

SYNTHESIS AND CHARACTERIZATION OF BIOMASS-DERIVED ACTIVATED CARBON FROM PALM SEEDS FOR THE ADSORPTIVE REMOVAL OF METRONIDAZOLE FROM AQUEOUS SOLUTIONS: INFLUENCE OF OPERATIONAL PARAMETERS

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ABSTRACT

Pharmaceutical contaminants, especially antibiotics such as metronidazole, pose a growing environmental threat due to their persistence in aquatic systems and resistance to conventional wastewater treatment. This study investigates the synthesis, characterization, and adsorption performance of biomass-derived activated carbon prepared from palm kernel shells and chemically modified through acrylonitrile grafting for the removal of metronidazole from aqueous solutions. The adsorbent was characterized using FTIR, SEM-EDS, and XRD analyses, which confirmed successful chemical modification and the development of a porous, nitrogen-rich carbon structure. Batch adsorption experiments were conducted to evaluate the effects of key operational parameters, including pH, agitation speed, contact time, and temperature. The results demonstrated that adsorption was most effective under acidic conditions (pH 2), at moderate agitation (250 rpm), and elevated temperatures (45°C), with a maximum adsorption capacity of 45.83 mg/g achieved after 15 minutes. Kinetic revealed that the process followed pseudo-first-order kinetics, suggesting a predominantly physical adsorption mechanism. These findings highlight the potential of chemically modified palm-based adsorbents as low-cost, sustainable alternatives for the efficient removal of pharmaceutical contaminants from water.

Keywords: Metronidazole Adsorbent Kinetics Model Operational Parameters

INTRODUCTION

Pharmaceuticals have increasingly been identified as emerging contaminants in water systems due to their continuous input and limited removal by conventional wastewater treatment processes. Antibiotics, in particular, are widely used in human and veterinary medicine and are often excreted as active metabolites into wastewater streams (Kümmerer, 2009). Their presence in aquatic environments, even at trace levels, can lead to antibiotic resistance and toxicological effects in aquatic organisms (Baquero., 2008. Aus der et al., 2016)

Metronidazole, a synthetic nitroimidazole antibiotic, is commonly prescribed for treating anaerobic bacterial and protozoal infections. It is considered moderately persistent in aquatic environments and exhibits a low sorption affinity to sludge, contributing to its mobility in water systems (Ternes., 2004). Studies have reported its detection in treated wastewater, surface water, and even groundwater at concentrations ranging from nanograms to micrograms per liter (Zhou et al., 2017; Kümmerer, 2009). As

metronidazole is not fully degraded in conventional treatment plants, there is a growing need for advanced, sustainable removal technologies.

Advanced Treatment Methods for Antibiotic Removal

Various technologies have been investigated for the removal of metronidazole and other antibiotics from water, including advanced oxidation processes (AOPs), membrane filtration, and biological treatment. AOPs, such as ozonation, Fenton reactions, and photocatalysis, can effectively degrade pharmaceuticals, but they often involve high energy input, costly reagents, and may generate toxic by-products (Ahmad et al., 2017). Membrane-based systems offer high removal efficiencies but are expensive and prone to fouling (Wang et al., 2009). Biodegradation processes have also been explored, but antibiotics tend to inhibit microbial activity, which limits the efficiency of biological treatments (Jelic et al., 2011).

Among these, adsorption has emerged as a cost-effective, efficient, and scalable method, particularly suitable for treating dilute concentrations of micropollutants (Foo and Hameed., 2010). Activated carbon is widely recognized for its superior adsorption properties due to its large surface area, tunable pore structure, and chemical stability.

Biomass-Derived Activated Carbon as a Sustainable Adsorbent

Commercial activated carbon is effective but expensive and derived from non-renewable resources like coal and peat. The development of low-cost adsorbents from agricultural wastes has gained momentum due to their economic and environmental advantages. Biomass such as coconut shells, rice husks, banana peels, sawdust, and palm kernel shells has been investigated as precursors for activated carbon production (Ioannidou and Zabaniotou., 2007; Tan et al., 2008; Babel and Kurniawan., 2003). Palm seeds, an underutilized by-product of the palm oil industry, are rich in lignocellulosic material and have demonstrated potential for high-yield activated carbon production (Daud and Ali., 2003). Activation, either physical or chemical (using agents such as KOH, ZnCl₂, or H₃PO₄), significantly enhances the surface area and pore volume of the carbon, making it suitable for adsorption applications (Deng et al., 2010). The conversion of such agro-waste into functional adsorbents contributes to circular economy goals by turning waste into valuable materials for environmental remediation.

Effect of Operational Parameters on Adsorption

The efficiency of the adsorption process is influenced by several factors, including pH, contact time, agitation speed, temperature, and adsorbent dosage. These parameters govern the surface

interactions, electrostatic forces, and diffusion rates between the adsorbent and adsorbate.

pH affects the surface charge of the adsorbent and the ionization state of the adsorbate. For metronidazole, acidic conditions often favor adsorption due to increased electrostatic attraction between the protonated adsorbent surface and the anionic form of the drug (Mohan and Pittman., 2007).

Contact time determines the kinetics of adsorption. Many studies report that adsorption of antibiotics onto activated carbon is rapid initially due to the availability of active sites, followed by a slower phase as equilibrium is approached (Ho and McKay., 1999).

Agitation speed enhances the mixing and mass transfer rate. Higher agitation generally reduces the boundary layer thickness around adsorbent particles, improving adsorption rates (Crini and Badot., 2008).

Temperature can either enhance or reduce adsorption depending on whether the process is endothermic or exothermic. In many cases, increased temperature enhances diffusion and adsorption capacity, indicating endothermic behavior (Ahmad *et al.*, 2017).

MATERIALS AND METHODS

Palm kernel Shell Powder, Hydroxylamine, HCl, Acrylonitrile, Ammonium persulfate, Tetracycline, Ethanol.

Preparation of adsorbent

The adsorbent was produced according to the procedure adopted by Jaafar *et al.* (2020), with minor modifications. The palm kernel shell was obtained from a local vendor, which was washed thoroughly with deionized water to remove surface impurities. It was then dried under the sun for at least 48 hours and further dried in a vacuum oven at 100°C for an hour to eliminate moisture content. The palm kernel shell was crushed using a mortar mill and further sieved to obtain a desirable size. The final product was placed in a plastic container and stored in a silica gel desiccant for further experimental use.

Preparation of 20% v/v hydroxylamine solution and 20% v/v acrylonitrile

For every 100 ml of the final solution, 24g of hydroxylamine and 80 mL of distilled water (solvent) were used. A measuring cylinder or pipette was used to measure out the required volume of hydroxylamine and the appropriate volume of distilled water (solvent) separately. In a mixing container, the measured hydroxylamine was added first, followed by the distilled water. The solution was stirred gently to ensure that the hydroxylamine was completely mixed with the water. The container was labelled with the concentration and date, and stored according to safety guidelines, as hydroxylamine can be hazardous. The same procedures were used to prepare a 20% v/v solution of Acrylonitrile, using 20 mL of acrylonitrile and 80 mL of solvent (distilled water) to achieve a total volume of 100 mL.

Preparation of metronidazole solution with a concentration of 200 mg/L

For metronidazole solution with a concentration of 200mg/L, the required amount was calculated using the formula:

$$\text{Amount}(mg) = \text{desired concentration} \left(\frac{mg}{L} \right) \times \text{volume of solution (L)} \quad (1)$$

For 200 mg/L in 1 litre of metronidazole solution, it was calculated as $\text{Amount}(mg) = 200 \left(\frac{mg}{L} \right) \times 1 \text{ liter}$

$$\text{Amount}(mg) = 200 \frac{mg}{L} \quad (2)$$

An analytical balance was used to weigh out the calculated amounts. The weighed powders were added to a volumetric flask, and a small volume of distilled water (approximately 100 mL) was added to facilitate dissolution of the powders. The solution was stirred using a magnetic stirrer until completely dissolved. Once the powders were fully dissolved, distilled water was added to reach the final desired volume (1 litre). The solution was well-mixed by stirring gently.

Synthesis of acrylonitrile-grafted palm seed powder

The synthesis process was conducted in two distinct stages: chemical modification and grafting. In the first stage, 10 grams of palm seed powder were combined with 50 ml of acetic acid solution (20% v/v) and 1 ml of hydrochloric acid. The mixture was stirred at 80 °C for 2 hours. The chemically modified palm seed powder was then thoroughly washed with distilled water to ensure it was neutral, followed by washing with ethanol, and dried at 60 °C overnight. In the second stage, 10 grams of the modified palm seed powder were mixed with 50 ml of acrylonitrile solution (20% v/v) and 1 ml of an initiator. This mixture was stirred at 70 °C for 4 hours to complete the grafting process. The final product was washed thoroughly with distilled water to ensure neutrality, rinsed with ethanol to remove unreacted substances, and dried at 60 °C, resulting in acrylonitrile-grafted palm seed powder.

Characterisation of adsorbent

The prepared acrylonitrile-grafted palm seed powder was characterised to determine its structural, morphological, and crystallographic properties. Fourier Transform Infrared Spectroscopy (FTIR) was employed to identify the functional groups present and confirm the successful chemical modification and grafting. Scanning Electron Microscopy (SEM) was used to examine the surface morphology and textural properties of the adsorbent. Additionally, X-Ray Diffraction (XRD) analysis was conducted to evaluate the crystalline structure and phase composition of the material. These analyses provided insights into the physicochemical properties of the adsorbent, which were crucial for understanding its adsorption behaviour.

Adsorption experiments varying the agitation Speed

0.100 g of the adsorbent was added to 50 mL of a metronidazole solution with an initial concentration of 200 mg/L. The mixture was subjected to stirring at a speed of 250 rpm under controlled conditions, maintaining a temperature of 25°C and an initial pH of 2, for a contact time of 5 minutes. Following the stirring process, the solution was filtered using filter paper, and the absorbance of metronidazole was determined using a UV-Vis spectrophotometer. This procedure was subsequently repeated under identical conditions, keeping parameters such as temperature, adsorbent dosage, pH, and contact time constant while adjusting the agitation speed to 500 and 750 rpm.

Adsorption experiments varying pH

0.100 g of the adsorbent was added to 50 mL of a metronidazole solution with an initial concentration of 200 mg/L. The mixture was subjected to stirring at a speed of 250 rpm under controlled conditions, maintaining a temperature of 25°C and an initial pH of 2, for a contact time of 5 minutes. Following the stirring process, the solution was filtered using filter paper, and the absorbance of metronidazole was determined using a UV-Vis spectrophotometer. This procedure was subsequently repeated under identical conditions, keeping parameters such as rotation speed, temperature, adsorbent dosage, and contact time constant while

adjusting the pH levels to 7 and 10. The absorbance values obtained at each pH were recorded and analysed.

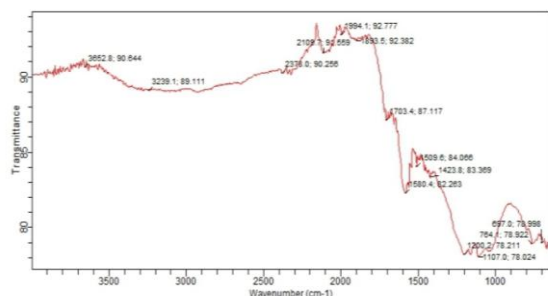
Adsorption experiments varying contact time

0.100 g of the adsorbent was added to 50 mL of a metronidazole solution with an initial concentration of 200 mg/L. The mixture was subjected to stirring at a speed of 250 rpm under controlled conditions, maintaining a pH of 7 and a temperature of 25 °C, for a duration of 5 minutes. Following the stirring process, the solution was filtered using filter paper, and the absorbance of metronidazole was determined using a UV-Vis spectrophotometer. This procedure was subsequently repeated under identical conditions, keeping parameters such as rotation speed, pH, adsorbent dosage, and temperature constant while varying the solution contact time to 10 minutes and 15 minutes, respectively. The absorbance values obtained at each contact time were recorded and analysed.

Adsorption experiments varying temperature

0.100 g of the adsorbent was added to 50 mL of a metronidazole solution with an initial concentration of 200 mg/L. The mixture was subjected to stirring at a speed of 250 rpm under controlled conditions, maintaining a pH of 7 and a temperature of 25 °C, for 5 minutes. Following the stirring process, the solution was filtered using filter paper, and the absorbance of metronidazole was determined using a UV-Vis spectrophotometer. This procedure was subsequently repeated under identical conditions, keeping parameters such as rotation speed, pH, adsorbent dosage, and contact time constant while varying the solution temperature to 35 °C and 45 °C, respectively. The absorbance values obtained at each temperature were recorded and analysed.

RESULTS AND DISCUSSION



Characterisation

Figure 1. FTIR (Fourier Transform Infrared Spectroscopy) analysis.

The FTIR spectrum of the palm seed powder was analysed to identify the functional groups present in the material. The spectrum revealed several characteristic peaks indicative of the material's chemical composition. A prominent peak at 3652.8 cm⁻¹ corresponds to O-H stretching vibrations, typically associated with hydroxyl groups. Another significant absorption was observed at 3239.1 cm⁻¹, representing N-H stretching vibrations, likely from amine groups.

Additional peaks were identified at 2376.0 cm⁻¹ and 2109.9 cm⁻¹, which may be attributed to C≡C or C≡N triple bonds. The absorption bands around 1509.6 cm⁻¹ and 1423.8 cm⁻¹ suggest aromatic ring vibrations. Furthermore, the sharp peaks in the region of 1000–1100 cm⁻¹ correspond to C-O stretching, characteristic of alcohols, esters, or ethers. These functional groups highlight the

organic nature of the material and its potential as an adsorbent, as they facilitate interactions with various adsorbates, including metronidazole.

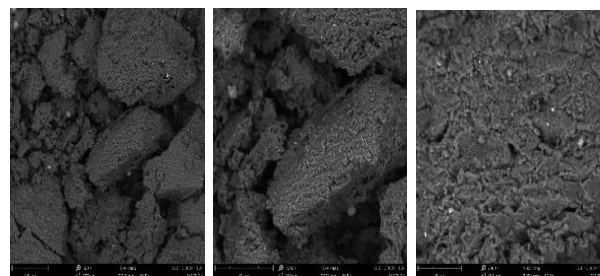


Figure 2. SEM images of the adsorbent Sample at magnifications of 2000× 1000× and 500×

The SEM images of the adsorbent Sample at magnifications of 2000× 1000× and 500× reveal a porous and heterogeneous surface morphology, characteristic of activated carbon derived from biomass. The micrographs display an interconnected network of pores with different sizes, indicating the effectiveness of the activation process in creating a porous structure. These pores are essential for adsorption applications, as they provide accessible sites for the adsorption of metronidazole molecules. The rough and irregular surface further suggests the presence of micro- and mesopores, which enhance the material's surface area and adsorption capacity.

The EDS analysis show that the sample mainly consist of carbon, with atomic concentration of 65.54% and a weight concentration of 59.06%. Nitrogen is also present at an atomic concentration of 30.52%, indicating that nitrogen were incorporated into the carbon matrix during the activation process. Such nitrogen-containing groups can enhance the adsorption performance by introducing active sites for interaction with adsorbates. Other minor elements identified include silicon, aluminium, and iron, which may be attributed to residual impurities from the raw material or activation process.

The porous structure observed in the SEM images, combined with the elemental composition from the EDS analysis, confirms that the material possesses favourable characteristics for adsorption applications. The high carbon and nitrogen content, along with the extensive pore network, make it suitable for capturing pollutants like metronidazole from aqueous solutions. These results align well with the expectations for biomass-derived activated carbon and underscore its potential as an effective adsorbent material.

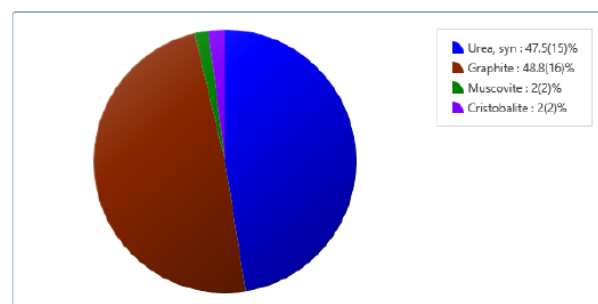


Figure 3a. Showing adsorbent's percentage compound composition

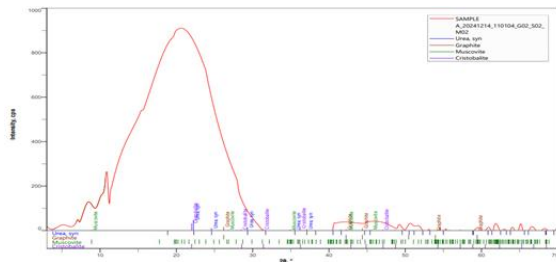


Figure 3b. Showing intensity against 2θ angle for synthesized adsorbent XRD analysis

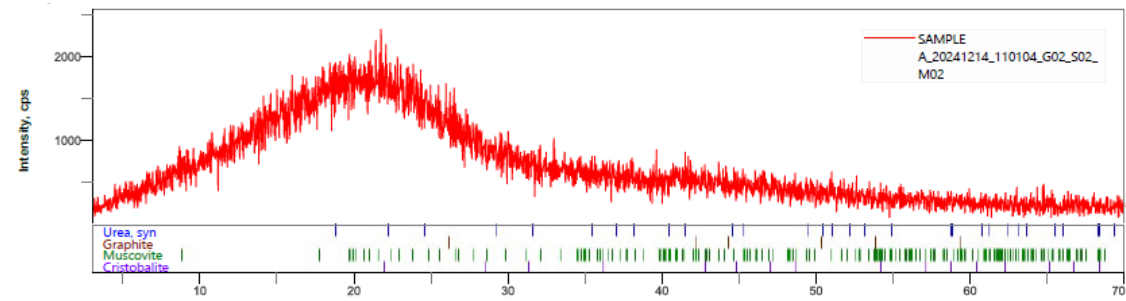


Figure 3c. Showing the crystalline structure and phase composition of the acrylonitrile-grafted palm kernel shell powder

The crystalline structure and phase composition of the acrylonitrile-grafted palm kernel shell powder were examined using X-ray diffraction (XRD). The XRD pattern showed a prominent peak with a d-spacing of 4.1626 Å, identified as Urea ($\text{CH}_4\text{N}_2\text{O}$), indicating successful chemical modification of the material. Other crystalline phases detected include Graphite (C) as the dominant phase, alongside smaller contributions from Muscovite ($\text{KA}_{12}(\text{Si}_3\text{Al})\text{O}_{10}(\text{OH}, \text{F})_2$) and Cristobalite (SiO_2). These phases highlight the structural transformation achieved through the preparation process.

Quantitative analysis of the XRD data revealed the following weight fractions: Urea: 47.51%, Graphite: 48.81%, Muscovite: 2.22%, Cristobalite: 2.29%. The material exhibits a semi-crystalline structure, characterized by sharp peaks associated with crystalline regions and a broad hump indicative of amorphous carbon. The graphitic domains in the material are particularly significant, as they facilitate strong π - π interactions with metronidazole molecules during adsorption. Meanwhile, the trace amounts of muscovite and cristobalite contribute to the material's structural stability and surface characteristics, enhancing its suitability for adsorption applications.

The combination of crystalline and amorphous phases provides a balance between porosity and structural integrity, which is crucial for adsorption. Furthermore, the integration of urea and graphite introduces functional groups that improve the chemical interaction between the adsorbent and metronidazole. These structural properties confirm the success of the modification and grafting processes, demonstrating the material's potential for effective adsorption.

pH Variation

The results in Table 1 indicate that the adsorption efficiency of metronidazole onto the chemically modified palm kernel shell-derived activated carbon is significantly influenced by the pH of the solution. The highest adsorption was observed at pH 2, with an absorbance value of 0.188, likely due to the protonation of the

adsorbent surface, enhancing electrostatic interactions with the negatively charged metronidazole molecules. At pH 7, a moderate adsorption efficiency was recorded, with an absorbance of 0.201, possibly due to a balance between protonation and deprotonation of the functional groups on the adsorbent. Conversely, the lowest adsorption was observed at pH 10, with an absorbance of 0.312, as the alkaline conditions caused deprotonation of the adsorbent surface and ionisation of metronidazole, thereby reducing the interaction between the adsorbate and the adsorbent. These findings highlight the critical role of pH in the adsorption process and suggest that acidic conditions are more favourable for the removal of metronidazole pollutants.

Table 1. Residual Concentration of Adsorbent at Different pH Levels

pH Level	Absorbance (Abs)	Residual Concentration (mg/L)
2	0.188	122.22
7	0.201	129.44
10	0.312	191.11

Agitation Speed Variation

The results in Table 2. show that the adsorption efficiency of metronidazole onto the chemically modified palm kernel shell-derived activated carbon is greatly affected by the agitation speed. The highest adsorption occurred at 250 rpm, with an absorbance value of 0.200, likely because of sufficient contact between the adsorbent and adsorbate. At 500 rpm, a moderate adsorption efficiency was recorded, with an absorbance of 0.180, probably due to increased turbulence and mixing, which slightly improved the mass transfer of metronidazole molecules to the adsorbent surface. In contrast, the lowest adsorption was observed at 750 rpm, with an absorbance of 0.160, as the high agitation speed likely caused a significant reduction in the interaction between the

adsorbate and the adsorbent, thereby decreasing the adsorption efficiency. These findings highlight the critical role of agitation speed in the adsorption process and suggest that lower agitation speeds are more favourable for the removal of metronidazole pollutants.

Table 2. Varying Agitation speed

Agitation speed (rpm)	Absorbance (Abs)
Blank	0.328
250	0.200
500	0.180
750	0.160

Temperature Variation

The calibration curve for metronidazole exhibited a strong linear relationship between concentration and absorbance, with a high correlation coefficient ($R^2 = 0.9619$), confirming the reliability and accuracy of the UV-Vis spectrophotometric method for quantifying metronidazole in solution. Temperature was found to significantly influence adsorption behavior in Table 3. As the temperature increased from 25°C to 45°C, a corresponding decrease in solution absorbance was observed, indicating enhanced adsorption of metronidazole onto the adsorbent. This increase in adsorption capacity at elevated temperatures suggests that the process is endothermic, likely driven by physical interactions such as van der Waals forces and π - π stacking. The improvement in adsorption performance with temperature may be attributed to increased molecular mobility and kinetic energy, which promote more effective interaction and diffusion of metronidazole into the adsorbent's porous structure. These findings highlight 45 °C as the optimal temperature for adsorption in this study and reinforce the potential of modified palm kernel shell as an efficient adsorbent for metronidazole removal from aqueous media.

Table 3. Showing Adsorption capacity at various temperatures

Temperature (°C)	Absorbance (Abs)	Residual Concentration mg/L	Adsorption Capacity mg/g
Blank	0.328	200	0.00
25°C	0.232	146.67	26.67
35°C	0.217	138.33	30.83
45°C	0.201	129.44	35.28

Adsorption results

The absorbance values obtained from the UV-Vis spectrophotometer for different contact times, Residual Concentration calculated using the calibration curve equation, and Adsorption capacity in Mg/g derived using $q_t = C - C_t V/m$ are presented in Table 4.

Where:

C_0 = Initial concentration (200 mg/L)

C_t = Residual concentration at time (given in the table: 160.56, 130, and 108.33 mg/L)

V = Volume of the solution (50 mL or 0.05 L)

m = Mass of adsorbent (0.100 g)

Table 4. Varying Contact Time

Contact Time (minutes)	Absorbance (Abs)	Residual Concentration (mg/L)	Adsorption Capacity (mg/g)
5	0.257	160.56	19.72
10	0.202	130	35
15	0.163	108.33	45.83

Contact Time Variation

The adsorption study demonstrated that the removal of metronidazole using acrylonitrile-grafted palm seed powder was efficient, with adsorption capacity increasing from 19.72 mg/g at 5 minutes to 45.83 mg/g at 15 minutes, highlighting the rapid uptake of the pharmaceutical contaminant. The process followed pseudo-first-order kinetics, as evidenced by the better fit of the experimental data to the pseudo-first-order model ($q_e = 79.01 \text{ mg/g}$, $k_1 = 0.0578 \text{ min}^{-1}$) compared to the pseudo-second-order model, which yielded unrealistic values. This suggests that the adsorption mechanism is likely dominated by physical interactions, with rapid adsorption occurring in the initial stages due to the availability of active sites on the modified adsorbent. The results underscore the potential of the adsorbent for effective and time-efficient removal of metronidazole from aqueous solutions.

Kinetics Models

To describe the adsorption behaviour of metronidazole on acrylonitrile-grafted palm seed powder, two kinetic models were used:

Pseudo-First-Order Model

The pseudo-first-order equation is expressed as:

$$q_t = q_e(1 - e^{-k_1 t}) \quad (3)$$

Where:

q_t = Adsorption capacity at time (mg/g)

q_e = Equilibrium adsorption capacity (mg/g)

k_1 = Rate constant for pseudo-first-order adsorption

t = Contact time (min)

Using the experimental data and curve fitting: $q_e = 79.01 \text{ mg/g}$

$k_1 = 0.0578 \text{ min}^{-1}$

Pseudo-Second-Order Model

The pseudo-second-order equation is expressed as:

$$q_t = \frac{k_2 q_e^2 t}{1 + k_2 q_e t} \quad (4)$$

Where:

- k_2 = Rate constant for pseudo-second-order adsorption (g/mg · min) . (5)

Using the experimental data and curve fitting:

- $q_e = 33.52 \text{ mg/g}$
- $k_2 = -226612.05 \text{ g/mg} \cdot \text{min}$ (unrealistic due to poor fitting)

Conclusion

This study successfully demonstrated the feasibility of utilizing acrylonitrile-grafted palm kernel shell-derived activated carbon as an effective adsorbent for the removal of metronidazole from aqueous solutions. Characterization analyses confirmed the development of a porous, nitrogen-functionalized adsorbent

structure capable of interacting with pharmaceutical molecules. Adsorption experiments revealed that optimal removal occurred at pH 2, moderate agitation speeds, and elevated temperatures, with a maximum adsorption capacity of 45.83 mg/g within 15 minutes. The adsorption process followed pseudo-first-order kinetics, indicating a primarily physical adsorption mechanism driven by van der Waals forces and π - π interactions. The enhancement of adsorption at higher temperatures further suggests that the process is endothermic. Overall, the findings underscore the potential of valorizing agro-waste materials, such as palm seed shells, into high-performance adsorbents for water treatment applications. This approach supports circular economy principles and offers a sustainable, cost-effective solution for mitigating pharmaceutical pollution in aquatic environments.

Conflict of Interest

We declare that there are no known competing financial interests or personal relationships that could have influenced the work reported in this paper. All authors have contributed to the research and preparation of the manuscript, and there are no financial or personal relationships that could inappropriately influence the work described in this study.

Acknowledgement

We strongly acknowledge the financial support received from the Tertiary Education Trust Fund (TETFUND) Institutional-Based Research (IBR) Grant through Kaduna State University Research Centre, which made this research possible.

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