SYNTHESIS, CHARACTERIZATION AND SORPTION PROPERTIES OF A SORBENT SYNTHESIZED USING SLAG AND RED MUD: ARSENIC REMOVAL FROM SPIKED AQUEOUS SOLUTIONS

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ABSTRACT

The aim of the present study was to synthesize a sorbent, for arsenic removal from aqueous solutions, reusing two waste materials (slag and red mud). The sorbent was prepared after chemical and thermal treatment, during which amorphous silica sol and FeOOH sol were produced simultaneously and form Fe-Si complexes on the surface of the slag. To characterize the sorbent Powder X-ray diffraction (XRD); Fourier transform infrared (FTIR) spectroscopy and Scanning Electron Microscopy (SEM-EDX) were applied. The surface area (BET method) and the Point of Zero Charge (PZC) of the sorbent were determined.). The sorption efficiency of the sorbent produced was investigated with kinetic and equilibrium studies, performed in batch conditions. The concentration of arsenic in solutions was determined by electro thermal atomic absorption spectroscopy (GF-AAS). The results of the study showed that with the described process, using metallurgical wastes, iron oxyhydroxides were "loaded" onto slag producing an effective sorbent for arsenic removal. Kinetic experiments proved that equilibrium was achieved within 15 hours, while the maximum adsorption capacity as evidenced by equilibrium experiments was 16.14 mg g^{-1}. Data proved to fit better to the Langmuir equation.

Keywords: red mud, slag, iron oxyhydroxides, sorption, arsenic, waste water

1. Introduction

Several anthropogenic activities, such as mining, electric power generation from fossil fuels, ore smelting, and intensive cultivation, introduce arsenic in aquatic environments or soil. Regardless its source once mobilized arsenic tends to accumulate in living organisms, resulting in various diseases or disorders (Fu and Wang, 2011). Arsenic being toxic and carcinogen, is a hazard of prime interest and increasing significance; as far as surface and ground water are concerned, it can be considered among the chief pollutants. Several techniques have been proposed for arsenic removal from aqueous systems aiming in reducing its concentration in order to minimize impacts and meet strict legislative standards (WHO, 1993). Conventional techniques for the arsenic removal from aquatic environment such as ion exchange, chemical precipitation, membrane techniques, filtration, electrolytic or liquid extraction, and reverse osmosis (Fu and Wang, 2011; Qdais and Moussa, 2005; WHO, 1993) are in several cases technically complicated and/or economically unfavourable. Among the alternative techniques that have been developed for arsenic removal from aqueous systems sorption is very popular since it is both effective and inexpensive. Among the numerous substances (Dakiky *et al.*, 2002; Gode and Pehlivan, 2005; Jochova

et al., 2004; Karabulut *et al.*, 2000; Kwon *et al.*, 2010; Lacour *et al.*, 2001, Pehlivan and Arslan, 2007) that have been tested as sorbents, iron oxides and oxyhydroxides, such as amorphous hydrous ferric oxide (FeOOH), goethite (a- FeOOH), and hematite (a-Fe_2O_3), proved to be very effective (Raul *et al.*, 2014, Gupta *et al.*, 2010; Hua *et al.*, 2012; Thirunavukkarasu *et al.*, 2002). However, most iron oxides are very fine materials being, therefore, difficult to separate from the aqueous phase after treatment. To overcome this difficulty several researchers investigated the possibility of "loading" iron oxides and oxyhydroxides onto sand or slag Ilic *et al.*, 2014, Martinez-Cabanas, 2015, Zhang and Itoh, 2005, Thirunavukkarasu *et al.*, 2002) producing in that way a sorbent that can be easily separated from the aqueous phase.

The aim of this study is to test the "loading" of FeOOH on the surface of metallurgical slag, using red mud as iron source and producing in that way an effective sorbent, easy to handle. Both metallurgical slag and red mud are common metallurgical wastes. Some researchers have proposed a number of applications in order to recycle them, such as environmental remediation, metal recovery, and production of various materials including building materials, polymers and ceramics (Amorim *et al.*, 2012; Bhatnagara et al, 2011; Costa *et al.*, 2010; Liu *et al.*, 2009; Liu and Zhang, 2011; Paramguru et al, 2005; Pontikes and Angelopoulos, 2013). The advantage of the procedure proposed in this study is quite obvious since it combines recycling with the synthesis of a high added value material, efficient for environmental remediation, contributing in that way to sustainable development.   .

2.   Materials and methods

2.1 Materials

The slag sample used in this study was electric arc furnace steel slag from Larco GMM S.A., Greece while the red mud sample used as iron source for the sorbent synthesis was obtained from Aluminium S.A., Greece. Both slag and red mud are characterized, according the Commission decision 2000/532/EC, as non-hazardous wastes. The metallurgical slag was crashed and sieved to a uniform size (0.25- 0.5mm) and dried at 105 °C for 24h before use. The red mud was treated with concentrated HCl and HNO_3 acids to produce the solution used as iron source for sorbent synthesis. By this digestion all red mud components except silica are dissolved and Fe(III) is expected to be the iron species in solution. All reagents used were of analytical grade. The chemical analysis of the two raw materials was performed by X-ray fluorescence (XRF) spectroscopy (S2 Ranger, Bruker). Crystalline compounds in slag were identified with an X-ray diffractometer (XRD) model D8 Advance of Bruker AXS, with application of Cu Kα radiation and nickel filter (U=35kV, I=35mA). The XRD scans were performed between 2 and 70 2θ°, with increments of 0.02° s^{-1}. DIFFRAC plus Evaluation software system by Bruker AXS and JCPDS database were applied for data processing and identification of crystalline components. Morphology, texture, and composition of the metallurgical slag were studied by scanning electron microscopy (SEM–EDX, Jeol 5400) equipped with an energy dispersive X-ray analyzer (INCA energy 300). High vacuum evaporation (JEE-4X, Jeol) was applied to form a thin carbon film to make the surface of the specimens electrically conductive. The specific surface area of the slag was determined with Nova 2200 apparatus (Quantchrom), according to the BET method.

2.2 Synthesis of the sorbent

The sorbent (S-RM) was prepared after chemical and thermal treatment, described elsewhere (Fu *et al.*, 2007; Swedlund and Webster, 2005; Zahng and Itoh, 2005) using as iron source the solution produced after acid treatment of the red mud. During the procedure amorphous silica sol and FeOOH sol are produced simultaneously forming, after treatment, Fe-Si complexes on the surface of the slag. The procedure followed was: 50 g of slag were contacted with 100 mL of 1M NaOH for 2 h and aged at 80 °C for 48h. Then an iron solution, produced by the dissolution of red mud, containing 1 M Fe was added until the pH value of the slurry was ~7. The slurry was magnetically stirred for 2h and aged for 12h at room temperature, then filtrated and heated at 105 °C for 2h and then at 550 °C for 1h. Upon cooling the

produced sorbent was vacuum-dried at 105 °C for 24h and stored in a capped bottle. For comparison reasons another sorbent (S-I) was prepared using a 1 M FeCl₃ solution as iron source, and following the same procedure; in addition, for the same purpose, amorphous FeOOH was prepared mixing 1 M NaOH and 1 M FeCl₃ solutions, aging for 12h and drying at 105°C for 24h.

2.3 Sorbent characterization

In order to compare S-RM with S-I their morphology, texture and composition were studied by scanning electron microscopy (SEM) using the equipment described for the slag, and crystalline compounds present in sorbents were determined by Powder X-ray diffraction (XRD) following the procedure described for the slag. In addition and for the same purpose Fourier transform infrared (FTIR) spectroscopy was applied and the complexes and the iron oxyhydroxides synthesized were identified. The FTIR spectra were obtained, in the range between 400 and 4000 cm^{-1}, using the Spectrum 1000, spectrometer by Perkin Elmer, while the samples were pressed in discs after mixing with KBr. The specific surface area of the sorbents was determined with Nova 2200 apparatus (Quantchrom), according to the BET method. The point of zero charge of the sorbents was measured based on the pH metric titration described by Babic *et al.* (1999).

2.4 Sorption experiments

The sorption capacity of the sorbents synthesized was examined with kinetic and equilibrium studies. The effect of contact time, and metal initial concentration on the sorption process were studied. All experiments were conducted in batch conditions using spiked aqueous solutions as sorbate. Na_2HAsO_4 (analytical grade) was used to prepare sorbates and As(V) is expected to dominate in the solution. All experiments were carried out in duplicate and mean values were used. Duplicate data differed from mean values by less than 2%.

2.4.1   Kinetic experiments

Batch experiments were conducted, for both sorbents (S-I, S-RM), at room temperature (25 °C), in plastic containers, under continuous stirring. Sorbent dosage used was 5g l^{-1} and arsenic initial concentration was 100 mg l^{-1}. At different time intervals (0.2, 0.5, 1, 2, 12, 15, 24, and 48h) the aqueous phase was withdrawn and filtered and the residual arsenic concentration, in the aqueous phase, was determined by flame atomic absorption spectroscopy (AAnalyst 100, Perkin Elmer). The analysis of arsenic in solutions produced after 48h treatment of the sorbent samples with deionized water, showed that there was no leaching from the sorbents. Initial pH and pH values throughout the experiments were determined using a pH meter (model InolabLevel1, by WTW).

2.4.2   Sorption Isotherms

Sorption isotherm studies were conducted by varying the arsenic initial concentration from 1 to 100 mg l^{-1} As (1, 3, 5, 10, 20, 50, 80, 100 mg l^{-1}). The sorption conditions were kept the same as in sorption kinetics and 15h was chosen as contact time. This time was chosen since it was proved, by the kinetic experiments, to be sufficient for reaching equilibrium. The residual arsenic concentration in the aqueous phase was determined by electro thermal atomic absorption spectroscopy (HGA 800, AAnalyst 100, Perkin Elmer). MATLAB software was used to test the fitting of experimental equilibrium data to Langmuir and Freundlich equations.

3. Results and discussion

3.1 Raw materials characterization

The results of the XRF analysis for the raw materials are presented in Table 1. The metallurgical slag mainly contains Ca, Si, Al, and Fe. It is, therefore, anticipated that contacting the slag, which contains 35% SiO_2, with NaOH results in the formation of Na_2SiO_3, while the addition to the slurry of the acidic iron solution leads to the formation of Si-Fe surface complexes "loading" in that way iron oxyhydroxides on the metallurgical slag (Thirunavukkarasu *et al.*, 2002; Zhang and Itoh, 2005). The red mud containing 42%

Fe_2O_3, on the other hand, can after dissolution produce an iron solution that can be used as the iron precursor for the sorbent formation.

Table 1. Content (% oxides) of main elements in Slag and Red Mud.

	CaO	SiO$_2$	Al$_2$O$_3$	MgO	Fe$_2$O$_3$
Slag	41	35	10	10	3
Red mud	19	15	21	2	42

The surface area of the slag proved being 0.13 m^2 g^{-1}. The surface morphology of the slag as evidenced by SEM characterization (Figure 1a) is glassy and in accordance with the extremely low surface area measured. Fayalite is the mineral identified by XRD since its three more intense peaks (d=2.830 Å, d=2.500 Å, d=1.775 Å) are present in the corresponding XRD pattern. (Figure 2a).
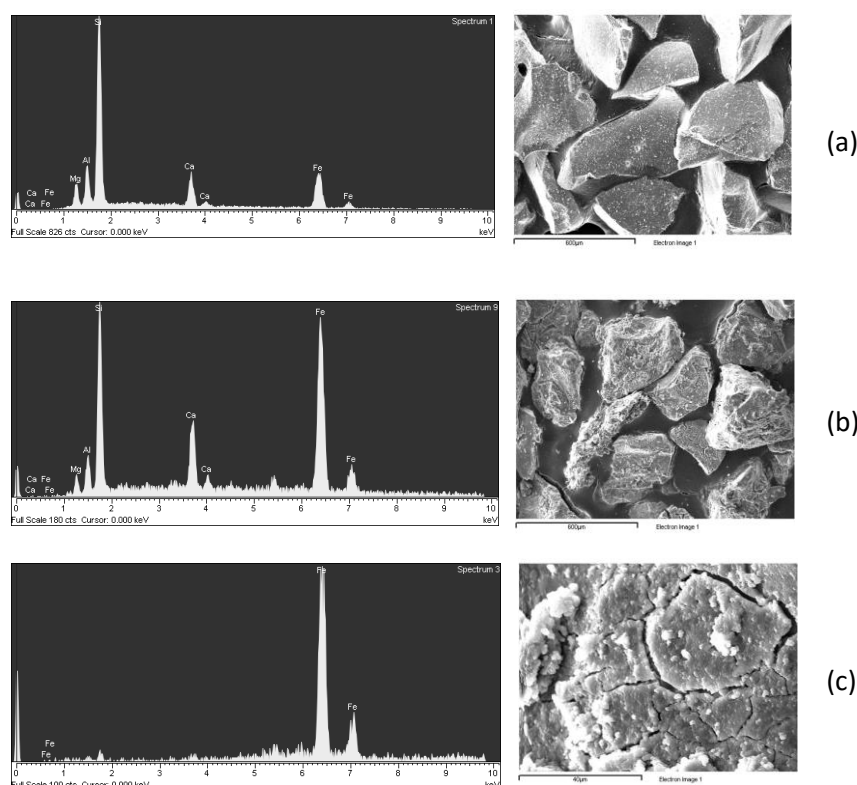

Figure. 1 SEM secondary image of the slag (a) and the sorbent S-RM (b and c) with corresponding EDS spectrum

3.2 Sorbent characterization

The morphology, texture and composition of the sorbents S-RM and S-I are identical as illustrated in Figures 1b and 1c, where the iron porous material loaded on the slag is depicted. The results of the mineralogical analysis of the sorbents (Figure 2b) shows that apart from fayalite, goethite (d= 4.184 Å, d=2.693 Å, d=2.451 Å) is also present in the sorbents confirming that iron oxyhydroxides were successfully loaded onto the metallurgical slag. FTIR spectra obtained for slag, S-RM, S-I and oxyhydroxide (FeOOH) are presented in Figure 3. As it is well known (Bartzas and Komnitsas, 2010; Music *et al.*, 1993; Strongin *et al.*, 2010) the broad band centred at 3450 cm^{-1} that appears in the samples of the iron loaded slag and the FeOOH, can be assigned to the stretching vibration of –OH, while the IR band at 1637cm^{-1} corresponds to the bending vibration of molecular water and hydrogen bonds. The bands between 400 and 600cm^{-1} can be attributed to Fe-O bonds and, in the case of S-RM and S-I, to Fe-O-Si bonds. The oxyhydroxide sample clearly contains goethite since its typical bands at 890 and 798 cm^{-1} are present in the corresponding spectra while the broad band centred at 1050cm^{-1}, present in S-RM and S-I spectra, can be

attributed to the overlapping of Fe-O-Si and ferrihydrite bands. The point of zero charge (PZC) and the surface area of the sorbents are reported in Table 2.

Table 2. Point of zero charge and surface area ($m^2 g^{-1}$) of the sorbents

	PZC	Surface Area ($m^2 g^{-1}$)
S-I	8.4	12
S-RM	8.1	10

The PZC measured was 8.4 and 8.1 for S-I and S-RM, respectively, and is in accordance with values reported in literature for iron oxides and oxyhydroxides (Strongin *et al.*, 2010). The surface area measured was 10 and 12 $m^2 g^{-1}$ for S-RM and S-I, respectively, which is within the range reported (Strongin *et al.*, 2010; Zahng and Itoh, 2005) for both natural and synthetic iron oxides (2-230 $m^2 g^{-1}$). The elevated surface area of the sorbents compared to that of the slag can be attributed to the porous oxyhydroxides that have been loaded onto the slag during the synthesis process.

The above mentioned results point to the conclusion that with the proposed procedure iron oxides and oxyhydroxides were 'loaded' onto slag and that the sorbent prepared in this study, using red mud as the iron precursor, proved to have similar characteristics with the one that was prepared, for comparison purpose, using $FeCl_3$ as iron source.

3.3 Sorption experiments

3.3.1 Kinetic experiments

Arsenic removal, by the sorbent S-RM, as a function of contact time is presented in Figure 4. The kinetic experiments showed that sorption capacity (mg As g^{-1} sorbent) increased rapidly with increasing contact time, while the equilibrium was achieved within 15h for all the batch experiments. Removal capacity proved to be 91% and 78% for S-I and S-RM sorbent, respectively. This is a significant difference, in accordance with the lower surface area measured for S-RM, which should be attributed to the fact that the sorbent was prepared using red mud as iron source. The pH value fluctuated throughout the experiments around 5.8. The effect of pH on the sorption process was not investigated. At pH 5.8 $H_2AsO_4^-$ predominates while the surface of the sorbent, given its PZC at 8.1 or 8.4, is positively charged (Stumm and Morgan, 1995), it is therefore anticipated that experimental conditions favour $H_2AsO_4^-$ adsorption on the sorbent.
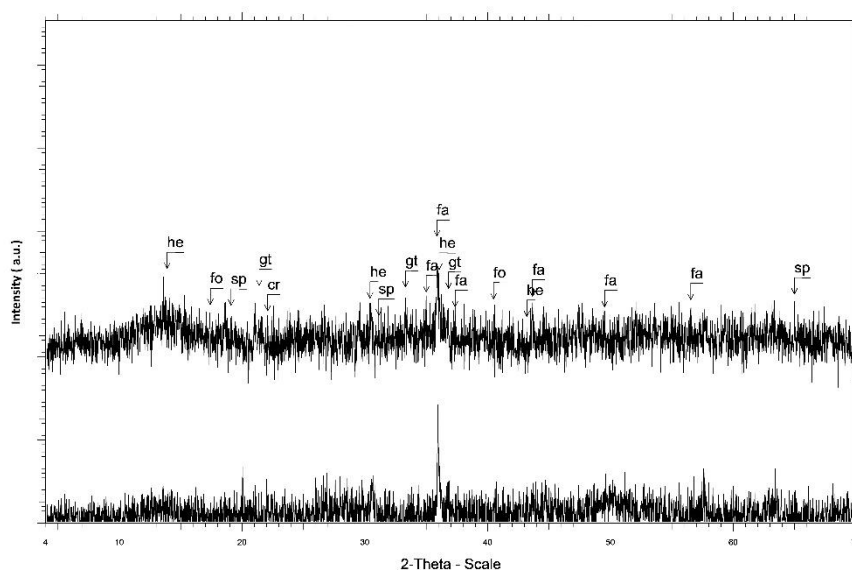

Figure 2. XRD analysis of S and S-RM. (gt: goethite, fa: fayalite, fo: forsterite, cr: cristobalite he: hedenbergite sp: spinel)
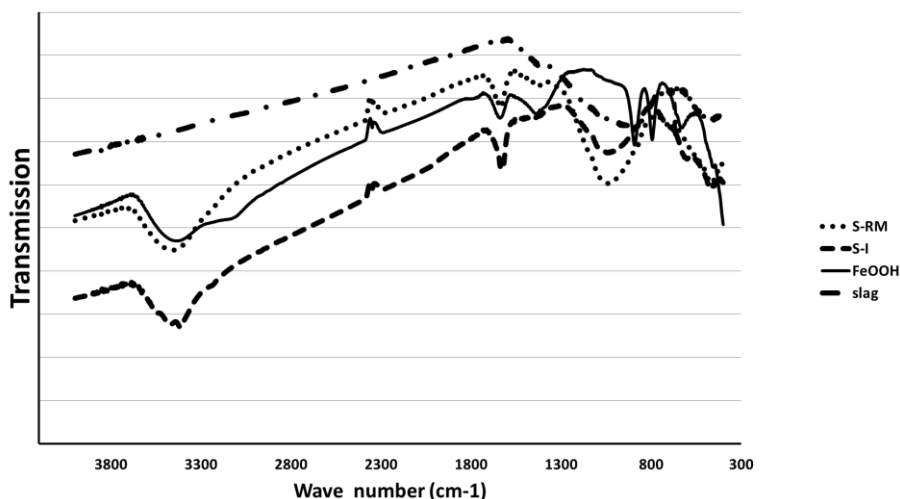
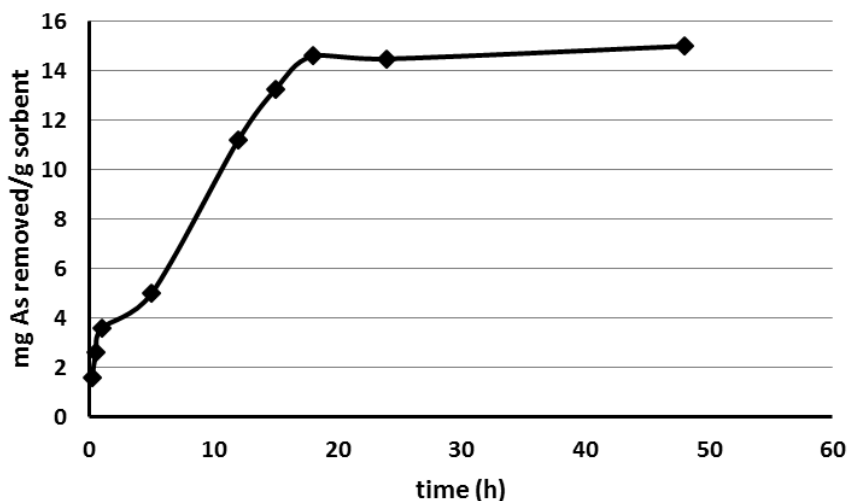**Figure 3.** FT-IR spectra of sorbents S-RM and S-I, FeOOH and slag

Figure 4. Sorption kinetics of arsenic sorption by sorbent S-RM. Experimental conditions: sorbent dosage 5g l⁻¹, pH 5.8, temperature 25 °C, As initial concentration 100 mg l⁻¹

3.3.2 Sorption isotherms

The results of the equilibrium experiments are illustrated in Figure 5. The sorption isotherm may be considered of H or L type according to the classification of Giles *et al.* (1974), indicating that S-RM shows high affinity for the metals studied. To analyze the experimental equilibrium data and to obtain the maximum adsorption capacity of the sorbent, the Langmuir (Langmuir, 1918) and Freundlich (Freundlich, 1926) models are tested.

The equations used are:

$q = Q_{max}bC_e/(1 + bC_e)$ (Langmuir)

$q = KC_e^{1/n}$ (Freundlich)

where q is the amount of arsenic adsorbed per unit mass of sorbent, C_e is the cation concentration at equilibrium, Q_{max} is the maximum adsorption capacity; *K* is related to the adsorption capacity and b, n are constants related to adsorption intensity. For a non-linear regression, MATLAB software was used.
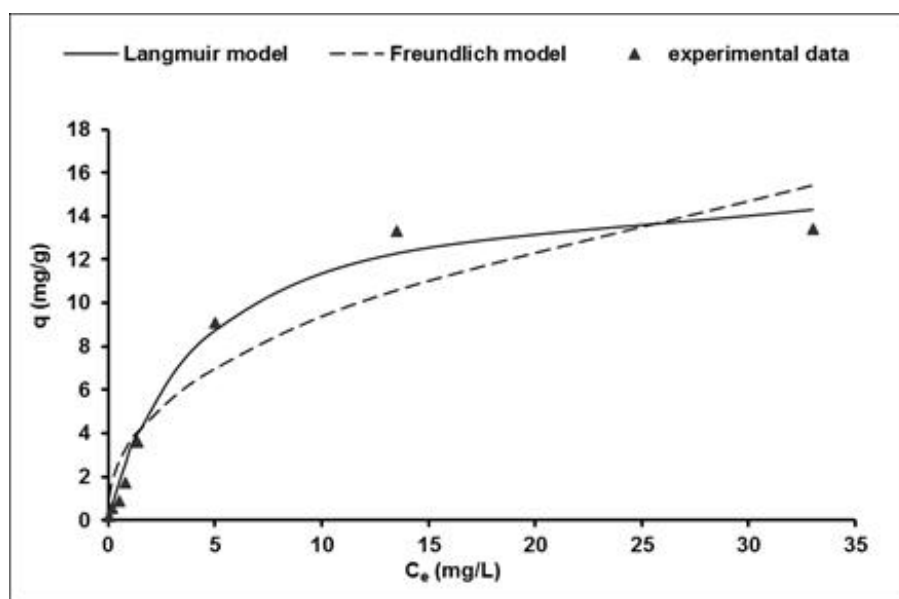
Figure 5 Sorption isotherm: predicted data vs experimental data. Lines represent predicted data by Langmuir and Freundlich models and symbols correspond to experimental data.

In Figure 5 both Langmuir and Freundlich fits for As are presented and it is concluded that experimental data of the present work is better fitted to the Langmuir equation than the Freundlich one, since the correlation coefficient calculated for the Langmuir equation is higher than that for the Freundlich equation (Table 3). This suggests that the arsenic sorption by the synthesized sorbent follows the Langmuir model and that the adsorption process could be described by the formation of a monolayer coverage of the adsorbate on adsorbent surface. The maximum adsorption capacity calculated by the Langmuir equation is 16.14 mg g^{-1}. This adsorption capacity is comparable to those reported in literature for various arsenic sorbents and higher than sorption capacities exhibited by other waste materials that have been proposed as arsenic sorbents (Martinez-Cabanas *et al.*,2015, Ilic *et al.*, 2014, Raul *et al.*, 2014, Salem Attia et al, 2014, Hua *et al.*, 2012; Huijie *et al.*, 2009; Jovanovic and Rajakovic, 2010; Mohan and Pittman, 2007; Thirunavukkarasu *et al.*, 2002; Zahng and Itoh, 2005).

Table 3. Langmuir and Freundlich constants from sorption isotherms at 25 °C.

Langmuir			Freundlich		
Qmax (mg g^{-1})	B (L mg^{-1})	r^2	K	n	r^2
16.14	0.24	0.985	3.53	2.37	0.894

4. Conclusions

The results of the present study show that iron oxyhydroxides are successfully loaded onto the metallurgical slag, leading to the synthesis of an effective and easy to handle sorbent for arsenic removal from aqueous systems. Red mud can be used as iron source, since the two sorbents S-RM and S-I synthesized in the present study, have similar characteristics and performance. The kinetic experiments show that equilibrium is achieved within 15h and the maximum sorption capacity calculated by the Langmuir equation is 16.14 mg g^{-1}. The experimental data is better fitted to the Langmuir equation than the Freundlich one suggesting that the arsenic sorption by the synthesized sorbent could be described by the formation of a monolayer coverage of the adsorbate on adsorbent surface. The sorption capacity, 16.14 mg g^{-1}, of S-RM is comparable to those reported in literature for various arsenic sorbents and higher than sorption capacities exhibited by other waste materials proposed as arsenic sorbents.

Work is in progress with arsenic speciation and desorption studies. Additionally, the effect of pH on the sorption process has to be investigated in order to identify the sorption process mechanism and optimize

the whole procedure to design pilot scale or field scale projects. Nevertheless the advantage of the procedure proposed is quite obvious since it combines recycling with the synthesis of a high added value material, efficient for environmental remediation, contributing in that way to sustainable development.

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