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ORIGINAL RESEARCH ARTICLE

Removal of ceftriaxone and ciprofloxacin antibiotics from aqueous solutions using graphene oxide derived from corn cob

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ABSTRACT

BACKGROUND AND OBJECTIVES: Preliminary studies on the exploration of carbonaceous materials from agricultural waste and their use as adsorbents for antibiotic removal have shown the potential to address a new threat to human health due to antibiotic residue. Therefore, this study developed and synthesized graphene oxide from corn cob for its efficiency in removing ceftriaxone and ciprofloxacin.

METHODS: The Hummers methods were used to synthesize graphene oxide from corn cobs. Graphene oxide was characterized using Fourier transform infrared, scanning electron microscope-energy dispersive x-ray, and x-ray diffraction instruments. During the synthesis process, antibiotic adsorption tests were extensively conducted by exploring four variables, namely dosage of adsorbent, potential hydrogen, concentration, and contact time.

FINDINGS: The result showed that graphene oxide from corn cob effectively removed 47 percent of ceftriaxone and 92.62 percent of ciprofloxacin. Furthermore, to ensure optimum use of the adsorbents, antibiotics ceftriaxone and ciprofloxacin weighing 40 milligrams and 20 milligrams. This is in addition to the initial concentrations of 14 and 2 parts per million, the potential of hydrogen 4, and contact times of 50 and 40 minutes, respectively.

CONCLUSION: In conclusion, adsorbents made from corn cobs are better at the removal of ciprofloxacin from water than the antibiotic ceftriaxone. The difference in molecular structure affected the percentage of antibiotic adsorption onto graphene oxide derived from corn cob. This study underscores the potential of the derived material as a promising adsorbent for efficiently removing ciprofloxacin from aquatic environments. The use of agricultural waste as advanced materials to address antibiotic residue pollution provided additional environmental pollution.

NUMBER OF REFERENCES	NUMBER OF FIGURES	NUMBER OF TABLES
55	11	3

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INTRODUCTION

Antibiotics are widely recognized for bacteriostatic properties, which effectively prevent the proliferation of bacteria and reduce the risk of death caused by bacterial infections in both humans and animals (Catteau *et al.,* 2018). However, its increasing application poses challenges in the aquatic environment, as antibiotic residues, particularly from commonly used antibiotics like ceftriaxone and ciprofloxacin (Zhu *et al.,* 2015), cannot be easily degraded. This situation contributed to the development of bacterial resistance to antibiotics (Ben *et al.,* 2019). Ceftriaxone, widely adopted for its significant effectiveness, broad range of action, and minimal risk of adverse effects, is commonly used in various medical settings, including clinics, animal husbandry, and fisheries. It is used in the treatment of diverse bacterial infections, such as pneumonia, bone, abdominal, skin, and soft tissue, including urinary tract infections (Ayele *et al.,* 2018). Despite its efficacy, the environmental impact of ceftriaxone is significant, specifically when used inappropriately or when exceeding the Maximum Residue Limit (MRL). Guo *et al.* (2017) reported that ceftriaxone treatment in mice significantly affected parameters like the histological cross-section of the distal small intestine, body weight, spleen index, immune globulin, and cytokines. This treatement is also used to treat mastitis in cows with inappropriate usage capable of contaminating antibiotic residues, negatively impacting the aquatic environment. This contamination can inhibit the development and genotoxicity of living creatures in these waters. In a research conducted by Chowdhury *et al.* (2022), it was reported that incorrect usage exceeding MRLs of antibiotics, particularly ceftriaxone, can have negative impacts on aquatic ecosystems, inhibiting the development and genotoxicity of living creatures in these waters. On the contrary, ciprofloxacin, a globally recognized fluoroquinolone, is extensively used in aquaculture, poultry farming, and clinical settings owing to its minimal toxicity, broad antibacterial spectrum, and limited development of bacterial resistance (Zhu *et al.,* 2019). The introduction of ciprofloxacin into water resources, even at low concentrations, has been associated with significant consequences such as impacting photosynthesis, altering the morphological structure of algae, and disrupting the balance of aquatic ecosystems. Furthermore, due to its widespread use and incomplete metabolism in humans and animals, ciprofloxacin residues were detected in diverse wastewater. The wastewater was generated from the pharmaceutical industry in Patancheru, India, with concentrations ranging from 28.000 to 31.000 grams per liter (g/L), hospitals in Switzerland recorded concentrations of >10 g/L. At the same time, residential towns around WWTPs in the same country reported levels between 0.255 and 0.568 g/L. Additionally, agricultural land in central China recorded concentrations ranging from 0.020 to 0.100 g/L (Honarmand *et al.,* 2022). This widespread occurrence proved ceftriaxone and ciprofloxacin are significant water pollutants that demand urgent research attention (Wakejo *et al.,* 2022). The effective removal techniques are important due to the importance of addressing antibiotic residues in the aquatic environment. Several methods, including membrane removal (Nasrollahi *et al.,* 2022), degradation (Ahmad *et al.,* 2021), electrochemical techniques (Orimolade *et al.,* 2023), and adsorption (Amari *et al.,* 2021), have been developed for antibiotic removal. The adsorption method was considered an alternative technique characterized by low cost, easy design, and operational convenience (Ehzari, *et al.,* 2022; Samimi and Nouri, 2023). This method focuses on developing efficient adsorbent materials, with numerous research reporting the successful use of various substances, including natural clays as adsorbents for the removal of humic acid from aqueous solutions (Gueu *et al.,* 2018), agricultural waste for the elimination of paracetamol (Osobamiro *et al.,* 2022), and amorphous zirconium for the removal of phosphate from water (Nuryadin and Imai, 2021). Carbon nanostructured materials are considered adsorbents due to its large surface area and excellent adsorption capacity, such as carbon nanotubes (CNTs), nanoparticles (NPs), graphene, and graphene oxide (GO) (González *et al.,* 2016). GO has become a significant focus in water treatment due to its unique properties, particularly its dispersibility in water facilitated by functional groups. However, this substance, which is conventionally synthesized from highly pure graphite is expensive and difficult to obtain from non-renewable sources (Bheel *et al.,* 2023). Tohamy *et al.* (2020) stated that the applicability of GO obtained from agricultural wastes is considered as low-cost

adsorbent for Ni(II) in aqueous solution. This promoted various efforts to explore other agricultural waste materials, such as cassava peel, rice grain, soybean pulp, cotton seeds, straw, peanut shells, and corn cobs, as alternative low-cost adsorbents (Akhavan *et al.,* 2014; Samimi and Mansouri, 2023). According to Wang *et al.* (2023), corn cobs are mainly composed of the following elements carbon, hydrogen, and oxygen. The carbon content, constituting 48.12 percent (%) of the mass of corn cobs, indicates its potential as a viable raw material for the production of natural graphite and GO (Liu *et al.,* 2014). Over the last seven years, corn production in Lampung Province, based on data from the Central Bureau of Statistics (BPS), has shown a consistent upward trend. In 2021, the harvested area expanded to 10.8 hectares, producing a substantial corn production of 2.518 million tons. The increase in corn production, led to a significant rise in waste products, due to inappropriate usage. Therefore, the use of renewable, and cheap corn cob is perceived as a promising precursor for GO in producing adsorbent materials for antibiotic removal. The high carbon content in corn cobs depicts its potential as an effective and sustainable solution for this application. However, research on the specific capabilities of GO derived from corn cobs as an adsorbent material for removing antibiotics, particularly ceftriaxone and ciprofloxacin, remains limited. The main objective of this research is to assess the adsorption efficiency of GO derived from corn cobs in eliminating ceftriaxone and ciprofloxacin from water solutions. It aimed to achieve maximum adsorption efficiency by systematically adjusting major parameters in the evaluation of ceftriaxone and ciprofloxacin adsorption. These parameters including adsorbent mass, potential of hydrogen (pH), initial adsorbate concentration, and contact time, were carefully manipulated. By determining the influential factors, the present research provides practical guidance for optimizing the adsorption process in wastewater treatment and other environmental media. Optimization is important for environmental protection and preventing the negative impacts of antibiotics in the ecosystem. The optimal conditions can also reduce operational costs and the environmental impact of the process. The resulting GO was characterized using techniques such as x-ray diffraction (XRD), Fourier transform infrared (FTIR), Raman, and ultraviolet-visible (UV-Vis) spectroscopy, as well as scanning electron microscope-energy dispersive x-ray (SEM-EDX) to confirm its nature. Therefore, this research aimed to determine the optimal conditions for using corn cob-based adsorbent to remove residues of antibiotics, specifically ceftriaxone and ciprofloxacin, from wastewater. The research included synthesizing GO, material characterization, and antibiotic adsorption tests with variations in parameters such as adsorbent dosage, pH, concentration, and contact time. This research was conducted at the Analytical Chemistry and Instrumentation Laboratory, Department of Chemistry, Faculty of Mathematics and Natural Sciences, University of Lampung in 2022. In addition, the characterization analyses were carried out in collaboration with the laboratory partner.

MATERIALS AND METHODS

The main material used for the preparation of GO was corn cobs obtained from local farmers in Lampung Province, Indonesia. Standard ceftriaxone and ciprofloxacin were supplied by Hexpharm Jaya, Indonesia. The chemicals used in the process included iron(III) chloride hexahydrate ($FeCl_3.6H_2O$), 37% concentrated hydrochloric acid (HCl), and 95 to 97% sulfuric acid (H_2SO_4), as well as 30% hydrogen peroxide (H_2O_2), were purchased from Supelco Sigma Aldrich. Additional reagents, such as 1.06498 of sodium hydroxide (NaOH) pellets, acetic acid (CH_3COOH) 695092 ACS reagent, methanol (CH_3OH) 179337 ACS reagent, ethanol (C_2H_6O), potassium permanganate ($KMnO_4$) 1.05082 ACS reagent, PhEur and barium chloride ($BaCl_2$) were supplied by MerckTM.

Synthesis of graphite from corn cob

The corn cob residue was sliced into small segments and thoroughly washed multiple times to remove dust and impurities. Subsequently, the segments were exposed to sunlight for two to three days and later dried in an oven at 100 degrees Celsius (°C) for 1.5 hours (h). The dried corn cobs are crushed into powder and carbonized in a furnace at 350 °C for 2 h. After cooling in a desiccator, the charcoal was finely pulverized using a mortar for the next stage. Next, 5 grams (g) of carbonized charcoal were added to 500 milliliters (mL) of distilled water and then stirred using a magnetic stirrer at 600 revolutions per minute

(rpm). To this solution, 4 mL of $FeCl_3.6H_2O$ was added at a rotation speed of approximately 900 rpm at room temperature. The acidity (pH) of the mixed solution was carefully adjusted to approximately pH = 2 by gradually adding 1 molar (M) of HCl, followed by stirring at 60 °C for 5 h. The resulting solution was centrifuged to separate the supernatant from the graphite precipitate. The precipitate was washed with distilled water until neutral, and then it was gradually oven-dried.

Synthesis of GO with modified hummers

The widely used Hummers method for synthesizing GO includes $KMnO_4$ and $NaNO_3$. Presently, a more environmentally friendly approach, known as the modified Hummers method, has been developed which eliminated the use of $NaNO_3$, increased $KMnO_4$ usage, in a mixture of H_2SO_4 and H_3PO_4, to reduce the production of toxic gases (Santamaría-Juárez *et al.,* 2019). The detailed procedures for the modified Hummers method are outlined as follows: Initially, 1 g of graphite sourced from corn cobs is placed into a beaker within a fume hood. Furthermore, 23 mL of concentrated H_2SO_4 enhances graphite reactivity and speeds up the oxidation process with $KMnO_4$. Stir the mixture using a magnetic stirrer and place it in an ice bath at 0 °C for 30 minutes. Gradually introduce 3 g of $KMnO_4$ while maintaining the temperature below 10 °C to prevent temperature fluctuations and potential explosions. Raise the mixed solution to 35 °C and stir for an additional 30 minutes, then gradually add 46 mL of distilled water and increase its temperature to 98 °C, maintaining it for 15 minutes. The oxidation process was halted by adding 140 mL of distilled water to 10 mL of a 30% H_2O_2 solution and stirred for 10 minutes. Wash the resulting suspension repeatedly with a 5% HCl solution to remove the sulfate content, then test with $BaCl_2$. Repeatedly wash with distilled water until reaching pH = 5, and separate the solution from the precipitation using centrifugation at 5000 rpm for 10 minutes. Disperse the produced precipitate in 450 mL of distilled water and sonicate for 2 hours, facilitating the exfoliation of graphite oxide sheets into a single-layer GO form. Filter the solution, and dry the precipitate obtained in an oven at 60 °C for 5 h to form GO (Chen *et al.,* 2013).

Characterization of GO

The functional groups in GO were identified through Fourier-transform infrared spectroscopy (FTIR), using an Agilent Technologies FTIR 630 Cary type machine. This analysis aimed to identify the bond configuration in the synthesized GO. Simultaneously, an examination of the phases present in both graphite and GO samples was conducted using X-ray diffraction (XRD). The XRD analysis was conducted using a 2013 PANalytical X'Pert Powder PW3040/60 X-ray diffractometer in the Netherlands, equipped with a nickel (Ni) filter and generated monochromated copper-potassium (Cu-K) radiation (Ångström [Å] = 1.54060) at 40 kilovolts (kV) and 30 milliamperes (mA). The scanning process occurred in step mode, covering 2 theta angles from 0° to 80° at a rate of 2° per minute. To further investigate the GO, the surface morphology and quantitative composition were analyzed using scanning electron microscopy with SEM-EDX (EVO® MA 10).

Optimization of antibiotic adsorption

Approxiamely 40 milligram (mg) of GO was put into a glass beaker, and 20 mL of a 14 parts per million (ppm) ceftriaxone standard solution was added. The pH of the solution was adjusted from 2 to 5 using either 0.1 M HCl or 0.1 M NaOH (Samimi and Shahriari-Moghadam, 2021). After stirring for 30 minutes, the stirrer was turned off to ensure the effective dispersion of GO in the antibiotic solution. Stirring speed plays a crucial role in the adsorption process, influencing the distribution and interaction between the adsorbent (GO) and the adsorbate (antibiotic). An insufficient stirring speed may result in uneven distribution of the adsorbent mass, potentially reducing the efficiency of the adsorption process. Subsequently, GO was separated from the solution through centrifugation at 10,000 rpm for 15 minutes, aiming for a swift separation of the filtrate from GO. The filtered solution was then analyzed using a UV-Vis spectrophotometer at a wavelength of 268 nanometers (nm). For the optimization process, variations in adsorbent mass, adsorbate concentration, and contact time, were implemented. The same method and procedure were applied for the optimization of ciprofloxacin antibiotics. The experiment was conducted with three replications, and the relative standard deviation (RSD) for pH (0.63% to 3.10%), concentration (1.88% to 1.48%), adsorbent mass (1.68% to 2.83%), and contact time (0.88% to 2.50%), showed good precision for

adsorption testing, with %RSD values < 5% for a 95% confidence level.

RESULTS AND DISCUSSION

Preparation and Charazterization of GO

Fig. 1 shows the processing sequence of corn cobs selected as raw materials for the production of GO. The process started with the drying of corn cobs, followed by crushing, leading to the generation of GO using the physically modified Hummers method (Chen *et al.,* 2013), and this led to the production of a powdered black susbstance. The synthesis of GO using the modified Hummers method takes ± 12 h before it can be used for the adsorption of antibiotics like ceftriaxone and ciprofloxacin. The preparation for the adsorption process lasts for an additional 3 h.

The GO obtained was further characterized using FTIR, SEM, and XRD instruments, as shown in Fig. 2. The generated FTIR spectrum in Fig. 2(b) showed distinctive features, including a hydroxyl group at a wavelength number of 3183 per centimeter (1/cm). Furthermore, absorption at wave numbers of 1897/cm, signified the presence of C-H bending groups (aromatic compounds), while the appearance of the C=C (cyclic alkene) group found in the 1580/cm spectrum indicated the existence of C-O bonds. The GO spectrum showed two absorption peaks, one at 1700/cm, indicating it contains carboxylic (-COOH) groups, and another at 1029/cm, depicting the presence of epoxy (C-O-C) functional groups. This consistency of functional groups in the FTIR spectrum with those in pure GO led to effective preparation (Chen *et al.,* 2015). The XRD results in Fig. 2(c) showed that the GO spectrum formed peaks at 10.62° and 23.42°, consistent with the research conducted by Özgan and Eskalen (2020), who reported two prominent ones at 10° and 23°. The SEM image of GO in Fig. 2(a) showed a thin sheet shape, without pores

Fig. 1: Preparation of GO: (a) corn cob, (b) crushed and (c) GO derived corn cob

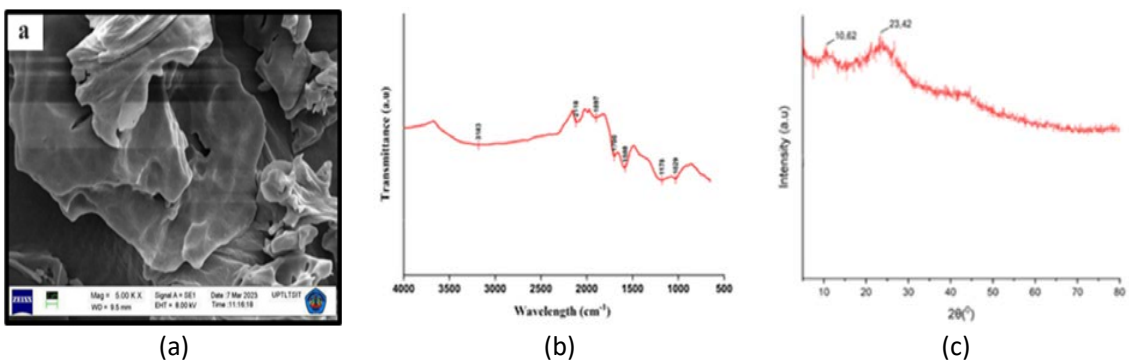


Fig. 2: (a) SEM image of GO, (b) FTIR spectrum, and (c) XRD spectrum

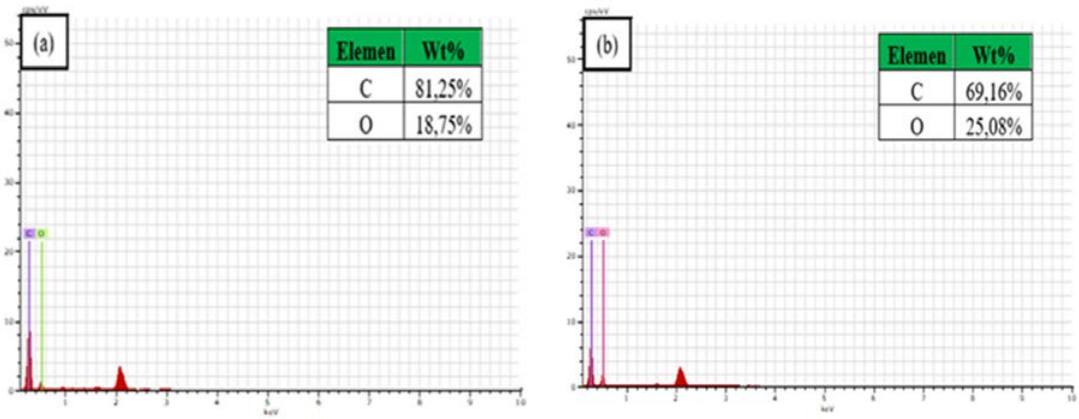


Fig. 3: EDX spectrum results on adsorbents: (a) graphite and (b) GO.

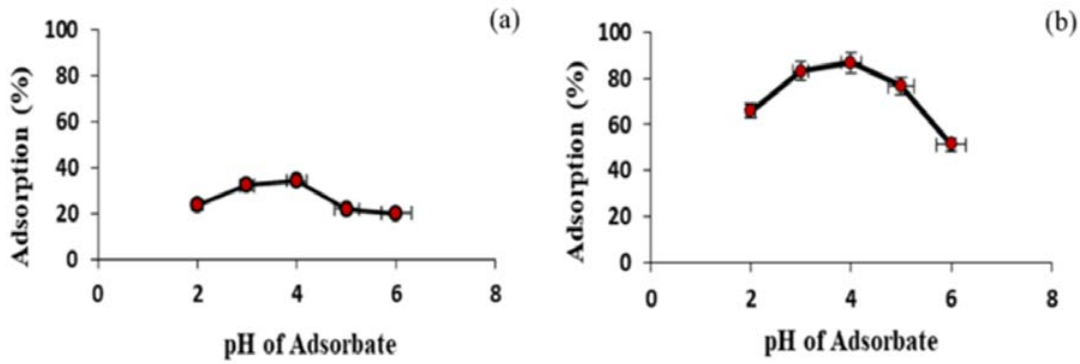


Fig. 4: Adsorption test results based on the effect of the adsorbate's optimum pH (a) ceftriaxone, and (b) ciprofloxacin

or wrinkled areas. In practice, GO has a relatively large surface that resembles a thin curtain. This confirmed the successful exfoliation procedure applied during the oxidation process and sonication, in line with the research preliminary (Naeini *et al.,* 2020).

The SEM characterization can be enhanced by using EDX to determine the elemental composition of the adsorbent. The results of GO adsorbent, illustrating the main elements as carbon (C) and oxygen (O), are shown in Fig. 3. Based on Fig. 3(a), the atomic composition of carbon (C) in graphite is 81.25%, indicating a higher number of elements. Conversely, in Fig. 3(b), the atomic percent (C) for GO is 69.16%, accompanied by oxygen percent (O) value of 18.75% and 25.08%, in graphite and GO, respectively. An increase in the oxygen percent indicates the successfully oxidation of GO, and this is in accordance with the adopted method, which mandates an increase in the content of this element in graphite oxide for the formation of GO (Kigozi *et al.,* 2020). Therefore, the combined SEM surface and EDX spectrum confirm the successful production of GO, presenting a smooth surface and sheet-like structure with high percentages of carbon (C) and oxygen (O) elements.

Adsorption optimization

Effect of pH adsorbate

Figs. 4(a) and 4(b) shows the results of the adsorption tests investigating the optimal pH effect for ceftriaxone and ciprofloxacin adsorption. The results showed that the adsorption efficiency of both ceftriaxone and ciprofloxacin compounds by GO reached its maximum level at pH = 4.

Ceftriaxone and ciprofloxacin underwent cationic and anionic transitions depending on the acidity,

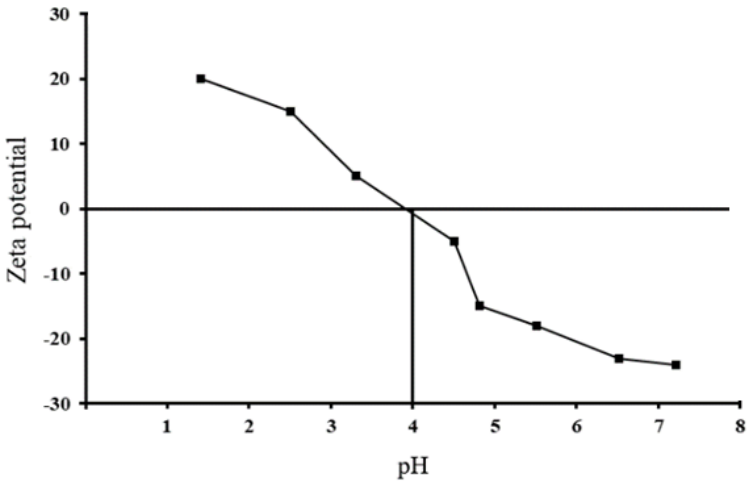


Fig. 5: Point of zero charge potential of GO

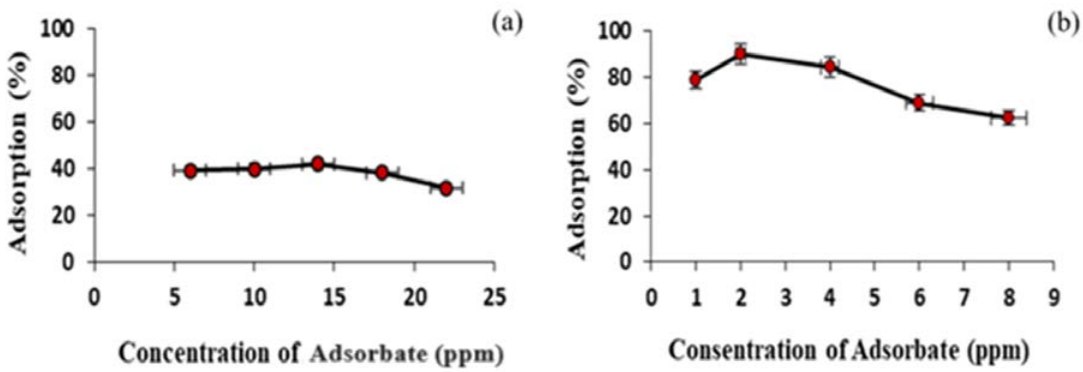


Fig. 6: Adsorption test to asses the influence of the optimal concentration of (a) ceftriaxone and (b) ciprofloxacin

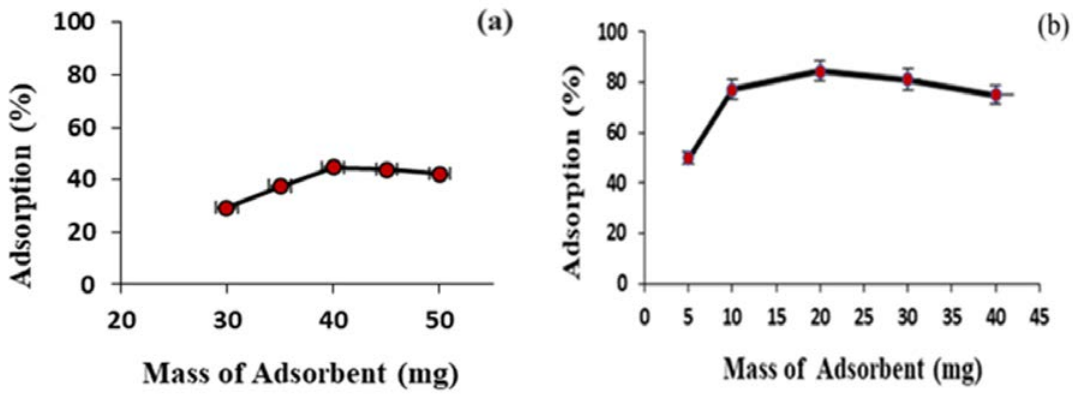
with protonation of the dimethyl-ammonium group resulting in a positive charge, while the loss of a proton from the carbonyl system led to a negative charge. GO point of zero charge (PZC), typically falls between 3.5 and 4, as shown in Fig. 5 (Li *et al.,* 2023).

The point of zero charge potential of GO is positively and negatively charged below and above the PZC (Li *et al.*, 2023). This difference in charge led to a strong electrostatic attraction between the oppositely charged ceftriaxone and ciprofloxacin and the functional groups of GO. The process led to a high adsorption capacity, in line with previous research (Yu *et al.*, 2016). As the pH increases to 7.0, GO decresaes, thereby enhancing its electronegativity and at 7, both GO and antibiotics carry negative charges, thereby intensifying the electrostatic repulsion (Chen *et al.*, 2015).

Effect of initial adsorbate concentration

Fig. 6 shows the results of the adsorption test, examining the impact of the optimal concentration of the adsorbates, specifically (a) ceftriaxone and (b) ciprofloxacin.

In Fig. 6(a), the optimal adsorbate concentration of 14 ppm, achieved a 41.95% adsorption rate. However, at concentrations of 18 and 22 ppm, the adsorption percentage decreased, indicating potential saturation of the adsorbent, and reduced absorption efficacy. Higher adsorbate concentrations tend to saturate more adsorbent pores, leading to a subsequent reduction in adsorption capacity. The prolonged contact between the GO adsorbent and ceftriaxone solution, which leads to increased adsorbate adsorption until equilibrium is reached, is in line with previous research (Tohamy *et al.*, 2020). In the



(a) (b)

Fig. 7: The outcomes of the adsorption test, focusing on the influence of the optimal adsorbent mass, are presented in (a) for ceftriaxone and (b) for ciprofloxacin.

adsorption process of ciprofloxacin by GO, optimal performance was observed at a concentration of 2 ppm, achieving a significant adsorption rate of 90.05%, as shown in Fig. 6(b). Meanwhile, at higher concentrations of 4 and 6 ppm, the adsorption rates gradually reduced. Lower adsorbate concentrations covering fewer active sites, decreasing the likelihood of adsorbate saturation in GO pores. The long contact time between the adsorbent and adsorbate increased the adsorbate amount on the surface of the adsorbent, which led to saturation and decrease in adsorption capacity (Yadav *et al*., 2018).

Effect of adsorbent mass

Fig. 7 shows the results of the adsorption test, examining the impact of varying adsorbent mass on both (a) ceftriaxone and (b) ciprofloxacin. The findings showed a connection between the adsorption rate and the mass of GO in ceftriaxone compounds. The optimal adsorbent mass condition was detected at 40 mg, achieving a 44.65% adsorption rate. However, within the range of 30 mg to 40 mg adsorbent mass, there was an increase in the levels of adsorbate absorbed. At 40 mg, the adsorbent mass reached its peak adsorption efficiency, attaining a point where the adsorbate absorbed is equal to the amount left in the solution, thereby maximizing the adsorption capacity. This enhancement in adsorption was attributed to the rise in active sites on the surface of the adsorbent, facilitating increased adsorbate absorption. Conversely, at a mass of 45 mg, there was a decrease in adsorption efficiency due to denser adsorbent particles, resulting in overlapping events during the process as shown in Fig. 7a. The increased density diminishes the active sites on the adsorbent, reducing its capacity to absorb adsorbate. This phenomenon is also similar to the findings reported in preliminary research (Beifing Lv *et al*., 2021).

Fig. 7(b) shows the relationship between the adsorption rate and the mass of GO in ciprofloxacin compounds. It is evident that the most effective adsorption process of ciprofloxacin compounds by GO occurred at 20 mg adsorbent mass at a rate of 84.55%. However, the use of adsorbent masses of 30 and 40 mg resulted in a decrease in the adsorption rate. This decline was attributed to a larger adsorbent mass causing increased density, leading to overlapping events between the particles. Consequently, these active sites were not maximized during the adsorbate absorption process (Bhaumik *et al.,* 2012), which is in accordance with preliminary research by (Arias *et al.,* 2020).

Effect of contact time

The results of the adsorption test, in respect to examining the impact of the optimal adsorbent mass for ceftriaxone (a) and ciprofloxacin (b) over different time intervals are shown in Fig. 8. The ceftriaxone adsorption test revealed a positive correlation between contact time and the adsorption of antibiotics, with a gradual increase as time progressed. This was attributed to the prolonged

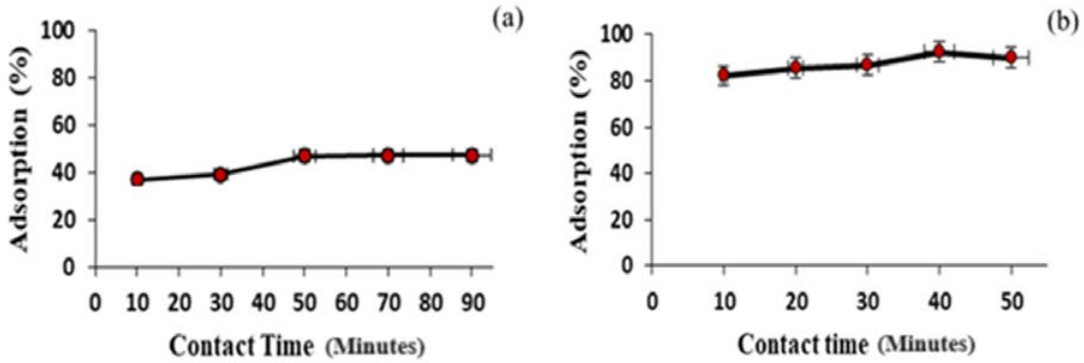


Fig. 8: The outcomes of the adsorption test, focusing on the influence of the optimal time for adsorbent mass, are presented in (a) for ceftriaxone and (b) for ciprofloxacin

Table 1: Adsorption capacities of different biosorbents

Biosorbent	Analyte	Adsorption level (%)	Contact time	Sources
Palm kernel Shell	Paracetamol	76.6	80 min	Osobamiro *et al.*, 2022
Calcium / iron (Ca/Fe)-layered double hydroxides (LDHs) from eggshell	Tetracycline	40-45	90 min	Abed *et al.*, 2023
Activated carbon from vine wood	Cephalexin	80	8 h	Pouretedal *et al.*, 2014
GO/Fe_3O_4-$SrTiO_3$	Cefotaxime	80	180 min	Nodeh and Saresti, 2016
Magnetic rod-like hydroxyapatite and MIL-101(Fe) metal–organic framework nanocomposite	Ciprofloxacin	26	25 min	Beiranvand *et al.*, 2022
Kaolin-Fe_3O_4	Cefixime	60	60 min	Azzouzi *et al.*, 2022
Corn cob	Ceftriaxone	47.04	50 min	The current study
Corn cob	Ciprofloxacin	92.62	40 min	The current study

contact time between the adsorbent and the adsorbate, enhancing its ability to be properly binded. At contact times ranging from 50 to 70 minutes, the percent adsorption value stabilized at 47%, with the most efficient contact time identified at 50 minutes as shown in Fig. 8a. Examining the outcomes at varying contact times in Fig. 8(b), the adsorption of ciprofloxacin by GO reached optimal conditions at 40 minutes, achieving a 92.62% adsorption rate. However, a reduction in adsorption rate was observed at a contact time of 50 minutes. This was attributed to the interaction between GO and ciprofloxacin compounds, leading to less-than-optimal GO adsorption power due to saturation in the adsorbent. The crowded surface area of the adsorbate causes ciprofloxacin compounds to desorb again (Yusof and Malek, 2009). This pattern is in line with the the findings reported in previous research (Asman *et al.*, 2016).

The adsorption level results in this research were compared with findings from several published investigations, as shown in Table 1. The GO adsorbent material sourced from corn cobs showed a significantly high adsorption capacity for the antibiotic ciprofloxacin. However, its adsorption capacity for ceftriaxone was found to be moderate compared to other biosorbents. The highest removal efficiency, reaching approximately 92.62%, was achieved using GO derived from corn cobs. According to Tohamy *et al.* (2020), GO derived from sugar cane bagasse showed the highest removal efficiency for Ni (III), reaching approximately 85.06%. Both sets of results were consistent with the Langmuir isotherm model, although it showed a significant difference in the removal efficiency of ceftriaxone. The maximum removal achieved with GO was 47%, which is lower than the highest one attained with an activated carbon-based TiO_2 composite, reaching 96.6% (Abdullah *et al.*, 2023). In addition, the isotherm model followed that of Freundlich in both cases.

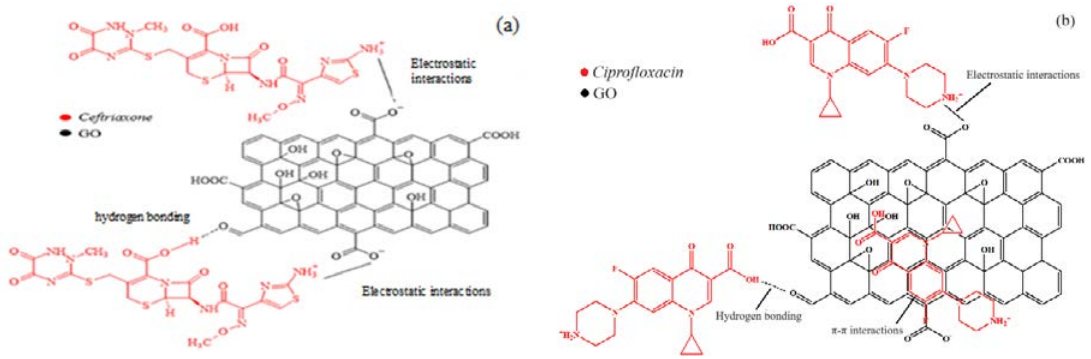


Fig. 9: Adsorption mechanism (a) ceftriaxone and (b) ciprofloxacin.

Table 2: Kinetics parameters for the adsorption of ceftriaxone and ciprofloxacin on GO.

Adsorbent	Adsorbate	Kinetic models			
		Pseudo-first-order		Pseudo-second-order	
		K_1	R^2	K_2	R^2
GO	Ceftriaxone	0.040	0.808	3.544	0.998
GO	Ciprofloxacin	0.015	0.734	0.150	0.848

Adsorption mechanism

Based on the results of the adsorption process, it was observed that the antibiotic ceftriaxone had a lower percentage compared to ciprofloxacin. This variation in adsorption behavior is due to the specific interaction mechanisms between ceftriaxone and ciprofloxacin on the GO surface, as shown in Fig. 9. The adsorption mechanisms that occur between GO and both antibiotics are hydrogen bonding, electrostatic and π-π interactions. Meanwhile, the benzene and nitrogen-heteroaromatic rings attached to the fluorine as well as ciprofloxacin and ceftriaxone groups, respectively can engage in π interactions.

The benzene ring contained in GO functions as a π electron donor, while the aromatic one in ciprofloxacin acts as an acceptor in electron interactions. The presence of two C=O groups and one OH, facilitates the formation of hydrogen bonds with oxygen-containing groups found on GO (Gamoń *et al.,* 2022). However, due to its aromatic benzene ring, ciprofloxacin showed a higher propensity for engaging in π-π interactions. In contrast, the structural characteristics of ceftriaxone makes it less likely to participate in π-π interactions, due to the lack of aromatic benzene rings. The patterned structure of the ceftriaxone molecule also hindered its ability to form strong and specific bonds with the GO surface, resulting in weaker interactions and observed lower adsorption percentage. Molecules with ordered structures, such as ciprofloxacin, tend to form stronger bonds with the solid or liquid surfaces due to its organized interaction sites (Zhang *et al.,* 2017). The smaller molecular shape, enhances its efficiency in reaching active adsorption sites. This is in line with the research conducted by Arias *et al.* (2020), which stated molecule size and shape significantly impact absorption efficiency, with smaller ones easily reaching adsorption sites. The influence of molecular structure on adsorption capacity arises from the ability of molecules with more active sites to form additional adsorption bonds, resulting in a greater percentage (Kuroki *et al.,* 2019).

Adsorption kinetics and adsorption isotherm

The simulation of adsorption kinetics was conducted using the pseudo-first and second-order models, as shown in Table 2 and Fig. 10.

The higher R^2 values, was used to evaluate the fit of the data and ensure it is line with either the pseudo-first or second-order models (Samimi and Shahriari-Moghadam, 2023). Examining the results of the adsorption kinetics for ceftriaxone and ciprofloxacin

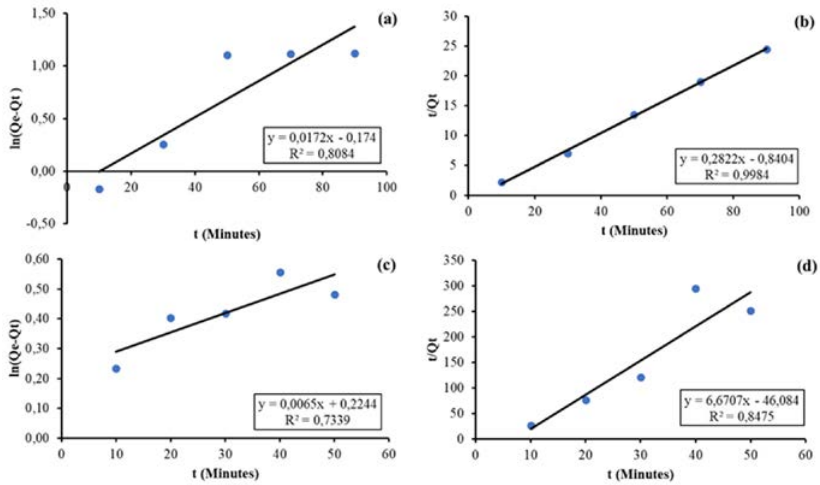


Fig. 10: Kinetic model graph of (a) pseudo-first-order ceftriaxone; (b) pseudo-second-order ceftriaxone; (c) pseudo-first-order ciprofloxacin and (d) pseudo-second-order ciprofloxacin

compounds, it was observed that the pseudo-first-order model poorly characterized antibiotic adsorption, due to its significantly small regression value. Conversely, the pseudo-second-order model established a more accurate and linear relationship, as reflected in its higher regression values. This supports the notion of a pseudo-second-order kinetic model for the adsorption process. In line with other research, Tang *et al.* (2013), reported excellent outcomes for the pseudo-second-order model in the adsorption of antibiotics such as ciprofloxacin and norfloxacin on reduced graphene oxide-M (RGO-M) surfaces. The formulas for pseudo-first and second-order reactions are expressed in Eqs. 1 and 2 (Samimi, 2024).

$$\log(q_e - q_t) = log(q_e) - \frac{K_1 t}{2,303} \quad (1)$$

$$\frac{t}{q_t} = \frac{1}{h} - \frac{t}{q_e} \quad (2)$$

$$t\frac{1}{2} = \frac{1}{K_2 q_e^{\,2}}$$

Where;

q_e and q_t are the amounts of Ceftriaxone and Ciprofloxacin adsorbate at equilibrium and time (t), respectively, measured in milligrams per gram (mg/g); K_1: is the pseudo-first-order rate constant per minute (/min); t1/2: the time required for the adsorption to take up half as many compounds as its equilibrium values per minute (min); h: K_2qe^2 is the initial adsorption rate as milligrams per minute (mg/min); K_2 is the pseudo-second-order rate constant in /min.

The Langmuir equation model using Eq. 3 (Samimi *et al.*, 2023).

$$\frac{C_e}{q_e} = \frac{1}{K_L Q_{max}} + \frac{1}{Q_{max}} C_e \quad (3)$$

Where;

C_e: is the equilibrium concentration of adsorbate (mg/L).

q_e: unit mass of adsorbent (mg/g).

K_L: Langmuir constant related to the measure of affinity of the adsorbate for adsorbent (L/mg).

Q_{max}: amount of adsorbate per unit mass of adsorbent (mg/g).

The Freundlich model is represented using Eq. 4 (Azimi *et al.*, 2019; Mohadesi *et al.*, 2023).

$$\log q_e = log\, \mathrm{K}_F + \frac{1}{\mathrm{n}} \log C_e \quad (4)$$

Where;

q_e: unit mass of adsorbent (mg/g).

C_e: is the equilibrium concentration of adsorbate

Table 3: Parameters of the ceftriaxone and ciprofloxacin adsorption isotherm on GO

Antibiotics	Adsorption Isotherm	Parameter 1	Parameter 2	R^2
Ceftriaxone	Langmuir	K_L = 1.274 L/mg	Qe = 3.021 mg/g	0.6177
	Freundlich	K_F = 2.116 (mg/g).(L/mg)$^{1/n}$	n = 1.243	0.9156
Ciprofloxacin	Langmuir	K_L = 1.445 L/mg	Qe = 2.065 mg/g	0.9101
	Freundlich	K_F = 3.235 (mg/g).(L/mg)$^{1/n}$	n = 1.854	0.7415

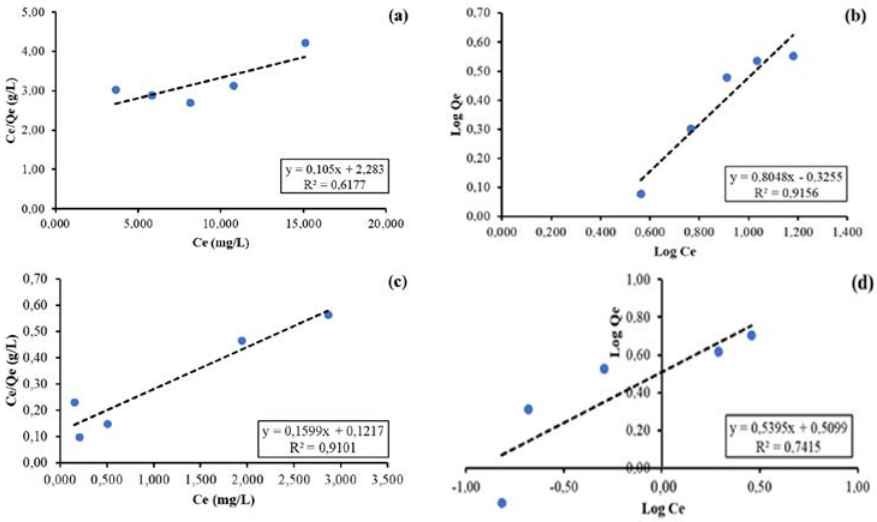


Fig. 11: Isotherm model graph (a) langmuir ceftriaxone; (b) freundlich ceftriaxone; (c) langmuir ciprofloxacin and (d) freundlich ciprofloxacin

(mg/L).

K_F: is the Freundlich constant for adsorption capacity (mg/g).

n: Freundlich index, which describes the degree of adsorption and surface heterogeneity.

A larger n value indicates stronger adsorption (Samimi and Safari, 2022), in addition the equilibrium data were subjected to further analysis using the Langmuir and Freundlich isotherm models, as shown in Table 3 and Fig. 11. The regression value (R^2) in Table 2 shows that in the adsorption process, ciprofloxacin has the highest R^2 value, following the Langmuir model (R^2) of 0.9101. However, ceftriaxone tends to adhere more closely to the Freundlich model (R^2) of 0.9156. The presence of chemical bonds showed that the adsorption of ciprofloxacin included chemical adsorption. These chemical bonds found in ciprofloxacin and GO include hydroge, and π-π bonds, as well as electrostatic interactions. The result obtained also showed that chemical adsorption interactions are less effective in the adsorption of ceftriaxone (Asman *et al.*, 2016).

CONCLUSION

In conclusion, the current study investigated the adherence of ceftriaxone and ciprofloxacin to graphene oxide (GO) derived from corn cobs, revealing intriguing and conspicuous findings. GO from corn cobs were subjected to FTIR analysis, identifying functional groups, mainly hydroxyl and carboxyl, enhancing the interaction potential of active sites on GO with other molecules. The SEM result showed that a smooth surface had been formed, constituting a thin monolayer sheet on the obtained GO. However, through careful experimentation, the ideal conditions for the adsorption of ceftriaxone and ciprofloxacin compounds were discovered. Overall 40 mg and 20 mg of adsorbent masses performed effectively with antibiotic concentrations of 14 ppm and 2 ppm, a pH level of 4, and contact times of 50 minutes and 40 minutes, respectively. These findings focused on the importance of precise experimental optimization in enhancing the removal efficiency of these antibiotics. The adsorption test for ciprofloxacin and ceftriaxone followed a pseudo-second-order model. Ciprofloxacin

and ceftriaxone had a significant removal efficiency of 92.62%, and 47%, respectively. The adsorption isotherms were in line with the Langmuir Model (R^2 = 0.9101) for ciprofloxacin, while ceftriaxone tended to follow the Freundlich Model (R^2 = 0.9156). This study reported the potential of the derived material as a promising adsorbent for efficiently removing ciprofloxacin from aquatic environments. Although these results were promising, it was evident that more work needed to be done to enhance the effectiveness of the adsorbent in eliminating ceftriaxone, allowing for broader applications with minimal environmental impact. The present study contributed to the scientific understanding of adsorption processes and also showed the potential of using adsorbents derived from agricultural waste for sustainable water treatment. This alternative method addressed both antibiotic residue pollution and the challenges associated with agricultural waste, presenting a promising path for eco-friendly water treatment practices, and contributing to the resolution of interconnected environmental issues.

AUTHOR CONTRIBUTIONS

Rinawati designed the field experiment, organized the study and discussion, and contributed to the preparation of the manuscript. M.D. Imelda conducted FTIR and SEM data analysis and interpreted the results. D.R. Mythia performed data optimization analysis, XRD, and kinetic data analysis. A. Rahmawati prepared tables and figures and interpreted the results. A.A. Kiswandono supervised the experiment and contributed to the preparation of the manuscript. F.H. Latief implemented neural network analysis and prepared related text and figures. S. Mohamad participated in the interpretation of results and contributed to manuscript preparation.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interest regarding the publication of this manuscript. The ethical issues, including plagiarism, informed consent, misconduct, data fabrication and/or falsification, double publication and/or submission, and redundancy, have been completely observed by the authors.

ABBREVIATIONS

%	Percent
°C	Degrees Celsius
[Å]	Ångström
$BaCl_2$	Barium chloride
BPS	Badan pusat statistik (in indonesian) / central bureau of statistics
C_2H_6O	Ethanol
CH_3COOH	Acetic acid
CH_3OH	Methanol
/cm	Per centimeter
CNTs	Carbon nanotubes
Cu-K	Copper-potassium
Eqs.	Equation
$FeCl_3.6H_2O$	Iron(III) chloride hexahydrate
Fig.	Figure
FTIR	Fourier transform infrared
g	Grams
g/L	Gram per liter

GO	Graphene oxide
H_2O_2	Hydrogen peroxide
H_2SO_4	Sulfuric acid
h	Hour
HCl	Hydrochloric acid
K	Constant
K_2qe^2	The initial adsorption rate as milligram per gram.minutes
$KMnO_4$	Potassium permanganate
kV	Kilovolts
L/mg	Liter per milligram
M	Molar
mA	Milliamperes
mg	Milligrams
mg/g	Milligram per gram
mg/g.min	Milligram per gram.minutes
min.	Minute
/min	Per minute
mL	Milliliters
MRL	Maximum Residue Limit
NaOH	Sodium hydroxide
Ni	Nickel
nm	Nanometer
NPs	Nanoparticles
pH	Potential of hydrogen
ppm	Parts per million
PZC	Point of zero charge
q_e	The amount of ceftriaxone and ciprofloxacin adsorbate at equilibrium
q_t	the amount of ceftriaxone and ciprofloxacin adsorbate at time
R^2	Regression
RGO	Reduced graphene oxide
rpm	Revolutions per minute
RSD	Relative standard deviation
SEM-EDX	Scanning electron microscope-energy dispersive x-ray
t	Time
UV-Vis	Ultra violet-visible
XRD	X-ray diffraction

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