ADSORPTION OF INDIGO CARMINE DYE FROM AN AQUEOUS SOLUTION BY TAMARIND SHELL POWDER: ISOTHERM, KINETIC AND THERMODYNAMIC STUDIES

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Abstract

The aqueous adsorption of Indigo carmine dye onto Tamarind shell powder was investigated. Contact time, initial dye concentration (20–200 mg/L), particle size (53, 75, 105, 125, and 152 m), pH (2.0–8.0), and temperature (283, 293, 303, 313, and 323 K) were all tested. The Langmuir, Freundlich, and Temkin models were used to analyze the equilibrium adsorption data of IC dye on Tamarind shells. The highest adsorption capacity (q_m) was discovered to be 19.7628 mg/g. Tamarind shell powder had a reasonable ability to remove the dye. The kinetics of IC dye adsorption closely followed the pseudo-first and second-order rate expressions, demonstrating that intraparticle diffusion is important in the adsorption mechanism. Isotherms have also been used to calculate thermodynamic parameters such as free energy, enthalpy, and adsorption entropy. The fact that the enthalpy change was positive (14.74329 J/mol) indicated that the adsorption was an endothermic process. The results show that Tamarind shell powder can be used as an adsorbent material for the adsorption of IC dye from aqueous solutions.

Keywords: Biosorption, Contact time, sorbent size, pH, concentration, Isotherms, kinetics, and thermodynamics.

1.0 Introduction:

Many hazardous dyes are present in effluents from industries such as dyeing, paper and pulp, textile, and so on **[1, 2]** and must be eliminated. Various dyes used in these industries are light and oxidation stable, as well as resistant to aerobic digestion. The brightest class of soluble dyes used by the textile industry is basic dyes **[3]**. Synthetic dyes are widely utilized in a variety of sectors, including textile dyeing and paper manufacturing. Every year, about 7 x 105 tons of these dyes are manufactured worldwide. During such dyeing procedures, it is believed that 10–15 percent of the dyes are lost in the dye effluent. For a variety of reasons, dyeing processes are a key environmental concern for the textile industry: Dyeing is a water-intensive process; large amounts of salt are frequently required to improve dye fixation on the
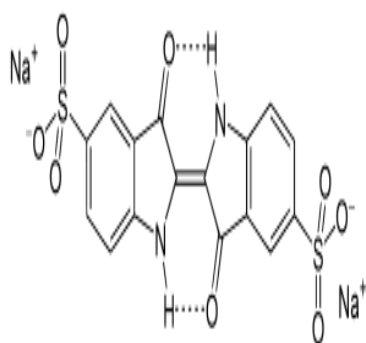
textile material; many dyes contain heavy metals as either a dye component or as a contaminant; and unfixed dye releases high doses of colour, as well as salt and metals, into mill effluent [4]. The dyes disrupt biological activity in bodies of water. They are also problematic since they have the potential to be mutagenic and carcinogenic [5]. The discharge of azo dyes into water bodies poses health and environmental hazards, a few synthetic dyes have been studied to determine their potential toxicity. The findings indicate that these dyes are hazardous to a range of species, including aquatic animals [6]. It has been observed that dye toxicity can occur as a result of either the parent compound's direct effect or its intermediate metabolites such as naphthalene, benzidine, and other aromatic amines [7, 8]. Some studies have found that wastewater created by the textile sector has a significant quantity of Total Organic Carbon (TOC), a high salt content, and pH extremes [9, 10]. Another issue related to recalcitrant character is oxidative stress, which is given by chromium in textile dyes and causes significant harm to plant growth and development, particularly photosynthesis and CO_2 absorption [11]. Whereas dyes have been known to humans since ancient times [12], it was not until the late nineteenth century that synthetic versions, along with intermediary chemicals that cause a high incidence of bladder cancer, such as benzidine and 2-naphthylamine, began to be created [13]. Textile dyes, in general, cause diseases ranging from dermatitis to central nervous system disorders [14], or they may be linked to the substitution of enzymatic cofactors, which results in the inactivation of the enzymatic activities themselves [15]. Textile dyes are acutely hazardous when consumed or inhaled, particularly when exposed to dust [16], causing skin and eye discomfort [17]. Contact dermatitis, allergic conjunctivitis, rhinitis, occupational asthma, and other allergic responses can occur in employees who manufacture or handle reactive colours [19]. The latter is the consequence of the development of a conjugate between human serum albumin and the reactive dye, which functions as an antigen, resulting in immunoglobulin E (IgE) antibodies that bind with histamine [18, 19]. Textile dye genotoxicity [20] is the largest possible long-term danger to human health [21]. Tiwari, Tripathi, and Gaur [22] allude to the presence of research done in Allium cepa root cells displaying chromosomal abnormalities, demonstrating, for example, the significant genotoxic effects of textile dyes. There is a critical need to develop treatment techniques capable of eliminating pollutants from textile waste. Various approaches are used, each with advantages and disadvantages. Their decision is based on effluent characteristics, available technology, and economic considerations. This section describes the use of physical and chemical techniques in the treatment of textile effluents for environmental safety.

2.0 EXPERIMENTAL PROCEDURE

The current study is a batch experiment on the biosorption of IC dye from aqueous solutions onto tamarind shells waste powder biosorbent.

2.1 Structure and properties:

Indigo carmine, also known as 5,5′-indigodisulfonic acid sodium salt, is an organic salt derived from indigo by aromatic sulfonation, which makes the chemical water-soluble. It is safe to use as a food colouring. Its E number is E132. It also serves as a pH indicator. Indigo carmine solutions are commonly used in obstetric surgery to identify amniotic fluid leakage. Intravenous injection of indigo carmine is frequently used in urologic surgery to highlight sections of the urinary system.

| a) Chemical bottle | b) Chemical Structure | c) Sample of Indigo carmine |

Fig. 2.1 Indigo Carmine dye

2.2 Preparation of the biosorbents

Seetammapeta, Mandal in Srikakulam, Andhra Pradesh, India, was where the tamarind shells were gathered. The gathered Tamarind shells and groundnut shells waste was washed many times with distilled water until the dirt particles were cleaned. Following a thorough washing with distilled water, the biosorbents were sun-dried until crisp, chopped into little pieces, powdered, and sieved. Without any further pretreatments, 53, 75, 105, 125, and 152 µm size powders were utilized as biosorbents in the current investigation.

Fig. 2.2 Biosrobent

2.3 Preparation of IC stock solutions:

Indigo Carmine dye powder was used to create stock solutions. All of the necessary solutions are made with double distilled water. To make 1000 mg/L IC stock solution, dissolve 1.0 g of IC in 1.0 L of distilled water. Dilutions of this stock solution are used to create samples with varying amounts of IC. Proper dilutions are used to create 10 mg/L IC solution. In a 1000 ml volumetric flask, 10 ml of 1000 mg/L IC stock solution is taken and brought up to the mark with distilled water. Dye solutions with concentrations of 20 mg/L, 50 mg/L, 100 mg/L, 150 mg/L, and 200 mg/L were produced. The pH of the solution is adjusted by adding the appropriate quantities of 0.1 N HCl and 0.1 N NaOH.

2.4 Dye Adsorption Experiments in Batch

The amount of dye decolorized and the percentage of dye removal were calculated using the equation Dye Removal percent = $(C_o - C_e)/C_o$ 100.

Dye uptake $q_e = (C_o - C_e)$ V/W

Where q_e denotes the amount of dye decolorized at equilibrium, C_o (mg/L) is the initial IC concentration, and C_e (mg/L) is the IC equilibrium concentration. V (L) denotes the volume of the IC solution, and W (g) denotes the weight of the powder.

2.5 EquilibriumStudies

This was done in a batch process by adding a pre-weighed amount of Tamarind shell powder and Groundnut shell powder to a known volume of an aqueous solution for a predetermined time interval. The procedures used to assess various parameters such as agitation time, pH, and initial concentration of Indigo Carmine in aqueous solution, dosage, and temperature.

2.6 Ultraviolet-Visible Spectroscopy

Various molecules, according to this technique, hold brilliant or clear light. When the light of explicit repeat is passed through the models, the degree of transport light radiation is concluded. As part of the recurrence, this spectrophotometer examination records the force of ingestion (A) or optical thickness (O.D). Absorbance is related to the engaging species' way length, L, and concentration, c.

3.0 Results and Discussion

The equilibrium adsorption isotherm is critical in the design of adsorption systems. The isotherm depicts the relationship between the amount of dye adsorbed at a particular temperature, the pH, particle size, and the liquid phase of the dye concentration. One of the most important parameters required to understand the behavior of the adsorption process in the adsorption isotherm in any adsorption study. The shape of an isotherm not only provides information about the dye molecules' association with adsorption but also replicates the likely

mode for adsorbing dye molecules. The most familiar way of obtaining an adsorption isotherm is to measure the concentration of the dye solution before and after the adsorption experiments. Several attempts have been made to determine dye uptake. In a batch mode of operation, experimental data are generated to investigate the effect of various parameters on the removal of IC dye from an aqueous solution using Tamarind shell powder as a biosorbent. The effect of various parameters on the biosorption of IC dye was investigated. The current study includes several experimental runs.

3.1 The Influence of agitation time:

Figure 3.1 shows the percent biosorption of IC dye by Tamarind shell powder as a function of agitation time. The 48 percent biosorption is gradually increased during the first 40 minutes of agitation, and their plots are found, and the biosorption is more or less constant.
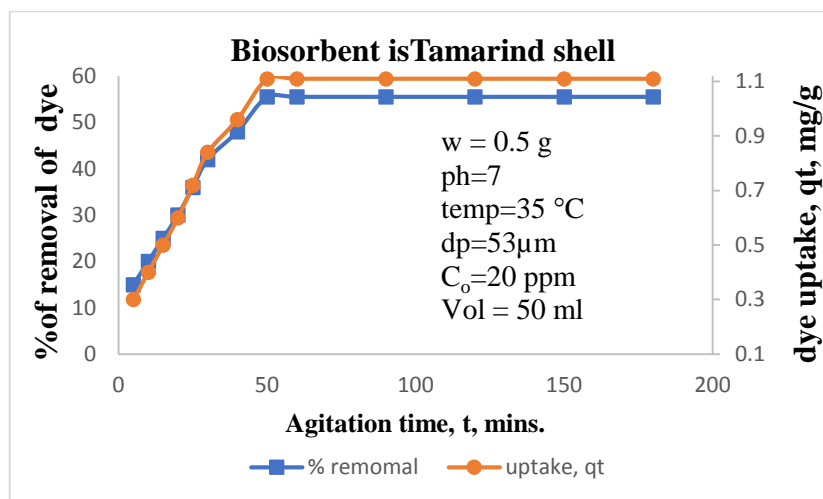

Biosorbent isTamarind shell

w = 0.5 g
ph=7
temp=35 °C
dp=53µm
C_o=20 ppm
Vol = 50 ml

(x-axis: Agitation time, t, mins.; left y-axis: %of removal of dye; right y-axis: dye uptake, qt, mg/g; legend: % remomal, uptake, qt)

Fig: 3.1 The impact of agitation time on the percentage of IC dye biosorption

As a result, the equilibrium agitation time is 50 minutes, with a 55.5 percent biosorption rate. The percent biosorption is increased from 15% to 55.5 percent during the agitation period of 5 to 180 minutes, and dye uptake is 0.28 to 1.28 mg/g. The experiment is typically made with 50 mol of aqueous solution adding 10 g/L of 53 µ size biosorption. The initial percentage rate of biosorption is higher. It is kept high because the biosorbent provides enough surface area for the biosorption of IC dye by Tamarind shell powder **[23-24].**

3.2. The Influence of the size of biosorbent:

Figure 3.2 depicts the percentage biosorption of IC dye by Tamarind shell powder as a function of biosorbent size. The percent biosorption drops from 55.5 to 31 percent as the biosorbent size grows from 53 to 152 µm, and dye uptake decreases from 1.11 to 0.0.62 mg/g as the particle size decreases. The biosorbent surface area increases as the particle size decrease **[25-26]**.
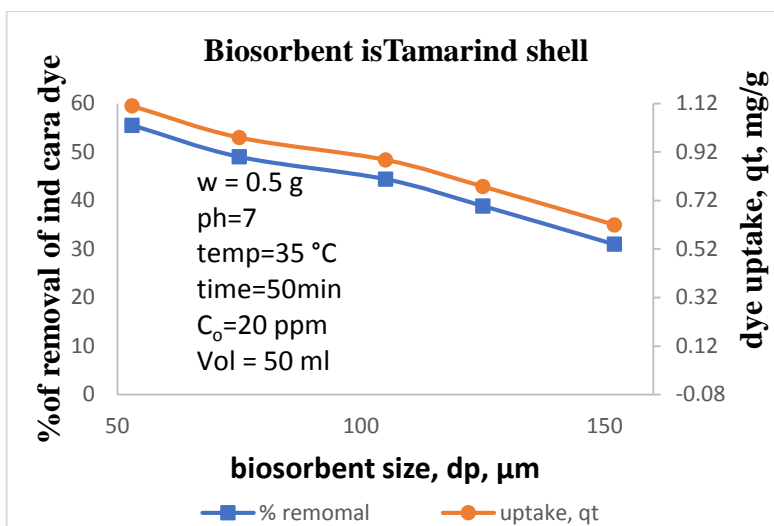
Fig: 3.2 Impact of biosorbent size on percentage IC dye biosorption

3.3 The Influence of pH in aqueous solution:

The figure in fig. 3.3 above is drawn between the pH of an aqueous solution and the biosorption of IC dye by Tamarind shells powder. Figure 3.3 shows a significant rise in percent biosorption as pH is increased from 2 to 8. As the pH climbed from 2 to 6, the percent biosorption increased from 45.5 to 55.50 percent.
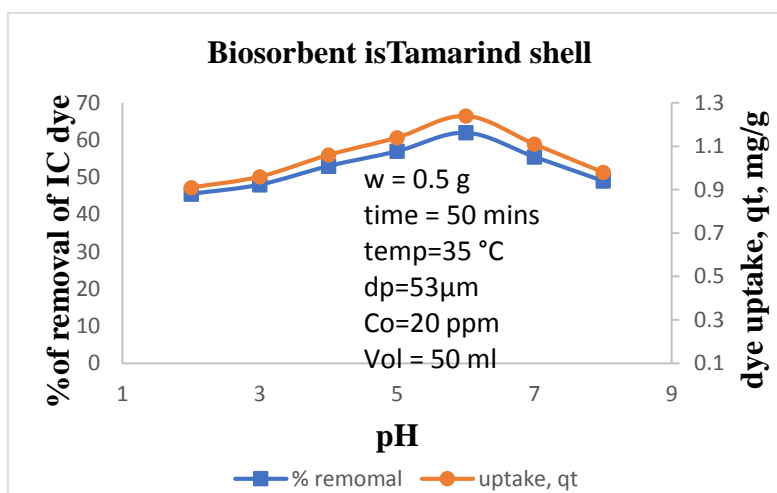

Fig: 3.3 The effect of pH on the percentage biosorption of IC dye

As the pH is increased from 6 to 8 the percent biosorption decreases. In the pH range of 2 to 6, the extent of biosorption increases from 45.5 to 62 percent. The acquired result suggests that the chemical interactions between the ions may have been swapped. Because of the role of ion exchange in biosorption **[27-28]**.

3.4. The Influence of the initial concentration of the solution, C_0 (mg/L):

Figure 3.4 depicts the influence of the initial concentration of IC dye by Tamarind shell powder in the aqueous solution on the percent biosorption at the optimum agitation time.

While raising the IC dye concentration from 20 to 200 mg/L, the percent biosorption decreases from 62 to 47 percent (1.24 to 9.4 mg/g).
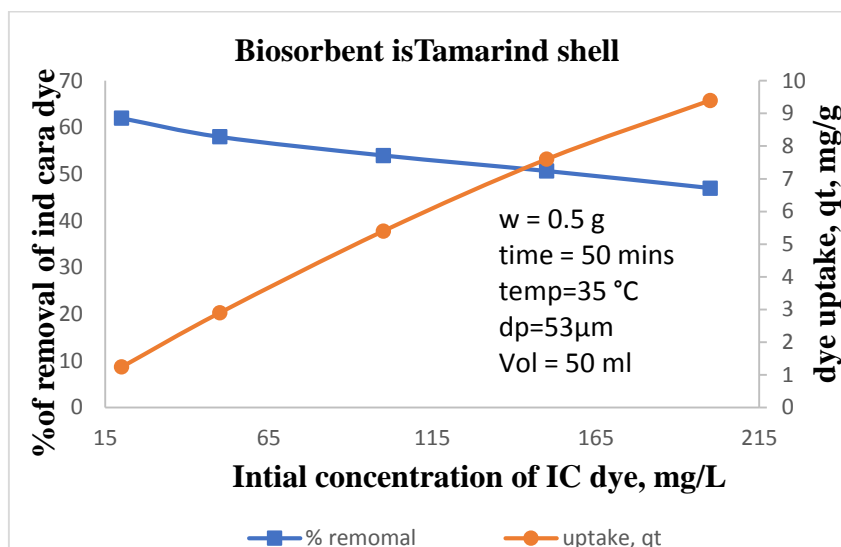

Fig: 3.4 The impact of initial concentration on percentage biosorption of IC dye

Tamarind shell powder removes a lower percentage of IC dye from larger concentrations of IC dye in aqueous solutions. The behavior can be attributed to the constant number of accessible active sites on the adsorbent and increased concentration **[29-30]**.

3.5. The Influence of dosage of biosorbent:

Figure 3.5 depicts the biosorbent dosage from aqueous solution and its fluctuation in percent IC dye biosorption by Tamarind shells powder. The dosage is increased from 10 to 60 g/L, and the percentage biosorption is increased from 62 to 89 percent, along with an increase in the biosorbent and an increase in the aqueous face.
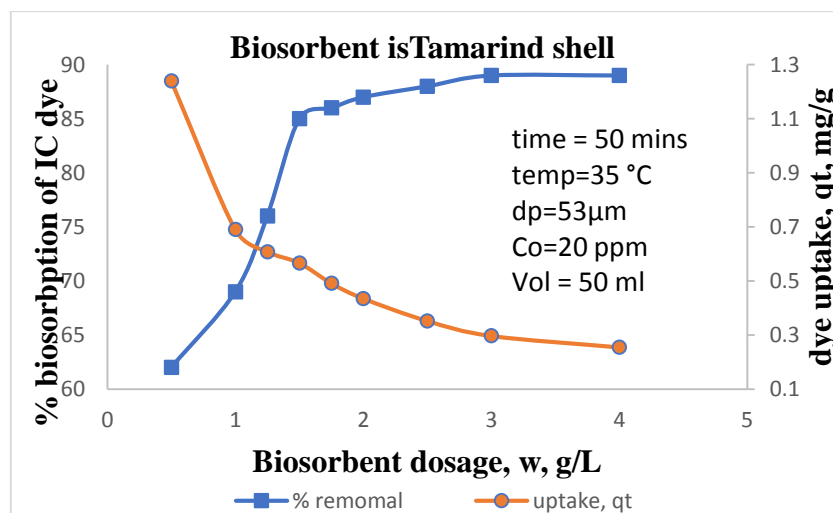

Fig: 3.5 The impact of biosorbent dosage on the percentage biosorption of IC dye

This is because the amount of biosorbent is increased, and there are more active sites available for dye uptake. The dosage is increased from 30 to 60 g/L, but the improvement in percent biosorption (88 to 89 %) is not appreciated **[31-32].**

3.6. The Influence of temperature, T, (K):

The temperature had a significant effect on the equilibrium dye uptake. Figure 3.6 depicts the effect of temperature changes on IC dye uptake. The graph shows that the adsorption capacity of Tamarind shell powder for the IC dye increased with temperature.
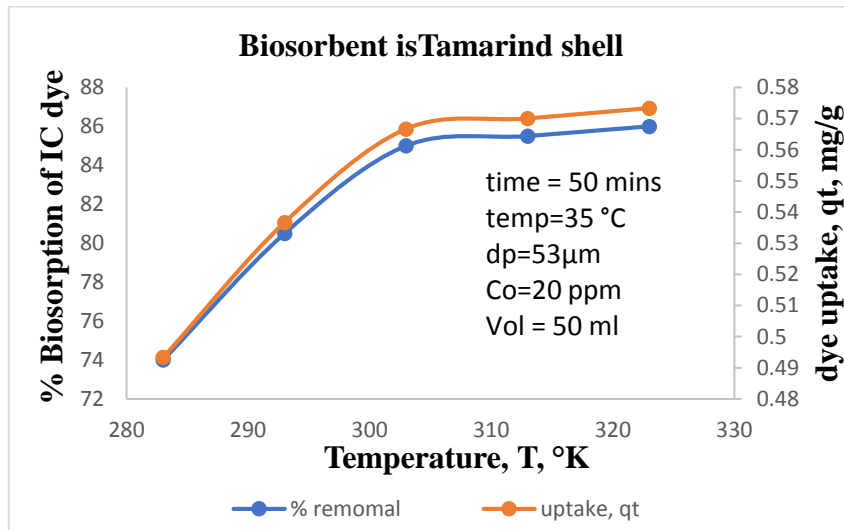

Fig: 3.6 The impact of temperature on the percentage biosorption of IC dye

Along with the temperature, a rise in the mobility of the big dye ion may result in a temperature range of 283 to 323 K. Surface active sites interact by increasing the number of molecules that may also demand energy. Furthermore, the inside structure of Tamarind shell powder allows big dyes to permeate with temperature increase, potentially causing swelling **[33-34]**.

3.7 Investigation of Isotherms:

3.7.1 Langmuir isotherm:

The Langmuir isotherm is the most common basic two-parameter equation. This straightforward isotherm is based on the following assumptions:

- Adsorbate is chemically adsorbed at a predefined number of locations.
- Each site can only store one type of adsorbate
- All sites are energetically comparable there are no interactions between the adsorbate species

The Langmuir equation is hyperbolic, and the equation is:

$$q_e/ q_m = K_L C_e / (1+K_L C_e) \qquad \text{-------- (3.1)}$$

The preceding equation can be rearranged as follows:

$(C_e/q_e) = 1/(K_Lq_m) + C_e/q_m$ ‑‑‑‑‑‑‑‑ (3.2)

The slope $1/(K_{Lqm})$ and the intercept $(1/q_m)$ can be calculated from the plots of (C_e/q_e) and C_e. The separation factor $[R_L = 1/(1+K_LC_e)]$ can be used to further analyze the Langmuir equation.

$0 < R_L < 1$	indicates	favorable adsorption
$R_L|>|1$	indicates	unfavorable adsorption
$R_L = 1$	indicates	linear adsorption
$R_L = 0$	indicates	irrepressible adsorption

In fig. 3.7, the Langmuir isotherm is drawn between C_e/q_e and C_e for the existing data. The final equation is

$(C_e/q_e) = 0.0506 C_e + 6.0054$ ‑‑‑‑‑‑‑ (3.3)

The (correlation coefficient of 0.9914) confirms that IC dye ions are strongly bound to the surface of Tamarind shells powder.
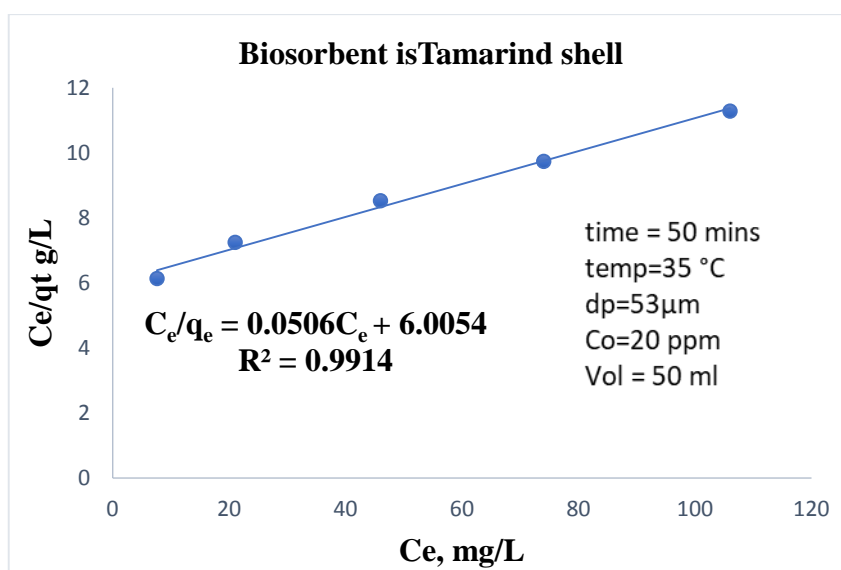

Fig: 3.7 Langmuir isotherm for biosorption

3.7.2 Freundlich isotherm:

Freundlich proposed an empirical adsorption isotherm equation that can be used for low and intermediate concentration ranges. Freundlich's isotherm is symbolised by

$q_e = K_f C_e^n$ ‑‑‑‑‑ (3.4)

Where K_f, mg/g denotes the adsorption capacity at dye equilibrium concentration and n denotes the degree of adsorption dependence.

Taking Ln on both sides, we get

$\ln q_e = \ln K_f + n \ln C_e$ ‑‑‑‑‑‑ (3.5)

In fig. 3.8, the Freundlich isotherm is drawn between $\ln C_e$ and $\ln q_e$, obtaining the following equation.

$$\ln q_e = 0.776 \ln C_e - 1.3261 \qquad \text{------ (3.6)}$$

The correlation coefficient of the equation is 0.9976. The 'n' value of 0.776 indicates that biosorption is favourable, fulfil the condition of $0 < n < 1$.
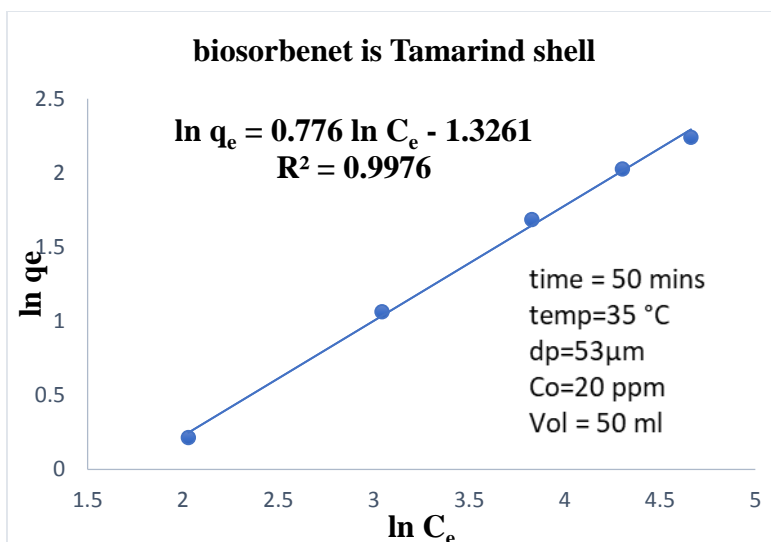

Fig. 3.8. Freundlich isotherm for biosorption

3.7.3 Temkin isotherm:

Temkin and Pyzhev isotherm equation describes the behavior of many adsorption systems on heterogeneous surfaces and is based on the equation:

$$q_e = RT \ln (A_T C_e)/b_T \qquad \text{------ (3.7)}$$

The linear form of the Temkin isotherm is

$$q_e = (RT/ b_T) \ln (A_T) + (RT/b_T) \ln (C_e) \qquad \text{------ (3.8)}$$

Where $\qquad A_T = \exp [b (0) \times b (1) / RT]$

$\qquad$ Slope, $b (1) = RT/ b_T$

$\qquad$ Intercept, $b (0) = (RT/ b_T) \ln (A_T)$

Figure 4.9 depicts a plot of q_e and $\ln C_e$. The IC dye biosorption equation is

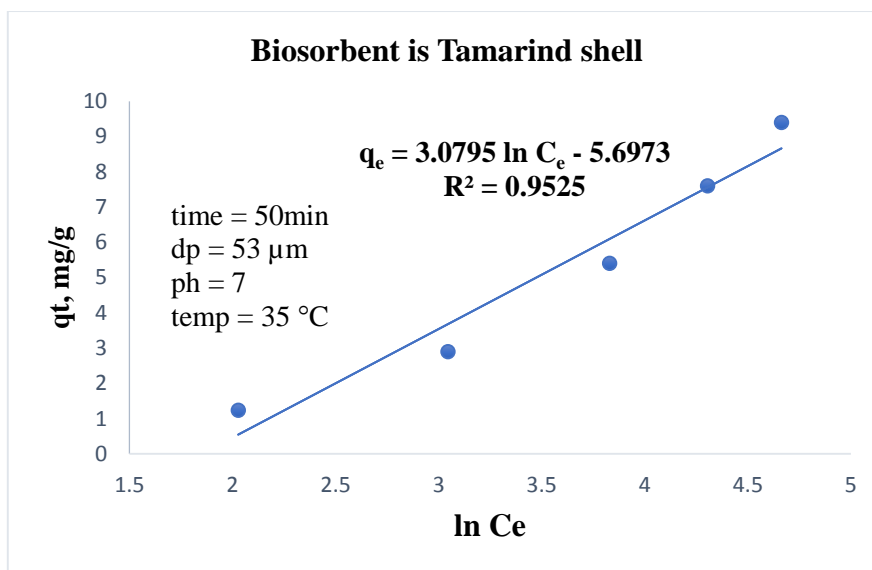$$q_e = 3.0795 \ln C_e - 5.6973 \qquad \text{------ (3.9)}$$

Fig:3.9 Temkin isotherm for % biosorption of IC dye

Table - 3.1 compiles the isotherm constants. Biosorption data are better represented by the Langmuir isotherm ($R^2 = 0.9914$) than by the Freundlich isotherms ($R^2 = 0.9976$) and Temkin ($R^2 = 0.9525$) **[35-36]**.

Table - 3.1 Isotherm constants

Langmuir isotherm	Freundlich isotherm	Temkin isotherm
q_m = 19.76285 mg/g	K_f = 0.26551 mg/g	A_T = 0.15722 L/mg
K_L = 0.008426	n = 0.76	b_T = 818.036
R^2 = 0.9914	R^2 = 0.9976	R^2 = 0.9525

3.8 Kinetics of biosorbtion

3.8.1 Lagergren First order Kinetics

Figures 3.10 and 3.11 depict a Lagergren first-order plot and a pseudo-second-order kinetics plot for IC dye biosorption. The rate constant values for first and second-order rate equations are summarised in Table-3.2 **[37-38]**. It should be noted that first-order rate equations satisfactorily explained the biosorption interactions with a higher correlation coefficient.

$log(q_e-q_t)$ = - 0.0212 t + 0.0746, R^2 = 0.9688   ---------(3.10)

Bioorbent = Tamarindshell

$$\log(q_e-q_t) = -0.0212\, t + 0.0746$$
$$R^2 = 0.9688$$

dp= 53 µm
ph=7
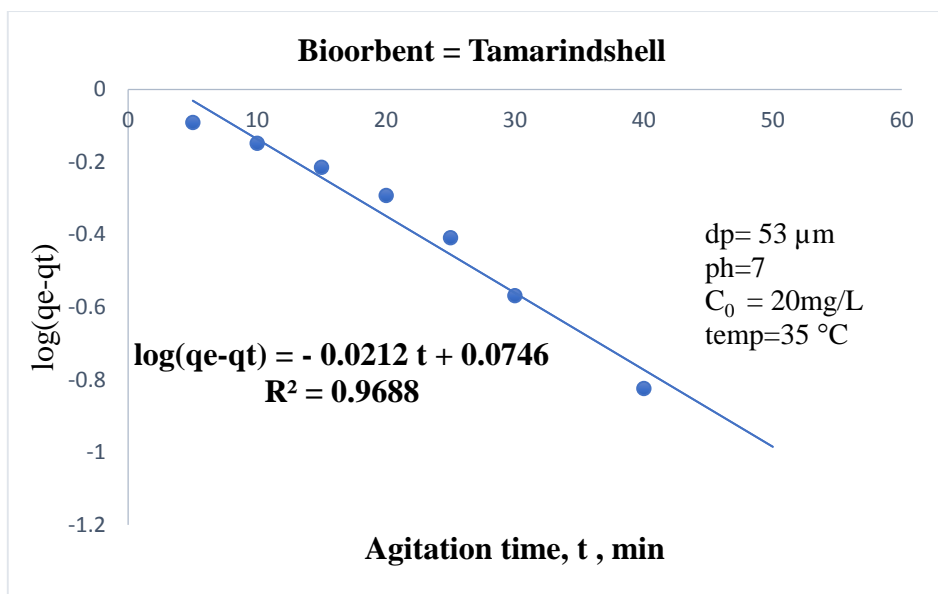C_0 = 20mg/L
temp=35 °C

Agitation time, t , min

Fig. 3.10 first-order kinetics for % biosorption of IC dye

3.8.2 Pseudo Second-order Kinetics

Figure 3.11 depicts a pseudo-second-order kinetics plot for IC dye biosorption, which is plotted between t and t/qt. The linear regression equation that was obtained is shown below.

$t/q_t = 0.6415\, t + 17.727$, $R^2 = 0.9016$ ------------ (3.11)

Biosorbent = Tamarind shells

$$t/q_t = 0.6415\, t + 17.727$$
$$R^2 = 0.9016$$

dp= 53 µm
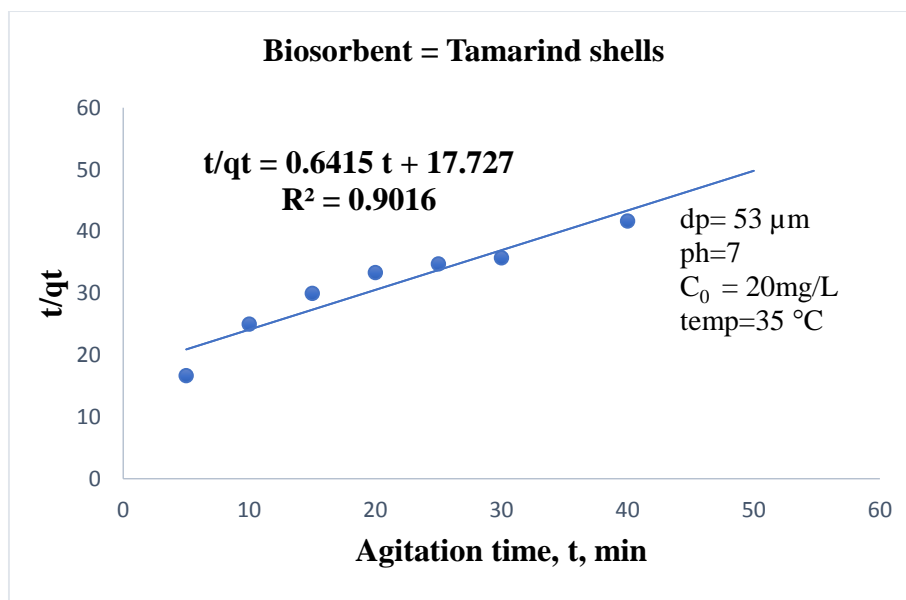ph=7
C_0 = 20mg/L
temp=35 °C

Agitation time, t, min

Fig. 3.11 Second-order kinetics for % biosorption of IC dye

Table – 3.2 Equations and rate constants

Order	Equation	Rate constant	R^2
Lagergren first order	$\log(q_e - q_t) = -0.0212\ t + 0.0746$	$0.04882\ min^{-1}$	0.9688
Pseudo second order	$t/q_t = 0.6415\ t + 17.727$	1.0774 g/ (mg-min)	0.9016

3.9 Thermodynamics :

Temperature affects decolorization. Overall, temperature dependence is linked to three thermodynamic boundaries, namely, change in enthalpy of decolorization ((H), change in entropy of decolorization ((S), and change in Gibbs free energy (G). Figure 3.12 depicts Van't Hoff's plot. Based on the data, the Gibbs free energy change (ΔG) for IC dye biosorption is calculated to be -10001.11 J/mol. The presence of a negative ΔG value indicates that biosorption is thermodynamically feasible and spontaneous. 14.7433 kJ/mol.K is the ΔH parameter. The positive ΔH indicates that biosorption is endothermic. For IC dye biosorption, the ΔS parameter is found to be 33.0556 J/mol K**.** The presence of a positive ΔS value indicates an increase in randomness at the solid interface during biosorption **[39-40]**.

Biosorbent = Tamarind shells

$$\log(q_t/Ce = -0.764/T + 1.7264$$
$$R^2 = 0.8696$$

time = 50 mins
dp= 53 µm
ph=7
C_0 = 20mg/L

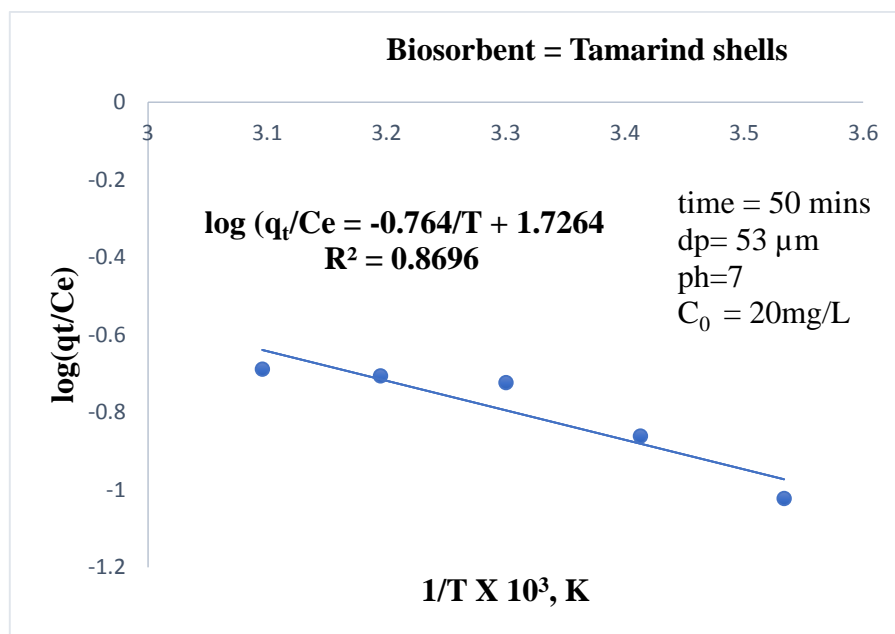y-axis: **log(qt/Ce)**

x-axis: **1/T X 10³, K**

Fig. 3.12 Vant Hoff's plot for biosorption

4. CONCLUSIONS:

The IC dye biosorption equilibrium agitation time is 50 minutes. With an increase in pH from 2 (45.5%) to 6, the percentage of IC dye biosorption from the aqueous solution increases

significantly (55.55 percent). The percent biosorption decreases from 45.5 to 62 percent as the pH rises from 6 to 8. 1.5 g or 30 g/L (0.566667 mg/g) is the best dosage for biosorption. At 303 K, the maximum uptake capacity of 19.76285 mg/g is achieved. According to the kinetic studies, the decolorization of IC dye is described by both Lagergren first order (R^2 = 0.9688) and pseudo-second-order (R^2 = 0.9016) kinetics. The experimental data is accurately represented by the Langmuir (R^2 = 0.9914), Freundlich (R^2 = 0.0.9976), and Temkin (R^2 = 0.9525) isotherms. The investigation also reveals the endothermic nature of biosorption as ΔH is positive (14.743299), irreversible nature of biosorption as ΔS is positive (33.05562), and spontaneity of biosorption as indicated by negative ΔG (ΔG = −10001.1136 J/mole).

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