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Study of ciprofloxacin adsorption using activated carbon from compressed wood: Evaluation of performance and efficiency in pollutant removal

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ABSTRACT

Adsorption processes have become promising methods to removal various emerging pollutants with friendly ecosystem side effects and low-cost features. Activated carbon (AC) was a suitable choice due to the abundance of numerous effective precursors. This study deals with compressed wood precursor and KOH as activator agents that offer adsorption capacity of ciprofloxacin antibiotic (CIP) (q_{max} = 206 mg/g) which exceeds many precursors in literature and good characterization analysis of scanning electron microscopy (SEM), nitrogen adsorption-desorption isotherms and X-ray diffraction (XRD). Different parameters affecting the process such as concentration, AC dose and contact time were all studied. Isotherm and kinetic models were investigated which fitted with Freundlich isotherm model and pseudo second order that describing the adsorption of CIP on AC. Also, fine regeneration results in using thermal methods even in three cycles with adsorption capacity (q_e = 127.6 mg/g compared to 178.4 mg/g by first cycle) according to the same parameter.

Keywords: ciprofloxacin, compressed wood, activated carbon, adsorption isotherm, kinetics.

INTRODUCTION

The emergence of contaminants in wastewater can raise affairs within the scholarly community and urged vast investigations into basic sources (Martinho et al., 2022; Al-yaqoobi et al., 2024) . Antibiotic contaminations are deemed to be main sources of pollution of contamination in water, harmful and hard to decompose (Liu et al., 2023). Antibiotics are developed to stop or treat the bacterial diseases. According to its behavior mechanism, antibiotics are grouped into several categories, for occurrence, polypeptides, tetracyclines, fluoroquinolones, etc. (Falyouna et al., 2022). Ciprofloxacin is one of more important antibiotics, 1-cyclopropyl-6-fluoro 1,4-dihydro-4-oxo-7-(piperazinyl)quinolone-3-carboxylic acid (CIP), which associated with fluoroquinolone group (Pollap et al., 2020). Ciprofloxacin (CIP) is widely applied in animal and human healthcare to remedy many bacterial diseases, for occurrence, respiratory, auditory canal, urinary tract, skin, bones, etc. (Przybylska et al., 2021). Ciprofloxacin is important antibiotics for the cure of bacterial infections that caused by different types of bacteria like Gram-positive and Gram-negative bacteria (Malakootian et al., 2021). Therefore, this antibiotic is applied for treatment many diseases, such as digestive, respiratory system and urinary tract infections (Pollap et al., 2020). CIP can arrive the ecosystem through several ways. One of these ways, discharge of wastewater cure plants is the important way for CIP to access the proximate rivers and ponds structures (Peñafiel et al., 2021). furthermore, the surplus CIP will expelled by animal, this secreted could be used as fertilizers, that causes contamination of the ground and finally CIP can leach into groundwater through soil filtration (Antonelli et al., 2020). Various strategies were employed to eliminate CIP, for example, adsorption (Arif et al., 2022), Advanced oxidative processes (AOPs) (Al-Buriahi et al., 2022), filtration technology (Olasupo et al., 2022), biodegradation (Cai et al., 2022).

Adsorption is recognized as an efficient, cost-effective, ecofriendly and simple processing technology for CIP elimination (Khalil et al., 2021).

In the adsorption process, interaction energy between the adsorbent and the adsorbed species can be distinguished whether adsorption is Physical (physisorption) or chemical (chemisorption). The formation of strong bonds such as ionic or covalent occurs in chemisorption, which is generally selective and irreversible. In comparison, hydrogen bonding, van der Waals forces and polar interactions are characterized as physisorption that is commonly nonselective and reversible (Mashkoor and Nasar, 2020). Various types of adsorbents were generated to dispose of CIP from water, including porous graphene (PG) (Khalil et al., 2020), biochar activated with carbon dioxide (Arif et al., 2022; Abd and Al-Yaqoobi, 2023; Abd and Al-Yaqoobi, 2025), self-floating silica (Hu et al., 2022), fish scale-based biochar (Dou et al., 2022), etc. Activated carbon is prominent for its features including porosity and large surface area that made it efficient adsorbents in addition to various applications (Gayathiri et al., 2022). In recent years, many types of adsorbents were synthesized to eliminate CIP like, cyclodextrin nano sponges (Rizzi et al., 2021), activated carbon with iron oxide nanoparticles (Al-Musawi et al., 2021), tannin foam loaded with iron (III) ions (Hao et al., 2021), polystyrene plastic (Yilimulati et al., 2021). Activated carbon is one of the favorite adsorbents for its feature, which can be derived from various precursors such as Kiwi peels (Gubitosa et al., 2022), corncobs (Oday and Al-Jendeel, 2024), Date pit (Mansour et al., 2021), cigarette wastes (Ahmed et al., 2024). The main objective of the present work is to study the best conditions for the removal of CIP by adsorption onto activated carbon (AC) prepared by KOH activation, also equilibrium isotherm and kinetics were studied.

MATERIALS AND METHODS

Chemicals

The sample of compressed wood was obtained from local carpentry workshops, Potassium hydroxide (KOH) with purity of 98% (India) and CIP from Dar Al Dawa Development &Investment Co. Ltd., Amman, Jordan.

Activated carbon synthesis

The collected compressed wood was washed with water three times to eliminate adhering dirt, then dried it in drier furnace at 60 °C for 24 hours to evaporate moisture and then grinding it by (Versatile grinding machine) to appropriate size. The result is used for preparation of Activated carbon (Raheem et al., 2024). Then, compressed wood put in the reactor that placed in a furnace to pyrolysis gradually and carbonize with an increment of 10 °C/min until the temperature reach 500 °C and held at this temperature (carbonization temperature) for 3 hrs. After that, the compressed wood leaved until reached to an ambient temperature. The result was mixed with KOH in 1:3 weight ratio as activator (Liu et al., 2023) then put it at room temperature for 24 hr to impregnate, after that it was dried by oven (Model IH-100, England) for 24 hours at 60 °C these samples were washed with large amount of distilled water to neutral pH.

Adsorption experiments

An adsorption study was conducted using AC to remove antibiotic (CIP) from aqueous solution, and the effect of concentration, AC dose, time and incubation temperature was done. Activated carbon (0.1 g) was placed in 50 ml of CIP (100–500 mg/L) solutions. Then, the mixture was performed at ambeint temperature with stirring at 200 rpm for 24 hr. After that, the mixture was filtered using filter paper and measured the absorbance with an ultraviolet spectrophotometer (UV-2900) at wavelength of 274 nm. The equilibrium adsorption amount (qe, mg/g) and removal rate (R%) (Abbas and Abbas, 2021) of CIP were determined by the following Equations 1 and 2

$$q_e = \frac{(C_0 - C_e) \times v}{m} \tag{1}$$

$$R\% = \frac{C_0 - C_e}{C_0} \times 100 \tag{2}$$

where: C_0 and C_e – CIP concentrations at initial and equilibrium time (mg/L), V – volume of sample (L), m – mass of AC (g).

ISOTHERM AND KINETIC MODELS

Isotherm models

Results of batch experiments were analyzed by three models: Langmuir, Freundlich and Temkin using the following linear equations (Aljendeel et al., 2023):

- Langmuir:

$$\frac{1}{q_e} = \frac{1}{q_m k_l C_e} + \frac{1}{q_m} \quad `(3)$$

- Freundlich:

$$\ln q_e = \ln k_f + \frac{1}{n} \ln C_e \quad (4)$$

- Temkin:

$$q_e = B \ln A + BLnCe \quad (5)$$

where: C_e – concentration of CIP at equilibrium (mg/L) qe – adsorption capacity of CIP at equilibrium (mg/g), q_m – maximum adsorption capacity, K_l – (L/mg), K_f – (mg/g (L/mg) $^{1/n}$) – adsorption constant of the two models, n – parameter signifies the affinity of the adsorbate to the adsorbent, A – (L/g) constant of Temkin isotherm related to equilibrium energy, B: (J/mol) Heat of adsorption.

Adsorption kinetic

To conclude, more information concerning the adsorption behavior, Pseudo first order (PFO), Pseudo second order (PSO) and Intra-particle diffusion (IPD) model were selected according to the following equations (Khalaf and Rashid, 2024):

- PFO:

$$\ln(q_e - q_t) = \ln q_e - k_1 t \quad (6)$$

- Pseudo second order (Al-Jubouri et al., 2022):

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (7)$$

- IPD (Hummadi, 2021)

$$q_t = k_3 t^{\frac{1}{2}} + C \quad (8)$$

where: q_e – CIP adsorption capacity at equilibrium, q_t – adsorption capacity at time t, both in mg/g. k_1 (min $^{-1}$) and k_2 (g/(mg ×min)) are the constants of the models.

RESULTS AND DISCUSSIONS

Scanning electron microscope (SEM)

The AC structure was identified by using SEM. The heterogenous structure obviously appears and the dense micropores penetrate inside the cluster. formation of porous channels and a hierarchical structure attributed to slow pyrolysis (Foorginezhad et al., 2024). The volatile compounds occupied vascular was gasification leaves hollow tunnels (Serafin et al., 2023) with long systematic and repeated cracks. Figure 1 shows the images of AC.

N_2 adsorption-desorption isotherm

From nitrogen adsorption-desorption isotherms Figure 2, KOH Activation AC matches with IV isotherms according to the International Union of Pure and Applied Chemistry (IUPAC). Which shows that the activated carbon mostly has mesopores, according to hysteresis loops (Yagmur et al., 2020). A distinct hysteresis at elevation pressure indicates a progressive formation of mesopores, as well as allocation of pore sizes (Togibasa et al., 2021). The sharp increase in adsorption after inflection point (which indicate to completion of first monolayer) refers to multilayer adsorption behavior.

X-ray diffraction (XRD)

The amorphous and graphite structure appeared in XRD analysis according to (Figure 3) notable peak between 20–29° 2θ angles which corresponding to (002) plane appointed to amorphous structure (Yagmur et al., 2020), while the sharp peak of 2θ = 31.5 indicates to a slide increasing in crystallinity. The presence of a wide amorphous peak and narrow crystalline peaks indicate a mixed amorphous and crystalline structure (Kwaśniewska et al., 2021).

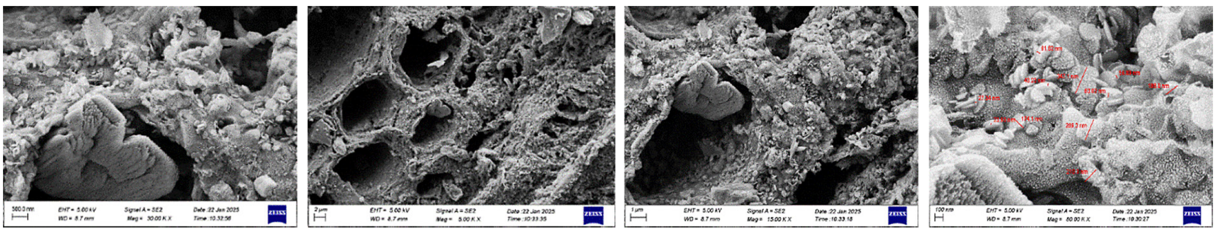

Figure 1. SEM images

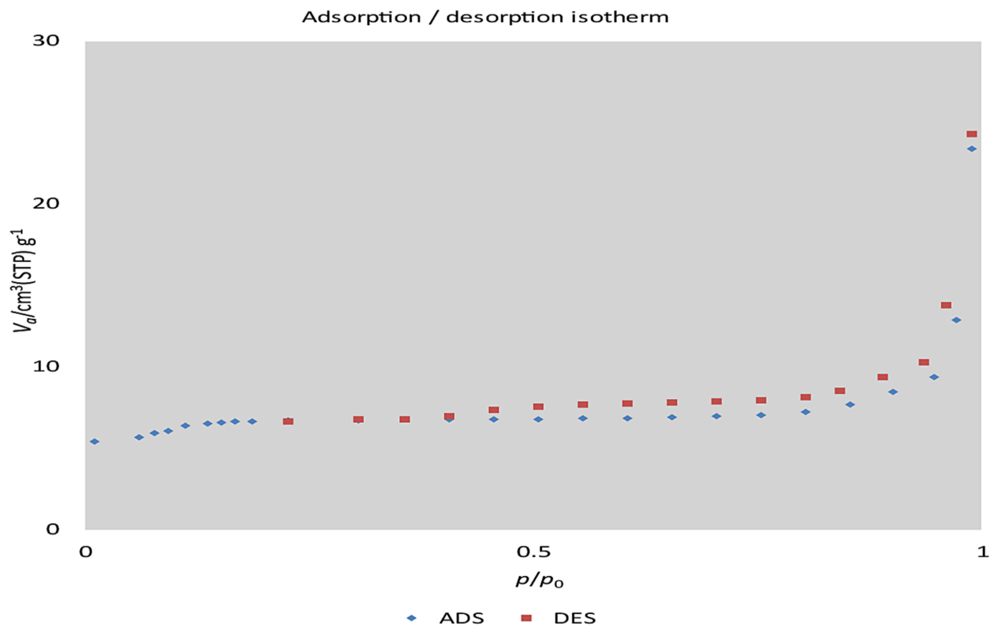

Figure 2. N_2 adsorption-desorption isotherm

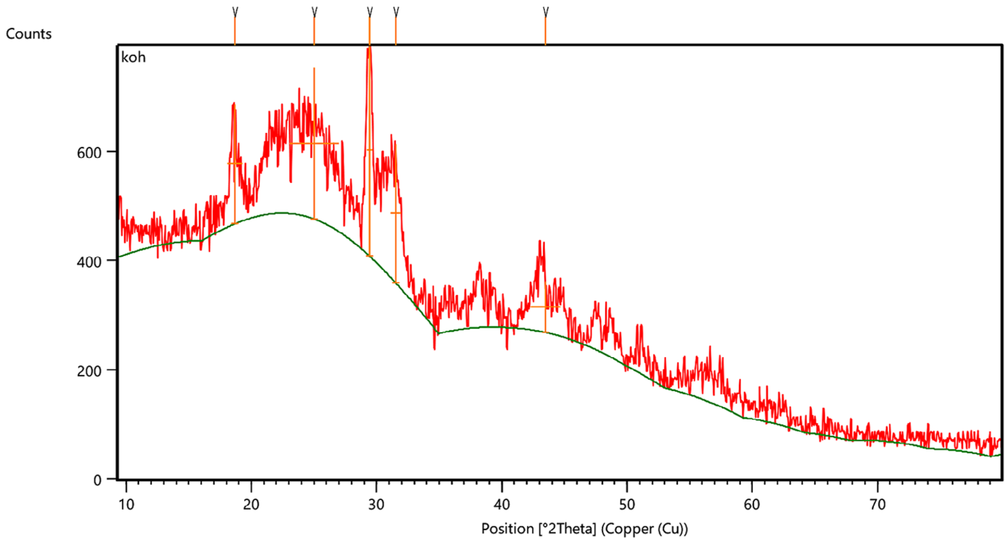

Figure 3. XRD pattern

Adsorption performances

Effect of contact time and adsorption kinetics

the adsorption capacity of AC for CIP increased slightly. The adsorption capacity of AC for CIP reached 169 mg/g within 6 hr. The kinetic model was fitted with linear form equations according to Figure 4, and the important parameters are obtained from the slope and the interception, as shown in Table 1. The fitting of the intraparticle diffusion model with (R^2 = 0.996) positive intercept which relevant to boundary layer thickness was large value correspond to a significant effect on the adsorption. Also, nonzero interception indicates that the adsorption process controlled by intra-particle diffusion in addition to film diffusion (Dharmarathna and Priyantha, 2024) and pseudo-second order (R^2 = 0.993) could describe the adsorption process. compared to (R^2 = 0.97) in pseudo- first order. The adsorption process consisted of bulk diffusion, boundary layer diffusion, and IPD. Initially, because of concentration gradient, CIP molecules reached the boundary layer and then distributed through the film to the surface of AC.

Table 1. Kinetic parameters for CIP adsorption on AC with initial concentration = 200 ppm at ambient temperature

Pseudo-first order model			
qe,exp(mg/g)	*qe,cal*(mg/g)	K_1(1/h)	R^2
169.42	83.93	0.37	0.97
Pseudo-second order model			
qe,exp(mg/g)	*qe,cal*(mg/g)	K_2(g/mg.h)	R^2
169.42	175.43	0.014	0.993
Intra-Particle diffusion model			
qe,exp(mg/g)	*C*(mg/g)	K_3(mg/g hr $^{1/2}$)	R^2
169.42	87.98	33.41	0.996

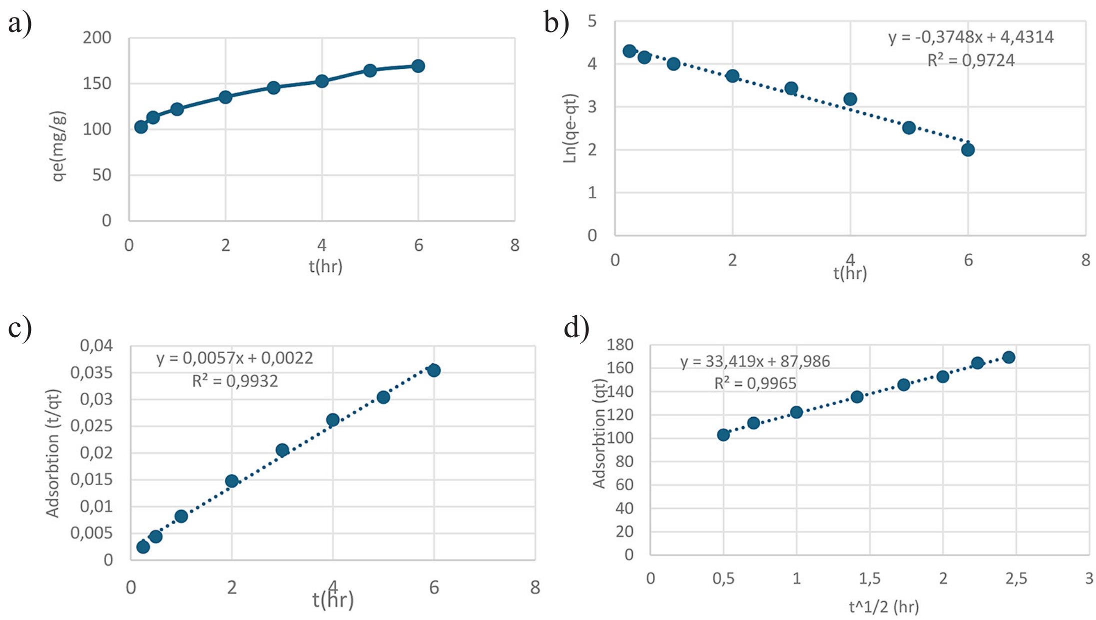

Figure 4. (a) time effect, (b) pseudo- first order, (c) pseudo- second order, (d) intraparticle diffusion model

Effect of concentration and isotherm models application

The adsorption behavior CIP at different initial concentrations were studied (Figure 5). the adsorption of CIP directly proportional with initial concentration. Because of high concentration gradient. The parameters relevant to the fitting of isotherm models are inserted in Table 2. It could be observed the correlation coefficient (R^2) of the Freundlich model (R^2 = 0.99) was much higher than that of the Temkin model (R^2 = 0.95) during the adsorption of AC on CIP. While Langmuir model also gives a suitable parameters value (Figure 7). It inferred that the Freundlich model fitted appropriately the experimental data that agree with Gubitosa et al. (Gubitosa et al., 2022), and the CIP molecules adsorbed heterogeneously on the surface of the AC.

Effect of AC dosage

The effect of AC dosage on the elimination of CIP was investigated within a dosage range of 0.012 to 0.1 g/50 mL aqueous solution, with an initial concentration of 200 mg/L for antibiotics, for 6 hours. As shown in Figure 6, the adsorption efficiency (%) of AC increased by increasing

Table 2. Isotherm parameters for CIP on AC at ambient temperature

Isotherm	Langmuir			Freundlich			Temkin		
Parameters	q_m	K_L	R^2	K_F	*n*	R^2	*A*	*B*	R^2
CIP	200	0.065	0.97	21.73	1.97	0.99	0.417	53.79	0.95

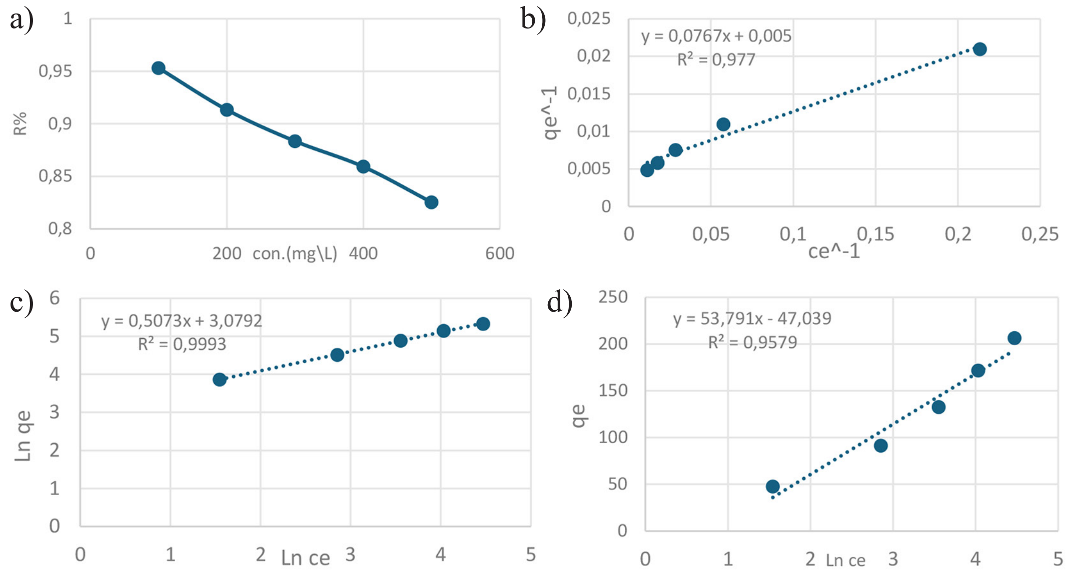

Figure 5. (a) concentration effect, (b) Langmuir (c) Freundlich (d) Temkin models

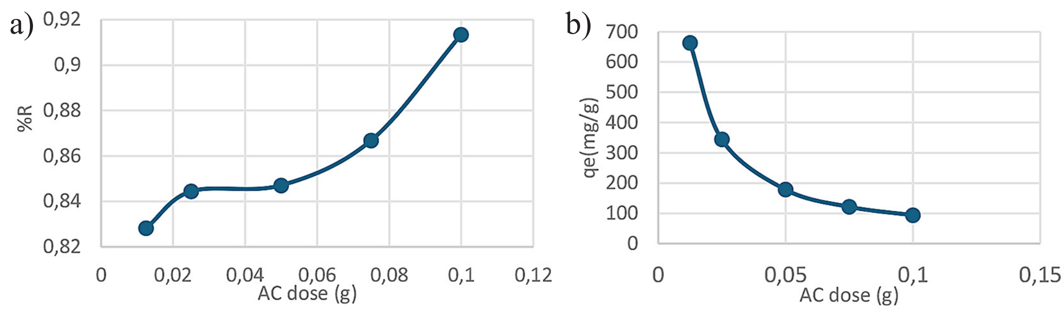

Figure 6. (a) AC dose effect with R%, (b) AC dose effect with adsorption capacity

the amount of dosage. The highest adsorption efficiency was observed at 0.1 g for CIP (93%). This pattern can be due to the large number availability of active binding sites with increasing AC dosage, which improved adsorption efficiency at higher dosage amount. When the dosage increases, the dispersion of CIP molecules through the active sites may decrease, mass transfer leading to higher CIP loading onto the AC, that leads to saturation of active sites. In other hand, the small dosage of AC obtained higher value of adsorption capacity (662 mg/g).

Regeneration of AC

While the conventional regeneration methods that use acidity or alkalinity agents lead to

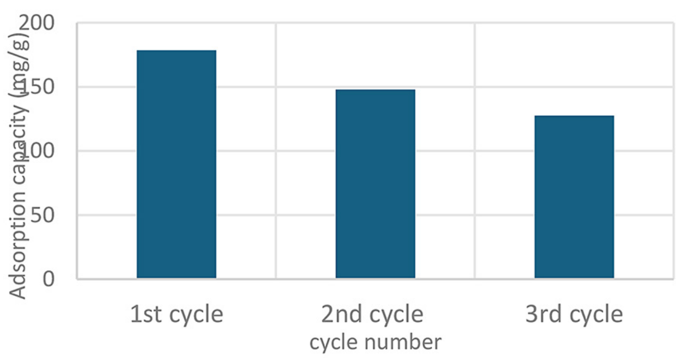

Figure 7. Adsorption of CIP by AC After 3 cycles

secondary contaminants, the pyrolysis method (Liu et al., 2023) could be an efficient method with high results. The adsorption experiments were carried out with initial concentration 200 ppm, fixed rpm, 0.05g AC dose/50mL sample and ambient temperature. The adsorbed AC was heated to 500 °C at a rate of 10 °C/min in a crucible tube and remain at this temperature for on hour to pyrolyzed. The second cycle achieved 147.88 mg/g adsorption capacity and 73.9% removal efficiency, while the third cycle achieved 127.6 mg/g and 63.8% respectively compared to 178.4 mg/g and 89% in first cycle according to Figure 7.

CONCLUSIONS

Compressed wood was carbonization followed by KOH activation to produce base-activated carbon for effective elimination of CIP. The AC produced was confirmed by various tests to characterization (SEM, BET and XRD). CIP removal capacity (mg/g) and efficiency (%) were affected by different parameters resembles, initial antibiotic concentration, time and AC dosage that investigated in this study. Modelling studies asserted the composition of multilayer of CIP over the AC surface and the involvement of physisorption as proven by Freundlich isotherm, also the results shows the fitting with pseudo-second order kinetics. Thermal regeneration up to 3 cycles refers to the effectiveness of AC synthesized from compressed wood for removal of CIP.

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