ICAM2009
11th International Conference on Advanced Materials
Rio de Janeiro Brazil
September 20 - 25

Characterization of retorted shale for use in heavy metal removal

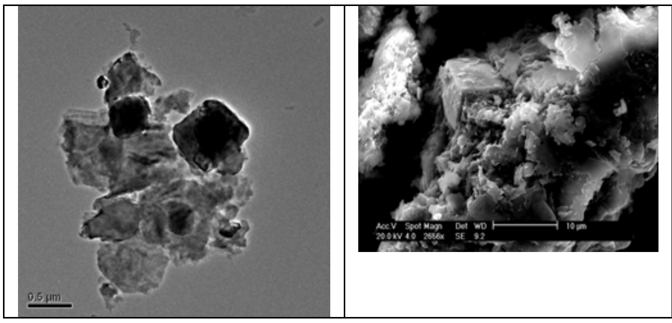
P. M. Pimentel[1], R. M. P. B. Oliveira[1]* D. M. A. Melo[1], Ch. Leroux[2], M. J. Anjos[3] G. G. Maldonado[4] and M. A. F. Melo[1]

(1) PPGCEM-Universidade Federal do Rio Grande do Norte, Natal, Brazil, e-mail: rosane@ufrnet.br
(2) IM2NP UMR CNRS 6242-Université du Sud Toulon-Var, 83957 La Garde, France.
(3) COPPE-Universidade Federal do Rio de Janeiro, Rio de Janeiro, Brazil.
(4) IMA-Universidade Federal do Rio de Janeiro, Rio de Janeiro, Brazil.
* Corresponding author.

Abstract- There has been a growing interest in the use of cost-effective materials to remove heavy metals from wastewater. In the present investigation, retorted shale, a solid residue of the thermal transformation of oil shale, was characterized with the objective of preparing heavy metals adsorbents. The fine residue was characterized by different techniques including, thermogravimetric analyses, BET, XRD, SEM/EDS and TEM. The characteristics of the powder were appropriate for adsorption. Batch adsorption studies *in produced water were performed.* The SRTXRF technique was use to determined the elements concentration.

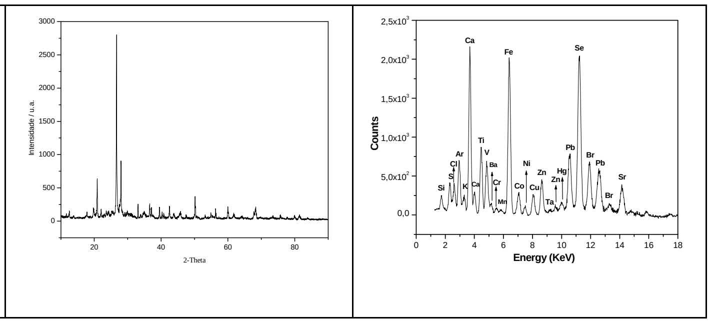
The present paper is an attempt to explore a possibility to utilize retorted shale (RS) to remove lead ions from waste water. This material is waste product obtained of pyrolysis (550 °C) of oil shale. Since RS holds several functional groups they would have high potential for heavy metals adsorption. In addition, the technological development for the beneficial use of this material is needed, once oil shale is abundant in 31 countries [1]. The RS was characterized by different techniques including particle size, thermogravimetric analyses, BET, X ray fluorescence, FTIR, XRD, SEM/EDS and TEM. The characteristics of the retorted shale were as follows: surface area (BET), 65.083 $m^2 g^{-1}$; density, 1.139 g cm^{-3} and average particle size, 19.1 μm, pore size 32.5 Å and chemical composition: SiO_2 (55.6%), Al_2O_3 (11.4%), TiO_2 (0.6%), Fe_2O_3 (8.6%), MgO (1.8%), CaO (3.5%), Na_2O (1.6%), K_2O (2.85%) in addition to minor components including MnO, V_2O_5, SrO, CuO, ZnO, ZrO_2, NiO and sulfur. It is observed that retorted shale is mainly composed by acids, bases and amphoteric oxides, which assure the presence of active groups of mineral species and organic residues on the grain surface, suggesting good adsorption behavior. TG curves for RS indicate a weight loss of approximately 16,3%. The RS degradation occurs in three stages. The first stage occurs up to 240 °C and corresponds to drying of RS. The second stage (240-400 °C) is due to the degradation of the organic matter and water interlayer between the fractions of clay minerals present in the samples. Above 550 ° C, decomposition of pyrite (FeS_2) and dehidroxilation of the fraction of clay minerals occur. In the XRD patterns, the following minerals have been identified: quartz, pyrite, plagioclase feldspar, gypsum, clay, minerals such as illite and montmorillonite. TEM and SEM images revealed the heterogeneity of the material, and clusters are observed with cubic forms characteristics of pyrite crystals.

The characteristics of the material were appropriate for adsorption. The applicability of retorted shale was demonstrated by treating with produced water samples (wastewater brought to the surface during the natural gas and crude oil production). The SRTXRF technique allows us to identify and quantify the elements presents in produced-water samples [2]. Removal was found to be approximately 40-70% for Cr, Fe, Co, Ni, Cu e Zn and over 80% for Hg and Pb. The present study thus reveals that the retorted shale can be used as an efficient adsorbent material for heavy metal removal from wastewater



(a) (b)

Figure 1: Image of retorted shale (**a**) TEM and (**b**) SEM



(a) (b)

Figure 2: (a) XRD of retorted shale (b) SRTXRF spectrum of produced-water sample

References

[1] Z. Al-Qodah, Water Res. 34 (17) (2000) 4295–4303.
[2] P. M. Pimentel, M.J. Anjos , D.M.A. Melo, M.A.F. Melo, L.M. Gonçalves Jr., C.N. Silva Jr., R.T. Lopes Talanta 74 (2008) 1231–1235