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Kinetics and adsorption studies on the removal of levofloxacin using coconut coir charcoal impregnated with Al_2O_3 nanoparticles

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ABSTRACT

Adsorption studies on removal of fluroquinolone antibacterial agent levofloxacin by means of alumina-doped coconut coir charcoal, a low-cost adsorbent prepared from coconut coir impregnated with alumina nanoparticles, were studied using a UV–Vis spectrophotometer. The adsorbent was prepared by combustion technique with *in situ* impregnation of alumina nanoparticles. The prepared adsorbent was characterized using X-ray diffraction, scanning electron microscopy and energy dispersive X-ray spectroscopy studies. Batch adsorption experiments were performed to study the effect of drug concentration, pH and adsorbent dosage. The optimal pharmaceutical molecule concentration, pH, adsorbent dose with removal percentage was found to be 3.6 mg/l (64%), 7 (56.6%) and 0.1 g (59.83%), respectively. The adsorption studies comprise both equilibrium adsorption isotherms and kinetics. The equilibrium data have been analyzed using Langmuir, Freundlich and Temkin isotherms. The adsorption isotherm data for levofloxacin were best represented by both Langmuir and Temkin isotherm.

Keywords: Adsorption; Levofloxacin; Coconut coir; Alumina; Kinetics; Nanoparticles

1. Introduction

Pharmaceuticals have become a vital concern due to presence of it in environment and are classified as the emerging contaminants [1–3].These pollutants may come in contact with marine environment through diverse pathways like excretion, hospital wastes, direct disposal, etc. Usually, the pharmaceutical compounds like fluroquinolone are not easily degradable at treatment plants [2]. Fluroquinolones are antibiotics which are generally used in treating human beings and animals. Because of the partial absorption of fluroquinolones, they are released into community sewage, which makes a route for antibiotics to enter into the marine environment. Fluroquinolones are found to be in various concentrations in both sewage treatment plant and industrial effluent treatments plants [1]. Hence, pharmaceuticals in the marine environment

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lead to serious effects such as contamination of water bodies, groundwater contamination, bacteria developing resistance to these pharmaceutical agents, thus affecting the health of human beings, flora and fauna [3]. Thus, with this point of view, there are a variety of techniques reported in the literature to treat these pharmaceutical molecules in the wastewater viz. chlorination [1,4], reverse osmosis [5], nanofiltration [5], ozonation [6], permanganate treatment [7], etc. But these methods have drawbacks in terms of economy, high power necessity, etc. Therefore, the use of adsorption technique holds good compared to other techniques because of its benefits such as low cost, efficient in decontamination, flexible and easy in process, etc. [2,8,12].

Nowadays, economy is been chief consideration; therefore, in case of using high cost commercial activated adsorbent like the zeolites, silica gel etc., low-cost adsorbents such as rick husk, coconut coir, saw dust, etc. are been greatly employed in the removal of pharmaceutical compounds from wastewater [9].

The purpose of this study is to explore the use of modified coconut coir as an adsorbent for removal of levofloxacin drug. The main interest of using coconut coir is because its easily accessible and coir as bio-sorbent material presents good potential due to high composition of lignin 35–45% and cellulose 23–43% [10].

This study has considered a particular pharmaceutical molecule named levofloxacin (Fig. 1) which is a antibiotic of fluroquinolone. It is also known as Levaquin, Tavanic, etc. Levofloxacin is used to treat bacterial infectivity like pneumonia, abdonominal infections, skin infections, etc. Molecular formula of the drug is $C_{18}H_{20}FN_3O_4$, molecular weight is 361.368 g/mol and λ_{max} is 295 nm [1].

In addition to kinetics and isotherms, outcome of pharmaceutical molecule concentration, pH and adsorbent dosage is also studied.

2. Experimental

2.1. Materials

Levofloxacin was obtained from Dr Reddy's laboratory, Hyderabad India. Stock solution of 1×10^{-4} M (36 mg/l) levofloxacin was prepared via dissolving a precisely weighed 0.018 g of levofloxacin (M.Wt. = 361.368 g/mol) in 500-ml volumetric flask by distilled water [11,12].

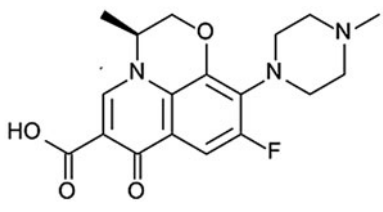

Fig. 1. Structure of levofloxacin drug.

2.2. Methods

2.2.1. Modification of coconut coir charcoal adsorbent

Coconut coir was obtained from the local market. The coir was cut in equal sizes to achieve uniform carbonization. The coir was then modified by impregnating Al_2O_3 nanoparticles by *in situ* combustion technique using aluminium nitrate and urea as precursors. This process was carried out by mixing 10 g of coconut coir with aluminium nitrate and urea in the ratio 1:2 in a crucible and then it was kept in a muffle furnace at a temperature of 400°C for about 15 min. After carbonization, the modified coir charcoal was powdered and then washed 3–4 times with distilled water to remove impurities and then it was kept in oven to dry at 100°C for a day [13].

2.3. Batch studies

Batch adsorption mode was adopted for this study. The experiment was carried out to investigate the effect of variation of LFC concentration, pH and adsorbent dosage, etc. using alumina-doped coconut coir charcoal (ADC) as an adsorbent. All the samples in test were kept for shaking at 200 rpm and were taken out from shaker at definite time interval and were centrifuged at 5,000 rpm for about 30 min so as to get clear supernatant solution. Absorbance of the solution was measured using UV spectrophotometer at λ_{max} 295 nm [2,11,12].

The percentage adsorption was calculated for all the parameters using the following equation [9,14]:

$$\text{Adsorption}\,\% = \frac{C_0 - C_f}{C_0} \times 100 \quad (1)$$

where C_0 = initial concentration of drug before adsorption, C_f = final concentration of drug after adsorption.

2.4. Adsorption isotherm and kinetics

Equilibrium studies were carried out for various initial concentrations of LFC and this information was used for Langmuir, Freundlich and Temkin isotherms so that to determine the suitability of isotherm model.

3. Results and discussion

3.1. Characterization study

3.1.1. Scanning electron microscopy

Scanning electron microscopy (SEM) is a tool to distinguish the surface morphology and physical properties of the adsorbent substances. It is helpful for determining the element form, porosity and suitable dimension distribution of the adsorbent. The surface description of the activated carbon was analyzed using a scanning electron microscope. The surface morphology of the ADC shows that the pore size is around 1 μm which shows that surface morphology was not much influenced by adsorption process [15] (Fig. 2).

3.1.2. X-ray diffraction

X-ray diffraction (XRD) is a method used in determination of crystallinity of a compound. Carbon was found in the XRD peak of the ADC (Fig. 3).

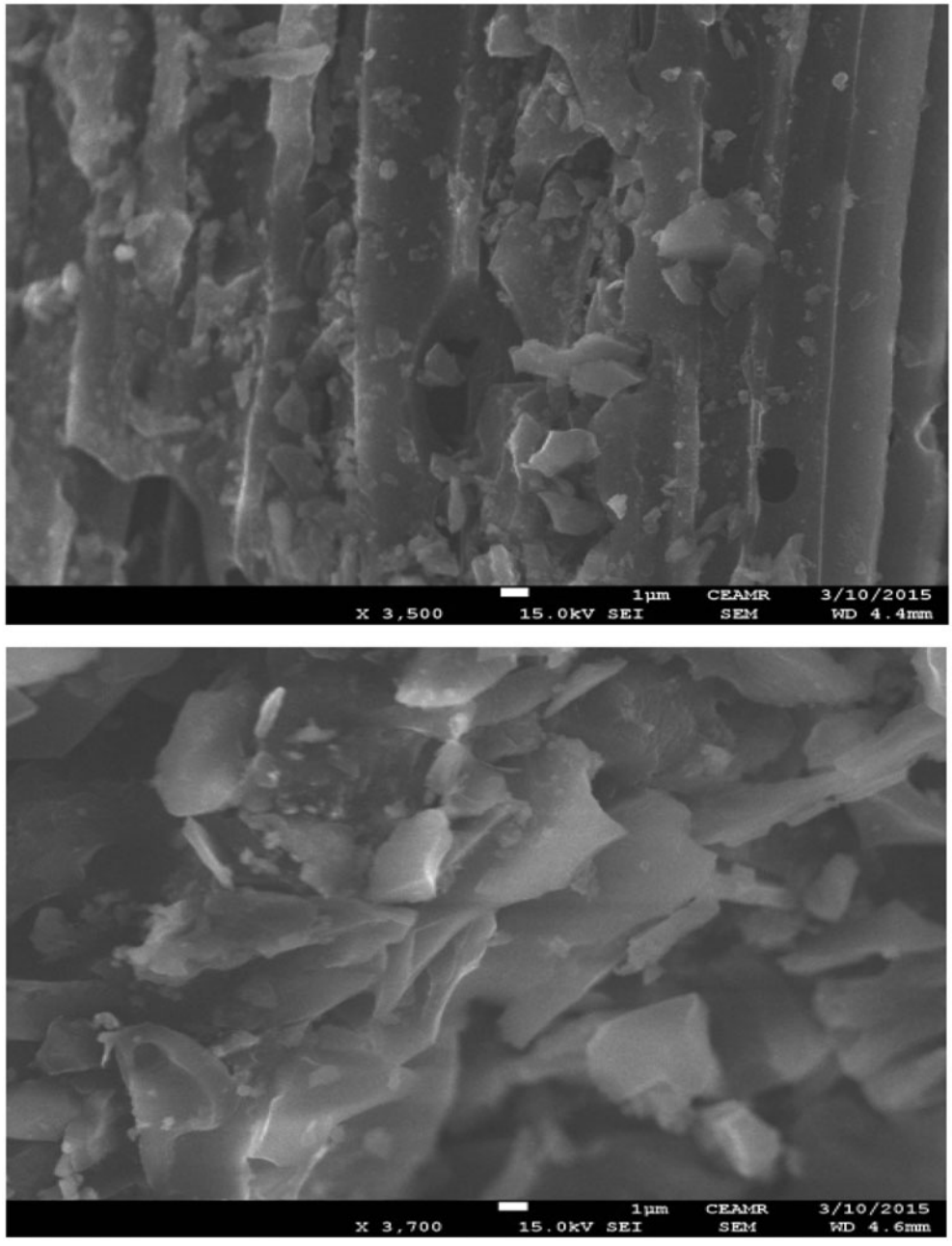

Fig. 2. SEM images of aluminium oxide nanoparticles doped on activated charcoal.

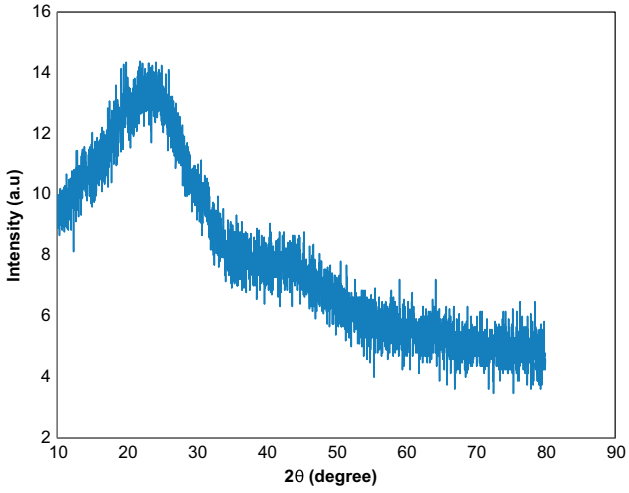

Fig. 3. XRD results showing aluminium oxide nanoparticles doped on activated charcoal.

3.1.3. Energy dispersive X-ray spectroscopy

Energy dispersive X-ray spectroscopy (EDS) is an analytical system used for elemental examination or chemical characterization of a sample [16]. The elemental composition of ADC was determined by EDS (Table 1) representing the percentage of Al that is 0.80%. This illustrates the doping of aluminium oxide nanoparticles on the coconut coir (Fig. 4).

3.2. Effect of LFC concentration

A constant dosage of ADC was used to study the influence of concentration of LFC and the experiment was carried out by varying the initial concentrations of the drug from 0.7 to 5 mg/l ($0.2–1.5 \times 10^{-5}$ M). Then, the mixture was agitated with ADC in a shaker for 1 h and was centrifuged at 5,000 rpm for 30 min to obtain clear supernatant and concentration of LFC was analyzed by spectrophotometer. The process was repeated till it attains equilibrium stage. Equilibrium time period for LFC drug was 300 min. From the initial concentration of drug and concentration at equilibrium, the % efficiency was computed for different LFC concentration [6,7]. From Fig. 5, it is clear that percentage adsorption was increasing up to 3.6 mg/l (1.0 M) beyond which there was no considerable increase. Therefore, optimal LFC concentration is 3.6 mg/l (1 M) with highest efficiency being 64%.

3.3. Effect of pH

The interaction study between adsorbent 0.1 g of ADC and drug concentration 3.6 mg/l (1 M) was carried out at different pH to find out optimal pH for maximum adsorption. pH of the solution was varied from 5.5 to 8.0 and prepared solutions were mixed for physical adsorption by keeping the solution in shaker. Solutions withdrawn at definite interval of time from shaker were centrifuged at 5,000 rpm for 30 min to obtain supernatant which is free from adsorbent particles and concentration of LFC analyzed by spectrophotometer. The process was repeated till the equilibrium was reached. From the original concentration of LFC and concentration of LFC at equilibrium, the % efficiency was calculated for different pH.

From Fig. 6, it is clear that adsorption is somewhat low at first, at low pH values of the solution. As the pH increases, the adsorption increased and reached a maximum value when pH of the solution is 7.0. After pH 7.0, the degree of adsorption decreases. LFC neutral species is dominant between pH 6 and 8 compared to LFC anion and LFC cation species. Therefore, it can be concluded that utmost LFC uptake is obtained at pH 7 with percentage adsorption being 56%. Similar outcome was reported by Mohanad J. Mohammed-Ridha 2014 that is optimal pH being 7, the sorption capacity of levofloxacin onto activated carbon, barley husk, egg shells [2].

3.4. Effect of adsorbent dosage

To evaluate the effect of adsorbent dosage that is ADC for removal of LFC drug, the experiment was carried out by changing the amount of ADC, which was varied from 0.02 to 0.2 g keeping other parameters constant. The solutions were agitated with different adsorbent dosage and then the solutions were kept in shaker for 1 h and then were centrifuged at 5,000 rpm for 30 min to obtain supernatant and concentration of LFC analyzed by spectrophotometer. From the original concentration of adsorbent dosage

Table 1
Element composition of modified coconut coir by EDS

Type	Element wt (%)			
Aluminium oxide nanoparticles doped on activated charcoal	C 78.00	O 20.00	Al 0.80	S 1.20

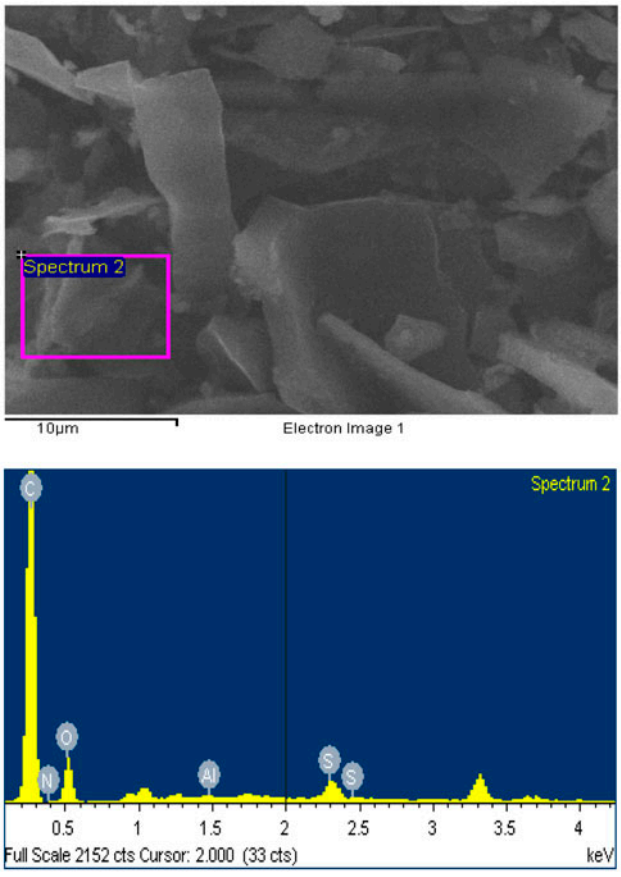

Fig. 4. EDS results showing aluminium oxide nanoparticles doped on activated charcoal.

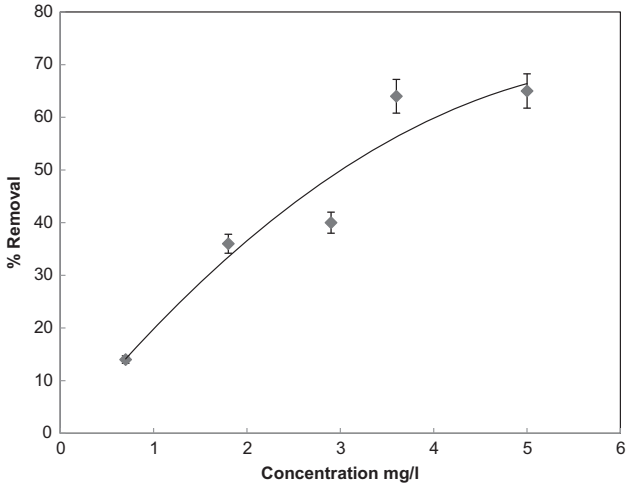

Fig. 5. Effect of initial concentration on adsorption of levofloxacin by ADC.

and concentration at equilibrium, the % efficiency was calculated for different adsorbent dosage [6,7]. From Fig. 7, it is clear that the adsorption efficiency

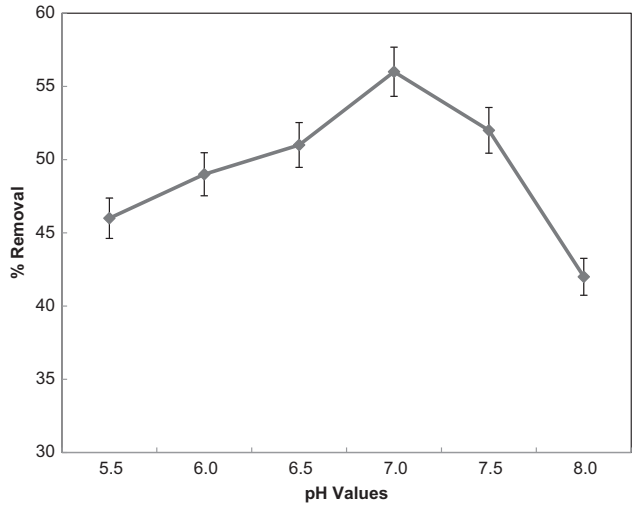

Fig. 6. Effect of pH on adsorption of levofloxacin by ADC.

of the ADC for removal of LFC drug increases with increasing the adsorbent quantity from 0.02 to 0.1 g with maximum percentage adsorption that is 59%, but further increase up to 0.2 g was found to decline the adsorption efficiency. This study can be explained by the number of active sites as the major reason for the differences. In fact, the total number of active sites increased with increasing adsorbent dosage. But further increase up to 0.2 g was found to reduce the adsorption efficiency. This may be due to the fact that as the adsorbent increase above the optimal amount, the active sites may be close with each other [17].

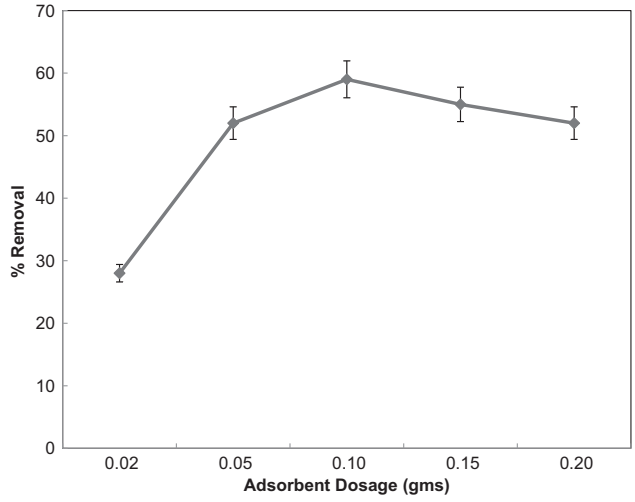

Fig. 7. Effect of adsorbent dosage (g) on adsorption of levofloxacin by ADC.

4. Adsorption isotherms

The equilibrium adsorption isotherm defines the interaction between adsorbate and adsorbent, and is essential in the design of adsorption systems [18]. The equilibrium adsorption isotherm was conducted for different concentrations of LFC pharmaceutical molecule with other parameters being constant.

The isotherms viz. Langmuir, Freundlich and Temkin isotherms were applied to study adsorption equilibrium.

Langmuir is based on statement that most favourable adsorption corresponds to a saturated single stratum of adsorbate molecules on adsorbent surface, energy of adsorption is stable; there is no transfer of adsorbate molecules in plane adsorbent surface [19].

Langmuir isotherm is given as:

$$\frac{C_e}{q_e} = \frac{1}{Q_o K_L} + \frac{C_e}{Q_o} \tag{2}$$

where C_e = equilibrium concentration mg/l, q_e = quantity of drug adsorbent onto modified coir at equilibrium mg/g, Q_o and K_L = Langmuir constants related to adsorption capacity and energy of adsorption. The essential features of the Langmuir isotherm may be expressed in terms of equilibrium parameter R_L, which is a dimensionless constant referred to as separation factor or equilibrium parameter.

$$R_L = \frac{1}{1 + K_L C_0} \tag{3}$$

where C_0 = initial concentration, K_L = the constant related to the energy of adsorption (Langmuir Constant). R_L value indicates the adsorption nature to be either unfavourable if $R_L > 1$), linear if $R_L = 1$, favourable if $0 < R_L < 1$ and irreversible if $R_L = 0$ [20].

According to Mckay et al., if the R_L values are between 0 and 1, then the adsorption is favourable [21]. The R_L values for Langmuir isotherm were found to be 0.18–0.03 for LFC pharmaceutical molecule concentrations 0.7–5.0 mg/l, which implies that adsorption of Levofloxacin on coconut coir activated charcoal is favourable adsorption as R_L values obtained at all initial concentrations lie between 0 and 1. Q_o and K_L are obtained from slope and intercept of Langmuir plot i.e. C_e vs. C_e/q_e. Fig. 8 of Langmuir isotherm shows correlation coefficient $R^2 = 0.983$.

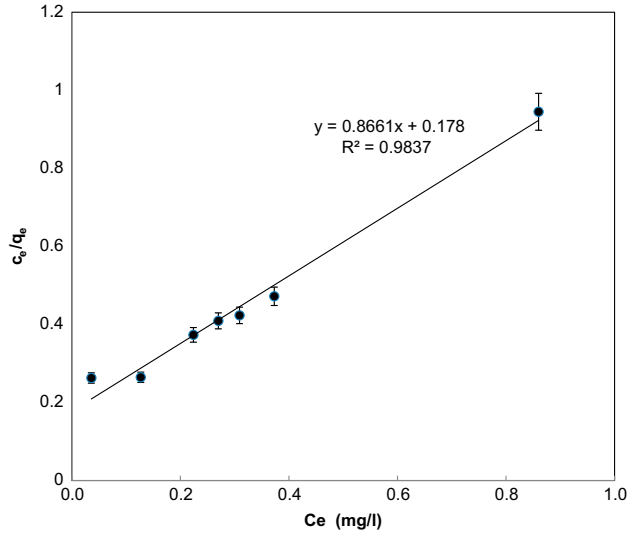

Fig. 8. The Langmuir isotherm model for the adsorption of levofloxacin on ADC.

4.1. Specific surface area

Monolayer coverage of the surface by the LFC can be used for the calculation of the specific surface area S according to equation:

$$S = \frac{Q_o N A}{M} \tag{4}$$

where S is the specific surface area, m^2/(g of adsorbent), Q_o monolayer sorption capacity, g of LFC/(g of adsorbent), N is the Avogadro number, 6.022×10^{23}, A is the cross sectional area of LFC, m^2, M is the molecular weight of LFC. The molecular weight and cross sectional area of LFC are 361.36 g/mol and 487.80×10^{-20} m^2, respectively, in a closed-packed monolayer [22,23].

Freundlich isotherm equation describes the surface heterogeneity and the exponential distribution of active sites and their energies [22].

Freundlich isotherm is given as:

$$\log q_e = \log k_f + \frac{1}{n \log C_e} \tag{5}$$

where q_e and C_e = the equilibrium concentrations of drug on the adsorbents (mg/g) and liquid phases (mg/l). K_f and n are the Freundlich constant related to adsorption capacity and adsorption intensity, respectively. Freundlich isotherm data are shown in Fig. 9. K_f and $1/n$ are obtained from slope and intercept of Freundlich plot i.e. log q_e vs. log C_e. According to

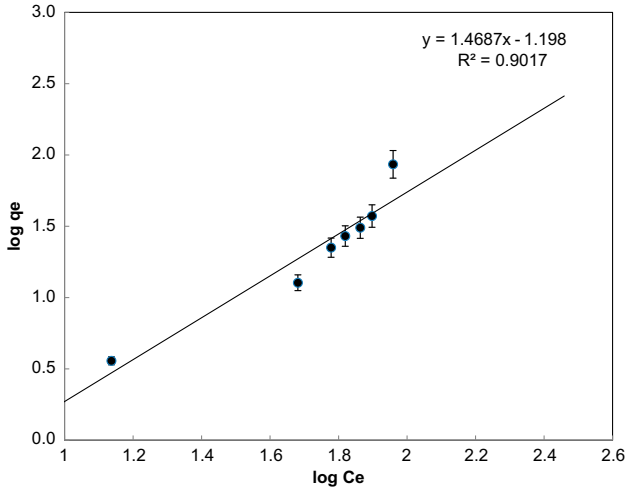

Fig. 9. The Freundlich isotherm model for adsorption of levofloxacin on ADC.

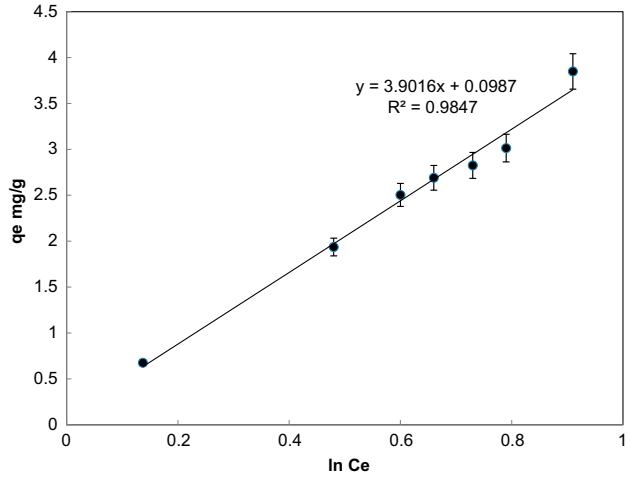

Fig. 10. The Temkin isotherm model for adsorption of levofloxacin on ADC.

Kadirvelu and Namasivayam [24], it has been shown that n values between 1 and 10 represent favourable adsorption. The numerical value of $1/n < 1$ indicates that adsorption capacity is only slightly suppressed at lower equilibrium concentration [25]. Freundlich isotherm shows correlation coefficient R^2 0.901 and value of n is 1.63; thus, value of n lies within the range which implies that ADC has high affinity for levofloxacin (Fig. 9).

Temkin isotherm includes a factor that takes into account the adsorbent–adsorbate interactions.

The isotherm is given by the following equation:

$$q_e = B \ln A_T + B \ln C_e \tag{6}$$

$$B = \frac{RT}{b_T} \tag{7}$$

where A_T = Temkin isotherm equilibrium binding constant (L/g), b_T = Temkin isotherm constant, R = universal gas constant (8.314 J/mol/K) T = Temperature at 298 K, B = Constant related to heat of sorption (J/mol) [26]. From Temkin plot that is q_e against ln C_e shown in Fig. 10, the following values were estimated: A_T = 0.94 L/g, B = 25.237 J/mol which is an indication of the heat of sorption indicating a physical adsorption process and the R^2 = 0.984. From Fig. 10, it is clear that adsorption follows the Temkin isotherm model. Langmuir, Freundlich and Temkin isotherms' data are presented in Table 2.

5. Adsorption kinetics

The study of adsorption kinetics of elimination of pharmaceutical compounds from wastewater is significant, as it provides insights into the reaction pathways and into the mechanism of adsorption reactions. It explains the solute uptake rate which manages the residence period of sorbate uptake at the solid–solution interface [2]. The kinetic study for the adsorption of pharmaceutical molecule was performed and the adsorption of LFC drug was complete in 300 min. The kinetic data were fixed in pseudo-first order and second order. Adsorption kinetic data of LFC are analyzed using the Lagergren first-order rate equation [27]:

$$\log (q_e - q_t) = \log q_e - \frac{k_1 t}{2.303} \tag{8}$$

Table 2
Parameters of Langmuir, Freundlich and Temkin adsorption models

Langmuir isotherm			Freundlich isotherm				Temkin isotherm			
K_L L/mg	Q_o mg/g	R^2	K_f mg/g	$1/n$	n	R^2	A_T L/g	b_T	B J/mol	R^2
6.4	1.15	0.983	8.05	0.613	1.63	0.901	0.94	98.17	25.237	0.984

Table 3
Pseudo-first-order and second-order rate constants for pH 5.5–7.5

pH	$10^3 k_1$ min^{-1}	k''_{app} L mol^{-1} min^{-1}
5.5	3.44	1,240
6	3.27	1,183
6.5	1.52	550
7	0.431	155
7.5	0.211	76

where q_e and q are the amounts of drug adsorbed (mg g^{-1}) at equilibrium and at time t (min), respectively, and k_1 is the Lagergren rate constant of first-order adsorption (min^{-1}). Values of q_e and k_1 were calculated from the plots of log ($q_e - q_t$) vs. time. Pseudo-first-order rate constants for different pH are as shown in Table 3.

6. Conclusion

The purpose of this study was to assess the performance of the low-cost adsorbent that is coconut coir. Conversion of coconut coir into ADC is cost effective and also efficient in removal of levofloxacin drug. Characterization study was carried out that is SEM, XRD and EDS. Adsorption was found to be dependent on drug concentration, pH and adsorbent dosage. The uptake of drug increased with increase in time and equilibrium was attained at 300 min. The optimal pharmaceutical molecule concentration, pH, adsorbent dose with removal percentage was found to be 3.6 mg/l (64%), 7 (56.6%) and 0.1 g (59.83%), respectively. Compared to Freundlich isotherm, Langmuir and Temkin isotherms provided an excellent fit to equilibrium data. Kinetic studies were best presented by both pseudo-first and second order.

Acknowledgements

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