UNIVERSITI PUTRA MALAYSIA

DEVELOPMENT OF GLYCIDYL METHACRYLATE GRAFTED IRRADIATED FIBERS AND POLY(ACRYLONITRILE-CO-ACRYLIC ACID) MICROPARTICLES ADSORBENTS FOR THE REMOVAL OF p-NITROPHENOL

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By

ALMOHAMEDABAS SHIHAB EZZULDIN M SABER

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Doctor of Philosophy

December 2021

DEDICATION

Firstly, this thesis is dedicated to Allah (subhanah wa taala) who gave me everything.
To my dearest great father and my beloved great mother,
To my loving family; my wife and my sons.

Shihab Ezzuldin M.Saber Almohamedabas
September 2021

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Doctor of Philosophy

DEVELOPMENT OF GLYCIDYL METHACRYLATE GRAFTED IRRADIATED FIBERS AND POLY(ACRYLONITRILE-CO-ACRYLIC ACID) MICROPARTICLES ADSORBENTS FOR THE REMOVAL OF p-NITROPHENOL

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December 2021

Chairman : Professor Luqman Chuah Abdullah, PhD
Faculty : Engineering

p-Nitrophenol (PNP) is one of the most hazardous pollutants; this compound is extremely damaging to human well-being and additionally, leads to both environmental and economic burdens. Several strategies have been utilized for the removal of phenols from effluents. The adsorption separation technique is considered to be an effective method; it is broadly utilized for wastewater treatment.

Various adsorbent materials are used for the purification of phenols-contaminated effluent. However, they are subject to limitations due to their expense, high-energy requirement, relatively low adsorption capacities, slow kinetics and challenges related to their regeneration and recyclability. To overcome these challenges, novel fibrous and microparticle based adsorbents have been designed and employed for PNP adsorption from aqueous solution.

Fibrous-based adsorbents were prepared by radiation-induced graft polymerization (RIG); glycidyl methacrylate (GMA) was grafted onto polyamide 6 (PA6) and natural cotton (Cot) substrates in order to form (PA6-*g*-GMA) and (Cot-*g*-GMA) fibers, respectively. The extent to which GMA was grafted on PA6 and cotton fibers was found to be markedly influenced by the absorbed dose of radiation and the reaction time of grafting. The optimal parameters were established so as to attain the required degree of grafting (DG) which tuned to 200% at 25 kGy absorbed dose and 30 minutes for PA6 whilst 10 kGy and 50 minutes for cotton fibers. A functionalization strategy was run with trimethylamine (TMA) to obtain TMA-(PA6-*g*-GMA) and TMA-(Cot-*g*-GMA). Redox polymerization (RP) of acrylonitrile (AN)/acrylic acid (AA) as poly(AN-*co*-AA) was employed so as to create microparticle-based adsorbents. A range of AA ratios were integrated into the polyacrylonitrile chain and additionally

functionalized with an amidoxime (AO) moiety in order to generate AO-poly(AN-*co*-AA) adsorbents.

The created adsorbents were evaluated so as to verify the copolymerization and functionalization processes and to describe the impact of preparation on the adsorbent's physiochemical properties utilizing a range of analytical strategies, including Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD) analysis, Field emission scanning electron microscopy (FESEM), Brunauer–Emmett–Teller (BET) surface area, pore size assessment, Thermogravimetric (TG-DTG) analyzer and point of zero charge (pHpzc).

Adsorption studies for PNP removal were conducted. The factors encompassing adsorbent dose, solution pH, temperature, initial PNP concentration and contact time were demonstrated to impact adsorption performance; this was optimized in depth. The adsorption process showed that the proportion of PNP removal increased when the adsorbent dose and PNP initial concentration were increased. The process of PNP adsorption was negatively affected by temperature, where a lower temperature was clearly preferable for greatest PNP adsorption. The adsorption was found to be pH-dependent; an increase in pH from 3.0 to 5.0 caused an increased in PNP removal, i.e. from 46.79% to 82.81% for TMA-(PA6-*g*-GMA) and from 49.31% to 85.33% for TMA-(Cot-*g*-GMA) whilst from 34.8% to 80.6% by changing the pH from 3 to 7. A pH of 5.0 was associated with maximum removal of PNP onto fibrous adsorbents and pH of 7 onto AO-poly(AN-*co*-AA) adsorbent.

The function of the adsorbents pertaining to kinetics, equilibrium, isotherm, and thermodynamics of PNP adsorption from aqueous solutions was assessed employing relevant models. Non-linear Pseudo-first order (PFO), Pseudo-second order (PSO), Elovich and Intraparticle diffusion (IPD) models were utilized to study the adsorption kinetics; PNP adsorption on all adsorbents was demonstrated to follow to Pseudo-second order model. While non-linear Langmuir, Freundlich, Temkin and Redlich-Peterson models offered data on the adsorption isotherms; in which, Redlich Peterson most closely described the equilibrium results, followed closely by Langmuir isotherm and Freundlich isotherm models for the fibrous and microparticle-based adsorbents, respectively. The maximum adsorption capacities were TMA-(PA6-*g*-GMA), 176.04 mg/g; TMA-(Cot-*g*-GMA) 180.00 mg/g; and AO-poly(AN-*co*-AA), 143.06 mg/g. Thermodynamic evaluation demonstrated that the adsorption was a spontaneous and exothermic process. Lastly, the specific high regeneration efficiency of the adsorbents was revealed.

The data from this study imply that fibrous adsorbents exhibit a higher adsorption capacity and more rapid kinetics than microparticle-based adsorbents. However, the latter have markedly higher adsorption capacity than alternative adsorbents described in previous studies. Therefore, it can be believed that the designed adsorbents are encouraging materials for the removal of PNP from water and wastewater.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

PENGHASILAN BAHAN GENTIAN TERADIASI DICANGKUK GLISIDIL METAKRILAT DAN PENJERAP MIKROZARAH POLI(AKRILONITRIL-KO-AKRILIK ASID) BAGI PENYINGKIRAN p-NITROFENOL

Oleh

ALMOHAMEDABAS SHIHAB EZZULDIN M SABER

Disember 2021

Pengerusi : Profesor Luqman Chuah Abdullah, PhD
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p-Nitrofenol (PNP) adalah antara pencemar yang merbahaya; sebatian ini sangat memudaratkan kepada manusia dan tambahan pula, boleh membebankan kepada kedua-dua alam sekitar dan ekonomi. Pelbagai strategi telah digunakan untuk menyingkirkan fenol dari efluen. Teknik pemisahan penjerapan telah dipertimbangkan sebagai kaedah yang efektif; ia telah digunakan secara meluas untuk rawatan sisa air. Pelbagai bahan penjerap telah diguna pakai untuk penulenan efluen yang mengandungi fenol. Walaubagaimanapun, bahan–bahan ini tertakluk kepada kekangan disebabkan oleh perbelanjaan, keperluan tenaga yang tinggi, kapasiti penjerapan yang rendah secara relatif, kinetik yang perlahan dan cabaran yang berkaitan dengan penjanaan semula dan kebolehan kitar semula. Untuk mengatasi masalah ini, bahan penjerap baharu berasaskan gentian dan mikropartikel telah direka dan digunakan untuk penjerapan PNP daripada larutan akueus.

Penjerap berasaskan gentian telah dihasilkan menggunakan pempolimeran cangkuk dengan dorongan radiasi (RIG); glisidil metakrilat (GMA) telah dicangkuk ke atas substrat polyamida 6 (PA6) dan kapas semulajadi (Cot) untuk menghasilkan gentian (PA6-*g*-GMA) dan gentian (Cot-*g*-GMA), masing-masing. Pelanjutan cangkukan GMA ke atas PA6 dan gentian kapas didapati telah dipengaruhi oleh dos radiasi yang diserap dan masa tindak balas cangkukan. Parameter yang optimum telah dibina bagi mencapai darjah cangkukan (DG) yang dikehendaki iaitu sebanyak 200% pada dos serapan 25 kGy dan 30 minit untuk PA6, manakala 10 kGy dan 50 minit untuk gentian kapas. Strategi kefungsian telah dijalankan menggunakan trimetilamina (TMA) untuk menghasilkan TMA-(PA6-*g*-GMA) dan TMA-(Cot-*g*-GMA). Pempolimeran redoks (RP) menggunakan akrilonitril (AN)/akrilik asid (AA) sebagai poli(AN-*co*-AA) telah dijalankan untuk menghasilkan penjerap berasaskan mikropartikel. Pelbagai nisbah AA telah diperkenalkan ke dalam rantaian poliakrilonitril dan tambahan pula difungsikan dengan kumpulan berfungsi amidoksim (AO) untuk menghasilkan penjerap AO-poli(AN-*co*-AA).

Penjerap yang dihasilkan dinilai untuk mengesahkan proses kopempolimeran dan kefungsian serta untuk menerangkan impak penyediaan terhadap sifat fisiokimia penjerap menggunakan pelbagai strategi analitikal, termasuklah penggunaan spektroskopi Inframerah Transformasi Fourier (FTIR), analisis pembelauan sinar-X (XRD), mikroskop imbasan elektron pancaran medan (FESEM), penganalisis kawasan permukaan Brunauer-Emmett-Teller (BET), penganalisis termogravimetri (TG-DTG) and penganalisis titik caj sifar (pHpzc).

Kajian ke atas penjerapan PNP telah dijalankan. Faktor merangkumi dos bahan penjerap, pH larutan, suhu, kepekatan awal PNP dan masa sentuhan telah menunjukkan kesan terhadap prestasi penjeraan; yang mana telah dioptimakan secara mendalam. Proses penjerapan menunjukkan bahawa nisbah penyingkiran PNP meningkat apabila dos bahan penjerap and kepekatan awal PNP meningkat. Proses penjerapan PNP dipengaruhi secara negatif oleh suhu, di mana suhu yang rendah lebih cenderung untuk penjerapan PNP yang tertinggi. Penjerapan didapati bergantung kepada pH; peningkatan pH dari 3.0 kepada 5.0 telah menyebabkan peningkatan penyingkiran PNP; contohnya daripada 46.79% kepada 82.81% untuk TMA-(PA6-*g*-GMA) dan daripada 49.31% kepada 85.33% untuk TMA-(Cot-*g*-GMA), manakala daripada 34.80% kepada 80.6% dengan mengubah pH daripada 3 kepada 7. pH 5.0 didapati menghasilkan penyingkiran maksimum PNP ke atas penjerap gentian and pH 7.0 ke atas penjerap AO-poli(AN-*ko*-AA).

Fungsi bahan penjerap berkaitan kinetik, keseimbangan, isoterma dan termodinamik untuk penjerapan PNP daripada larutan akeus telah dinilai menggunakan model yang bersesuaian. Persamaan model tidak linear Pseudo-tertib pertama (PFO), Pseudo-tertib kedua (PSO), Elovich and resapan intrapartikel (IPD) telah digunakan untuk mengkaji kinetik penjerapan; penjerapan PNP menggunakan semua bahan jerapan menunjukkan bahawa ia mengikuti model kinetik Pseudo-tertib kedua. Manakala, model tidak linear isoterma Langmuir, Freundlich dan Redlich Peterson memberikan data isoterma; di mana, Redlich Peterson paling hampir untuk menggambarkan keputusan keseimbangan, diikuti dengan model isoterma Langmuir dan isoterma Freundlich, untuk penjerap gentian dan mikropartikel, masing-masing. Kapasiti penjerapan maksimum bagi setiap bahan penjerap adalah TMA-(PA6-*g*-GMA), 176.04 mg/g; TMA-(Cot-*g*-GMA) 180.00 mg/g; dan AO-poli(AN-*ko*-AA), 143.06 mg/g. Penilaian termodinamik menunjukkan bahawa penjerapan adalah proses spontan dan eksotermik. Akhirnya, kecekapan penjanaan semula spesifik yang tinggi oleh penjerap telah diperlihatkan.

Data daripada kajian ini menunjukkan bahawa penjerap gentian mempunyai kapasiti penjerapan yang tinggi dan kinetik yang lebih pantas berbanding penjerap berasaskan mikropartikel. Walau bagaimanapun, penjerap mikropartikel dikenali mempunyai kapasiti penjerapan yang jauh lebih tinggi daripada penjerap alternatif seperti dijelaskan dalam kajian sebelumnya. Oleh yang demikian, dijangkakan bahawa penjerap yang direka adalah bahan yang menggalakkan untuk penyingkiran PNP daripada air dan air sisa.

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Shihab Ezzuldin M.Saber Almohamedabas
September 2021

This thesis was submitted to the Senate of the Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Doctor of Philosophy. The members of the Supervisory Committee were as follows:

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Declaration by Members of Supervisory Committee

This is to confirm that:

- the research conducted and the writing of this thesis was under our supervision;
- supervision responsibilities as stated in the Universiti Putra Malaysia (Graduate Studies) Rules 2003 (Revision 2012-2013) are adhered to.

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LIST OF ABBREVIATIONS

AA	Acrylic acid
AN	Acrylonitrile
AO	Amidoxime
BET	Brunauer-Emmett-Teller
Cot	Cotton
DG	Degree of grafting
DTG	Derivative Thermogravimetry
EB	Electron beam
EPA	Environmental Protection Agency
FESEM	Field emission scanning electron microscopy
FTIR	Fourier Transform Infrared Spectroscopy
GMA	Glycidyl methacrylate
KPS	Potassium persulfate
PA6	Polyamide 6
pHpzc	Point of zero charge
RIG	Radiation induced grafting
RP	Redox polymerization
PFO	Pseudo first order
PSO	Pseudo second order
IPD	Intraparticle diffusion
PNP	p-Nitrophenol
SBS	Sodium bisulphate
TMA	Trimethylamine

LIST OF SYMBOLS

A	Initial adsorption rate constant (mg/g min)
B	Desorption rate constant (g/mg)
b_T	Adsorption intensity(J/mol)
$C_\circ$	Initial concentrations
C_e	Equilibrium concentrations
C_{ip}	Constant related to boundary layer thickness (mg/g)
$\Delta G°$	Gibb's free energy change (kJ/mol)
ΔH^O	Enthalpy change (kJ/mol)
ΔS^O	Entropy change (J/mol K)
k_1	Pseudo-first-order rate constant (1/min)
k_2	Pseudo-second-order rate constant (g/mg min)
k_{ip}	Intra-particle diffusion rate constant (mg/g $min^{0.5}$)
k_d	the linear sorption distribution coefficient (q_e/C_e)
K_L	Langmuir constant (L/mg)
K_F	Freundlich constant (mg/g)$(L/mg)^{1/n}$
K_T	Temkin constant (L/g)
K_{RP}	Redlich-Peterson constant related to adsorption capacity (L/g)
α_{RP}	Binding site affinity (1/mg)
B	Isotherm exponent
M	Molecular weight (g/mol)
m	Mass of adsorbent (g)
$q_{e(cal)}$	Calculated adsorption capacity (mg/g)
$q_{e(exp)}$	Experimental adsorption capacity (mg/g)

q_{max}	Monolayer capacity (mg/g)
q_{em}	Average value of experimental adsorption capacity (mg/g)
R	Ideal gas constant of 8.314 J/mol K
RE	Removal efficiency
R_L	Separation factor
R^2	Correlation coefficient
SSE	Sum of square errors

CHAPTER 1

INTRODUCTION

1.1 Research Background

Following the acceleration of residential and industrialization expansion, global concerns relating to the pollution of the environment have become evident, with grave consequences (Zhang et al., 2020). The large-scale manufacture and broad use of differing key substances has led to a range of extremely noxious organic compounds being released into the planet's water bodies. Such products include pharmaceutical, phenols, pesticides, dyes, and personal care items, amongst others (Awfa et al., 2018; Tkaczyk et al., 2020).

Effluent waste products released by various industries often encompass phenolic compounds such as phenol, p-nitrophenol, and etc (Singh & Verma, 2018). These are toxic to humans and can cause a spectrum of medical conditions, from a simple headache to tumorigenesis or unexpected fatality. The global pollution of aquatic areas with phenolic waste has been identified as a present concern of rising magnitude. Indeed, phenolic compounds are deemed to be the principal pollutants of water-based ecosystems; even in trace quantities they are toxic to human, animal and vegetation species (Patel et al., 2020; Vasantha & Jyothi, 2020). The widespread utilization of phenolic compounds in effluents from both industry and urban areas facilitates their introduction into aquatic ecosystems. Examples include leachates arising from waste deposits, effluents discharged by oil refineries and pharmaceutical sites, together with overspill from the agricultural employment of pesticides (Osman, 2014; Othman et al., 2020). Thus, surveillance of these types of pollutants is mandatory in order to maintain a clean and hazard-free environment (Sushma & Yadav, 2020).

p-Nitrophenol (PNP) is a phenolic compound which is both poisonous and resistant to degradation. Huge quantities of PNP are released into effluents owing to its broad spectrum of use industrially, e.g. in pharmaceutical, agricultural industries, dyestuff and via its formation as a spin-off product effluent. PNP can precipitate grave damage to the environment. Its mutagenic properties and toxic effects on both kidney and liver means that it seriously impacts human well-being (Wang et al., 2017). PNP has been recognized in natural water and in effluent as a consequence of its extreme solubility; it is highly stable in aqueous solutions (Mei et al., 2020). Thus, such chemicals are unable to be liberated immediately into water systems without treatment.

Previously, municipal water treatment plants were utilized in order to clean wastewater produced by industrial enterprises. Such methods were reliant on biological activity and were generally noted to be inefficacious for the removal of the more impervious phenolic discharges. Currently, novel modes of treatment have been promoted; this

area is the subject of ongoing study and evolution. Present techniques for removing phenolic substances encompass biological degradation, oxidation, utilizing chemicals such as ozone, hydrogen peroxide or chlorine dioxide, adsorption onto synthetic and natural adsorbents, solvent extraction and membrane separation (Mohd, 2020; Shankar et al., 2020).

Biological destruction and chemical oxidative processes are highly responsive to the working context; when the former takes place without sufficient speed, the outcome can be difficult to anticipate. Thus, adsorption, of all the techniques alluded to, remains the method of choice. It has a more optimum endpoint, can be repeated with accuracy and is also cost-effective (Uddin, 2017; Awad et al., 2019).

The principal forms of adsorbents utilized involve activated carbon, activated alumina, silica gel, molecular sieve carbon, molecular sieve zeolites and polymeric adsorbents. Activated carbons are porous substances frequently employed for the purification of substances in chemical and pharmaceutical activities; they are additionally used within the environment for decontamination interventions. Industrially accessible activated carbons are mostly produced from coal, wood, or coconut shell. They are multi-purpose agents which can uptake a wide range of organic and inorganic materials from solution in both liquid and gas phases. A limitation is that the process is expensive, and the production and recycling of activated carbon is an intensive process. Thus, the hunt for additional options that offer efficacious adsorptive properties is ongoing, leading to additional studies evaluating both synthetic and naturally arising polymers that may offer more adaptable, multi-functional, pragmatic, and low energy solutions together with more optimal functional activity characterized by increased adsorption capacity, high-speed kinetics, and recyclability.

Over recent years, fibrous adsorbents have been the focus of attention as possible options to the more traditional adsorbents; they are inexpensive and their surface exhibits high activity. They have sufficient mechanical strength, the ability for surface chemical change, the capability to be reutilized, and are straightforward to employ (Gao et al., 2017; Khosravi Mohammad Soltan et al., 2021). Additionally, microparticle-based polymers are broadly used for the decontamination and separation of organic materials owing to their wide range of functionality. Thus, they are seen as a valid option to other conventional adsorbents such as activated carbon for the removal of particular organic compounds from polluted water (Gai et al., 2019; Ling et al., 2019).

Radiation-induced graft polymerization (RIG) has been the subject of considerable focus as it is recognized as an efficient method for the development of adsorbents for the removal or retrieval of a number of specific solutes from aqueous sources. Redox polymerization (RP) is a technique in general usage for the synthesis of polymers for a range of purposes. In the current study, two separate routes were utilized to create adsorbents for PNP elimination. Firstly, RIG was deployed to prepare fibrous adsorbents, i.e. glycidyl methacrylate (GMA), grafted onto synthetic polyamide 6

(PA6) and natural cotton (Cot) fibers, respectively, and then functionalized with trimethylamine (TMA). Secondly, RP was used to synthesize microparticle adsorbents from acrylonitrile (AN) /acrylic acid (AA) copolymers; amidoxime (AO) was used for copolymers functionalization.

1.2 Problem Statement

One of the derivatives of phenol, PNP which has been deemed to be a priority contaminant to the environment by the United States Environmental Protection Agency (USEPA) owing to its unremitting poisonous potential (Panagos et al., 2013; Fatima et al., 2019). Industrially, the annual production of PNP could hit several tons to meet the world demand because of its uses; inevitably, some leaches into the ecosystem. As per Malaysia's Environmental Quality Act, the permitted limits for phenolic substances in wastewater should not exceed 1.0 µg/L (Standard A effluent) and 1.0 mg/L for (Standard B effluent) (DOE, 2010; Shaarani & Hameed, 2010). Thus, multiple methods have been developed in order to purify wastewater including photocatalytic oxidation (Ojha et al., 2019; Rodríguez-Romero et al., 2019), electrolysis (Cheng et al., 2007; Zhang et al., 2020), adsorption (Nakhjiri et al., 2021; Rong & Han, 2019), oxidation (Chen & Shih, 2020; Faria et al., 2007), biodegradation (Wei et al., 2020) and membrane separation (Tan et al., 2019; Alshabib & Onaizi, 2019). Of the suggested methods, adsorption processes are generally the most practical owing to their efficiency and lower cost.

Employment of polymeric adsorbents, i.e. based on fibrous or particles structures is a promising strategy for the removal of organic pollutants by chemical or physical adsorption from contaminated solutions. However, due to the wide ranges of potential contaminants and adsorbents, respectively, it can be challenging to choose a proper adsorbent for a particular treatment context. In order to find a solution to this problem, it is essential to comprehend the overall adsorption procedure and to estimate the concentration of a specific substance that is adsorbed by a particular adsorbent. This, therefore, necessitates accurate predictive modelling and mechanistic understanding of major interactions occurring within the process, data that are largely not available. Moreover, despite the fact that regeneration of a spent polymer is achievable in ambient conditions with a minimal degree of loss, activated carbon, in contrast, is characterized by the need for an expensive heat-driven renewal technique, greater energy requirement and higher rates of attrition. These challenges have driven greater research efforts to develop adsorbent materials utilizing alternative methods for the treatment of water and wastewater.

Electron beam (EB) radiation for RIG offers an efficacious and practical way in which to graft a monomer onto a polymer substrate. Adsorbents created via this method are potentially superior options due to exhibits unique advantages including greater efficiency and fast, non-toxic to the environment and thus does not cause any further pollution associated with toxic chemical or catalysts. Furthermore, RP is in general usage. It enables multiple monomers to undergo polymerization and can be conducted in the presence of moderate conditions. A positive outcome of this research will offer

solutions to several difficulties linked with the development of industrial viable valid and proper functional adsorbents for the treatment of wastewater containing organic environmental contaminants.

1.3 Research Goal and Objectives

This study has two principal objectives.

The initial objective relates to the creation of the adsorbents. The absorbents that are fibrous based consist of GMA grafted onto PA6 and cotton fibers, respectively; these are then functionalized using TMA. The adsorbents that are microparticle-based comprise AN/AA copolymers, functionalized with AO. Both forms were evaluated in order to establish their properties to adsorb PNP from aqueous solutions.

The second main objective was to determine the adsorption properties of these adsorbents, including their optimization during the adsorption process, kinetics, isotherm, thermodynamics, and regeneration properties.

Detailed objectives of this research include:

1. To characterize the chemical and physical properties of TMA-(PA6-*g*-GMA), TMA-(Cot-*g*-GMA) and AO-poly(AN-*co*-AA) prepared under different reaction conditions.
2. To optimize and compare the preparation of fiber-based and microparticles-based adsorbents for PNP adsorption.
3. To evaluate adsorption performance of the TMA-(PA6-*g*-GMA), TMA-(Cot-*g*-GMA) and AO-poly(AN-*co*-AA) adsorbents for removal of PNP from aqueous solution.

1.4 Scope of the Study

To achieve the above research objectives, this research focuses on:

RIG and RP methods, respectively, were utilized for the synthesis of the PNP-selective adsorbents. The RIG process was performed in three-stages, i.e. irradiation, grafting and finally, chemical treatment with TMA. The first absorbent was generated by RIG of GMA onto PA6 fibers, utilizing a radiation dose range of 10-50 kGy and a reaction time of 20-180 minutes at 40 ˚C. The second adsorbent was created by RIG of GMA onto natural cotton fibers, using a radiation dose range of 5-50 kGy and a reaction temperature of 50 ˚C. The two irradiated grafted fibers were functionalized with TMA solution.

PAN-based copolymers were engineered using five varied ratios of AA, which were added to the PAN chain in order to increase the hydrophilic characteristics by the integration of a carboxyl moiety. Hydroxylamine hydrochloride (HH) was utilized to achieve chemical functionalization of the poly(AN-*co*-AA) by the AO. The physiochemical characteristics of the engineered adsorbents, encompassing morphology, surface chemical functional, elemental composition, structural, textural traits, thermal stability, and point of zero charge, were characterized using a number of strategies, including Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), field emission scanning electron microscopy (FESEM), Brunauer–Emmett–Teller (BET) evaluation, thermogravimetric analysis (TGA) and pHpzc assessment.

The adsorption studies were performed using a range of variables, e.g. adsorbent dose, initial solution pH, adsorption process temperature, initial PNP concentration and contact time. PNP adsorption kinetics and the adsorption mechanism for the adsorbents, non-linear models for Pseudo-first order, Pseudo-second order, Elovich and Intraparticle diffusion were investigated. Equilibrium isotherm studies were appraised with the use of non-linear isotherm adsorption models, i.e. Langmuir, Freundlich, Temkin and Redlich-Peterson models. PNP adsorption, thermodynamic properties, Gibb's free-energy change ($\Delta G°$), enthalpy change ($\Delta H°$) and entropy change ($\Delta S°$) were measured. The regeneration properties of the absorbents with respect to the desorption of PNP were additionally studied utilizing the eluents hydrochloric acid (HCl), nitric acid (HNO_3), sodium hydroxide (NaOH) and ethanol (EtOH).

1.5 Novelty and Contribution of Research Study

On both small and large scales, phenols and PNP contaminants are released into aqueous streams from oil and petrochemical plants, pharmaceutical industries, textile manufacturers, paint, and pesticide businesses. They are also generated as by-products from a number of industrial processes. Functionalized polymer-based adsorbents have a broad spectrum of heightened physical and chemical characteristics that make them encouraging materials for wastewater separation and decontamination techniques.

In the current research, TMA functionalized fibrous adsorbents have the potential ability to surmount the difficulties faced by other types of adsorbents in relation to adsorption capacities and kinetics. The method comprised a shortened RIG process based on inexpensive PA6 and easily accessible and renewable natural cotton fibers. At the same time, PAN-based microparticles, functionalized with AO, were formed under moderate conditions by RP. The evident novelty of the current study includes the engineering of the three adsorbents utilized for PNP adsorption, the comparison of their adsorption performance, delineation of their mechanisms of PNP uptake, and their regeneration abilities. This study provides data to determine the potential industrial feasibility of the designed adsorbents. The latter could significantly diminish both the financial burden of wastewater purification and the accrual of solid waste. The majority of industrial effluents contain more than one type of organic contaminants; thus, the use of adsorbents can be expanded to remove additional soluble organic compounds and in particular, substances that contain hydroxyl and nitro groups.

1.6 Thesis outline

This thesis has been organized into six chapters and covers all the detail aspects of this research study.

Chapter 1 covers the background, problem statement, research objectives, scopes, and novelty of the study.

Chapter 2 presents the detailed and up to date literature review on phenolic compounds contaminants especially PNP in wastewater, sources, toxicity effects and various treatment technologies for their removal. The theoretical backgrounds of adsorption study for the adsorbate-adsorbent system are explained. The description of the latest information of various adsorbents' preparation methods provided as well.

Chapter 3 shows the overall research methodology and procedures. All materials and chemicals related to mentioned goals are offered. The second part showed the engineering of the adsorbents followed by characterization approaches of the adsorbents including FTIR, XRD, FESEM, BET, pHpzc and TG-DTG. The last part includes adsorption studies in detail.

Chapter 4 contains the results and discussion and description of the adsorbents, preparation, characterizations, and their performance for PNP adsorption.

Chapter 5 presents overall conclusions and the recommendations for future studies.

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