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Adsorption of Pharmaceuticals Compound Livofloxacine In Aqueous Solutions Using UPR As Surfactant

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ABSTRACT
An activated carbon- unsaturated polyester resin applied as an adsorbent for the effective exclusion of a pharmaceutical substance from wastewater Because of its high adsorption efficiency, and rapid adsorption kinetics, a activated carbon-unsaturated polyester resin applied as an adsorbent for the effective exclusion of a pharmaceutical substance from wastewater. This composite was characterized using several analytical techniques, including X-ray diffraction (XRD), scanning electron microscopy (SEM), and analysis. The efficacy of the composite adsorption was studied using a type of antibiotic drug (levofloxacinee) as the adsorbate. Adsorption parameters such as the adsorbent dosage, initial drug concentration, pH of the solution, and adsorption kinetics were investigated. The results demonstrated that the adsorption equilibrium was rapidly attained after 6 min with almost complete elimination (99.97%) of the levofloxacine. A batch system was used to determine the adsorption kinetics and isotherm of levofloxacine onto adsorbent unsaturated polyester resin, and the obtained results corresponded to pseudo-second-order-kinetic and Langmuir isotherm model, respectively. The nanocomposite exhibited an adsorption capacity of 101.01 mg g−1, with a negligible loss in efficiency. Therefore, this adsorbent could be used as an efficient platform to remove contaminants from wastewater.

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INTRODUCTION

Pharmaceuticals with analgesic, antipyretic, antibiotic, and anti-inflammatory actions are the most common class of pollutants with hazardous effects on human health among the various types of water organic pollutants [1]. These have a high consumption rate, a low water solubility, and a high mobility in water, all of which indicate that they have a major harmful influence on the ecosystem. Pharmaceuticals and their metabolites are excreted into the wastewater treatment plants in cities [2]. Some of these molecules are destroyed during the sewage treatment process, while others are removed through chemical or biological means. The majority of medications are both poisonous and carcinogenic. As a result, pharmaceuticals constitute the only form of significant pollution in the globe [3]. Pharmaceuticals are a new class of micropollutants that enter the environment through both direct and diffuse sources, such as production effluents and waste disposal [4]. According to the World Health Organization (WHO), around a quarter of the diseases that humans face today are caused by long-term exposure to environmental pollution, such as air, soil, and water contamination. Pharmaceuticals have been found in a wide range of environmental samples around the world, including wastewater treatment plant effluents, surface water, ocean, groundwater, soils, and sediments, which is linked to their bioresistant nature [5].

Water-soluble antibiotics are a major sort of organic environmental contaminant, as antibiotics were overused and abused due to a lack of early advice and regulatory measures [6]. These compounds move via aquatic systems and the food chain once released into the environment, where they persist and enrich the air, water, and soil [7]. Antibiotic use has had a significant influence on ecosystems and human health, and the problem of antibiotic removal demands immediate attention [8].

Levofloxacine (LEVO) is a newer antibiotic that belongs to the fluoroquinolones (FQs), a class of synthetic broad-spectrum antibiotics. The first and second generation quinolones are effective against gram-negative bacteria, but the third and fourth generation quinolones also exhibit activity against gram-

positive bacteria. In 2003, the second-generation quinolone ciprofloxacin was the most commonly given quinolone in Europe. Levofloxacine and moxifloxacin, both 3rd generation quinolones, are currently the most commonly prescribed antibiotics [9].

The capacity of germs to become resistant to levofloxacine can directly increase the rate of human death. It increases the risk of health problems, lowers the effectiveness of treatment, and raises healthcare expenses. Levofloxacine can potentially kill aquatic species if there is an excessive amount of the antibiotic in the water. During the growth phases of a fish embryo, levofloxacine can cause death within 24 hours. When these aquatic species become part of the food chain, hazardous compounds can be biomagnified and bioaccumulated. Levofloxacine pollution in the aquatic environment can prevent algal growth. According to research, this class can also be found in aquatic species like fish and shrimp, which can serve as a source of food for higher levels of the food chain. Furthermore, levofloxacine-containing wastewater used in agriculture could have an effect on plant physiology and growth. Drinking water laced with antibiotics has triggered a hunt for more effective water treatment procedures due to the potential for major health and environmental implications.

Adsorption, catalytic degradation, biodegradation, photocatalytic degradation, and enhanced oxidation have all been investigated in recent years to reduce antibiotic contamination in the environment [10]. The commonly used adsorption approach [11] has the advantages of ease of operation, flexibility, low energy consumption, high removal rates, low secondary pollution, and cheap adsorbent renewal cost among these methods. Antibiotics have been removed from water using a variety of absorbent materials. However, these adsorbent materials' weak adsorption efficiencies, low adsorption capacities, unacceptable recyclability, secondary contamination issues, and high costs have severely limited their practical utilization. As a result, innovative, high-efficiency, low-cost adsorbents for removing antibiotics from water are urgently needed. In this study, the adsorbent is unsaturated polyester resin (UPR). Batch adsorption was used to investigate the effects of pH, adsorbate concentration, adsorbent dosage, contact time, and temperature. Langmuir, Freundlich, Dubinin-Radushkevich, and Tempkin adsorption isotherm models provided a plausible mechanism for the ongoing adsorption process as well as thermodynamic parameters. Under the studied experimental circumstances, thermodynamic characteristics revealed that the adsorption of levofloxacine onto UPR was possible, spontaneous, and endothermic.

MATERIAL AND METHODS

Sigma donated the levofloxacine (St. Louis, Missouri). $C_{18}H_{20}FN_3O_4$ has a molecular weight of 361.37 and a melting point of 260 to 270°C (decomposition) (Figure 1). All other compounds were of analytical quality and were used exactly as they were given to us. All of the chemicals employed in the nanoparticle synthesis, such as ferrous chloride tetrahydrate ($FeCl_2 \cdot 4H_2O$), ferric chloride hexahydrate ($FeCl_3 \cdot 6H_2O$), and NaOH, were of analytical grade. All of the water used in the studies was sterile. Merck and M/s Naphtha Resins, Bangalore, India, provided the adsorbent UPR. All of the reagents utilised were analytical reagent grade, and the research was conducted using distilled water.

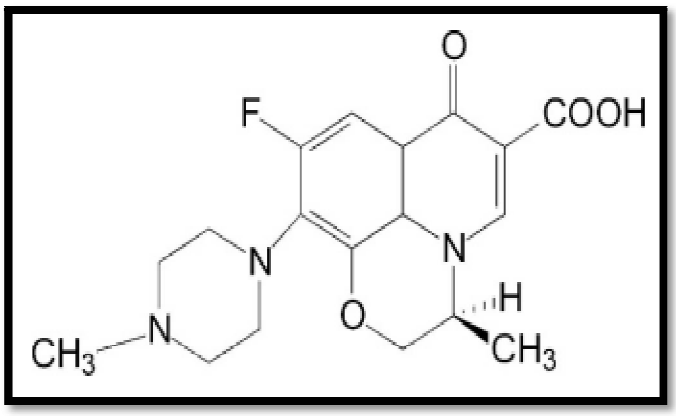

Fig.1: Chemical structure of levofloxacine

Reagents and solutions

The levofloxacine working standard solution was made by accurately weighing 100 mg of the levofloxacine reference substance, then transferring it to a 100 mL volumetric flask, dissolving it, and diluting it to volume with water to reach a concentration of 100 mg/mL. To generate multiple solutions at required concentrations from the stock solution, a dilution ratio of Na_2CO_3 (1 M) and 2 mL F-C reagent

(1:1) was utilised. Volumes of working standard solution equivalent to 0.4 mg/mL were put into a succession of 50 mL volumetric flasks. Water was used to fill each flask to the required capacity. Britton-Robinson (B-R) buffers in the pH range 2.5 to 12.0 were made by adding 0.4 M NaOH solution to a stock solution of 2.14 mL phosphoric acid, 2.3 mL acetic acid, and 2.472 g boric acid. After mixing, the solutions were agitated and left overnight to reach equilibrium.

Apparatus

A decibel DB1011 digital pH metre with a glass electrode was used to determine the pH of the test fluid. The absorbance measurements were acquired using a systronics spectrophotometer (166) spanning the wavelength range of 340 to 990 nm after it was previously calibrated using buffers of known pH in acidic and alkaline medium. During the research, a Sartorius CP224S analytical balance (Gottingen, Germany) and a Frontline FS 4 ultrasonic cleaner (Mumbai, India) were employed.

Procedure

For thermodynamic analysis, adsorption isotherms, and adsorption kinetics, adsorption experiments were conducted by altering pH, contact time, adsorbent dose, temperature, and beginning drug concentration. Levofloxacine model working solutions were used in the equilibrium experiments. Experiments were carried out at temperatures of 30, 40, and 50 degrees Celsius. The researchers utilised a variety of levofloxacine solutions with starting concentrations ranging from 0.1 to 0.6 mg/L. In a conical flask, 30 mL of model solutions were mixed with fix adsorbent mass. To establish equilibrium, the conical flask was stirred on a water bath shaker at an ideal pH and temperature. At 30°C, the mixture was stirred. The duration of interaction ranged from 5 to 30 minutes. The supernatant was filtered through Whatmann filter paper (No. 1) at a predetermined period, when equilibrium was assumed to have been reached, and the amount of medication adsorbed was measured spectrophotometrically at max 670 nm. All of the experiments were repeated two times.

The following mass balance relationship was used to compute the amount of medication adsorbed qe (mg g-1):

$$qe = (Co - Ce)\, W / V \quad (1)$$

Where Co and Ce are the initial and equilibrium concentrations (mg L-1) of drug solutionrespectively; V is the volume (mL); and W is the mass (g) of the adsorbent.

RESULTS AND DISCUSSIONS

Adsorbent characterization

As illustrated in Fig.2, the UPR was examined using a scanning electron microscope (SEM). Surface texture and porosity are visible in a SEM image of UPR. A Zeiss EVO 50 apparatus was used for SEM. Graphite monochromatic with Cu Ka radiation (k = 1.5406) was used to perform powder X-Ray diffraction (XRD) measurements on a Diffractometer system XPERT-PRO X-Ray powder diffract metre (Fig. 3). The phase characterisation was done using an X-Ray diffraction (XRD) pattern at two different temperatures ranging from 10° to 70°. The XRD pattern shows a prominent diffraction peak at 2 = 10°, indicating that the particles are crystalline.<

Effect of amount of adsorbent

Adsorption was carried out at various temperatures to optimise the adsorbent dose for the removal of levofloxacine from its aqueous solutions (30, 40 and 50°C). At constant pH and adsorbate concentration, the adsorbent dose was changed from 0.66 to 3.33 g/L. Figure 4 shows that adsorption rises with increasing the amount of adsorbent from 0.66 to 3.33 g/L at all temperatures. This is owing to the fact that raising the adsorbent dosage increases the number of accessible adsorption sites, resulting in better adsorption. However, it was discovered that as the adsorbent dose was increased, the efficiency did not improve linearly. The % adsorption remains constant after then, even while the adsorbent dosage in the adsorption system increases due to the unavailability of the adsorbate. The uptake capacity did not change considerably as the amount of adsorbent was increased.

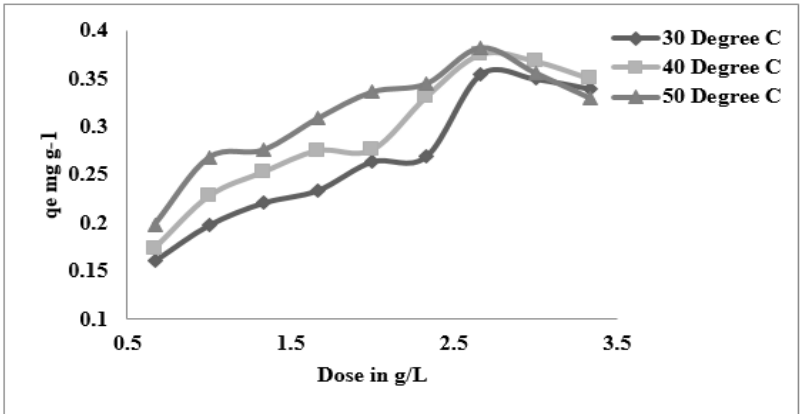

Fig.2: Effect of amount of adsorbent for the removal of rifabutin at 0.35mg/mL at pH 10.9 and different temperatures

Effect of adsorbate concentration

The rate-limiting step in the reaction is defined by the dependence of the adsorbate concentration on the rate of adsorption. The initial drug concentration and the rate of elimination have a direct relationship. Adsorption tests for UPR were conducted at pH 11.5, with concentrations ranging from 0.1 to 0.6 mg/mL and a fixed dose of adsorbent of 0.4 mg/mL. The percentage of drug removal reduces as the concentration of adsorbate increases, according to the study. The rate of adsorption falls from 82.6 percent to 61.5 percent. It was discovered that as the drug concentration is increased, drug removal reduces, despite the fact that the amount of drug adsorbed increases (Fig. 3).

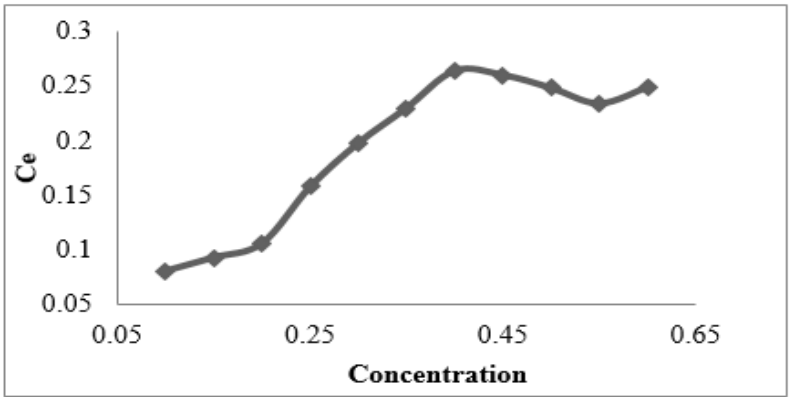

Fig. 3: Effect of concentration for the removal of rifabutin over MNA at 1.66 g/L at pH 10.9 and 30°C temperature

Effect of temperature

For the adsorption process, temperature is a crucial factor. The adsorption of medication was shown to be larger at higher temperatures than at lower temperatures. Adsorption tests for UPR were conducted out at various temperatures, including 30, 40, and 50 degrees Celsius. The rate of drug uptake increased as the temperature increases, showing that the process was endothermic in nature. The greatest adsorption for UPR was observed at 50°C, and adsorption followed the order 30, 40, 50°C, as shown in Fig. 4. The increase in temperature increases the mobility of the large drug ion while simultaneously causing a swelling effect within the adsorbent's internal structure, allowing the large drug molecule to penetrate further. As a result, adsorption capacity should be mostly determined by chemical interactions between functional groups on the adsorbent surface and the adsorbate, and should increase as temperature rises.

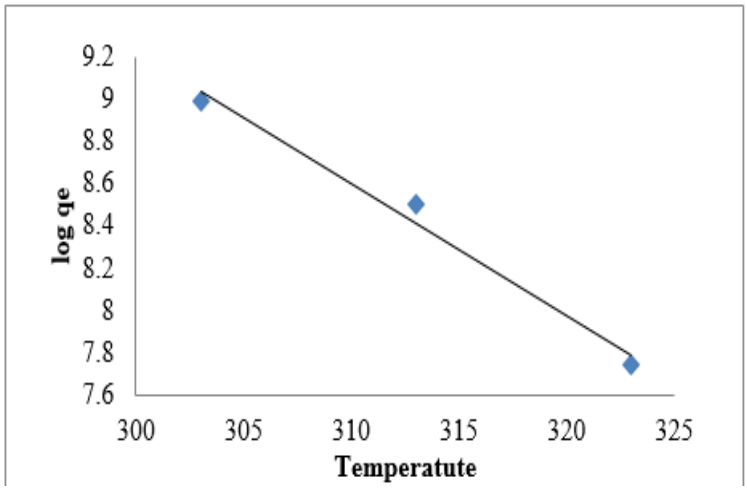

Fig. 4: Effect of temperature for the removal of rifabutin over MNA at 1.66 g/L at pH 10.9 and different temperatures

Effect of contact time

At the optimum initial concentration of drug, 0.5 mg/mL, the influence of contact time (10 to 120 min) on the amount of drug adsorbed was examined. As demonstrated in Fig. 5, the degree of rizatriptan benzoate elimination (in terms of qe) by UPR increases and reaches a maximum value as contact duration increases. Based on these findings, the equilibrium time in adsorption studies was set at 80 minutes. The adsorption of levofloxacine on UPR enhances the elimination of levofloxacine from aqueous solutions over time, until equilibrium is reached.

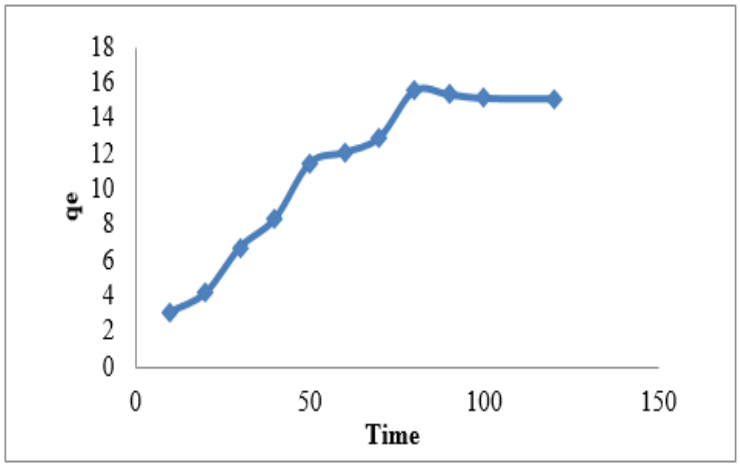

Fig.5: Effect of contact time for the removal of rifabutin over MNA at 1.66 g/L at pH 10.9 and 30°C temperature

Effect of pH

As demonstrated in Fig.6, the percent removal of the drug levofloxacine over the entire pH range from 2 to 12 demonstrates a shift in the percent removal of the drug levofloxacine over the entire pH range. This shows that the drug has a strong interaction with both adsorbents, and that either H+ or OH ions could alter the adsorption capacity. The highest uptake of medication occurs around pH 2 - 12 as seen in Figure 8. In the case of UPR, adsorption appears to increase as pH rises.

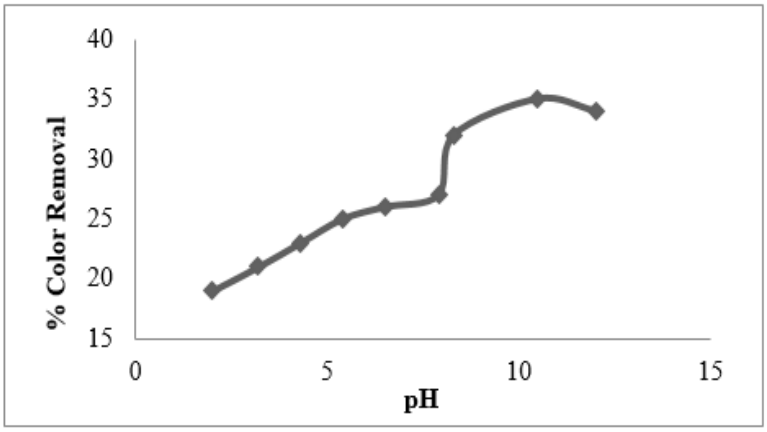

Fig.6: Effect of pH for the removal of rifabutin over MNA at 1.66 g/L at 30°C temperature and different Ph

ADSORPTION ISOTHERMS MODELING

The fraction of adsorbate molecules partitioned between the liquid and solid phases at equilibrium is described by an adsorption isotherm. Adsorption isotherms were used to predict the adsorption of levofloxacineonto UPR particles. Freundlich, Langmuir isotherms, and Tempkin analysis were used to examine the adsorption data. These isotherms can be used to calculate the total amount of adsorbent required to remove a specific amount of adsorbate from a solution.

Langmuir isotherm

The Langmuir equation is expressed as:

$1/q_e=1/Q^0 + 1/bQ^0\ Ce$ (2)

Where qe denotes the amount adsorbed (mol g-1) and Ce denotes the equilibrium concentration of the adsorbate (mol L-1). For maximal adsorption capacity and energy adsorption, the Langmuir constants are Q^0 and b, respectively. (See Table 2) The result of plotting 1/qe against 1/Ce is a straight line with slope $1/bQ^0$, demonstrating that Entacapone adsorption over silver nano particles follows the Langmuir isotherm. Another key aspect of the Langmuir isotherm may be explained in terms of a dimensionless constant separation factor RL, which revealed the Entacapone silver nano particle system's favourable nature as follows:

$RL = 1/(1 + b\ Co)$ (3)

Co (mmol L-1) is the initial Entacapone concentration, while b is the Langmuir constant. Entacapone has been reported to have RL values of 0.122 at various temperatures. RL values between 0 and 1 show good Entacapone adsorption onto adsorbent silver nano particle at the concentration measured. The Freundlich equation yielded lower correlation coefficients than the Langmuir equation (Fig. 7).

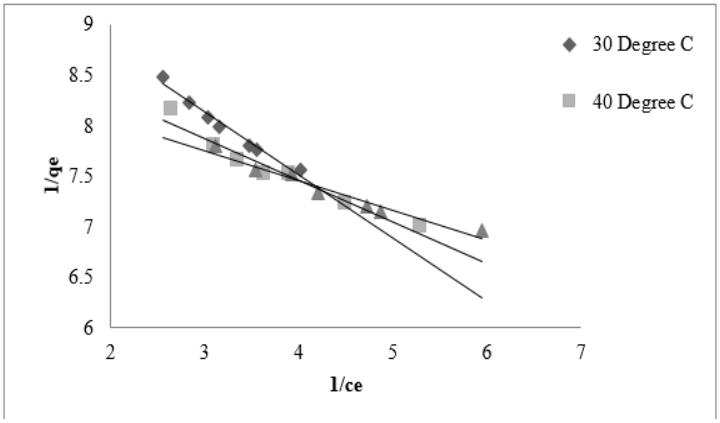

Fig. 7:Langmuir adsorption isotherms for adsorption of Levofloxacinee over UPR

Table – 1:Langmuir Constants for Levofloxacinee over UPR

Temp.(°C)	b (mol g^{-1})	Q°(L mol^{-1})	bQ°	R^2	%RSD
30 ◦ C	12.35	0.048	0.615	0.972	1.23
40 ◦ C	10.87	0.032	0.485	0.954	1.12
50 ◦ C	8.53	0.029	0.312	0.949	1.32

Freundlich isotherm

The Freundlich isotherm model, which describes the adsorption process, is one of the most well-known. This model is applicable to adsorption on heterogeneous surfaces with adsorbed molecules interacting, and the application of the Freundlich equation also shows that adsorption energy reduces exponentially as the sorption centres of an adsorbent are completed.

This isotherm is an empirical equation that can be used to represent heterogeneous systems, and it is written in linear form as follows [12]:

log qe = log KF + 1/n log Ce (4)

where, KF is the Freundlich constant related to the bonding energy. 1/n is the heterogeneity factorand n (g/L) is a measure of the deviation from linearity of adsorption. Freundlich equilibrium constants were calculated using the plot of log qe vs log Ce (Fig. 8). Adsorption is linear if n = 1, chemical if n 1, and physical if n > 1. 1.24-1.89 was revealed to be Freundlich's n value. 1st Table n between 1 and 10 indicates physical levofloxacine biosorption onto UPR. The R2 values are used to determine the goodness of fit of the experimental data to the isotherm models.

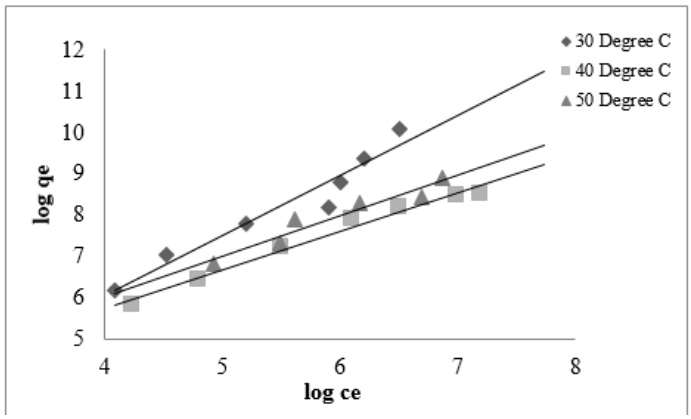

Fig. 8 Freundlich adsorption isotherms for adsorption of Levofloxacinee over UPR

Table – 2: Freundlich constants for the Levofloxacinee over UPR

Temp. (°C)	K_f	N	R^2	%RSD#
30 ◦C	21.43	5.314	0.942	1.86
40 ◦C	19.87	4.038	0.980	1.42
50 ◦C	18.72	3.163	0.931	1.38

Tempkin isotherm

Tempkin and Pyzhev investigated the heat of sorption as well as the sorbent-sorbate interactions. They claimed that as a result of these interactions, the heat of sorption of all molecules in the layer dropped linearly with coverage (Fig.11) [13] The Tempkin isotherm Equation (v) reads as follows:

Qe = (RT/bT) ln (ACe) (5)

where RT/bT = B (J/mol), where B is the Tempkin constant for heat of sorption, and A (l/g) is the equilibrium binding constant for maximal binding energy. T (K) is the absolute solution temperature, and R (8.314 J/mol K) is the universal gas constant.

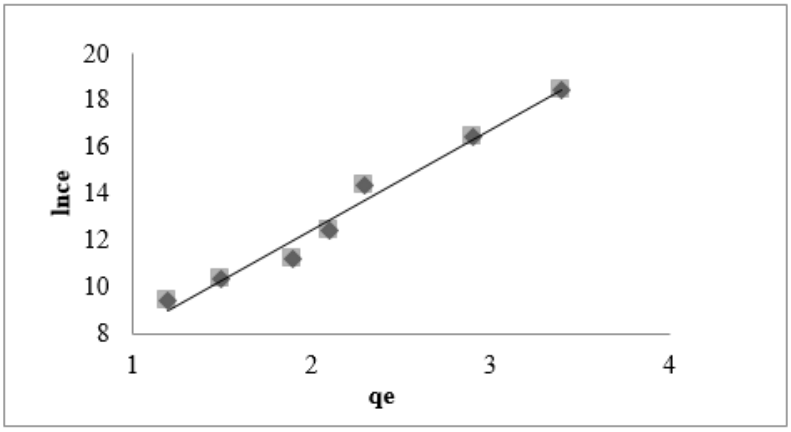

Fig. 9: Tempkin adsorption isotherm for adsorption of Levofloxacinee over UPR

Table – 3: Tempkin constants for the Levofloxacinee over UPR

Temp.(° C)	B(J mol^{-1})	A (L g^{-1})	B	R^2
30 ° C	19.236	815.89	2432	0.979

5. ADSORPTION THERMODYNAMICS

Thermodynamics assumes that entropy change is the motivating factor in an isolated system where energy cannot be generated or lost. The following equations were used to estimate thermodynamic parameters such as change in free energy (G°) (kJ mol1), enthalpy (H°) (kJ mol1), and entropy (S°) (J K1 mol1):

$\Delta G^o = -RT \ln b$ (6)

$\Delta H^o = -R (T_2T_1)/ (T_2-T_1) \ln (b_2/b_1)$ (7)

$\Delta S^o = (\Delta H^o - \Delta G^o)/T$ (8)

Where b, b1, and b2 are the equilibrium constants at various temperatures derived from the slopes of straight lines produced from Langmuir adsorption isotherms at various temperatures, R (8.314 J mol K1) is the universal gas constant, and T (K) is the absolute solution temperature. The values of the thermodynamic parameters derived using the preceding equations are shown in the table (Table 5). The value of H° was positive, indicating an endothermic sorption reaction [14].

Table 4: Thermodynamics parameters of Levofloxacinee over UPR

	Δ G°(kJmol^{-1})			ΔH° (kJ mol^{-1})	ΔS° (Jk^{-1}mol^{-1})
Adsorbent	30°C	40°C	50°C	30°C	30°C
UPR	-21.46× 10^4	-29.83× 10^4	-32.53×10^4	29.15 × 10^4	58.42

6. ADSORPTION KINETIC STUDIES

Adsorption kinetics are highly dependent on the sorbent material's physical and/or chemical properties, as well as sorbate species, which influence the sorption mechanism. Several models have been suggested to investigate the governing mechanism of sorption processes such as mass transfer and chemical reaction: pseudo first-order kinetics, pseudo second-order kinetics, Elovich, intraparticle diffusion, and Liquid film diffusion kinetics [15]. Lagergren [16] proposed the pseudo-first-order kinetic model for sorption of solid/liquid systems, which is expressed by Equation (9):

$\log (qe - qt) = \log qe - kad \times t/2.303$ (9)

where qe and qt (mg/g) represent the amounts of adsorbate adsorbed at equilibrium and at any time, t (h), respectively, and kad (1/h) represents the adsorption rate constant. where qe denotes the quantity adsorbed at equilibrium and qt denotes the amount adsorbed at any given time t. At 30, 40, and 50 degrees Celsius, the adsorption kinetics were studied with varying contact times. It's one of the key factors in determining sorption efficiency. To further understand the behaviour of this low-cost adsorbent, the kinetics of drug removal were investigated in this study. The graph of log (qe - qt) versus t shows straight lines, indicating that the adsorption process in each case follows first-order rate kinetics (Fig. 13). Table 6 shows the kad values calculated from each Lagergren plot for each system. The pseudo-

second-order kinetic model's correlation coefficients are 0.95, indicating a poor pseudo-second-order fit to the experimental data [17].

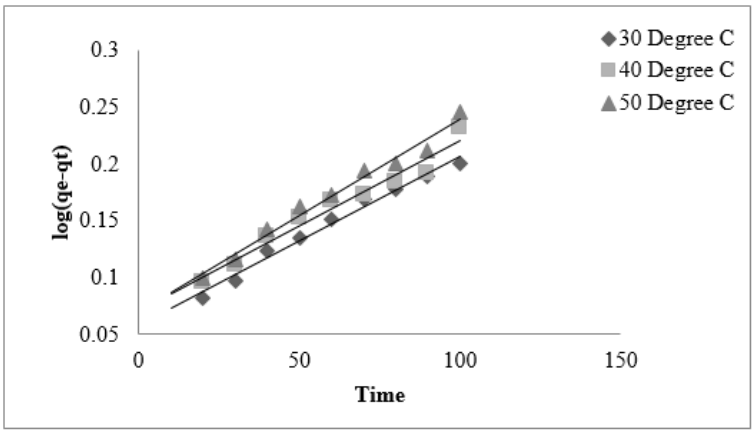

Fig.10: Lagergren pseudo first order plots for adsorption ofLevofloxacinee over UPR

Table – 5Rate constant k_{ad} for Levofloxacinee over UPR

Temp (°C)	k_{ad}	% RSD
30°C	0.413	0.984
40°C	0.527	0.964
50°C	0.702	0.981
# Average of three replicates measurement		

CONCLUSION

The current study aims to establish a versatile and dependable method for removing the antibiotic levofloxacine from wastewater. The current work has a key benefit in that UPR was utilised without any prior activation treatment, lowering adsorption costs. In light of these data, it is reasonable to conclude that the adsorbent is extremely effective at removing levofloxacine. This technology can effectively remove levofloxacine from wastewater and other effluents. The equilibrium, kinetic, and thermodynamic parameters of levofloxacine adsorption onto UPR were studied. The dynamical behaviour of levofloxacine adsorption on UPR at different temperatures accorded well with a pseudo first order kinetic model, indicating that chemisorption is the rate-limiting step. Freundlich, Langmuir, and Tempkin adsorption isotherms are also confirmed by the adsorption data.

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