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Utilization of Curry Neem Bark for the Effective Adsorptive Removal of Azithromycin from Aqueous Environment

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Abstract

Because of the extensive usage of antibiotics, especially azithromycin, they are persistent in aquatic ecosystems, which presents dangers to the environment and public health. This study uses a batch experimental approach to examine the effectiveness of azithromycin removal from wastewater. Numerous factors affecting the adsorption process were methodically investigated, including contact time, initial drug concentration, pH, temperature, and adsorbent dosage. FTIR and SEM techniques were used to characterize the adsorbent. Curry neem bark's effectiveness as a budget-friendly adsorbent for eliminating azithromycin was assessed. The findings demonstrated that adsorption fit the Langmuir isotherm model well and followed pseudo-second-order kinetics, suggesting monolayer adsorption. The best removal effectiveness (up to 78%) was obtained at pH 5 with an adsorbent dosage of 0.12 g and a contact period of 60 minutes. In order to reduce pharmaceutical contamination in water bodies, this study shows that batch adsorption is a practical and effective technique for extracting azithromycin from wastewater.

Keywords: Azithromycin**,** Curry neem bark, Antibiotic, Water, Environment.

Introduction

Antibiotics have received a lot of attention in recent decades as a major source of environmental pollution, especially in surface water, ground water, livestock farms, and wastewater plant effluents[1]. Since pharmaceuticals are widely used to treat human and animal diseases, they have become a major environmental issue in recent years[2]. Antibiotics, which are categorized as emerging contaminants, have been widely detected in natural water sources, raising concerns. The possible hazards to human health and the environment posed by antibiotic resistance make these medications especially concerning[3].

The widespread usage of macrolides to treat a variety of illnesses makes them stand out among the many different groups of antibiotics[4]. Macrolide medicines with different ring diameters, such as azithromycin, are categorized as second generation antibiotics[5]. Azithromycin (AZTH) is frequently administered to penicillin-allergic people to treat infections of the urinary system, respiratory tracts (both upper and lower), skin, ears, and oral, ocular, and intestinal tracts[6].

According to research on azithromycin, the body does not completely absorb it and it can be eliminated in wastewater in its main form. Moreover, this antibiotic's metabolites and transformation products in humans are unknown[7]. It has been determined that wastewater from facilities that produce antibiotics is another significant source and route via which antibiotics such as azithromycin are released into aquatic habitats. When azithromycin gets into aquatic settings, it can linger for a long time and may have an impact on aquatic ecosystems and creatures. Antibiotic-resistant bacteria could possibly arise as aazithromycin result of its presence, endangering the health of both people and animals. Furthermore, slow metabolism raises the possibility that sewage treatment facilities may not adequately degrade it[8].

There are numerous biological and physicochemical methods for getting rid of antibiotics. A number of cutting-edge technologies have been studied recently to eliminate these contaminants, including adsorption of contaminants onto carbonaceous or nano-magnetic adsorbents, membrane filtering methods (like reverse osmosis and nano-filtration), electrochemical treatments, chemical oxidation through ozone or ozone/hydrogen peroxide, and photocatalytic degradation[9]. Nonetheless, adsorption is widely recognized because of its benefits, which include simplicity, low toxicity, low energy cost, and great removal effects[10]. Numerous absorbents are employed to remove pollutants, including carbon nanotubes, charcoal, zeolite, chitosan, activated carbon, bentonite, and resin[11,12]. Cela-Dablanca et al.[13] reported successful removal of AZTH by oak ash, pine bark, and mussel shell. In this work dried curry neem tree bark powder (DCNBP) is used to eliminate AZTH from aqueous environment. The process provides a sustainable way to cleanse water by utilizing the bioactive compounds present in neem bark, which encourages

the formation of bioadsorbent. The goal of this work is to better understand the adsorption capacity and kinetics of these bio-adsorbents in order to help develop long-term strategies for lowering antibiotic contamination in water supplies.

Fourier-transform infrared spectroscopy (FTIR) along with scanning electron microscopy (SEM) was applied in this work to characterize the physicochemical properties of DCNBP. Kinetic models and adsorption isotherms were studied to understand the mechanics underlying the removal process. Natural material-based bio-adsorption has attracted a lot of attention as an economical and environmentally friendly substitute. Particularly, large surface area, porous structure, and functional groupings like hydroxyl, carboxyl, and amine groups that can interact with pharmaceutical compounds make DCNBP a widely accessible agricultural by-product, a promising adsorptive material. This study provide a low-cost, environmentally friendly way to reduce pharmaceutical pollution in water systems.

Materials and Methods

Chemicals: Analytical grade compounds were all employed in this experiment. We bought sodium hydroxide (NaOH, 96.0%), potassium permanganate (≥98.02%), potassium carbonate (K_2CO_3), ethanol (C_2H_5OH) (≥98.02%), acetone (C_3H_6O) (≥99.20%), and hydrochloric acid (HCl, 37%) from Sigma-Aldrich. Tablets of azithromycin ($C_{38}H_{72}N_2O_{12}$) were bought from a nearby pharmacy. All glassware was thoroughly cleaned with distilled water and acetone before to use.

Standard Solution preparation: Standard solutions containing 5, 10, 15, 20, and 25 mg/LAZTH were made by dilution of stock solution having a strength of 100mg/L with deionized water. The Labman double beam UV-Vis spectrophotometer model LMSPUV 1900 was used to measure absorbance at 547 nm.

Preparation of DCNBP: Curry neem (also known as sweet neem or karuveppilai) bark powder requires first harvesting and cleaning the bark by removing it from the tree and washing it with distilled water. The bark must next be completely dried (sun drying). After drying fully, it is ground into a fine powder using amechanical mixer grinder and stored in an airtight container.

Characterization of DCNBP: The morphology of DCNBP was studied using Scanning electron microscopy (SEM) equipment (Carl Zeiss UHR SEM model Gemini SEM 500 KMAT).

Batch Investigation: This study used curry neem bark, a natural bio-adsorbent, to remove antibiotics, specifically azithromycin, in a series of interrupted experiments. The experiments were conducted at room temperature (25°C ± 1) with 50mL samples. The investigation focused on several features, including pH levels (4–9), contact time (5-80 minutes), initial azithromycin concentrations (3–18 mg/L), amounts of bio-adsorbent (0.02–0.16g/L), and the adsorbents' recoverability and reusability. Each parameter was optimized independently while the other values remained unchanged. Each test was administered three times to ensure accuracy. Containers were cleaned with acid and then rinsed with distilled water to prevent contamination. The pH was changed using hydrochloric acid (0.1N) and sodium hydroxide (0.1N). The stock solution was made by ultrasonically dissolving 100 mg of azithromycin in 40 mL of ethanol, and it was subsequently diluted with 100 mL of pure water. This stock was then used to achieve the required concentrations in 250 mL samples. The adsorption capacity (q_e) and efficacy of antibiotic removal were calculated using the following formulas[14]:

$$R(\%) = \frac{(C_0 - C_e)}{C_0} \times 100 \quad (1)$$

$$q_e = \frac{(C_0 - C_e)V}{m} \quad (2)$$

where m is the absorbent mass in g, V is the solution volume in L, C_0 and Ce are the beginning and final azithromycin concentrations in mg/L, and R is the percentage of azithromycin removal efficiency. Excel was used to evaluate statistical data, and graphs were produced as a result. Additionally, adsorption kinetic and isotherm models were fitted to the data[14].

Kinetic Studies: The rate at which an adsorbent extracts antibiotic molecules from water is examined in kinetic studies for antibiotic removal via adsorption[15]. In adsorption studies, the two primary kinetic models used are pseudo-first-order and pseudo-second-order models[16]. In this work, UV spectroscopy data was analyzed using these models, and matching graphs were created. The pseudo-first-order and pseudo-second-order kinetic models are represented by equations (3) and (4), respectively:

$$ln(q_e - q_t) = lnq_e - k_1 t \quad (3)$$

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

where k_1 and k_2 are the rate constants for the first- and second-order reactions in min^{-1} and L $mg^{-1}min^{-1}$, respectively[17].

Isotherm Studies: Isotherms are used to calculate the equilibrium correlation between the adsorbent and the adsorbate under ideal conditions[18]. Finding a model that works well for the adsorption data provides important information about the adsorption process, including how the adsorbent and adsorbate interact[19]. The Freundlich and Langmuir models of adsorption are the most widely used[20]. The Langmuir model assumes a monolayer of adsorbate molecules on a homogeneous solid surface, while the Freundlich model is based on multilayer adsorption on a heterogeneous surface. The linear forms of the Langmuir and Freundlich isotherms are given by equations (5) and (6)[14].

$$\frac{C_e}{q_e} = \frac{1}{q_{max}K_L} + \frac{C_e}{q_{max}} \quad (5)$$

$$logq_e = logK_F + \frac{1}{n_f} logC_e \quad (6)$$

$$R_L = \frac{1}{1+K_L C_0} \quad (7)$$

Where K_F is the Freundlich constant, qmax is the maximum adsorption capacity (mg g^{-1}), nf is the heterogeneity factor, C_0 is the initial concentration of azithromycin, qe is the amount of AZTH adsorbed at equilibrium, and Ce is the equilibrium concentration of AZTH. The dimensionless constant R_L which shows the favorability of the adsorption process, is represented by equation (7).

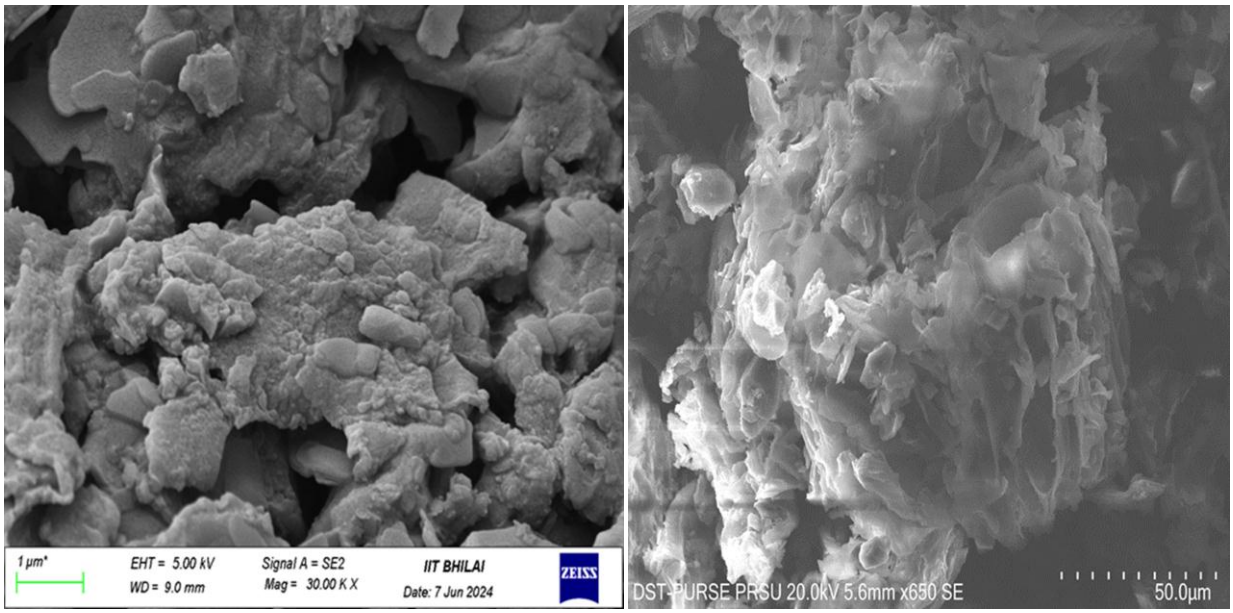
Results and Discussion

Characterization of the DCNBP: Analysis using scanning electron microscopy (SEM) was done to investigate the morphology of DCNBP. Figure-1 shows the SEM image of DCNBP before and after adsorbing AZTH. The SEM image of the DCNBP reveals that its granular structure is uneven, porous, and rough-textured. Because pores increase its surface area, DCNBP is a good adsorbent. Following AZTH adsorption, DCNBP's porous structure showed signs of becoming more compact.

Effect of pH: Protonation and deprotonation of DCNBP surface and speciation of AZTH are greatly affected by the pH of the medium. Impact of pH on adsorptive performance of DCNBP towards AZTH was studied in the pH range 4-9. Removal % as well as adsorption capacity increases on increasing the pH from 4 to 5. In the pH range 6-9 removal % and adsorption capacity decreases with increasing pH (Figure-2). Thus pH 5 was found optimum pH value for adsorptive removal of AZTH.

Effects of Initial Concentration AZTH: The initial concentration of AZTH is one of the important factors for effective adsorption. Regulating the concentration is crucial for proper availability of the adsorbent's active sites to the adsorbate. Impact of concentration of AZTH on DCNBP's adsorption capacity was studied in the concentration range 5-55 mg/L at pH 5 with stirring time 60 minutes and DCNBP dose 0.12g/L. DCNBP appears to be highly efficient at lower antibiotic dosages, as evidenced by the maximal clearance efficacy of 78% seen at a starting concentration of 35mg/L (Figure-3).

Impact of DCNBP Dose: The impact of DCNBP dose on AZTH adsorption was investigated in the range 0.02–0.16 g/50 mL as illustrated in Figure-4. Increasing the dosage from 0.02 to 0.12g clearly enhanced the amount of azithromycin that was adsorbed. Continue to drop from 0.13 to 0.14 grams. Nevertheless, the adsorption capacity reaches a plateau at 78 units after an adsorbent dose of around 0.12, signifying saturation at which point more adsorbent does not appreciably boost capacity. This pattern points to an ideal adsorbent dosage of 0.12; subsequent dose increases do not result in any gains in adsorption, most likely because the neem bark's active sites are saturated and the azithromycin adsorption remains unchanged. Therefore, for all of the batch adsorption studies, 0.12 g of neem bark powder was thought to be the ideal dosage[14].



(A) Before adsorption (B) After adsorption

Figure-1: SEM image of DCNBP (a) before adsorption (b) After adsorption.

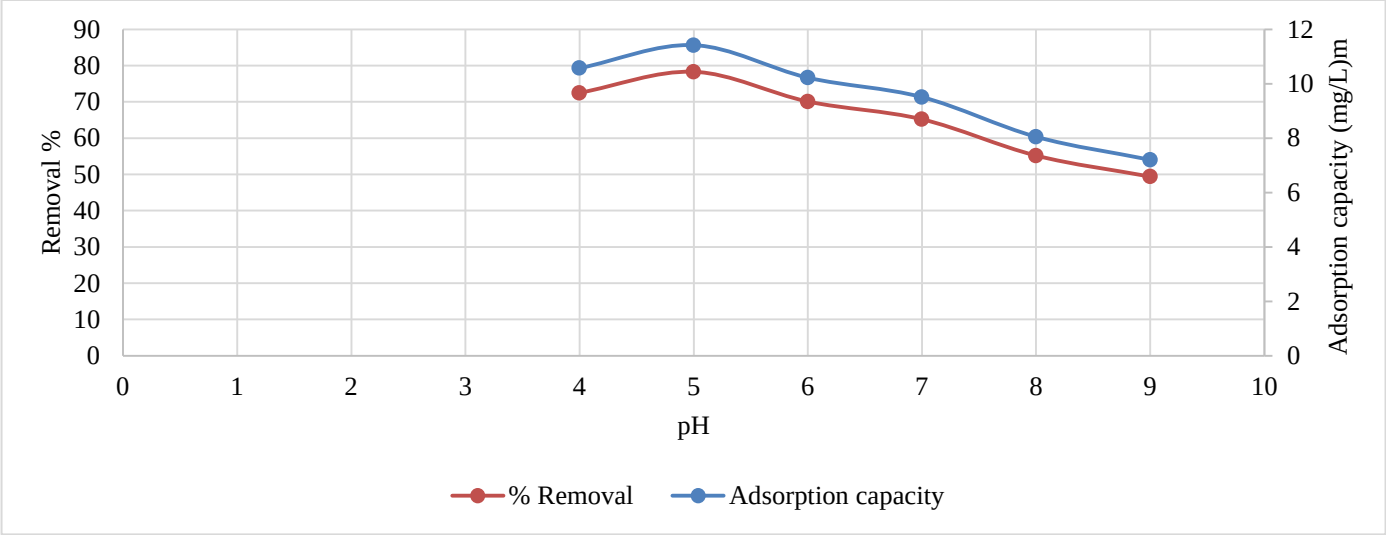


Figure-2: The impact of pH on DCNBP's ability to remove AZTH.

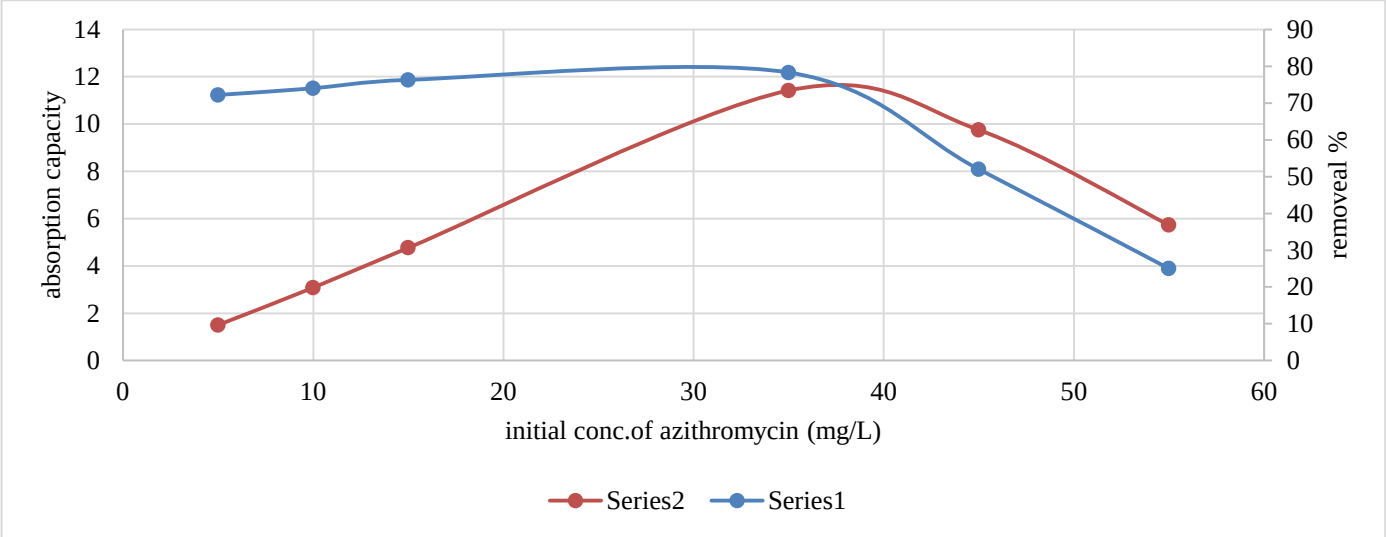


Figure-3: DCNBP's adsorption capability at different starting concentrations of AZTH.

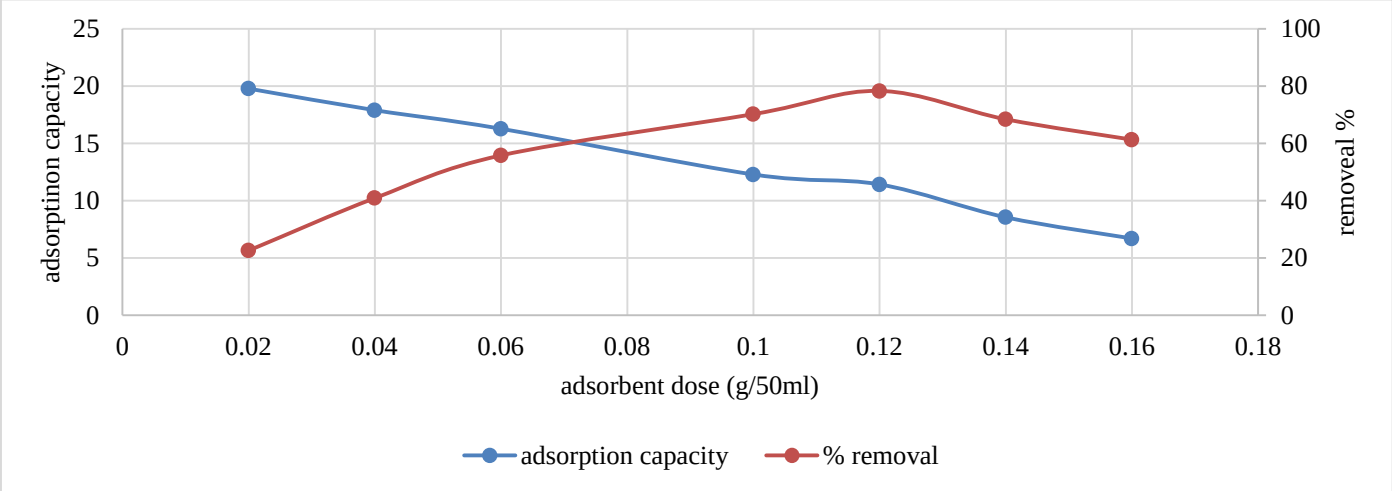


Figure-4: The adsorption capacity and removal % of AZTH at different adsorbent doses.

Effect of contact time on the removal of azithromycin by bio –adsorbent: The high surface area and huge volume of the adsorbent cause the adsorption process to proceed extremely quickly in the first stage (10–60 min)[21]. After 60 minutes, the removal efficiency reaches its maximum of 78%, and it remains there for 70 and 80 minutes subsequently. This plateau indicates that the removal efficiency does not considerably increase after 60 minutes, indicating that equilibrium has been reached. In the early adsorption process, the large concentration of active adsorption sites on the Curry neem bark surface is what causes the fast removal rate at the first stage.

Adsorption isotherm studies: The plot (Figure-6) of log C_e and log q_e had an R^2 value of 0.5154, which indicates that the fit was poor. The adsorption isotherm parameters were presented in Table-1.

Table-1: Adsorption isotherm parameters.

Langmuir isotherm model			Freundlich isotherm model		
KL	Q max	R^2	Kf	1/n	R^2
2.17	6.45	0.91	2.24	0.4147	0.51

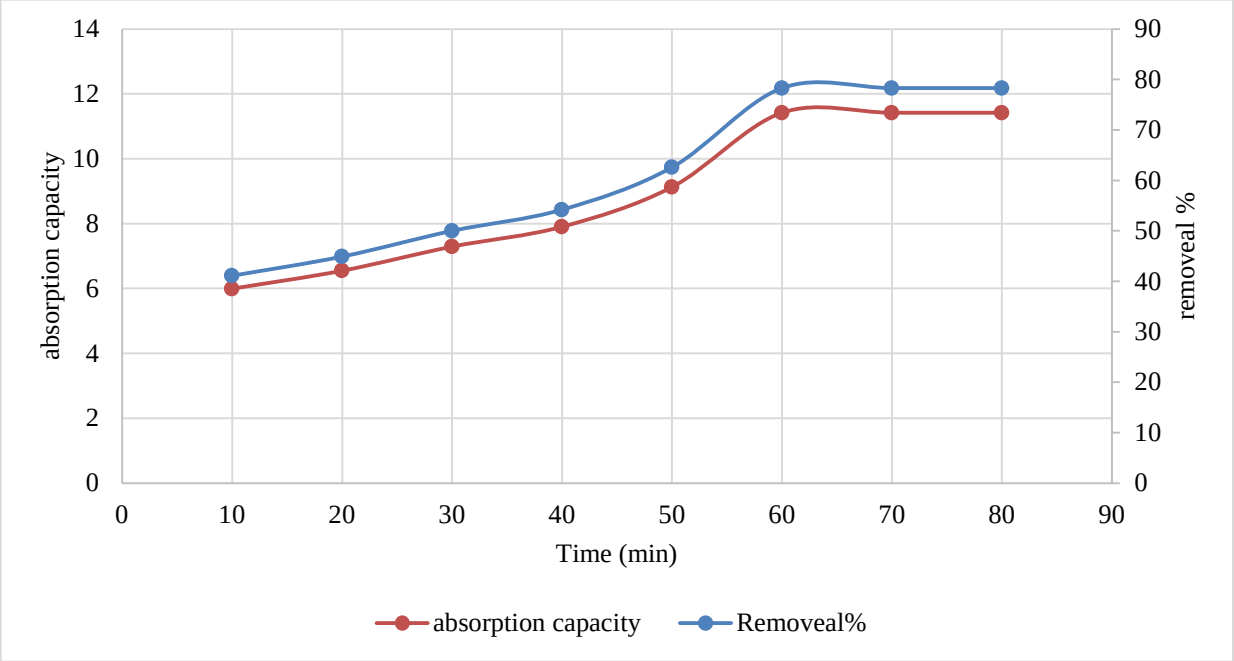


Figure-5: The effect of contact time on adsorption capacity and removal %.

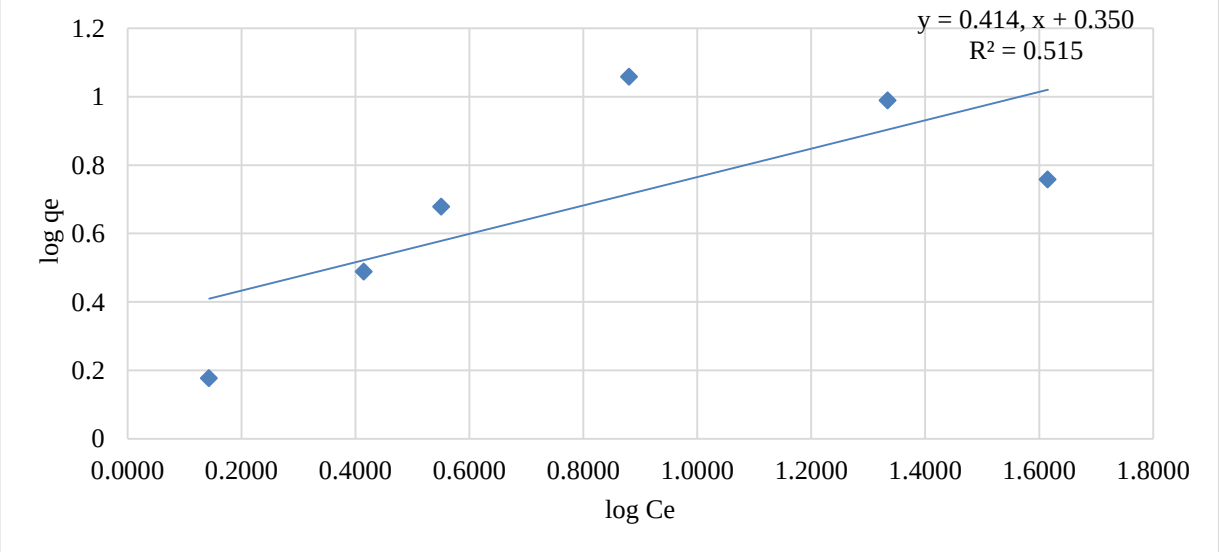


Figure-6: Freundlich adsorption isotherm plot of logQe versus logCe for azithromycin adsorption.

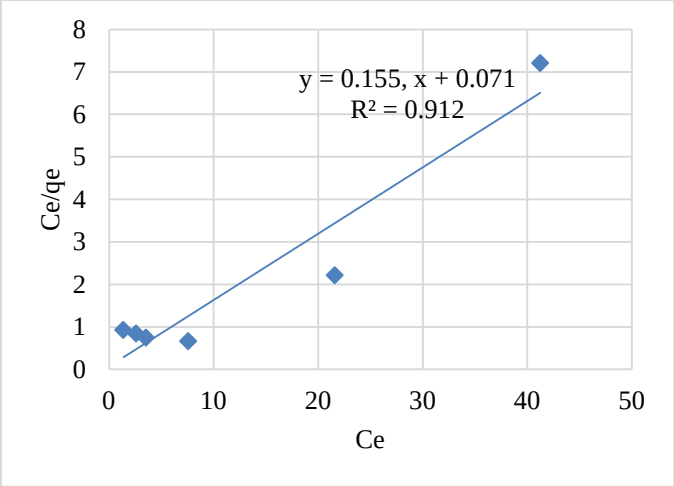


Figure-7: Langmuir adsorption isotherm plot for removal of AZTH by DCNBP.

The obtained R^2 value for the Langmuir model (Figure-7) was 0.9122, indicating that the adsorption data compatible with the model well. Comparing the correlation coefficients of the Langmuir and Freundlich isotherm models reveals that the latter has a higher correlation coefficient, indicating a better fit with the experimental data used in this study.

Kinetics studies: For kinetic profiling of AZTH adsorption by DCNBP, adsorption capacity and % removal were examined at various time intervals. Plots for pseudo first and second order models are presented in Figure-8 and 9, respectively. From both the models value of R^2 was found same but value of q_e obtained from the pseudo second order model was close to experimental value (Table-2). On this basis applicability of pseudo second order model is found better for adsorptive elimination of AZTH by DCNBP. This indicates that the adsorption rate is proportional to the square of the number of adsorbent's empty sites.

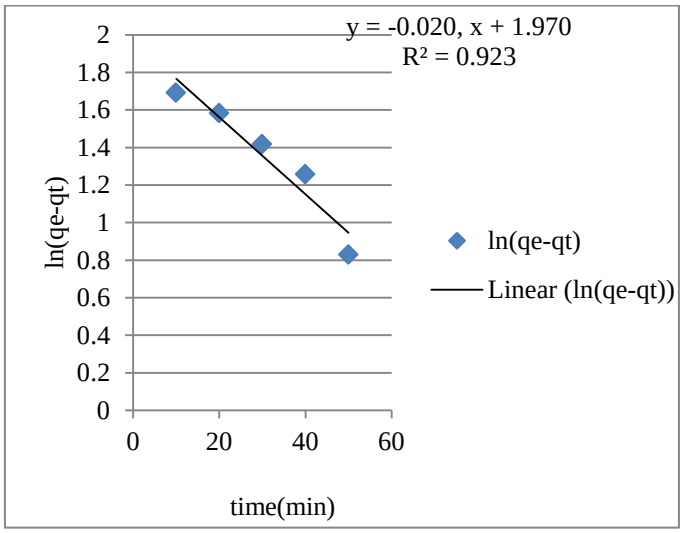


Figure-8: Kinetic analysis plot of t versus $\log(q_e - q_t)$.

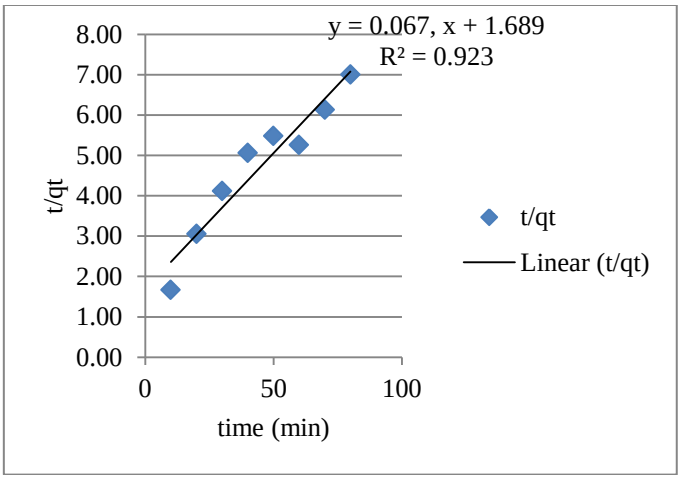


Figure-9: Kinetic analysis plot of t versus t/q_t.

Table-2: Adsorption kinetics parameters.

Name of applied model	Kinetic Parameters	Calculated values
Pseudo-first-order model	k_1 (min^{-1})	0.02
	R^2	0.923
	q_e (mg/g)	7.17
Pseudo-second-order model	k_2 (g/mg min) X 10^{-3}	2.659
	R^2	0.923
	Qe(mg/g)	14.92

Investigations of thermodynamics Adsorptive elimination using a natural bio-adsorbent was carried out at three different temperatures—298 K, 303 K, and 308 K—in order to examine the thermodynamic properties. Using equations (8) and (9) thermodynamic parameters were evaluated and presented in Table-3.

Table-3: Thermodynamic Parameters.

Adsorbate	ΔH^0 kJ/mol	ΔS^0 kJ/mol K	ΔG^0 kJ/mol		
			303K	308K	313K
Azithromycin	24.26	0.14	-18.16	-18.86	-19.56

$$\ln Kd = (\Delta S^0/R) - (\Delta H^0/RT) \quad (8)$$

$$\Delta G^0 = \Delta H^0 - T\Delta S^0 \quad (9)$$

V: AZTH solution's volume (mL), m: mass of DCNBP (g), K_d: distribution constant (mL/g), R: gas constant (J/mol K), T: temperature (Kelvin), and C_0 and Ce: starting and equilibrium concentration of AZTH (mg/L). Adsorption's Gibbs free energy

change (kJ/mol), enthalpy (kJ/mol), and entropy (kJ/mol K) are represented as ΔG^0, ΔH^0, and ΔS^0, respectively.

The presence of an endothermic reaction during the elimination process is indicated by the positive ΔH^0 value. A negative ΔG^0 value indicated that adsorptive elimination happens on its own. The positive values of ΔS^0 show that the randomness at the interface between the DCNBP and the AZTH increased during the elimination process.

Conclusion

This study demonstrates that DCNBP has the potential to eliminate AZTH contamination from aqueous solutions. Under the right conditions, removal efficiency can reach 78.3%. pH, AZTH concentration, and dosage all significantly affect the adsorption process. A lower initial concentration of 35 ppm and an optimal pH of 5 yield the best results because of the favourable interaction between the AZTH and DCNBP. Research on kinetics and isotherms provides insight into the adsorption processes, indicating adherence to a pseudo-second-order kinetic model and identifying AZTH adsorbs on DCNBP through chemisorption. The overall efficacy of DCNBP indicates their appropriateness for the remediation of antibiotic contamination.

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