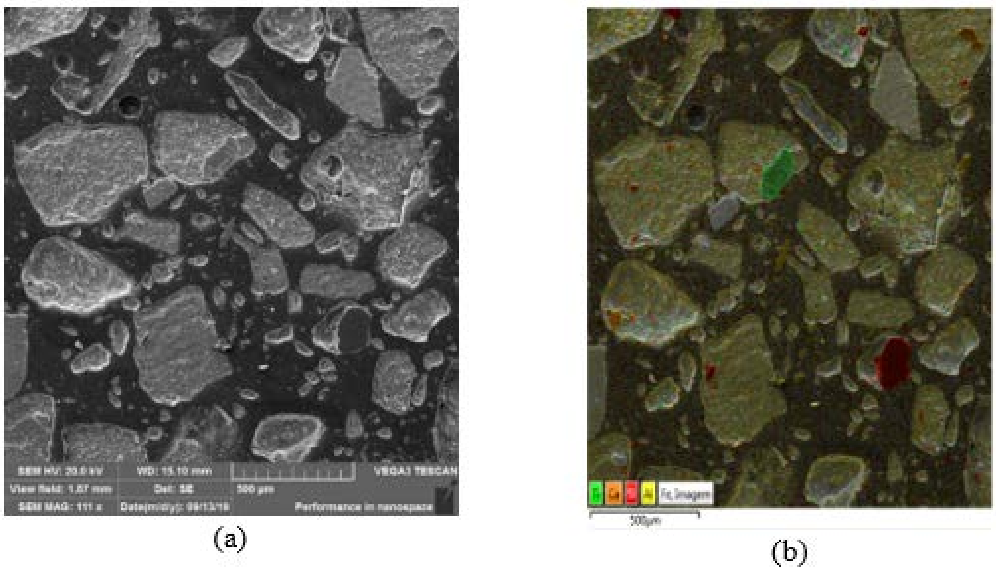

Figure 2 - (a) Bauxite Red Mud SAM image; (b) Chemicals determined by EDS.

Batch Testing: volumetric approach

These tests consisted of rotating BRM and ARD samples using different volumetric proportions in 200 mL polyethylene flasks, under 140 rpm for 21 hours.

The BRM sample came directly as pulp from the industry, and it was only submitted to a homogenization on a mechanical stirrer before rotating. The pulp and the ARD initial properties, before rotation, are depicted on Table 6.

After fulfilling the flasks with BRM and ARD, they were completely sealed and immersed in tap water, which was maintained at an average temperature of 25 ºC during rotation. The tests were run in duplicate using four solids percentage (10%, 30%, 60% and 80%), totalizing 8 flasks plus 1 flask with only ARD solution, called here as "blank".

Table 6 - BRM and ARD initial properties.

BRM Property	Value	Method
Bulk Density	1.395 g/cm^3	Volumetric
Solids Density	3.860 g/cm^3	ABNT-NBR 6508/84
Humidity Ratio by Volume	61%	Oven dried
Solids Percentage	39%	Oven dried
pH (Solid:Deionized Water = 1:2.5)	12.30	EMBRAPA (2017)
ARD Property	**Value**	
pH	1.85	
Eh	630 mV	
Electrical Conductivity	18605 mS/cm	
Temperature	19ºC	

Batch Testing: mass approach

The BRM sample preparation consisted of oven drying it at 134 ºC for 4.5 hours, followed by mechanical homogenization and sieving through a 600 μm mesh. The operational procedures for these tests were similar to the volumetric approach, except that the period of rotation was 30 hours and, instead of volumetric, mass proportions of dry solid (BRM) and solution (ARD) were applied, as presented on Table 7, which also presents the physico-chemical properties of the ARD before the contact with the BRM sample.

Batch Testing: Constant Soil:Solution approach

Considering the results obtained in the Mass Approach procedures, one polyethylene flask was filled with 66.7 g of dried BRM and 100 mL of ARD, making up a soil: solution ratio of 1:1.5. This flask was then rotated to 140 rpm for 30 hours. Aliquots of 2 mL of ARD were extracted from this suspension at predetermined times. After, these suspensions were centrifuged at 5.000 rpm and diluted with a HCl solution (1:1) for chemical analyses using the same method applied for the ARD initial characterization.

The efficiency on removing the chemicals from the ARD after contacting the BRM sample was calculated by the percent reduction in its concentration after rotation, referred here as *Extraction* (%), as shown in equation (1), where C_o and C_f refer to the ARD initial and final concentration, respectively.

$$Extraction\ (\%) = \frac{C_0 - C_f}{C_0} \times 100 \quad (1)$$

Table 7 - Solid: Solution ratio used for batch testing (mass approach) and the ARD properties.

Solid : Solution	BRM Dry Mass (g)	ARD Mass (g)	ARD Properties
1 : 1.5 (duplicate)	66.7	100.0	pH = 1.75
1 : 2.5 (duplicate)	40.0	100 .0	EC = 16,879 mS/cm
1 : 5.0 (duplicate)	20.0	100.0	Eh = 639 mV
1 : 7.0 (duplicate)	14.3	100.0	
"Blank"	0.0	100.0	

RESULTS AND DISCUSSIONS

Batch Testing: Volumetric Approach

Figure 3 depicts the pH evolution for the different BRM/ARD volumetric proportions. It can be observed that the neutrality was only obtained for the proportions of 60% and 80% of BRM. Assuming that the equilibrium has been established after 5 hours (see Figure 3), the pH variation was plotted against the BRM proportion (Figure 4), which reveals a linear trend (R^2 = 0.9807). The interpolation of the regression equation presented on Figure 4 states that the BRM proportion of 42.6% of BRM yields a pH = 7.

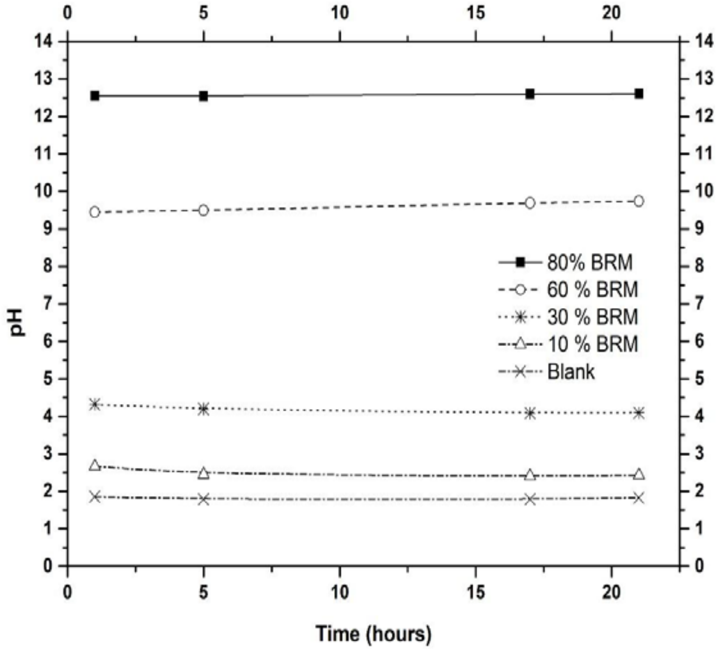

Figure 3 - pH evolution for different BRM proportions.

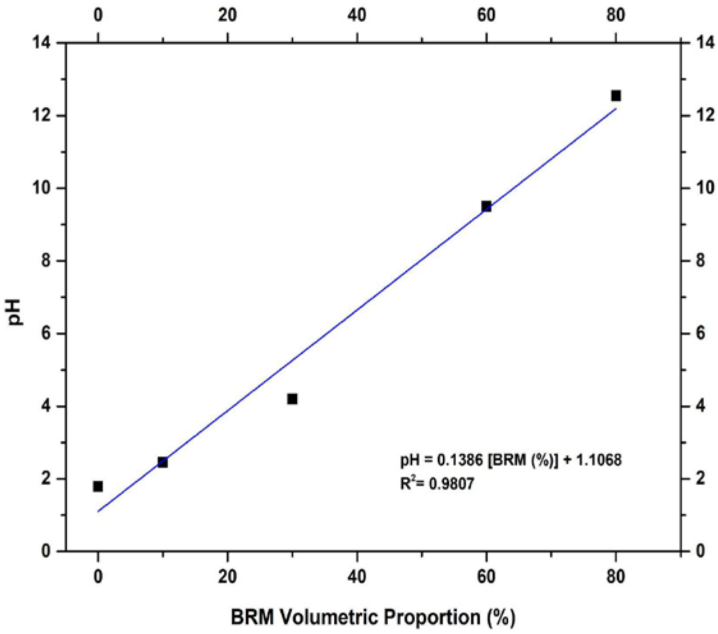

Figure 4 - BRM volumetric proportion versus pH for 5-hour batch results.

Batch Testing: mass approach

As shown in Figure 5, an almost steady pH was achieved within 24 hours of rotation for all solid: liquid ratio, and a pH above 7.0 was only observed for the 1:1.5 ratio. In terms of Eh (Figure 6), a significant reduction was observed only for 1:1.5 and 1:2.5 solid: liquid ratio, shifting the suspension from oxidative to reducing conditions. It can be seen also that Eh equilibrium was achieved within three hours of rotation.

Batch Testing: Constant BRM:ARD Ratio

Extraction data for the BRM(solid): ARD(liquid) ratio of 1:1.5 are presented in Table 8, along with the corresponding values of the Brazilian Effluent Standard CONAMA 430/2011. Extraction above 90% (94.83 to 100.00%) was found for all elements, exceptions made to Ca (35%) and Zn (29.99%). Effluent discharge unconformities were found for Fe and Zn. It is believed that these unconformities are not so significant, and a little dilution effort would bring these concentrations to conformity.

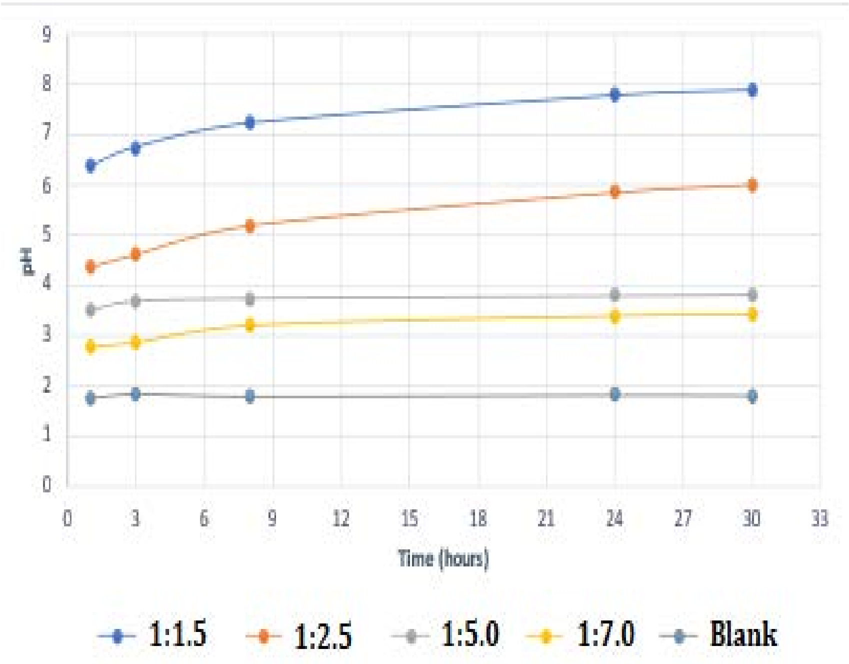

Figure 5 - pH evolution with different BRM (solid): ARD(liquid) mass proportions.

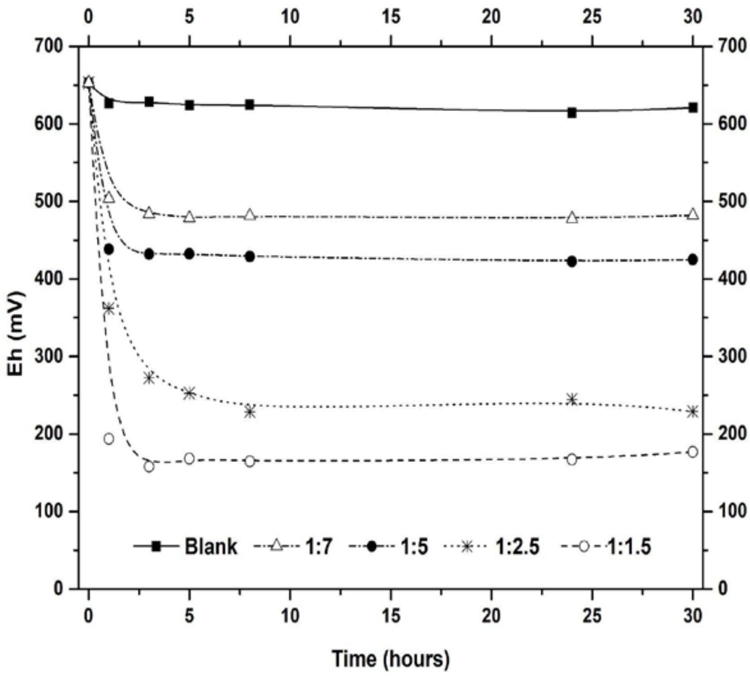

Figure 6 - Eh evolution with different BRM (solid):ARD(liquid) mass proportions.

Table 8 - Extraction data for ARD after contacting the BRM sample (BRM:ARD ratio of 1:1.5).

Element	**Initial Concentration C_o (mg/L)**	**Final Concentration C_f (mg/L)**	***Extraction* (%)**	**CONAMA 430/11 (mg/L)**
Al	1069.22	1.27	99.88	—
As	16.30	0.15	99.10	0.5
Ca	511.88	330.41	35.45	—
Fe	12784.29	54.46	99.57	15.0
Mg	54.75	2.83	94.83	—
Mn	8.40	0.30	96.42	1.0
Ni	18.97	0.00	100.00	2.0
Zn	8.50	5.96	29.99	5.0

Figure 7 presents the extraction evolution over 8 hours of rotation for all the chemical elements of Table 8. It can be observed that Al, As, Fe, Mn and Ni presented a complete extraction (100%) within the first hour of rotation, while the Mg concentration reached the equilibrium (extraction of 94.83%) after 3 hours of rotation.

As mentioned above, the Ca and Zn extraction amounts were lower than the others. The Ca extraction varied between 30 and 40% over 8 hours, while the extraction of Zn almost instantly reached 70 %, subsequently decaying to 30%. These variations may be related to precipitation of these cations as new solid phases.

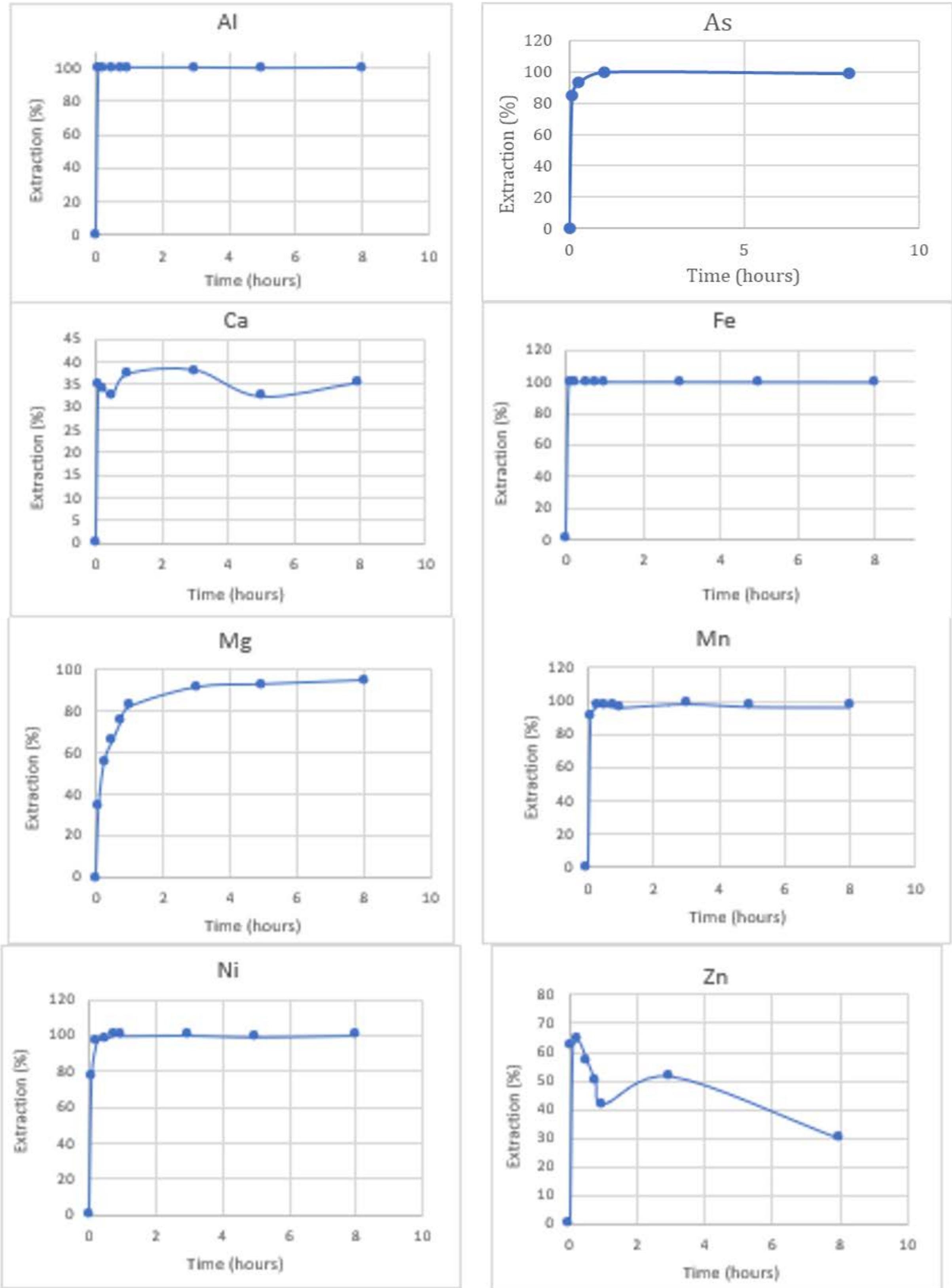

Figure 7 - ARD elemental extraction over 8 hours of batch testing.

Table 9 presents a comparison of the chemical characterization of the BRM sample before (Table 4) and after contacting the ARD solution. No significant changes were observed for the major oxides. As for the trace chemicals, while Cr and As disappeared from theses solids, a substantial increase in the Ni, Mg and Zn has taken place. As these variations can also be related to sample heterogeneities, it is not conclusive that reactions between the BRM solid sample and ARD liquid are responsible for these variations.

Table 9 - BRM chemical concentrations before and after contacting the ARD solution.

Element	Initial Concentration	Final Concentration
FeOOH + Fe_2O_3 (%)	66.07	65.25
SiO_2 (%)	16.29	15.45
Al_2O_3 (%)	11.90	12.02
CaO (%)	4.10	6.30
Na_2O (%)	0.64	—
Pb (ppm)	2639	1260
Mn (ppm)	1888	1760
Ni (ppm)	332	1020
Cr (ppm)	328	—
Zn (ppm)	112	1290
As (ppm)	170	—
Mg (ppm)	—	1290

CONCLUSIONS

As proposed, this paper assessed the efficiency of the neutralization and chemical extraction of an acid rock drainage (ARD) sample given by its mixture with bauxite red mud (BRM). Laboratory short-term batch tests were performed using volumetric and mass approaches and main results are presented next:

- Only the volumetric proportions of 60 % and 80 % of bauxite red mud (BRM) resulted in suspensions with pH values above 7.0. The linear correlation between the BRM volumetric proportion and the pH of the acid rock drainage (ARD) showed that 42.6% of BRM yields a pH=7;
- In terms of mass proportion, the BRM (solid): ARD (liquid) ratio necessary to elevate the pH above 7.0 was the 1:1.5;
- Using the solid (BRM): solution (ARD) ratio of 1:1.5 (weight basis) resulted in extraction amounts of over 90 % for all ARD dissolved ions, exceptions made to Ca (35 %) and Zn (30 %), showing the BRM efficiency as a treatment process to ARD;
- Particularly for the major oxides, the BRM sample presented no significant changes on its solid chemical composition after contacting the ARD solution.

For future research, some other BRM samples should be tested, as well as the chemical speciation of some key elements in the ARD (As, for instance) after contacting the BRM should be investigated.

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