








Adsorptive Removal of Tetracycline from Aqueous Media Using Biomass-Derived Activated Carbon from Palm Seeds

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Article Information

Article Type:

Research Article

Keywords:

Tetracycline removal, Biomass-derived activated carbon, Palm seed adsorbent, Acrylonitrile grafting, Water purification.

History:

Received: 22 June 2025

Revised: 25 September 2025

Accepted: 25 September 2025

Available Online: 6 November 2025

Citation: Ahmad Muhammad Sani, Abdulrahim Ibrahim, A. Abdulrazak, Alhassan Kabiru Usman, Abdullahi Usman Umar, Kamal Dan'azumi, Nathaniel Zaki Elisha, Adsorptive Removal of Tetracycline from Aqueous Media Using Biomass-Derived Activated Carbon from Palm Seeds, Kirkuk Journal of Science, 20(4), p. 1-9, 2025, <https://doi.org/10.32894/kujss.2025.161820.1220>

Abstract

This study demonstrates the efficient removal of tetracycline (96.4%) from aqueous solutions using acrylonitrile-grafted palm seed activated carbon, optimized at pH 10 and lower temperatures (25 °C). Batch experiments revealed that adsorption capacity peaked at 350 mg/L (70% removal) under vigorous agitation (750 rpm), while adsorbent dosage critically influenced performance, with 0.9 g achieving 92.1% removal. Characterization via FTIR, SEM, and XRD confirmed the material's porous structure (65.54% carbon, 30.52% nitrogen) and graphitic crystallinity, enabling strong π - π interactions. The exothermic process exhibited reduced efficiency at higher temperatures (7% at 45 °C) and larger adsorbent-adsorbate solution volume (20.1% at 150 mL). These results highlight the adsorbent's cost-effectiveness and sustainability for water treatment, offering a viable alternative to conventional methods. The adsorbent was chemically modified through grafting with acrylonitrile to enhance its surface functionality and adsorption capacity. These findings demonstrate that acrylonitrile-grafted palm seed activated carbon is a promising, eco-friendly, and efficient adsorbent for removing tetracycline from contaminated water, providing a sustainable alternative to conventional treatment technologies.

1. Introduction:

The occurrence of pharmaceutical contaminants, particularly antibiotics such as tetracycline, in aquatic environments is a growing global concern. Tetracycline is extensively used in human and veterinary medicine to treat a wide range of

bacterial infections, including respiratory tract infections (e.g., pneumonia, bronchitis), sexually transmitted diseases (e.g., chlamydia, syphilis), skin conditions (e.g., acne, rosacea), and zoonotic diseases (e.g., anthrax, brucellosis) [1], [2] skin conditions (e.g., acne, rosacea) [3].

Conventional water treatment processes, including coagulation, oxidation, and biological treatment, often fail to remove tetracycline and related micro pollutants [4] completely. Among advanced treatment technologies, adsorption stands out due to its high efficiency, simplicity, and cost-effectiveness [5]. Activated carbon is widely recognized for its high surface

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area, porosity, and adsorption capacity, making it ideal for removing organic contaminants [6]. However, the high cost of commercial activated carbon limits its practical application, especially in developing regions.

To overcome this limitation, researchers have focused on producing activated carbon from low-cost and renewable biomass materials such as coconut shells [7], rice husks [8], banana peels [9], and palm seeds [10]. Palm seeds are an agricultural byproduct rich in carbon content and are abundantly available in tropical countries. Utilizing palm seeds for activated carbon production not only adds value to agricultural waste but also offers an eco-friendly solution to environmental pollution [11].

This study investigates the synthesis of activated carbon from palm seeds and evaluates its performance in removing tetracycline under various operational parameters. The goal is to introduce the presence of pharmaceutical contaminants, particularly antibiotics like tetracycline, in aquatic environments, which is a growing global concern.

2. Material and Methods:

All chemicals used were of analytical grade:

Palm kernel shell powder (local supplier, Kaduna, Nigeria; washed and sun-dried). Hydroxylamine hydrochloride ($\text{NH}_2\text{OH}\cdot\text{HCl}$, 98.5% purity, Sigma-Aldrich, Germany). Hydrochloric acid (HCl , 37% purity, Merck, Germany). Acrylonitrile ($\text{C}_3\text{H}_3\text{N}$, 99% purity, Acros Organics, USA). Tetracycline hydrochloride ($\text{C}_{22}\text{H}_{25}\text{ClN}_2\text{O}_8$, $\geq 95\%$ HPLC grade, Sigma-Aldrich, Germany). Ethanol ($\text{C}_2\text{H}_5\text{OH}$, 99.8% purity, Merck, Germany). Acetic acid (CH_3COOH , 99.7% purity, Fisher Scientific, UK).

2.1 Preparation of the adsorbent:

The adsorbent was produced according to the procedure adopted by [12], with minor modifications of drying temperature to 100°C instead of 60°C . 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 before being 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 of 1.2 mm. The final product was placed in a plastic container to prevent possible sorption and/or degradation, then stored in a silica gel desiccant for further experimental use.

2.2 Preparation of solution (1):

20% v/v hydroxylamine solution and 20% v/v acrylonitrile was prepared. For every 100 ml of the final solution, 24 g 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.

2.3 Preparation of solution (2):

Tetracycline solution with a concentration of 200 mg/L was prepared. For the tetracycline solution with a concentration of 200 mg/L, the required amount was calculated as

$$\text{Amo}(\text{mg}) = 200 (\text{mg/L}) \times 1 \text{ liter}$$

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 (about 100 mL) was added to help dissolve 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.

2.4 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 0.5 M hydrochloric acid. The mixture was stirred at 80°C for 2 hours on a hotplate. 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 in a vacuum oven for 6 hrs. Resulting in acrylonitrile-grafted palm seed powder.

2.5 Characterization of the adsorbent:

The prepared acrylonitrile-grafted palm seed powder was characterized 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, as in Figure 1 below. 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 physico-chemical properties of the adsorbent, which were crucial for understanding its adsorption behavior.

2.6 Adsorption experiments varying the agitation Speed:

An amount of adsorbent 0.100 g was added to 50 mL of a tetracycline 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 tetracycline 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.

2.7 Adsorption experiments varying pH:

An amount of the adsorbent, 0.100 g, was added to 50 mL of a tetracycline 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 tetracycline was determined using a UV-Vis spectrophotometer at 357nm. 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 analyzed.

2.8 Adsorption experiments varying the solution volume:

To investigate the effect of solution volume on the adsorption of tetracycline, a 0.100g sample of the adsorbent was introduced into tetracycline solutions of varying volumes under controlled conditions. Initially, 50 mL of tetracycline solution with an initial concentration of 200 mg/L was prepared. The mixture was subjected to stirring at 250 rpm, maintained at a pH of 7 and a temperature of 25 °C, for a contact time of 5 minutes. After stirring, the solution was filtered using filter paper, and the residual concentration of tetracycline was determined by measuring the absorbance using a UV-Vis spectrophotometer. To study the effect of varying solution volumes, the procedure was repeated under identical conditions, except that the solution volumes were adjusted to 100 mL and 150 mL, respectively, while keeping all other parameters constant. The absorbance values obtained for each

volume were recorded and analyzed to evaluate the adsorption efficiency.

2.9 Adsorption experiments varying temperature:

An amount of the adsorbent, 0.100 g, was added to 50 mL of a tetracycline 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 Whatman filter paper, and the absorbance of tetracycline 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 analyzed.

3. Results and Discussion:

3.1 Characterization:

The spectrum revealed several characteristic peaks indicative of the material's chemical composition. A weak peak at 3652.8 cm^{-1} corresponds to O-H stretching vibrations, typically associated with hydroxyl groups. And the characteristic peak of the amine group (NH_2), in the range 3400-3300 cm^{-1} , was not observed; this might be due to the overlapping of the NH_2 and OH stretching vibration. Additional peaks were identified at 2376.0 cm^{-1} and 2109.9 cm^{-1} , which may be attributed to CC or CN 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 tetracycline. The SEM images in Figure 2 reveal a porous and heterogeneous surface morphology characteristic of activated carbon derived from biomass. The micrographs show an interconnected network of pores with varying sizes, indicating the effectiveness of the activation process in developing a porous structure. These pores are crucial for adsorption applications, as they provide accessible sites for the adsorption of tetracycline 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 accompanying EDS analysis is shown in the Figure (3a and b) indicate that the sample is predominantly composed of carbon, with a significant atomic concentration of 65.54% and a weight concentration of 59.06%. Nitrogen is also present at an atomic concentration of 30.52%, suggesting the incorporation of nitrogen functionalities into the carbon matrix during the activation process. Such nitrogen-containing groups can enhance the adsorption performance by introducing active

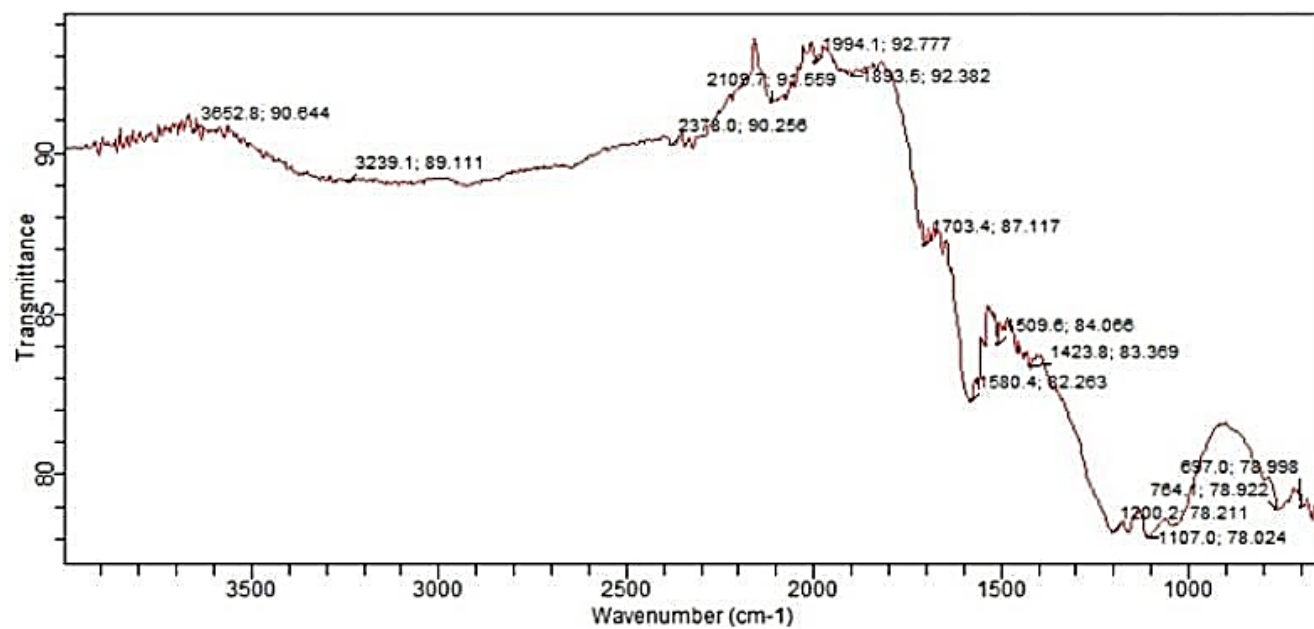


Figure 1. FTIR Spectroscopic analysis of acrylonitrile grafted palm kernel activated carbon.

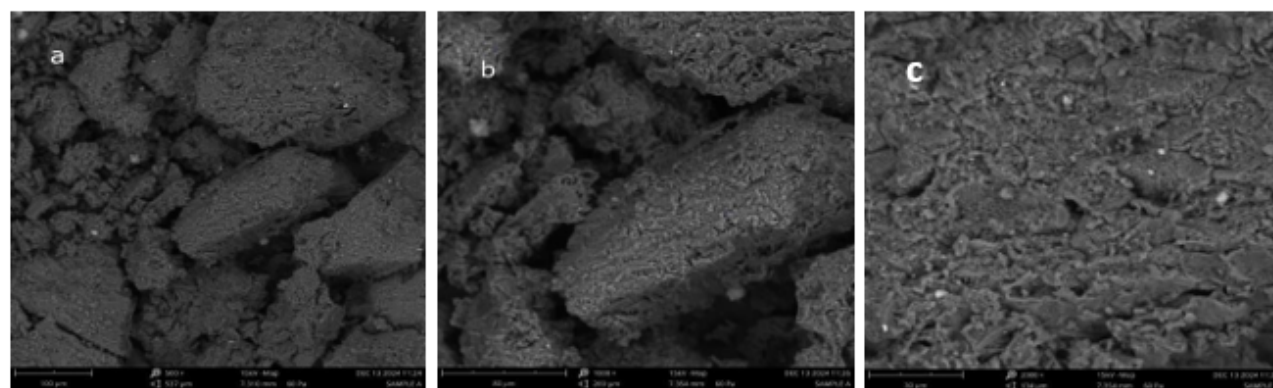


Figure 2. Showing SEM images of the adsorbent, Sample a) 100 μm , 500 magnification, b) 80 μm , 1000, and c) 30 μm , 2000 magnification.

sites for interaction with adsorbates. Other minor elements identified include silicon, aluminum, 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 determined by EDS analysis, confirms that the material exhibits favorable characteristics for adsorption applications. The high carbon and nitrogen content, along with the extensive pore network, make it suitable for capturing pollutants like tetracycline from aqueous solutions. These results align well with the expectations for biomass-derived activated carbon and underscore its potential as an effective adsorbent material. The crystalline structure Figure (3c) 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 -interactions with tetracycline 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 tetracycline. These structural properties confirm the success of the modification and grafting processes, demonstrating the material's potential for effective adsorption.

3.2 Effect of Adsorbent Dosage on Tetracycline Removal:

The removal efficiency of tetracycline increased significantly with an increase in the adsorbent dosage, as presented in Table 1. At a low dosage of 0.1 g, the removal efficiency was only 28.25%, but it increased to 65.75% at a dosage of 0.9 g. This enhancement in adsorption performance is attributed to the greater number of active sites and surface area available for interaction with tetracycline molecules as the quantity of adsorbent increases. The rise in removal percentage from 0.3

g (35.13%) to 0.5 g (42%) suggests a critical threshold at which the adsorption capacity sharply improves due to sufficient surface interaction and mass transfer. This trend is attributed to the increased number of active sites available for adsorption at higher dosages. Similar findings were reported for the adsorption of antibiotics onto various biomass-derived adsorbents [13]. However, the rate of increase diminishes beyond a certain dosage due to possible agglomeration and reduced effective surface area [13]. Table 1

Table 1. Variation of % removal of tetracycline with adsorbent dosage.

Adsorbent dosage (g)	Absorbance (Abs)	Removal of tetracycline (TC) %
0.1	0.258	28.25
0.3	0.247	35.13
0.5	0.236	42.00
0.7	0.219	52.63
0.9	0.198	65.75

3.3 Influence of pH on Adsorption Efficiency:

Solution pH plays a crucial role in the adsorption process by influencing both the surface charge of the adsorbent and the ionization state of tetracycline. As shown in Table 2, adsorption efficiency increased markedly from 35.13% at pH 2 to 96.38% at pH 10. This trend is likely due to the deprotonation of functional groups on the adsorbent surface at higher pH levels, leading to electrostatic attraction between the negatively charged adsorbent and the positively charged tetracycline species. The optimal adsorption at alkaline pH also indicates that palm seed-derived activated carbon may be more effective for treating basic wastewater effluents containing tetracycline. Likely due to the increased negative surface charge of the adsorbent and deprotonation of functional groups [14]. For this research, a pH of 7 was maintained, even though it varied to investigate the effect of its variation, as shown in Table 2. Tetracycline exists in different ionic forms depending on pH, and alkaline conditions favor the adsorption of positively charged species [15]. A similar pH-dependent trend was observed in tetracycline adsorption onto rice husk biochar and bamboo-derived carbon [16], [17].

3.4 Effect of Temperature on Adsorption Performance:

The adsorption efficiency decreased with increasing temperature, as detailed in Table 3. At 25 °C, the removal efficiency was 25.13%, which decreased to only 7.00% at 45 °C. This

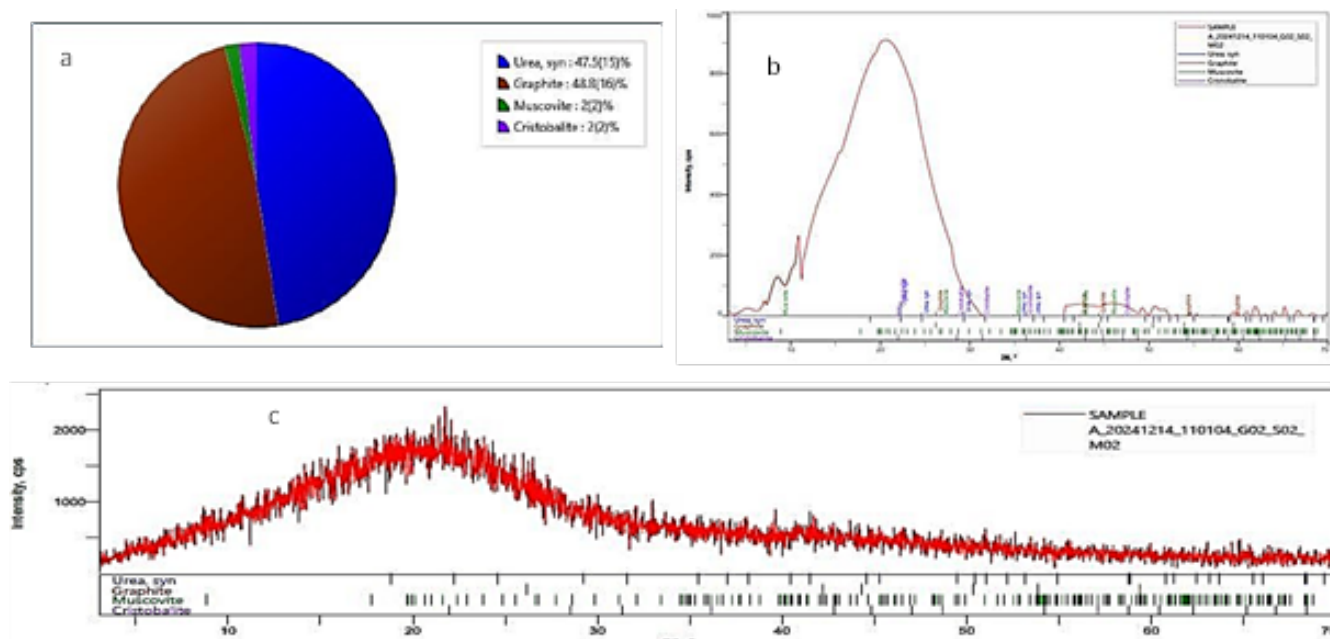


Figure 3. Showing XRD analysis: a) Pie Chart for relative abundance of mineral phase in palm kernel shell powder activated carbon, b) XRD Pattern of activated carbon, and c) Amorphous and Crystallinity of the adsorbent.

Table 2. Residual Concentration and Adsorption Efficiency at Various pH Levels.

pH	Absorbance (Abs)	Residual Concentration (mg/L)	Adsorption Efficiency (%)
2	0.247	129.75	35.125
7	0.204	76.00	62.00
10	0.149	7.25	96.375

inverse relationship suggests that the adsorption of tetracycline onto palm seed-based activated carbon is an exothermic process. The decrease in adsorption efficiency at higher temperatures may be attributed to the weakening of adsorptive interactions and possible desorption of adsorbed molecules due to increased thermal agitation. This suggests an exothermic process where increased thermal motion reduces the affinity between adsorbate and adsorbent [18]. Thermodynamic studies in similar systems have confirmed that adsorption of tetracycline is generally spontaneous and exothermic [19].

3.5 Impact of Agitation Speed on Adsorption Capacity:

As shown in Table 4, increasing agitation speed enhanced both the adsorption capacity (Q_e) and removal efficiency. At 250 rpm, the removal efficiency was 30%, which increased to 70% at 750 rpm. This trend indicates that agitation improves the diffusion of tetracycline molecules to the adsorbent surface and helps to overcome mass transfer resistance. It also

Table 3. Residual Concentration and Removal Efficiency at Different Temperatures.

Temperature (°C)	Absorbance (A)	Residual Concentration mg/L)	Removal Efficiency (%)
Blank	0.210	200.00	0.00
25°C	0.263	149.75	25.13
35°C	0.279	169.75	15.13
45°C	0.292	186.00	7.00

ensures uniform mixing and better contact between adsorbent particles and the solute. This improvement is due to reduced boundary layer thickness and increased external mass transfer rates. Higher agitation ensures better dispersion of particles and more efficient collision between solute molecules and active sites [20].

Table 4. The Adsorption capacity and removal efficiency of tetracycline are tabulated below.

Agitation Speed (rpm)	Adsorption capacity (Q_e) mg/L	Removal efficiency (%)
250	150	30%
500	300	60%
750	350	70%

3.6 Effect of Solution Volume on Adsorption Efficiency:

Table 5 illustrates that increasing the solution volume harmed adsorption efficiency. At a volume of 50 mL, 66.37% of tetracycline was removed, but this dropped to 20.13% at 150 mL. The decline in removal efficiency with increased solution volume may be due to a fixed amount of adsorbent becoming saturated quickly, resulting in reduced availability of active binding sites relative to the tetracycline molecules present in the larger volume. This can be attributed to the fixed amount of adsorbent becoming saturated quickly as the volume (and thus the total tetracycline content) increases [20]. Therefore, optimization of solution volume relative to adsorbent mass is critical for efficient adsorption.

Table 5. The Adsorption capacity and removal efficiency of tetracycline are tabulated below.

Solution Volume (mL)	Absorbance (Abs)	Residual Concentration (mg/L)	Adsorption Efficiency (%)
50	0.197	67.25	66.37
100	0.221	97.25	51.38
150	0.271	159.75	20.13

4. Conclusions:

Palm seed-derived activated carbon is a promising and sustainable adsorbent for removing tetracycline from aqueous solutions. The study demonstrated that removal efficiency is highly dependent on adsorbent dosage, solution pH, and agitation speed. At the same time, it decreases with higher temperatures and higher adsorbent-adsorbate concentration in the solution volume. The material is low-cost, eco-friendly, and offers a viable alternative to commercial activated carbon for antibiotic removal in water treatment systems.

Funding: This research was funded by the Institution-Based Under Tertiary Education Trust Fund through the Kaduna State University Research Centre.

Data Availability Statement: All of the data supporting the findings of the presented study are available from corresponding author on request.

Declarations:

Conflict of interest: The authors declare that they have no conflict of interest.

Ethical approval: This research did not include any human subjects or animals, and as such, it was not necessary to obtain

ethical approval.

Author Contributions: Ahmad Muhammad Sani proposed, defended the research proposal, and secured the research funds. Kamal Danazumi, Alhassan Kabir Usman, and Nathaniel Zaki Elisha collected the raw materials, prepared the samples, and conducted the optimal parameters analysis, respectively. A. Abdulrazak interpreted the results. Abdulrahim Ibrahim and Abdullahi Usman Umar wrote the manuscript and proofread it, respectively. .

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إزالة التتراسيكلين بالامتصاص من الوسائط المائية باستخدام الكربون النشط المشتق من الكتلة الحيوية من بذور النخيل

أحمد محمد ساني * ، عبد الرحيم إبراهيم ، عبد الرزاق ، الحسن كبير عثمان ، عبد الله عثمان عمر ، كمال دنارومي ، ناثانيل زكي إليشا
قسم الكيمياء البحتة والتطبيقية ، كلية العلوم الفيزيائية والحياة ، كلية الهندسة والعلوم والتكنولوجيا ، جامعة ولاية كادونا ،
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الخلاصة

توضح هذه الدراسة كفاءة إزالة التتراسيكلين (96.4%) من المحاليل المائية باستخدام كربون منشط مُطعم بأكريلونيتريل من بذور النخيل، مُحسّن عند درجة حموضة 10 ودرجات حرارة أقل (25 درجة مئوية). أظهرت تجارب الدفعات أن سعة الامتصاص بلغت ذروتها عند 350 ملغم (إزالة 70%) تحت التحريك القوي (750 دورة في الدقيقة)، بينما أثرت جرعة المادة المازة بشكل حاسم على الأداء، حيث حققت 0.9 غرام إزالة 92.1% . أكدت عمليات التوصيف باستخدام تقنيات تحويل فورييه للأشعة تحت الحمراء (FTIR)، والمجهر الإلكتروني الماسح (SEM)، وتقنية حيود الأشعة السينية (XRD) البنية المسامية للمادة (65.54% كربون، 30.52% نيتروجين) وبلوريتها الجرافيتية، مما يُتيح تفاعلات $\pi-\pi$ قوية. أظهرت العملية الطاردة للحرارة انخفاضاً في الكفاءة عند درجات حرارة أعلى (7% عند 45 درجة مئوية) وحجماً أكبر لمحلول المادة المازة (20.1% عند 150 مل). تُبرز هذه النتائج فعالية المادة الماصة من حيث التكلفة واستدامتها في معالجة المياه، مُقدمةً بديلاً عملياً للطرق التقليدية. عُدلت المادة الماصة كيميائياً من خلال تطعيمها بالأكريلونيتريل لتحسين وظائف سطحها وقدرتها على الامتصاص. وتُظهر هذه النتائج أن الكربون المنشط المُطعم بالأكريلونيتريل من بذور النخيل يُعدّ مادة ماصة واعدة وصديقة للبيئة وفعالة لإزالة التتراسيكلين من المياه الملوثة، مما يُوفر بديلاً مستداماً لتقنيات المعالجة التقليدية.

الكلمات الدالة: إزالة التتراسيكلين، الكربون النشط المشتق من الكتلة الحيوية، مادة ماصة لبذور النخيل، تطعيم الأكريلونيتريل، تنقية المياه..

التمويل: تم تمويل هذا البحث من خلال صندوق التعليم العالي القائم على المؤسسات من خلال مركز أبحاث جامعة ولاية كادونا.

بيان توفر البيانات: جميع البيانات الداعمة لنتائج الدراسة المقدمة يمكن طلبها من المؤلف المسؤول.

اقرارات:

تضارب المصالح: يقر المؤلفون أنه ليس لديهم تضارب في المصالح.

الموافقة الأخلاقية: : لم تتضمن هذا البحث أي تجارب على البشر أو الحيوانات، بالتالي لم يكن من الضروري الحصول على موافقة أخلاقية.

مساهمات المؤلفين: اقترح أحمد محمد ساني مقترح البحث ودافع عنه وحصل على تمويل البحث. قام كمال دنارومي، والحسن كبير عثمان، وناثانيل زكي إليشا بجمع المواد الخام، وإعداد العينات، وإجراء تحليل العلامات الأمثل، على التوالي. أ. عبد الرزاق فسر النتائج. كتب عبد الرحيم إبراهيم وعبد الله عثمان عمر المخطوطة وقاما بمراجعتها على التوالي.