International Journal of Scientific Research in Chemical Sciences
Vol.10, Issue.4, pp.17-23, August 2023
E-ISSN: 2455-3174
Available online at: www.isroset.org

Research Paper

Kinetic modelling of *p*-Nitrophenol ions adsorption onto activated and non-activated carbon from macadamia nutshells in a model solution

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Received: 10/Jun/2023; **Accepted**: 17/Jul/2023; **Published**: 31/Aug/2023

Abstract— Water contamination caused due to organic pollutants has been a significant issue because they have a tendency to build up within the organism to hazardous concentrations. Additionally, they are often resistant to degradation and, as a result, persist in the environment for prolonged periods. Among the most common phenol derivatives is *P*-Nitrophenol (PNP), which is one of the most common and toxic pollutants found in wastewater. The present study investigates the potential of utilizing activated carbon derived from macadamia nut shell as an effective means of addressing the presence of P-Nitrophenol (PNP) in wastewater, aiming at its remediation. To introduce, the nutshell underwent charring in a blast furnace operating at a temperature of 600°C. The resulting ash was subsequently activated and employed for the purpose of adsorbing PNP from an aqueous solution. The activated and non-activated adsorbents were employed in order to examine the kinetics of PNP ion binding from a homogenous water solution, utilizing a lot of experimental setup. Sorption behaviour of PNP ions on both unmodified and modified sorbents was assessed using spectrophotometric measurements. Subsequent analysis was performed to analyse the obtained data, with approximate first-order kinetics and second order kinetics. The correlation coefficients (R^2) provided strong evidence the complete set of experimental conditions conformed to the Pseudo-second-order kinetic model, with R2 figures above 0.9851. This finding explained how adsorption process of PNP ions involved a chemisorption mechanism. The modified adsorbent demonstrated higher experimental and calculated adsorption capacities compared to the unmodified adsorbent. Specifically, the modified adsorbent exhibited higher values when it came to the adsorption of PNP ions. The rate constants (k2) values were found elevated in the chemically altered adsorbent compared to the unaltered adsorbent. Among the PNP ions, the highest rate constant recorded was 5.130×10^{1} (mg g $^{-1}$min ^{1}). The findings of the study demonstrated the effectiveness of the adsorbents in removing PNP from wastewater. This promising approach holds the potential to mitigate pollution in the environment resulting from industrial activities, while also offering an economically and environmentally friendly solution.

Keywords— Adsorption, kinetics, macadamia nutshells, chemisorption, pseudo-first-order, pseudo-second order

1. Introduction

Water is an indispensable resource on our planet, playing a vital role in the survival of every living organism. At a global scale, according to the U.S. Geological Survey the production of phenol and acetone in the United States was approximately 4.4 billion pounds in 2020, to meet the global demand which was estimated to be around 11 million metric tonnes in 2019. The report also projected to annual increase of 2.6% in the demand for phenol from 2020 to 2027. This surge in demand is credited to the escalating need for phenol in diverse sectors including automotive, construction, electronics, and healthcare, as outlined in the report by [1]. Therefore, phenolic compounds remain a major problem in Africa continent and some other developing countries.

Water contamination poses a significant issue in Kenya, where an estimated 40% of the population relies on unclean water sources like stagnant wells, dams, rivers, streams, and ponds to meet their basic needs. These water bodies are plagued by bacterial contamination and pollution due to the existence of harmful metals and organic pollutants [2,3].
The industrial sector in Kenya discharges wastewater that contains phenolic compounds, which are highly toxic due to their classification as persistent pollutants based on scientific research. [4]. Consequently, there is a pressing need to eliminate these compounds from the water, and this has posed a significant challenge for environmental researchers and scientists working to safeguard the well-being of the ecosystem and aquatic organisms.

Macadamia nut shells are normally discarded as wastes but they are quite hard and can cause damage to lawnmowers and

other equipment if not properly disposed. Macadamia nut shell as a biological lignocellulose material, comprises of the complex polymer of a polysaccharide and hemicellulose component intimately bound to a complex polymer that provides rigidity and strength to the plant tissue which possesses a complicated chemical structure with both phenolic as well as alcoholic hydroxyl groups being present in the structure [5]. The main function of lignin is to impart rigidity and strength to the cell wall by acting as a structural matrix. Therefore, the method of preparation and the carbon precursor are the characteristics that will determine the texture and surface moieties of the material. Activation is done to increase the adsorbing power of an adsorbent and is often made to alter the physical characteristics like functional groups, coarseness, external area, external charge external energy and surface reactivity [6].

Kinetic studies are essential for understanding and controlling chemical reactions, developing new materials and chemical processes. Kinetic adsorption process depends on the shift of the adsorbate from the bulk of the solution to the interphase of the sorption medium and relies on the dispersion of the adsorbate arranging itself inside the sorbent pores [7]. Frequently employed kinetic models encompass the pseudo-primary-order and pseudo-secondary-order models, which help in the prediction of the rate and the extent of adsorption under different conditions. Pseudo-primary-order model involves a situation where one reactant controls the rate of reaction, acting as the rate-determining step. In contrast, the pseudo-second-order model encompasses a scenario where both reactants actively participate in the reaction, influencing the rate of reaction collectively. [8].

2. Related Work

Related studies have explored the enduring toxicity of halogenated phenolic compounds, investigating their effects on wildlife and humans, elucidating the burdens these compounds impose [4].

Similarly, Oginni *et al*., (2019) [5] used KOH activation to enhance activated carbon by creating porous structures, amplifying surface area, and modifying adsorption properties. This process involves chemical reactions and heat, contributing to improved adsorptive performance for diverse applications.

Moreover, additional investigations were conducted regarding the adsorption and equilibrium analyses concerning phenol and PNP. These studies focused on activated magnetic carbon derived from cauliflower waste, expanding the understanding of its potential for environmental remediation through efficient adsorption processes. [9].

With similar approach, an advanced comparison of the adsorption process of industrial dyes onto adsorbents derived from grape cores in aqueous environments examined the effectiveness of grape cores-based adsorbents in treating water contaminated with industrial dyes [10].

Similar conclusions drawn from kinetic, equilibrium, and thermodynamic investigations pertaining to PNP removal have yielded valuable insights into the viability of utilizing a composite made from clay and Cocos nucifera shell-based adsorbents for water treatment. This approach, integrating natural and modified adsorbents, has advanced the creation of effective and eco-friendly techniques for eliminating PNP from polluted water sources [11].

3. Materials and methods

The study's methodology encompassed a comprehensive array of materials and techniques. The materials employed were carefully selected to meet the research's objectives, while the methods applied were rigorously designed to ensure accurate data collection and analysis

Research design

This study encompassed random selection, preparation of samples, chemical alteration, instrumentation, maximization experiments, steady state, and reaction rates experiments for adsorption of PNP ions.

Chemicals, Reagents and Solvents

The existing methods utilized in this research were formulated with purified water, and substances employed were of superior analytical quality. The PNP substance utilized possessed a purity level of 98%. Furthermore, hydrochloric acid with a concentration of 37%, sodium hydroxide with a concentration of 98%, acetic acid with a purity level of 99.7%, and sodium acetate with a purity of 98% were also employed. All chemicals were procured from Kobian Limited, Kenya.

Preparation of concentrated and diluted solutions

To create the initial solutions with a concentration of 1000 µg/l of PNP, distilled water was utilized. Subsequent concentrations were attained by progressively diluting the original stock solutions. To adjust the pH levels, solutions of the concentrations of sodium hydroxide and hydrochloric acid used were both employed.

Modification of the sorbents

Adsorbents can be chemically modified to enhance their properties or customize them for specific applications. This modification can involve various methods, including the use of bases, acids, oxidizing agents, or reducing agents. Surface functional groups on adsorbents play a crucial role in adsorption processes. Modifying adsorbents can influence these surface functional groups by introducing or altering chemical moieties on the surface. Reducing agents, which have the ability to donate electrons to other substances, can be used as one type of modifying agent. When reducing agents are utilized to modify adsorbents, they have the capability to introduce fresh functional groups or alter the ones already present on the surface. As a consequence, the oxidation state of the adsorbents undergoes changes. In this study, the surface modification was accomplished through the utilization of KOH for surface reduction.

The objective of this research was to create high-quality activated carbons with a well-developed microspore architecture within the particles for the removal of PNP from wastewater. To achieve this, macadamia agricultural waste was used as the carbon source and treated with KOH, which permeated the particles through interconnected pores. The process involved heating approximately 300 g and dried macadamia waste in platinum containers in the muffle furnace at 600°C for 2 hrs to yield biochar. The biochar was then modified using a method described in [12] &[13].

Adsorption optimisation Experiments

The trials were employed to enhance the effectiveness of certain factors such as pH level, duration of contact, primary concentration of phenolate ions, and the amount of adsorbent used. To conduct the sorption experiments, screw top containers with a volume of 100 mL were utilized and placed on a mechanical reciprocating shaker model. The shaker operated at a speed of 170 rotations per minute. In order to optimize the impact of these parameters on the absorption process, the remaining factors were kept constant while the specific parameter being studied was systematically varied [2 & 10].

To analyze all the parameters, the UV-Vis spectrophotometer (Analytik Jena, Model Specord 200) was engaged. The spectrophotometer measured the absorption of light at a wavelength of 400 nm, which corresponded to the maximum absorption point [10 & 11].

The quantity of phenolate ions taken up per unit mass of adsorbent was determined using equation 1.

$$qe = (co - \frac{Ce}{\mathrm{m}}) \quad (1)$$

Where the adsorption capacity, denoted as qe, is a measure of the amount of phenolate ions that can be attached to the adsorbent. The overall amount of the solution of phenolate ions, is represented as V in litres, and the mass of the adsorbent, represented as m in grams, are also taken into account in the calculation.

Kinetic modelling

Kinetic modelling involves employing various rate equations to elucidate the process of metal ion absorption, thereby discerning its mechanism. It serves as a valuable instrument for comprehending and enhancing intricate systems, finding extensive utility across diverse domains[15].

Pseudo-first-order

Pseudo-first-order is an expression utilized in chemical kinetics to describe the reaction rate, in the case where only one reactant controls the overall rate, can be described using a pseudo-first-order approach. This approach assumes that the reaction occurs under conditions where the reaction kinetics are not hindered by the relative proportion of a reactant with a higher concentration. Still, it's noteworthy to understand that this assumption may not hold true in all cases. The pseudo-first-order approach should be employed only when the concentration of the reactant with the higher concentration is significantly greater than that of the reactant with the lower concentration. In cases where this assumption does not hold true, a different rate equation may be necessary to describe the reaction kinetics [16], and also vary with the magnitude of occupied sites [17]. Equation 2 provides the linearized form of the given mathematical model.

$$Ln\ (qe - qt) = Ln\ qe - k1t \quad (2)$$

Where qt' refers to the amount of ions that have been adsorbed in milligrams per gram at a specific time 't.' The rate constant for the Pseudo-first-order reaction is denoted as 'k1,' which influences the kinetics of the process. The values of 'k1' and 'qe' can be obtained by constructing a plot of the natural logarithm of the difference between 'qe' and 'qt' against time."

Pseudo-second-order

Apparent-second-order model of kinetic behaviour describes the rate of a chemical reaction by modelling reactions where the rate-controlling factor is thought to involve the adsorption of one or more reactants onto the surface of a catalyst or other adsorbent material. Equation 3 provides the linearized form of the given equation.

$$\frac{t}{qt} = \frac{1}{k2qe2t} + \frac{1}{qet} \quad (3)$$

Where k_2 denotes the second rate constant [18] while the value of q_e and k_2 are calculated by plotting t/q_t against time *t.*

Studies on the dynamic adsorption process

A measurement of 0.1 grams was taken for both the unaltered and adapted adsorbent, which was subsequently placed into plastic bottles. A volume of 20 mL was used. comprising 20 mg/L of PNP compound buffered at pH 4 was added. They were then agitated for 5, 10, 20, 30, 40, 60, 90, 120, 150 and 180 minutes at room temperature. The samples underwent filtration, and the concentrations of the supernatant solutions were determined.

4. Results and discussion

FTIR Results

Results for the FTIR spectrum of non-activated and activated carbon figures showed by [13] indicated functional groups capable of binding to PNP ions. The FT-IR investigation affirmed investigation affirmed the occurrence of amine (-NH2) and nitro (NO2) functional clusters in the UMNS adsorbent, indicating their existence. Conversely, the MMNS adsorbent displayed hydroxyl (-OH) and carboxyl (-COOH) groups, which were advantageous for the process of adsorption. The findings aligned with the intended purpose of activating carbon derived from macadamia nutshell using KOH [13].

From figure 3, FTIR (Fourier Transform Infrared) results revealed significant interactions within the compound. π-π interactions between aromatic moieties were evident, enhancing stability. Electrostatic attractions between partially charged ions further contributed to the complex's structural

integrity. Hydrogen bonding involving hydrogen atoms played a crucial role in forming secondary structures. These interactions collectively shaped the compound's conformation and properties. All these information was clearly illustrated [13]

SEM Results

Scanning Electron Microscopy (SEM) analysis exhibited notable findings. The unmodified adsorbent displayed substantial cavities, suggesting its inherent porosity. In contrast, the modified sample showcased an augmented porosity, attributed to cellulose and lignin breakdown during treatment. Moreover, the SEM images depicted PNP ions adsorbed onto these modified structures, effectively obstructing pores. Remarkably, this adsorption was corroborated by significant shifts in FTIR peaks of the modified sample, indicating successful adsorption occurrence. Thus, SEM revealed a morphological transformation, enhanced porosity, and confirmed the effective adsorption of PNP ions onto the modified adsorbent [13].

Kinetic studies

The kinetic isotherms formed connections within the two utilized models, aiding in determining adsorption capacity and rates effectively.

The reaction kinetics were assessed using the Pseudo-first-order and Pseudo-second-order kinetic rate models, where various rate equations were investigated to model the kinetic data and ascertain the mechanism of ion assimilation by the adsorbents [15] as represented by equations 2 and 3, respectively.

These findings were further visualized in figures 1 and 2. Results for the Pseudo-first-order and Pseudo-second-order obtained were presented in the Table 1

Table 1: Kinetics of PNP ions adsorption by UMNS and MMNS adsorbents

Kinetic models	Adsorbent	Parameters	Values
Pseudo first order	UMNS	k_1 (mg/g min^{-1})	0.0089
		$q_{e(calc)}$ (mg/g)	0.7023
		$q_{e(exp)}$(mg/g)	1.7630
		R_2	0.4319
	MMNS	k_1 (mg/g min^{-1})	0.0085
		$q_{e(calc)}$ (mg/g)	0.7432
		$q_{e(exp)}$ mg/g)	2.0010
		R_2	0.3999
Pseud second order	UMNS	k_2 (mg/g min^{-1})	0.0048
		$q_{e(calc)}$ mg/g	1.8790
		R_2	0.9907
	MMNS	k_2 (mg/g min^{-1})	0.0513
		$q_{e(calc)}$ (mg/g	2.2200
		R_2	0.9851

In the pseudo-first-order reaction, UMNS demonstrated a slower adsorption rate (k1) than MMNS. The adsorption mechanism in Figure 3 indicated chemisorption, involving chemical reactions between the material surface and adsorbate, favoring pseudo-second-order behavior.

The quantity adsorbed (qe) in both unmodified and modified samples was comparatively lower in the pseudo-first-order than in the pseudo-second-order, confirming the latter as the preferred model. Smaller R2 differences between experimental and calculated values validated pseudo-second-order as the superior model.

From Table 1, experimental and calculated *qe* values of PNP ions for UMNS and MMNS adsorbents in pseudo-second-order were nearly identical implying a chemisorption adsorption mechanism. Still, adsorption rate (k_2) was high in MMNS (5.130×10^{-2} mg/g min^{-1}) than in UMNS with (4.834×10^{-2} mg/g min^{-1}) for pseudo-second-order.

The rate of adsorption recorded higher values (0.051 mg/g) than those obtained in a similar approach [19] with 0.005 mg/g, meaning that this study was the best alternative. The adsorption capacity data for qe_{cal} were higher in the MMNS than UMNS with values 1.879 mg/g and 2.220 mg/g respectively. These findings demonstrated that the chemical alteration not only enhanced the velocity of adsorption but also increased the capacity for adsorption. This was due to the change in the structure to highly microporous one with the formation of hydroxyl functional groups on the activated carbon surface [5], by Abdel-Ghani *et al*., (2016) and Dhorable *et al*., (2016).

Greater adsorption rate values within the pseudo-second-order model suggest its superiority and also signify that chemical modification enhanced the adsorption process.

Figure 1 illustrates a pseudo-first-order plot depicting the adsorption of PNP ions onto both activated and non-activated carbon materials.

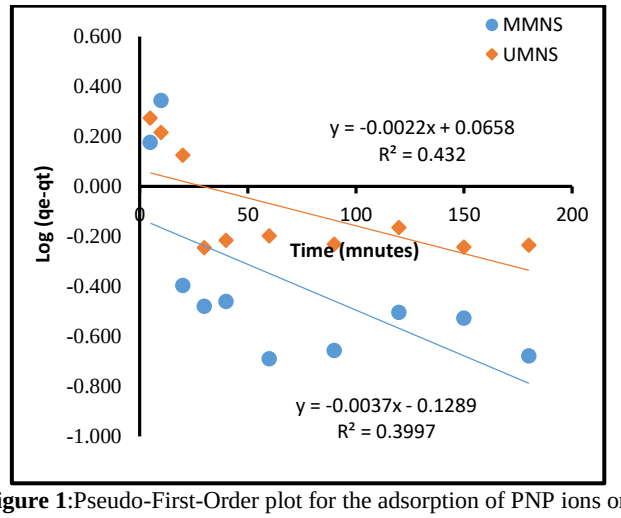

Figure 1:Pseudo-First-Order plot for the adsorption of PNP ions on MMNS & UMNS

Based on Figure 1, the PNP ions exhibited low R-squared values when explored employing the pseudo-first-order approximation. Additionally, the calculated and experimental values diverged significantly, indicating that the data did not align with this particular model.

While the pseudo-first-order plot in Figure 1 provides insight into the adsorption of PNP ions onto activated and non-activated carbon, it's important to note that this model might not be the most suitable fit for the adsorption process. The pseudo-first-order kinetics assumes a linear rate equation, suggesting a constant fraction of adsorbate is being adsorbed per unit time.

However, many adsorption systems exhibit more complex behavior, where the rate of adsorption might not follow a simple linear pattern. In cases involving intricate interactions, multilayer adsorption, or complex surface phenomena, alternative kinetic models like pseudo-second-order or intra-particle diffusion kinetics could provide a more accurate representation of the adsorption process. Therefore, the choice of kinetic model should be carefully considered based on the specific adsorption system and its underlying mechanisms.

Pseudo-second-order plot for the adsorption of PNP ions on MMNS & UMNS was obtained as shown in figure 2.

From figure 2, Pseudo-second-order kinetics yielded an $R^2 >$ 0.98. This phenomenon can be designated to a chemisorption process where the rate-limiting step involves the exchange of electrons between surface sites that are rich in electrons and ions that are deficient in electrons [20].

The selection of an appropriate kinetic model to describe an adsorption process is crucial in understanding and optimizing the process. The pseudo-second-order kinetic model has gained significant attention and acceptance due to its ability to provide a more accurate representation of adsorption behavior compared to other models.

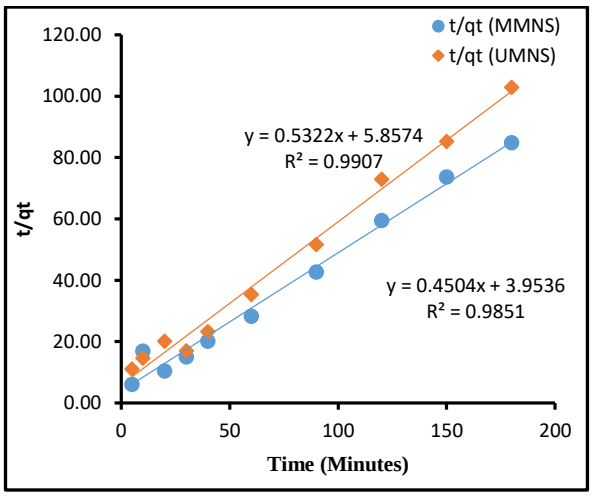

Figure 2:Pseudo-second-order plot for the adsorption of PNP ions on MMNS & UMNS

This model is widely used in various fields including wastewater treatment, pollutant removal, and material synthesis, where adsorption processes play a pivotal role. In this investigation, pseudo-second-order kinetic model was based on the assumption that the rate-limiting step of the adsorption process involved chemisorption, where the adsorbate molecules were strongly attached to the adsorbent surface through chemical bonds.

This assumption is often valid for many adsorption systems, especially those involving the removal of pollutants from aqueous solutions. In chemisorption, the adsorption capacity of the adsorbent material was not solely governed by the availability of surface sites but also by the affinity and strength of the chemical interaction between the adsorbate and the adsorbent.

Other factors such as the physicochemical characteristics of the system, experimental conditions, and additional analyses were typically considered to provide a comprehensive understanding of the chemisorption process in this investigation.

Adsorption mechanism of PNP adsorption onto MMNS was elaborated as shown in figure 3.

The mechanism of interaction between a modified adsorbent and an adsorbate can vary depending on the nature of the modification and the specific characteristics of the adsorbent and adsorbate involved. Modified adsorbents refer to materials that have undergone some form of treatment or alteration to enhance their adsorption properties. In this study, the modification involved changes to the surface chemistry as pointed out in FTIR, and morphology depicted in SEM results, or structure of the adsorbent material. The interactions between modified adsorbents and adsorbates can be broadly categorized into physical and chemical interactions.

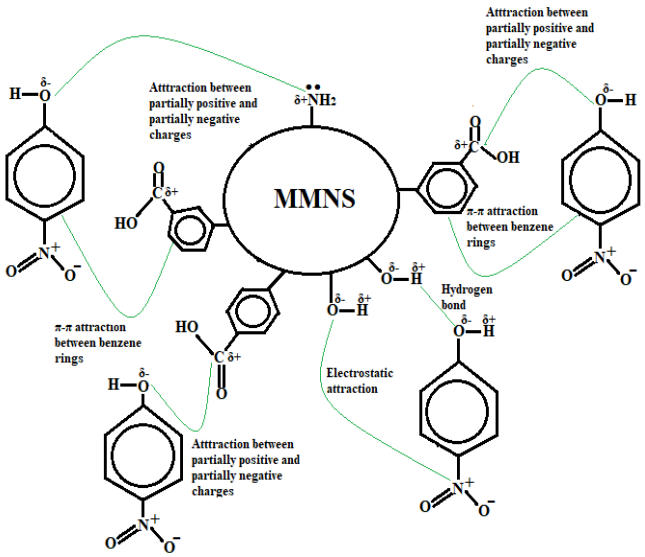

Figure 3:Mechanism of PNP adsorption onto MMNS
Source: [17–19]

Based on the data presented in Table 1, experimental and calculated *qe* values of PNP ions for UMNS and MMNS adsorbents in pseudo-second-order were nearly identical implying a chemisorption adsorption mechanism. Still, adsorption rate (k_2) was high in MMNS (5.130×10^{-2} mg/g min^{-1}) than in UMNS with (4.834×10^{-2} mg/g min^{-1}) for pseudo-second-order. The rate of adsorption recorded higher

values (0.051 mg/g) than those obtained in a similar approach [19] with 0.005 mg/g, meaning that this study was the best alternative. The adsorption capacity data for qe_{cal} were higher in the MMNS than UMNS with values 1.879 mg/g and 2.220 mg/g respectively. These findings demonstrated that the chemical alteration not only enhanced the velocity of adsorption but also increased the capacity for adsorption. This was due to the change in the structure to highly microporous one with the formation of hydroxyl functional groups on the activated carbon surface [5], by Abdel-Ghani *et al*., (2016) and Dhorable *et al*., (2016).

5. Conclusions

The FT-IR results confirmation showed that the UMNS adsorbent contained amine ($-NH_2$) and nitro (NO_2) functional groups, while the MMNS adsorbent contained hydroxyl (-OH) and (-COOH) groups in addition which are favourable for adsorption. This supports the objective of activating carbon derived from macadamia nutshell using KOH. Therefore, the modification significantly improved the adsorption properties of the material. The parameters of pH, contact time, sorbent dosage on PNP ions concentration highly affected the kinetic modelling with the chemisorption process that demonstrates an escalated rate that follows second-order kinetics, surpassing the expected behaviour with elevated values of 5.130×10^2 mg/g min $^{-1}$, PNP ions in the modified adsorbent. In general, it can be concluded that the macadamia nutshell wastes have the potential to effectively remove PNP ions from any industrial wastewater when modified with KOH.

Initially, the proposed mechanism for PNP adsorption was the electron donor mechanism. However, during the adsorption process, it was noted that other mechanism such as π-π interaction between the benzene ring and the solvent through hydrogen bonding mechanism also played a role. These additional mechanisms contributed to the overall adsorption process and helped to enhance the adsorption efficacy of the material. These observations highlight the complexity of adsorption processes and the need for a thorough understanding of the underlying mechanisms in order to optimize the performance of adsorbent materials for specific applications; See figure 3 [17–19].

Data Availability

The information will be accessible via specified avenues. The writer will guarantee this by either placing it in a respected data library or granting access upon inquiry. This approach will enhance openness, replicability, and the chance for additional assessment or confirmation by fellow experts, thus fostering cooperation and enriching the progress of scientific knowledge.

Conflicts of interest

The writers assert no conflicting concerns associated with this research and agree to submit the work to the Journal "International Journal of Scientific Research in Chemical Sciences'' The work has not been published to another journal.

Funding source

The funding for the author comes from a distinguished scholarship granted by Dedan Kimathi University of Technology.

Author's contribution

The authors' investigation into the adsorption properties of activated carbon derived from macadamia nutshell brings not only scientific advancements but also potential benefits to society. By successfully enhancing the adsorption capacity through chemical modification with KOH, the author's work paves the way for highly efficient and cost-effective removal of PNP ions from industrial wastewater. This holds significant promise for addressing environmental pollution and ensuring cleaner water resources. Furthermore, the utilization of macadamia nutshell waste as adsorbent showcases the potential for sustainable waste management practices, reducing waste accumulation and promoting a circular economy. Overall, the author's contribution contributes to both scientific understanding and practical applications, ultimately benefiting society by tackling water pollution and promoting sustainable solutions.

Acknowledgment

I express my gratitude to the supervisors for their invaluable guidance throughout this study, and I would also like to extend my thanks to Dedan Kimathi University of Technology, United States International University, and Geoprobe Limited for their significant contributions to this research.

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