

MODIFICATION OF ION EXCHANGE RESIN WITH MAGNETITE  
FOR CHROMIUM REMOVAL

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## **DEDICATION**

This thesis is dedicated to mother and step father who always giving me encouragement, moral support, and love to keep moving forward. To my father, thank you for your support. To my lovely siblings, you are the light of my life. To Laila, thank you for everything and I have a beautiful journey with you.

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## ABSTRACT

A hybrid adsorbent was developed by introducing magnetite nanoparticles ( $\text{Fe}_3\text{O}_4$ ) onto the surface of ion exchange resin (cation and anion exchange resins) to improve the adsorption performance for chromium removal from aqueous solution. A rough surface was observed on the surface of both magnetite-loaded cation resin (IRN77- $\text{Fe}_3\text{O}_4$ ) and anion resin (IRN78- $\text{Fe}_3\text{O}_4$ ), revealed by field-emission scanning electron microscope (FESEM). In addition, Fe-O bond was detected on Fourier transform infrared spectroscopy (FTIR) spectra on the surface of both magnetite-embedded ion exchange resins (MIER). From energy-dispersive x-ray spectroscopy (EDX) analysis, 8.2 % and 5.2 % of iron elements was identified on the surface of IRN77- $\text{Fe}_3\text{O}_4$  and IRN78- $\text{Fe}_3\text{O}_4$ , respectively. A larger surface area compared to the unmodified resin was detected for MIER by using a Brunauer–Emmett–Teller (BET) surface area analyzer. Batch adsorption tests with chromium ion were performed with both modified and unmodified resins at various conditions. The equilibrium study identified that optimum conditions for Cr(III) and Cr(VI) adsorption were at pH 3, 1 hour reaction time, and 0.3 g of adsorbents. The adsorption capacity for Cr(III) using IRN77- $\text{Fe}_3\text{O}_4$  increased up to 17% with a faster removal rates than IRN 77. On the contrary, the IRN78- $\text{Fe}_3\text{O}_4$  was discovered to have a lower adsorption capacity of chromium (VI) than unmodified resin due to pH change during adsorption. The maximum adsorption capacity of Cr(III) and Cr(VI) removal using IRN77- $\text{Fe}_3\text{O}_4$  and IRN78- $\text{Fe}_3\text{O}_4$  was remarked at 69.2521 and 77.2798 mg/g, respectively. Furthermore, the adsorption kinetics for both resins fitted well to the pseudo-second-order model. Based on the experimental results, it was found that ion exchange and electrostatic attraction were dominant mechanisms for chromium removal. Lastly, sorption-desorption studies were performed to evaluate reusability for both modified resins. The adsorption capacity for both MIER can achieve maximum removal efficiency of more than 90% for five cycles of usage. Modified ion exchange resin with magnetite nanoparticles could be a promising adsorbent in the remediation of chromium and potentially used in actual field applications to remove other heavy metal contaminants.

## ABSTRAK

Bahan penjerap hibrid telah dibangunkan dengan menambah nanopartikel magnetit ( $\text{Fe}_3\text{O}_4$ ) di permukaan resin pertukaran ion (resin pertukaran kation dan anion) bagi menambah baik prestasi penjerapan kromium dalam larutan akueus. Permukaan resin yang kasar telah dikenal pasti pada permukaan resin kation ( $\text{IRN77-Fe}_3\text{O}_4$ ) dan resin anion ( $\text{IRN78-Fe}_3\text{O}_4$ ) melalui analisa mikroskop elektron pengimbas pelepasan medan (FESEM). Selain itu, ikatan Fe-O juga telah dikenal pasti menggunakan spektroskopi transformasi inframerah Fourier (FTIR) bagi kedua-dua resin pertukaran ion yang telah tertanam dengan magnetit (MIER). Daripada analisis spektrometer penyebaran tenaga sinar-X (EDX), 8.2 % dan 5.2 % unsur ferum telah didapati ada pada permukaan  $\text{IRN77-Fe}_3\text{O}_4$  dan  $\text{IRN78-Fe}_3\text{O}_4$ . MIER juga didapati mempunyai luas permukaan yang lebih besar berbanding resin sebelum diubah suai melalui analisa permukaan Brunauer–Emmett–Teller (BET). Ujian penjerapan kromium secara berkelompok telah dijalankan dalam pelbagai keadaan dengan menggunakan kedua-dua resin yang telah dan belum diubahsuai. Kajian keseimbangan mengenal pasti bahawa keadaan optimum untuk penjerapan Cr(III) dan Cr(VI) adalah pada pH 3, 1 jam tempoh proses penjerapan, serta 0.3 g dos bahan penjerap. Kapasiti penjerapan Cr(III) dengan menggunakan  $\text{IRN77-Fe}_3\text{O}_4$  telah meningkat dengan kadar 17 % dan bahan ini mempunyai kadar penyingkiran yang lebih pantas berbanding IRN 77. Manakala, kapasiti penjerapan Cr(VI) menggunakan  $\text{IRN78-Fe}_3\text{O}_4$  adalah lebih rendah berbanding resin yang tidak diubah suai kerana pH larutan meningkat semasa proses penjerapan berlangsung. Kapasiti penjerapan maksimum bagi Cr(III) dan Cr(VI) menggunakan  $\text{IRN77-Fe}_3\text{O}_4$  dan  $\text{IRN78-Fe}_3\text{O}_4$  masing-masing telah dikenal pasti pada 69.2521 dan 77.2798 mg/g. Selain itu, penjerapan kinetik bagi kedua-dua resin telah didapati menepati model tertib-kedua-pseudo. Berdasarkan keputusan eksperimen, telah didapati bahawa proses pertukaran ion dan daya tarikan elektrostatik adalah mekanisma yang dominan bagi menjerap kromium dari larutan. Akhir sekali, analisa penjerap dan nyahjerap telah dijalankan menggunakan kedua-dua bahan penjerap ini untuk menilai kadar kebolegunaan semula. Kedua-dua MIER mempunyai kecekapan penjerapan yang melebihi 90 % dengan kitaran penjerapan dan nyahjerap sebanyak 5 kali. Pengubahsuaian pada permukaan resin pertukaran ion menggunakan nanopartikel magnetit berpotensi sebagai bahan penjerap yang baik untuk menjerap ion kromium dan bahan ini juga boleh digunakan untuk menjerap bahan cemar logam berat yang lain.

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## LIST OF ABBREVIATIONS

AAS	-	Atomic adsorption spectroscopy
BET	-	Brunauer-Emmett-Teller
CNT	-	Carbon nanotubes
DFT	-	Density functional theory
EDX	-	Energy-dispersive x-ray spectroscopy
FESEM	-	Field-Emission Scanning Electron Microscope
FTIR	-	Fourier transform infrared spectroscopy
FO	-	Forward osmosis
GA	-	Genetic Algorithm
GO	-	Graphene oxide
HCl	-	Hydrochloric acid
IAEA	-	International Atomic Energy Agency
IER	-	Ion exchange resin
MIER	-	Modified ion exchange resin / magnetite ion exchange resin
MNP	-	Magnetic nanoparticles
MOF	-	Metal-organic frameworks
NaOH	-	Sodium hydroxide
nZVI	-	Nano zero-valent iron
RO	-	Reverse osmosis
RTP	-	TRIGA PUSPATI Reactor
UF	-	Ultrafiltration
UTM	-	Universiti Teknologi Malaysia
PFO	-	Pseudo-first-order
PSO	-	Pseudo-second-order

## LIST OF SYMBOLS

$C_0$	-	Initial concentration
$C_e$	-	Equilibrium concentration
$k_1$	-	PFO rate constant
$k_2$	-	PSO rate constant
$K_F$	-	Freundlich constant
$K_L$	-	Ratio of the adsorption rate and desorption rate
$n$	-	Heterogeneity factor
$q_e$	-	Chromium ion adsorbed at equilibrium
$q_t$	-	Adsorption capacity at time
$q_m$	-	Maximum adsorption capacity
$R^2$	-	Linear regression
$R_L$	-	Separation factor or equilibrium parameter
$k_{id}$	-	Intraparticle diffusion rate constant
$t$	-	Time
$V$	-	Volume of solution
$W$	-	Adsorbent mass

# CHAPTER 1

## INTRODUCTION

### 1.1 Problem Background

Water is an essential substance that humans are using for drinking and other daily routines. Rapid growth in human population, industrialization, urbanization and agricultural activities contribute to water pollution. The world population is expected to increase to an average of 9.8 billion by 2050, leading to increasing global demand for agricultural and energy production (UNESCO, 2018). This global water demand will significantly increase over the next two decades, and some countries are already facing water scarcity due to limited surface water resources. Increasing wastewater generation due to rapid industrialization and dwindling of water resources can cause catastrophe to human and other living organisms if not resolved. According to IWA (2018), around 80% of all wastewater discharged into waterbodies can cause a detrimental effect on human health and environmental hazard. To overcome this problem, wastewater treatment and water recycling become global issue. Various toxic contaminants such as oil, dyes, inorganics ions, synthetic organic material and heavy metals have been released into the aquatic environments through various human activities. Among this pollutants, release of heavy metal such as chromium (Cr), cadmium (Cd), mercury (Hg), lead (Pb), nickel (Ni), and thallium (Tl) are potentially hazardous in combined or elemental forms.

Chromium is one of many heavy metals detected in waterbodies from natural and anthropogenic sources. This element can be present in various oxidation states from 0 to VI, in which three stable forms of chromium (Cr(0), Cr(III) and Cr(VI)) are commonly found in the environment. Metal processing, tannery, chromate production, stainless steel welding, dye production are industries with a massive contribution to chromium wastewater production (ASTDR, 2012). The toxicological effects of chromium on human health were studied among various researchers. Cr(VI) is a well-

known carcinogen and high solubility in water that can penetrate through the biological cell membrane, which can cause an allergic reaction and increasing the risk of cancer (Zhitkovich, 2011). Over the years, numerous conventional methods have been used for chromium removal from wastewater, such as chemical precipitation, ion exchange, electrolytic recovery, membrane separation, floatation and adsorption. These methods have their pros and cons. Among these methods, adsorption is reported to be more promising for chromium removal due to this method provides high adsorption efficiency, some adsorbent materials are low cost, and ease in availability and operation (Samuel *et al.*, 2019; Dubey and Gopal, 2007; Panda *et al.*, 2017; Lal *et al.*, 2020).

Magnetic nanoparticles, such as magnetite ( $\text{Fe}_3\text{O}_4$ ), nano zero-valent iron (nZVI), and maghemite ( $\gamma\text{-Fe}_2\text{O}_3$ ) have been widely used in various fields, including medical, industry and engineering application (Tahar *et al.*, 2018; Hu *et al.*, 2011; Chowdhury and Yanful, 2010; Mejia-Santillan *et al.*, 2018). The magnetic nanoparticles have sparked an immense interest as an adsorbent to remove heavy metals and contaminants in wastewater, such as arsenic, lead, and chromium (Chowdhury and Yanful, 2010; Hu *et al.*, 2011; Tahar *et al.*, 2018). The magnetic nanoparticles have unique properties such as high surface area-to-volume ratio, easy to synthesize, comparatively low cost, easily separated from wastewater using external magnet, and can regenerate by desorption with HCl or NaOH (Ilankoon, 2014). To ensure stability and prevent aggregation of this adsorbent, magnetite nanoparticles can be integrated with functionalized polymer, surface coating with novel molecules or inorganic materials (Shuang *et al.*, 2013; Chen *et al.*, 2011; Hu *et al.*, 2010).

Among heavy metal removal frameworks, ion exchange resins (IER) are important synthetic organic adsorbents. These resins can provide good thermal stability, good mechanical strength and stability when exposed to radiation, low-level sludge generation and can be regenerated (Darmograi *et al.*, 2016; Wang and Wan, 2015). Ion exchange resin has been used in the solid-phase extraction for radionuclides, heavy metals, pharmaceutical pollutants and anion removal from ground water, wastewater and other sources (Laili *et al.*, 2010; Kang *et al.*, 2004; de Heredia *et al.*, 2006; Taleb *et al.*, 2017; Li *et al.*, 2012). However, this synthetic

adsorbent suffers from inherent drawbacks such as low selectivity with other competing anions, resin fouling due to organic matter, a considerable cost for regeneration procedure on an industrial scale (Tandorn *et al.*, 2017; Shuang *et al.*, 2013).

Many studies have focused on enhancing the physical properties and adsorption performance of IER by introducing rare earth, metal oxide, and nano-adsorbent on the surface of the resin (He *et al.*, 2012; Tandorn *et al.*, 2017; Sadeek *et al.*, 2017). In recent years, polymeric-based iron oxide adsorbents gained more attention for wastewater treatment. Combination of these materials can provide an excellent mechanical property, improves adsorption efficiency, and specific affinity towards the removal of particular pollutant (Shuang *et al.*, 2013; Li *et al.*, 2017; Erdem Yayayürük and Yayayürük, 2019). This study aims to evaluate the potential use of magnetite incorporated with ion exchange resins (MIER) for chromium adsorption in an aqueous solution. In addition, the efficiency of adsorption and desorption of chromium was also studied using this adsorbent.

## **1.2 Problem Statement**

The surface modification of ion exchange resin with magnetic nanoparticles is essential to improve adsorption performance for heavy metal removal. A previous study demonstrated that anion exchange resin incorporated with  $\text{Fe}_3\text{O}_4$  enhanced the adsorption of humic acid and improved the antifouling performance of the adsorbent (Shuang *et al.*, 2013). They also determined that the reusability of anion resin also improved after modification with magnetite. There have been several studies on the adsorption of heavy metals from water by magnetic nanoparticles loaded resin. Li *et al.* (2017) developed a novel magnetic cation exchange resin for copper and nickel removal from an aqueous solution. The adsorption capacity for this adsorbent was high, but somehow it was difficult to develop this adsorbent since it was synthesized using suspension polymerization and hydrolysis reaction. A surface-modified IRA 420 with magnetite nanoparticles was designed to improve uranium extraction (Sadeek *et al.*, 2017). However, there was not enough information about the isotherm, kinetic and



regeneration study. Despite all these studies, a minimum effort has been made to study the combination of magnetite with commercialized ion exchange resin for chromium removal.

In this study, the chromium ion was selected as a model pollutant for magnetite-loaded ion exchange resin (MIER). The MIER was produced by *in-situ* coprecipitation synthesis of magnetite with commercialized nuclear grade ion exchange resin. Therefore, loading magnetite on the surface of IER should improve the adsorption performance for chromium removal.

### **1.3 Research Objectives**

This research aims to provide a methodology for developing modified ion exchange resin with magnetite, which can serve as a chromium ion removal from an aqueous solution. Thus, the objectives of this research are:

- (a) To evaluate the surface morphology and elemental composition of cation and anion exchange resin incorporated with magnetite nanoparticles ( $\text{Fe}_3\text{O}_4$ ).
- (b) To investigate the adsorption performance of modified ion exchange resins for chromium removal at various conditions (pH, contact time, and adsorbent dosage), adsorption isotherm, adsorption kinetics, and regeneration.

### **1.4 Scopes of Study**

To achieve the objectives, the scopes of study in this research are:

- (a) The ion exchange resins used in this study was the Amberlite IRN 77 (strong acidic cation exchange resin) and Amberlite IRN 78 (strong basic anion exchange resin)

- (b) The concentration of iron precursors was fixed at 0.1 M for the modification IER with magnetic nanoparticles by *in-situ* co-precipitation method in nitrogen condition at 25°C.
- (c) To evaluate the interaction between magnetite nanoparticles and IER, the field-emission scanning electron microscope (FESEM) was used to undergo morphological analysis of adsorbents. Surface functional groups on the surface of the adsorbent were determined by using FTIR analysis. Moreover, the EDX analysis was carried out to examine the elemental composition of adsorbents before and after loading with magnetite. Further analysis of modified resin was performed by Brunauer-Emmett-Teller (BET) surface area analyzer to compare surface area before and after modification. These elemental and surface area analyses were performed to clarify whether the magnetite nanoparticles were successfully immobilized on the resin's surface.
- (d) Batch adsorption experiments of cation and anion adsorbents were performed with Cr(III) and Cr(VI), respectively. Adsorption equilibrium of chromium ion was determined by adsorption experiments at various pH (2-10), time (10-300 minutes), and adsorbent dosage (0.05 – 1 g).
- (e) Langmuir and the Freundlich isotherms evaluated the maximum adsorption capacity of both unmodified and modified resin. The adsorption isotherm was investigated by adsorption of chromium ions at various concentrations with a fixed adsorbent mass, reaction time, and solution temperature.
- (f) Adsorption kinetics was performed with chromium at 50, 100, and 150 mg/L using modified IER. Kinetic data were evaluated using pseudo-first-order and pseudo-second-order kinetic models.
- (g) The desorption of chromium was using HCl as a stripping solution for magnetite-loaded cation resin (IRN77-Fe<sub>3</sub>O<sub>4</sub>) or NaOH for magnetite-loaded anion resin (IRN78-Fe<sub>3</sub>O<sub>4</sub>). Desorption-regeneration cycles were performed five times to evaluate the removal efficiency of MIER.

## 1.5 Significance of Study

This study explains the interaction between magnetite nanoparticles and ion exchange resin from the characterization analysis. This study also highlights the effective removal of chromium in an aqueous solution. The modification of IER with magnetite nanoparticles is one of many ways to improve the adsorption performance of the ion exchange resin. The magnetic nanoparticle increases the surface area and provides a lot more adsorption active sites for chromium removal. As the adsorption capacity of resin increases, the number of reaction cycles also increases, reducing the number of resins needed for industrial-scale water treatment applications. The equilibrium, isotherm, kinetic studies performed in this study provide sufficient information for the adsorption efficiency. The desorption and regeneration of modified IER represent the sustainability of adsorbent for heavy metal removal.

It can be suggested that both IRN77-Fe<sub>3</sub>O<sub>4</sub> and IRN78-Fe<sub>3</sub>O<sub>4</sub> would serve as a potential adsorbent for the removal of chromium and other heavy metals from an aqueous solution. In addition, these adsorbents also can be utilized in various applications, including radionuclides or heavy metal extraction and water purification systems.

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