

**UNIVERSITI TEKNOLOGI MARA**

**ADSORPTION OF  
Ce(III) AND Nd(III) IONS ONTO  
MONOSODIUM GLUTAMATE  
FUNCTIONALIZED CHITOSAN,  
XANTHATED CHITOSAN  
AND XANTHOGENATED  
CHITOSAN BEADS**

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**PhD**

**May 2020**

## AUTHOR'S DECLARATION

I declare that the work in this thesis was carried out in accordance with the regulations of Universiti Teknologi MARA. It is original and is the results of my own work, unless otherwise indicated or acknowledge as referenced work. This thesis has not been submitted to any other academic institution work or non-academic institution for any degree or qualification.

I hereby acknowledge that I have been supplied with the Academic Rules and Regulations for Post Graduate, Universiti Teknologi MARA, regulating the conduct of my study and research.

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Thesis Title : Adsorption of Ce(III) and Nd(III) Ions onto  
Monosodium Glutamate Functionalized Chitosan,  
Xanthated Chitosan and Xanthogenated Chitosan  
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## ABSTRACT

In this study, chitosan beads were modified with monosodium glutamate as well as sulphur and magnesium for the removal of precious rare earth metals ions, cerium(III), Ce(III) and neodymium(III), Nd(III) from an aqueous solution via batch sorption experiments. Three variants of modified chitosan beads were prepared which were monosodium glutamate functionalized chitosan (MSGC), xanthated chitosan (XC) and xanthogenated chitosan (XNC) beads. These adsorbents underwent several characterizations such as swelling and solubility test, determination of pH of aqueous slurry ( $\text{pH}_{\text{slurry}}$ ) and pH of zero point charge ( $\text{pH}_{\text{zpc}}$ ), fourier transform infrared spectroscopy (FTIR), field emission scanning electron microscopy coupled electron dispersive spectroscopy (FESEM-EDS), pore size and surface area analysis and ultimate analysis in order to study the efficiency in removing Ce(III) and Nd(III) ions from aqueous solutions. MSGC, XC and XNC beads showed significant swelling percentage yet insoluble in acidic, basic and neutral media. Meanwhile, the values of  $\text{pH}_{\text{slurry}}$  and  $\text{pH}_{\text{zpc}}$  were relatively close for all types of adsorbents. FTIR showed that sulphur was successfully added into XC and XNC beads. The uneven and non-porous chitosan beads were found to be rougher and more irregular as they were modified while EDS had strengthen the fact that sodium, sulphur and magnesium were added onto MSGC, XC and XNC beads. As the pore diameter of these beads lies between 11.70 and 39.58 nm, MSGC, XC and XNC beads can be classified as mesoporous. The ultimate analysis had again proved that sulphur had been induced into XC and XNC beads. For batch sorption study, the physicochemical parameters such as the pH, stirring speed, adsorbent dosage, and stirring time were determined as to ensure the adsorption occur at optimum conditions. The optimum pH for adsorption of Ce(III) and Nd(III) ions were 4 and the optimum stirring ranged from 300 to 600 rpm. Adsorption of Ce(III) ions required 0.015 to 0.025 g of MSGC, XC and XNC beads; while Nd(III) ions required 0.01 to 0.04 g of the same adsorbents. A rapid adsorption process was observed for both Ce(III) and Nd(III) ions where it reached the equilibrium state in 30 min of stirring time. Chemisorption was suggested as the rate limiting step as the experimental data for all adsorption system followed pseudo-second-order kinetics model. Based on the adsorption isotherm results, Langmuir model fitted well the experimental data with the maximum adsorption capacities ( $Q_{\text{max}}$ ) recorded for adsorption of Ce(III) ions onto MSGC, XC and XNC beads at 300 K were 167.5, 559.9 and 463.8  $\text{mg g}^{-1}$ , respectively. Meanwhile, the  $Q_{\text{max}}$  values recorded for adsorption of Nd(III) ions onto MSGC, XC and XNC beads were 68.3, 338.2 and 253.9  $\text{mg g}^{-1}$ , respectively. Based on thermodynamic study, adsorption of Ce(III) ions onto XC beads and adsorption of Nd(III) ions onto MSGC and XNC beads were endothermic nature of adsorption. Adsorption of Ce(III) and Nd(III) ions (excluding adsorption of Ce(III) ions onto XNC beads) recorded positive  $\Delta S^\circ$  which attributed to an increase of adsorbent-adsorbate interface. MSGC, XC and XNC beads were more selective to adsorb Nd(III) ions than Ce(III) ions in the binary study. The presence of  $\text{Na}^+$ ,  $\text{K}^+$  and  $\text{Cl}^-$  slightly reduced the adsorption of Ce(III) ions onto MSGC beads, meanwhile the presence of  $\text{Ca}^{2+}$  and  $\text{SO}_4^{2-}$  showed significant effect on adsorption of Ce(III) and Nd(III) ions onto MSGC, XC and XNC beads. The application of EDTA, HCl,  $\text{HNO}_3$ , and NaOH are unable to desorbed Ce(III) and Nd(III) ions from the adsorbents.

## ACKNOWLEDGEMENT

Allhamdullillah. My highest gratitude to Almighty Allah for giving such wonderful opportunity for me to pursue my study until this level. I dedicate this thesis to my beloved late father-in-law, Allahyarham Haji Zakaria Mustaffa who passed away during my final stage of thesis completion due to liver cancer. This thesis also a special tribute to my best friend Waheeba Ahmed Al-Amrani who always inspire me while persuing my study.

I will never reach this level without guidance from my main supervisor, Assoc. Prof. Megat Ahmad Kamal Megat Hanafiah, my co-supervisor and motivator Dr. Shariff Che Ibrahim and my helpful co-supervisor Prof. Wan Saime Wan Ngah. Million thanks to Mr. Ahmad Kambali Khalil, Mr. Rosmi, Mr. Zulfayusri, Mr. Zubir and Mr. Ayob for the technical support during the lab sessions.

Thank you to my most precious backbone, Mr. Mohd. Tarmizi Zakaria and my son Muhammad Tsaqif Fitri Mohd Tarmizi for their unconditional support, understanding, motivations and sacrifices.

Thank you to my parents, Dr. Md Ariff Abas and Mrs. Wan Sepiah Shiekh Ibrahim, mother-in-law, Mrs. Shamsiah Hj. Mohd and siblings for their understanding, love and courage during these years.

I most appreciate the intelligence discussion with my lab mates, Miss Dian Nashiela Fatanah, Dr. Siti Azimah Mutalib and Dr. Shahrul Nizam Ahmad during completing this thesis. Last but not least, thank you to Dr. Asma Assa'edah, Dr. Affaizza, Dr. Nik Fatini, Mrs. Rozaini and labmates for their warmth concern.

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