ID: 5026

Development of a Zeolite Modified by an Ion Exchange and Impregnation Process for the Recovery of Uranium from Aqueous Effluent

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ABSTRACT

In this study, adsorbent materials were developed by surface modification of zeolite NaX, using two processes, namely ion exchange and saline impregnation with $MnCl_2$ with the aim of improving the adsorption capacity uranyl ions in aqueous solutions. The synthesized and modified Faujasite X zeolite was characterized by X-ray diffraction, thermogravimetric analysis, Fourier transform infrared spectroscopy, laser particle size analyzer and nitrogen adsorption-desorption analysis. The textural properties, kinetics, adsorption equilibria and desorption cycle of NaX, NaX-exchanged and NaX-impregnated zeolites were studied to evaluate their potential in uranium adsorption. The experimental results show that the adsorption capacity of uranium by the zeolite NaX 24 mg/g was improved following the modification treatments. It is 30 mg/g and 45 mg/g for the NaX-impregnated and Na-X-exchanged materials respectively. The kinetic study showed that the uranyl ion recovery process is controlled by the pseudo-second order model and the equilibrium data of uranyl ion adsorption was best fitted by the Langmuir model for the three materials. Desorption with HNO_3 at 0.1N revealed the recovery of 80% of uranyl ions after one treatment cycle. The adsorption yields of uranyl ions by the NaX, exchanged NaX and NaX materials impregnated with a real solution titrating 114.9 mg/L in uranium, under optimal conditions, are 81.28%, 87.25% and 90 .73% respectively.

Keywords: zeolite NaX, ion exchange, saline impregnation, uranium.

1 Introduction

The presence of radionuclides in effluents constitutes a significant problem, their treatment has become a global concern; many techniques have been developed for their recovery. One of these techniques is adsorption on zeolite materials, following their high exchange capacity, large specific surface area and resistance to radiation, but adsorption capacity is not optimal because of the diffusional limitations imposed by their microporous structure. Various approaches have been proposed to generate additional porosity or property in zeolite crystals, such as (desilication, dealumination, use of polymers, solid templates, organic complex, ion exchange, impregnation….), either by post-synthesis or by intra-synthesis with the aim of improving the adsorption capacity of the zeolite with respect to uranyl ions which remain the most promising solution. With this in mind, the modification of an adsorbent making it possible to increase the external surface of the crystals and to generate large pores between the crystals (inter-crystalline porosity) or to create an additional network of pores within the crystals, most generally mesopores, while retaining the properties and advantages of the microporous network still present (intra-crystalline porosity), is essential. Our choice fell on the faujasite surface [3]. In this study, ion exchange and impregnation with $MnCl_2$ were used to modify NaX zeolite with the aim of increasing its uranyl ion adsorption capacity. The protocols for the hydrothermal synthesis of zeolite and the various experimental post-synthesis modification processes (ion exchange and impregnation) as well as the characterization of the adsorbents will be described. The parametric, kinetic, adsorption isothermal study will be carried out. A study on the regeneration of adsorbent materials by desorption process was carried out as well as the application of a real effluent.

DOI: 10.21467/abstracts.170 ISBN: 978-81-970666-1-0 (eBook)

2 Experimental

The synthesis of NaX zeolite by hydrothermal route [4], post-synthesis surface modification processes such as ion exchange and saline impregnation with $MnCl_2$ at 5% wt [5] have been carried out.

3 Results and Discussion

The characterization of the materials developed and modified showed that the NaX phase was well crystallized. A clear decrease in crystallinity for both modified materials without alteration of the basic structure was noticed. The appearance of two very visible lines for the NaX-impregnated material, which does not appear for NaX-exchanged in the XRD spectrum, is due to the quantity of $MnCl_2$ impregnated which is much greater than the quantity exchanged 'see figure 1). FTIR spectroscopy confirms the incorporation of manganese chlorides inside the pores of the materials following the appearance of new absorption bands at 923 and 925 cm^{-1} in the exchanged and impregnated materials. The textural measurements show an increase in the specific surface area and the pore volume of the NaX-exchanged material from 565 m^2/g to 612 m^2/g, on the other hand a decrease in the specific surface area as well as the pore volume for the NaX-impregnated zeolite (345m^2/g), which may be the result of blocked pores; this implies that through this impregnation process, the modified sites are distributed almost exclusively on the surface of the adsorbent and the $MnCl_2$ ions have barely penetrated into the pores. The thermogravimetric analysis shows thermal stability of the materials with different mass losses, which once again confirms the incorporation of manganese chloride inside the pores. The Si/Al ratio was determined for the fabricated and modified materials from the Fourier transform infrared spectrum using the double ring vibration frequency. The adsorption capacities are 45 mg/g, 30 mg/g and 25 mg/g for NaX-exchanged, NaX-impregnated and NaX respectively. The equilibrium isotherms of uranium (VI) adsorption on the three adsorbents are in perfect agreement with the Langmuir model and the adsorption of uranyl ions is correctly described by the pseudo-second order kinetic model. The study of desorption with HNO_3 at 0.1N revealed that the recovery of uranyl ions is of the order of 80% by the NaX, NaX-exchanged and NaX-impregnated materials for one treatment cycle. The adsorption yield of uranyl ions on the NaX, exchanged NaX and NaX materials impregnated with a real solution titrating in the 114.9 mgU/L is respectively of the order of 81.28%, 87.25% and 90.73%.

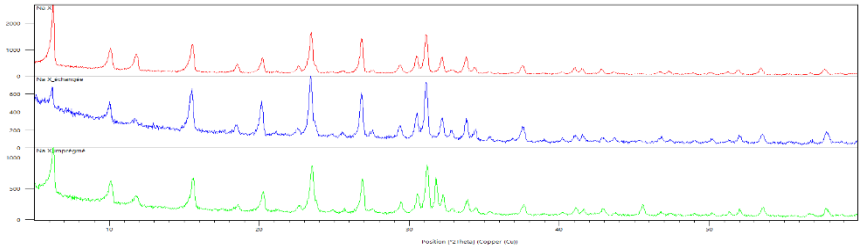

Figure 1. XRD pattern of NaX, Na-X-exchanged and NaX-impregnated samples

4 Conclusions

The objective of this study is to develop new ways of modifying the surface of a zeolite adsorbent material in order to increase its specific surface area and why not its adsorption capacity. Zeolite NaX is modified by ion-exchange and salt impregnation to improve uranium sorption capacity. The characterization and application of these materials were a major asset illustrating the originality of this research work. The use of materials modified by ion exchange and saline impregnation for the recovery of radioelements such as uranium has many advantages compared to the use of the mother zeolite, which demonstrates a better adsorption capacity towards the uranium.

ISBN: 978-81-970666-1-0
Series: AIJR Abstracts
DOI: 10.21467/abstracts.170

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ISBN: 978-81-970666-1-0

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DOI: 10.21467/abstracts.170