Ain Shams University
Faculty of Science
Chemistry Department

Development and performance evaluation of hybrid magnetic composites as solid phase extractants for some lanthanides

A Thesis Submitted by

Mohamed Abd El-Hameed Attia Mohamed

M. Sc. of science in chemistry (2015)

Assistant Lecturer

Nuclear Chemistry Department – Hot Laboratories Center

Atomic Energy Authority

For the Ph. D. Degree of Science in Chemistry

(Inorganic Chemistry)

To

Chemistry Department

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2019

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Head of Chemistry Department
Prof. Dr. Ibrahim Hussieny Ali Badr

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Prof. of Radiochemistry،
Hot Laboratories Center, Atomic Energy Authority.

Date of discussion	**Head of Chemistry Department**
23/4/2019	Prof. Dr. Ibrahim Hussieny Ali Badr

بِسْمِ اللَّهِ الرَّحْمَٰنِ الرَّحِيمِ

قَالُوا سُبْحَانَكَ

لا عِلْمَ لَنَا إِلا مَا عَلَّمْتَنَا

إِنَّكَ أَنْتَ الْعَلِيمُ الْحَكِيمُ

صَدَقَ اللهُ الْعَظِيمْ

سورة البقرة: آية ٣٢

Dedicated to

(The Spirit of My Father)

To

My Dear Mother, My Lovely Wife, My Children,

My Brother and My Sisters

All of those who **Love me**

Mohamed A. Attia

Acknowledgement

*First, I would like to thank **ALLAH,** most merciful, who blesses my effort, shows me the way and enables me to present this work in an acceptable style. On the top, **my parents**, **my wife**, **my children** and **the rest of my family.** None of the things I have done would be done without their endless support, patience and encouragement throughout my years of study.*

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Hydroxyapatite/$NiFe_2O_4$ superparamagnetic composite: Facile synthesis and adsorption of rare elements

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HIGHLIGHTS

- A superparamagnetic hydroxyapatite composite (CaHAP/NF) was successfully synthesized.
- The composite is a crystalline in nature, possesses a high porous structure and chemically stable at pH > 3.5.
- $^{152+154}Eu$ and ^{160}Tb radionuclides were effectively removed using the synthesized magnetic composite.
- REEs were sufficiently recovered using $FeCl_3$ and EDTA as eluents.
- The chromatographic separation of Eu(III) from Tb(III) was significantly attained using EDTA.

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ABSTRACT

A magnetic hydroxyapatite composite (CaHAP/NF) derived from calcium hydroxyapatite [$Ca_{10}(PO_4)_6(OH)_2$] and nickel ferrite [$NiFe_2O_4$] was successfully synthesized by a coprecipitation method. The synthesized composite was characterized using Fourier transform infrared spectroscopy (FT–IR), X-Ray diffractometer (XRD), thermogravimetric differential thermal analysis (TG-DTA), scanning electron microscopy (SEM) and vibrating sample magnetometer (VSM). Results clarify that the composite is a crystalline in nature, thermally stable up to 800 °C and possesses a high porous structure. The synthesized CaHAP/NF composite is a superparamagnetic material easily separated from aqueous solutions and would dissociate to some extent in strongly acidic conditions. The synthesized material was successfully applied as a solid phase for separation of Eu(III) and Tb(III) ions from aqueous solutions. The effect of various parameters (e.g. solution pH, equilibrium time and ionic strength) on sorption process was studied in static conditions. The synthesized sorbent could be considered as an efficient candidate for separation and recovery of Eu(III) and Tb(III). The sorption process was very fast initially, reached equilibrium within 6 h of contact and independent of ionic strength. The maximum sorption capacity values were 137.35 and 130.43 mg g^{-1} for Eu(III) and Tb(III), respectively. Desorption of Eu(III) and Tb(III) from loaded sample was studied using various eluents and maximum recovery was obtained using $FeCl_3$ and EDTA solutions. More importantly, both $FeCl_3$ and EDTA were individually applied as eluents in chromatographic separation of Eu(III) and Tb(III) in CaHAP/NF packed column and the best separation results were obtained by EDTA.

1. Introduction

Rare earth elements (REEs) consist of 17 elements of the periodic table including 15 lanthanides along with yttrium and scandium (Liatsou et al., 2015; Anastopoulos et al., 2016; Jacinto et al., 2018). They are further subdivided into light and heavy rare earth elements on the basis of their atomic number. REEs have unique properties and often termed as "seeds of technology" (Ponou et al., 2014). They are widely used in different applications such as metallurgy, electronics, alloys, fertilizers, lasers, magnets, superconductors, catalysis, chemical reagents and nuclear energy (Smith et al., 2016; Rychkov et al., 2018). The total demand for REEs was increased from 128,000 t in 2011 to 170,000 t in 2015, while it is expected to rise to 255,000 t in 2020 with a growth rate about 6–10%/year (Fernandez, 2017). Such expected great demand for rare earth elements is resulted mainly from their widespread applications in many fields of human life. With ever-

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