

Characterization and Adsorptive Performance of Dually Synthesized *Artocarpus heterophyllus* Fibrous Waste-Assisted Fe₂O₃-MgO Nanocomposite for Acridine Orange Removal: RSM Optimization Study

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Abstract: In this study, the *Artocarpus heterophyllus* fibrous waste assisted Fe₂O₃-MgO nanocomposite (AHFW/Fe₂O₃-MgO NC) was successfully fabricated through a combined precipitation–calcination approach and characterized using XRD, SEM, TGA, FTIR, BET. The synthesized AHFW/Fe₂O₃-MgO NC was assessed for its efficiency in Acridine orange (ACO) dye remediation, with process optimization performed using Response Surface Methodology (RSM). Systematic batch adsorption experiments indicated that optimal conditions—pH 9, AHFW/Fe₂O₃-MgO dosage of 0.34 g/L, initial ACO dye concentration of 20 mg/L, and agitation time of 40 min—resulted in a maximum removal efficiency of 88.63%. Kinetic modelling indicated that the adsorption process conformed closely to the pseudo-second-order model ($R^2 = 0.992$), implying that chemisorption is the rate-limiting stage. The equilibrium results were optimally characterized by the Langmuir isotherm ($R^2 = 0.995$), indicating monolayer adsorption on a uniform surface with a maximum adsorption capacity of 227.3 mg/g. RSM analysis predicted an optimum removal efficiency, which closely aligned with experimental findings, exhibiting a deviation of < 2%, thereby validating the robustness of the model. Overall, this study demonstrates that the dually synthesized *Artocarpus Heterophyllus* Fibrous waste-assisted Fe₂O₃-MgO nanocomposites present a sustainable, cost-effective, and scalable alternative for efficient wastewater treatment applications.

Keywords: *Artocarpus heterophyllus* fibrous waste; Fe₂O₃-MgO nanocomposite; Characterization, Acridine orange; RSM optimization; Wastewater treatment, sustainable nanocomposite.

1. INTRODUCTION

The swift growth of industrial operations, especially in the textile, printing, and biomedical industries, has led to the extensive release of synthetic dyes into water bodies, presenting considerable ecological and health hazards. Acridine orange (ACO), a cationic dye, is particularly concerning because to its poisonous, mutagenic, and carcinogenic properties, as well as its resistance to biodegradation attributed to its persistent aromatic structure (Bag et al. 2021). It is estimated that 10–20% of dyes utilized in industrial processes are lost during application and discharged into wastewater, significantly contributing to water contamination (Aliyeva & Azizkhanli 2024). Even at minimal concentrations, ACO can diminish light penetration, impede photosynthesis, and provoke genotoxic consequences via DNA intercalation (Aliyeva & Azizkhanli 2024).

Among numerous treatment approaches, adsorption has proven to be an effective and cost-efficient procedure, frequently attaining removal efficiencies exceeding 85–98% using improved adsorbents (El Meziani et al. 2025). Adsorbents made from biomass have garnered significant interest owing to their affordability, renewability, and environmental sustainability. Specifically, the fibrous waste of *Artocarpus heterophyllus* (jackfruit) constitutes a plentiful agro-residue abundant in cellulose, hemicellulose, and lignin, offering functional groups such as –OH, –COOH, and –NH₂ that enhance dye adsorption. Comparable lignocellulosic biomass materials have exhibited efficient adsorption characteristics governed by hydrogen bonding, electrostatic attraction, and π – π interactions (Onyango et al. 2025). Nevertheless, raw biomass frequently exhibits restricted surface area and a diminished number of active binding sites, hence constraining its adsorption efficacy.

Recent literature highlights that metal oxide-functionalized biomass adsorbents, especially Fe-based systems, significantly enhance adsorption performance by increasing surface reactivity, porosity, and active binding sites. For instance, Fe-impregnated biochar derived from agricultural residues exhibits improved adsorption behavior due to the formation of iron oxides and enhanced surface area (Dong et al. 2024). Magnetic Fe₃O₄-modified biomass adsorbents further improve separation efficiency and reusability, making them highly effective for wastewater treatment (Santhosh et al. 2020). Studies also report that Fe-modified biochar systems outperform pristine biomass due to enhanced electrostatic interactions and surface complexation mechanisms (Wang et al. 2020).

Despite these advancements, limited studies have explored *Artocarpus heterophyllus* fibrous waste assisted Fe₂O₃-MgO nanocomposite (AHFW/Fe₂O₃-MgO NC) for ACO removal. Moreover, comprehensive studies integrating structural characterization (XRD, FTIR, BET, TGA, SEM) with adsorption mechanism analysis through kinetic, isotherm, and thermodynamic modeling are scarce. The optimization and predictive modelling are performed using RSM. Therefore, the present study focuses on developing an *Artocarpus heterophyllus* fibrous waste assisted Fe₂O₃-MgO nanocomposite as a sustainable adsorbent, contributing to lignocellulosic waste valorization and effective wastewater remediation.

2. MATERIALS AND METHODS

2.1. Materials

Artocarpus heterophyllus fibrous waste, Ferrous sulphate heptahydrate, Magnesium dinitrate hexahydrate, NaOH pellets, Acridine Orange dye, and deionized (DI) water, ethanol.

2.2. Preparation of *artocarpus heterophyllus* fibrous waste extract

The gathered *artocarpus heterophyllus* fibrous waste were meticulously cleansed with deionized water to eliminate residual contaminants, thereafter chopped into smaller segments, and sun-dried for four days. The pre-dried material underwent additional oven drying at 60 °C for 24 hours to guarantee total moisture elimination. The desiccated fibrous waste was further pulverized with a mechanical grinder and sieved to achieve a consistent particle size of 150 µm. Subsequently, 10 g of the powdered material was dispersed in 100 mL of distilled water and heated at 75 °C for 1 hour under continuous magnetic stirring. After cooling and filtration, the obtained *artocarpus heterophyllus* fibrous waste extract was collected and stored in airtight containers for future nanocomposite synthesis and adsorption investigations. (Sowjanya et al. 2023; Ramasamy & Miranda 2022).

2.3 Synthesis of AHFW- Fe₂O₃-MgO nanocomposite

A sustainable *Artocarpus heterophyllus* fibrous waste assisted Fe₂O₃-MgO nanocomposite (AHFW/Fe₂O₃-MgO NC) was manufactured using precipitation-calcination utilizing 50 ml of fibrous waste extract. The fibrous waste extract was transferred into a beaker and subjected to continuous magnetic stirring. 0.1 M FeSO₄·7 H₂O (iron precursor solution) was added gradually, followed by the dropwise addition of 0.05 M Mg(NO₃)₂·6H₂O (the magnesium precursor solution) while maintaining an Fe:Mg molar ratio of 2:1. The pH was modified to 10 using NaOH, resulting in the precipitation of Fe(OH)₂ and Mg(OH)₂. The mixture was heated to 70 °C for 3 hours, aged for 24 hours, facilitating strong interaction between metal hydroxides and functional groups (–OH, –COOH) of the extract matrix. The formed precipitate was separated by centrifugation at 5000–8000 rpm for 10 min, repeatedly washed with distilled water and ethanol until neutral pH was achieved, and dried overnight at 80 °C. Finally, the dried material was calcined in a muffle furnace at 450 °C for 3 h to obtain the dually synthesized

Artocarpus heterophyllus fibrous waste-assisted Fe₂O₃-MgO nanocomposite with improved porosity. (Sahua 2024; Myneni et al. 2020).

2.4. Batch studies of ACO dye adsorption

Adsorption tests were performed using changes in the agitation time (min), dye initial concentrations (mg/L), AHFW/Fe₂O₃-MgO NC dosage (g/L), and solution pH and the range of the parameters covered in this present study is shown in Table 1. The contents are shaken for proper mixing in a temperature-regulated gyratory shaker and then centrifuged for 15 minutes at a remarkable speed of 4000 revolutions per minute (rpm) to achieve precise separation. A UV spectrophotometer fitted with a 1 cm quartz cuvette was used to measure the dye concentrations by detecting the maximum absorbance of ACO dye at 494 nm. The percentage of dye elimination (DE) was estimated using equation (1).

$$\%DE = \frac{(C_o - C_e)}{C_o} \times 100 \quad \dots (1)$$

where C_o and C_e are initial and equilibrium concentrations (mg/L) respectively, and w is the mass of the adsorbent (g/L). The uptake capacity of dye (q_e) presented in mg/g was calculated by using the following mass balance equation (2):

$$q_e = \frac{(C_o - C_e)}{w} \quad \dots (2)$$

2.5. Statistical design strategy for AHFW/Fe₂O₃-MgO NC – driven ACO dye removal

A systematic statistical strategy using RSM was utilized to optimize the removal of ACO dye by AHFW/Fe₂O₃-MgO NC. Central composite design (CCD) was selected to capture curvature effects by integrating factorial, axial, and centre points within an expanded design space. Preliminary screening established feasible operational limits for the process variables. Four influential factors—agitation time, AHFW/Fe₂O₃-MgO NC dosage, solution pH, and initial ACO concentration—were evaluated at coded and actual levels. The adsorption response was modelled using a second-order polynomial generated through least-squares regression. All statistical analyses, diagnostic checks, and optimization procedures were conducted in Design-Expert version 13.0, providing a reliable framework for predicting system behaviour and identifying optimal ACO removal conditions.

Table 1: Optimization of variables range covered in the present study.

Variables	AHFW/Fe ₂ O ₃ -MgO NC adsorbent	
	Acridine orange dye	
	Min(-1)	Max(+1)
Contact time, t (min)	5	55
Adsorbent dosage, w (g/L)	0.1	0.5
Solution pH	5	11
Initial dye concentration, C_o (mg/L)	10	100
Temperature, T (K)	303	318

3. RESULTS OR RESULTS AND DISCUSSIONS

3.1 Characterization Overview

The X-ray diffraction (XRD) pattern of the produced AHFW/Fe₂O₃-MgO NC, illustrated in Fig. 1, displays a broad diffraction profile, signifying a semi crystalline structure. A prominent peak at $2\theta = 20\text{--}25^\circ$ is attributed to the (200) plane of cellulose, but the shoulder at $15\text{--}17^\circ$ is associated with the (110) plane, hence validating the lignocellulosic structure (French 2014). The lack of pronounced, strong peaks indicates a disturbance in crystalline order resulting from Fe–O and Mg–O inclusion. Peak broadening further signifies the nanoscale

dispersion of metal species. These structural modifications augment surface heterogeneity and increase active adsorption sites, hence enhancing sorptive efficacy (Sun et al., 2016).

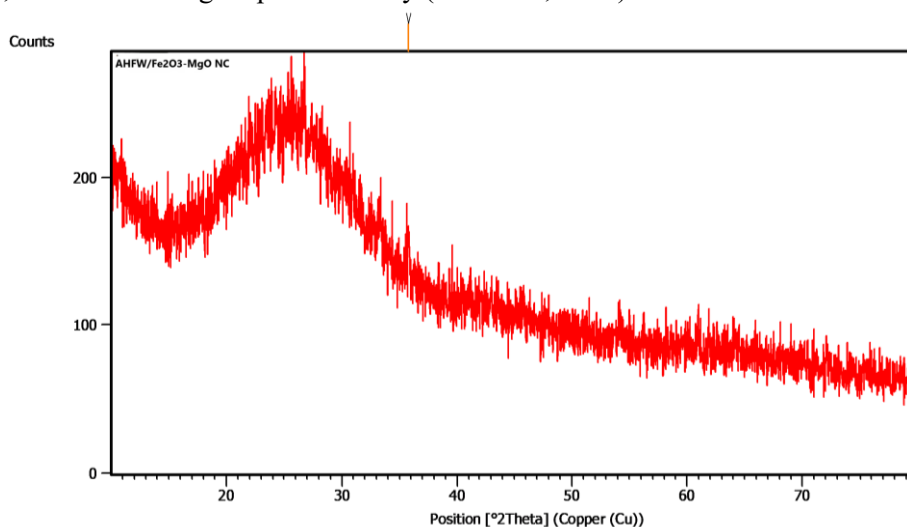


Fig. 3: XRD pattern showing broad halo ($2\theta = 20\text{--}35^\circ$) semi crystalline AHFW/Fe₂O₃-MgO structure.

Scanning electron microscopy (SEM) analysis (Fig. 2a-b) demonstrated that the produced Fe/Mg dually doped *Artocarpus heterophyllus* fibrous waste nanocomposite possesses a heterogeneous and highly porous surface shape characterized by loosely packed microclusters ranging from 21.84 to 27.86 nm in size. The micrographs reveal uneven fibrous networks adorned with uniformly dispersed metal oxide nanoparticles. The presence of Fe and Mg particles increases surface roughness and generates additional active sites, advantageous for adsorption and catalytic applications. The aggregation of nanoparticles was negligible, indicating efficient dispersion within the lignocellulosic matrix. The structural attributes align with enhanced physicochemical performance documented for analogous bio-based nanocomposites (Ramesh et al., 2021; Singh & Kumar, 2020).

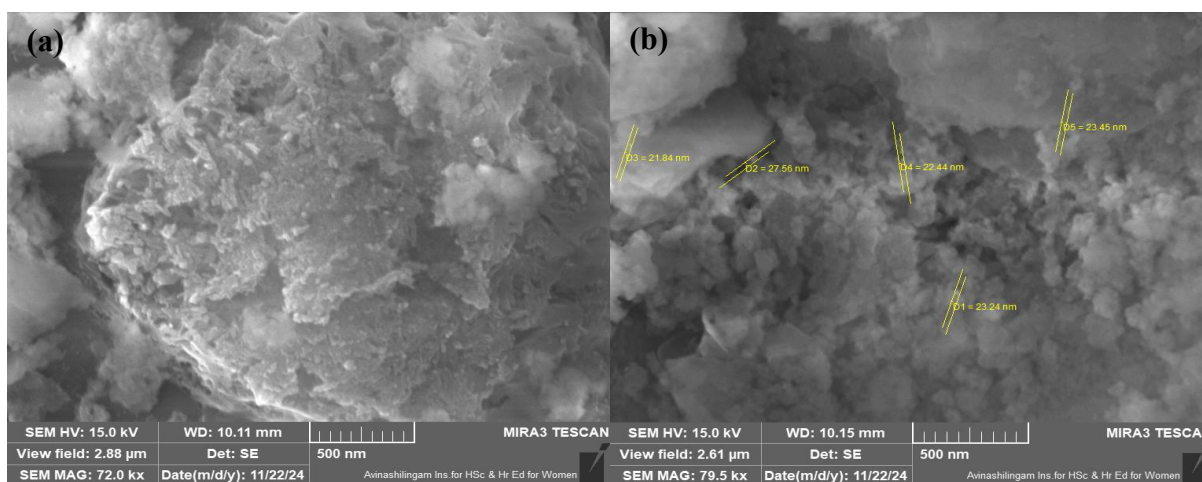


Fig. 2: a,b) SEM image showing porous, microclusters (loosely packed)

The FTIR spectra of the AHFW/Fe₂O₃-MgO NC to adsorption (Fig. 3) displayed characteristic peaks at approximately 3400 cm⁻¹ (–OH stretching), 2920 cm⁻¹ (C–H stretching), and 1030–1100 cm⁻¹ (C–O stretching), thereby affirming the lignocellulosic structure along with Fe–O (643.12 cm⁻¹) and Mg–O (957.62 cm⁻¹) bonds. Following the adsorption of acridine orange (Fig. 3), significant shifts and alterations in intensity of these bands were observed, signifying interaction between functional groups and dye molecules. Minor alterations in the –OH and C–O regions indicate that hydrogen bonding and electrostatic interactions are the primary mechanisms

at play. The spectral alterations validate the effective adsorption of acridine orange onto the modified surface (Kumar & Singh 2019).

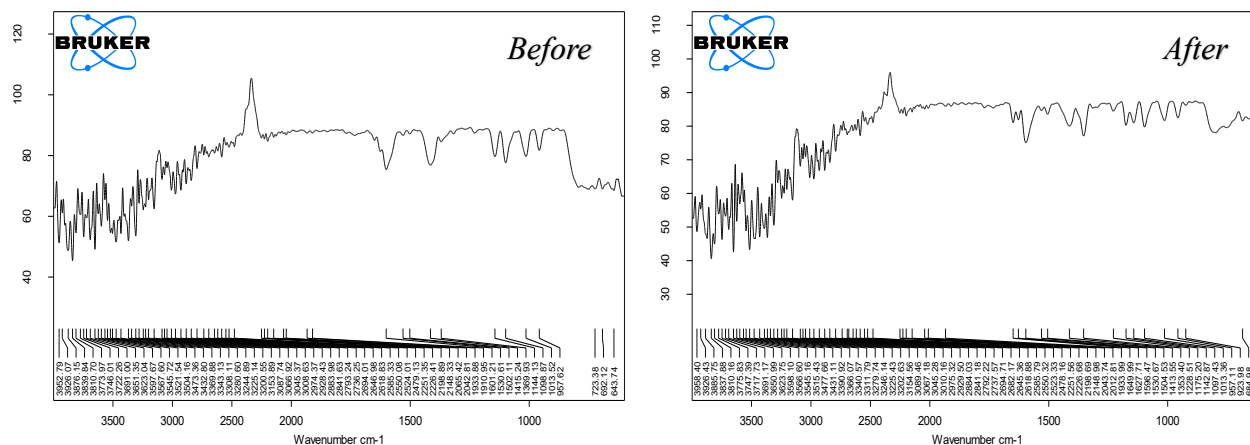


Fig. 3: FTIR spectra before and after ACO adsorption showing functional group interactions.

Thermogravimetric analysis (TGA) of the produced AHFW/Fe₂O₃–MgO NC (Fig. 4) demonstrated a multi-stage weight loss profile. The initial weight loss of about 3% below 150 °C is attributed to moisture evaporation, succeeded by a significant decomposition of 4–6% between 200–400 °C resulting from the degradation of hemicellulose and cellulose. A gradual reduction in weight at elevated temperatures signifies lignin degradation and improved thermal stability conferred by Fe₂O₃–MgO nanocomposite supported by AHFW derived bioorganic matrix. The enhanced stability facilitates its use in dye adsorption techniques, including the elimination of acridine orange (Kumar & Singh 2019).

The BET analysis of the AHFW/Fe₂O₃–MgO NC (Fig. 5) demonstrated a type IV isotherm with a pronounced hysteresis loop, signifying a mesoporous structure. The material demonstrated a BET surface area of 117.8 m²/g and a pore volume of 0.136 cm³/g post-doping, offering numerous active sites for adsorption. The BJH study verified a consistent pore size distribution within the mesoporous range. The textural features markedly augment the adsorption capacity for organic dyes, including ACO, by enhancing surface contacts (Kumar & Singh 2019).

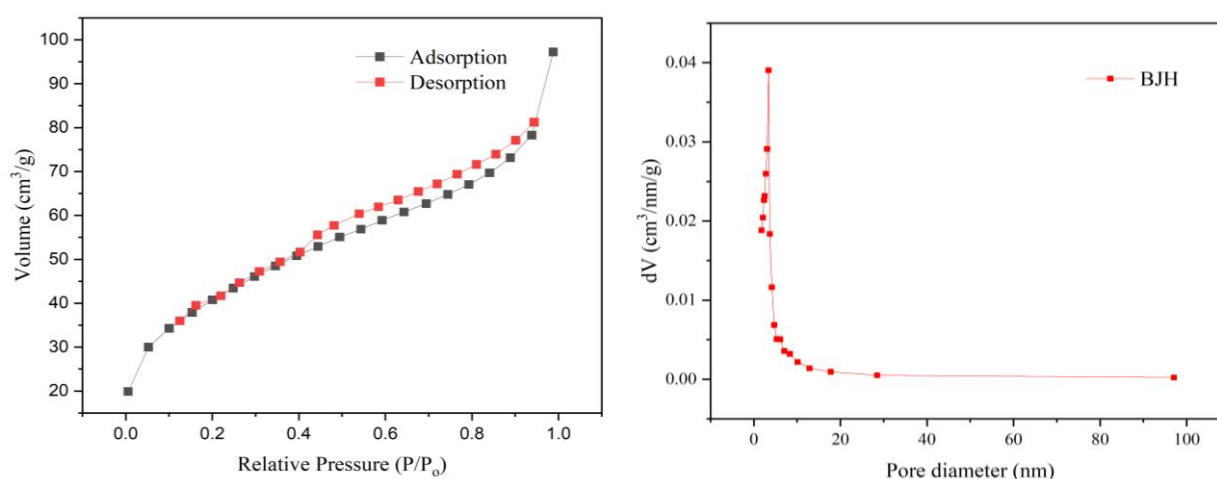


Fig. 5: BET isotherm showing mesoporosity and high surface area of AHFW/Fe₂O₃–MgO NC

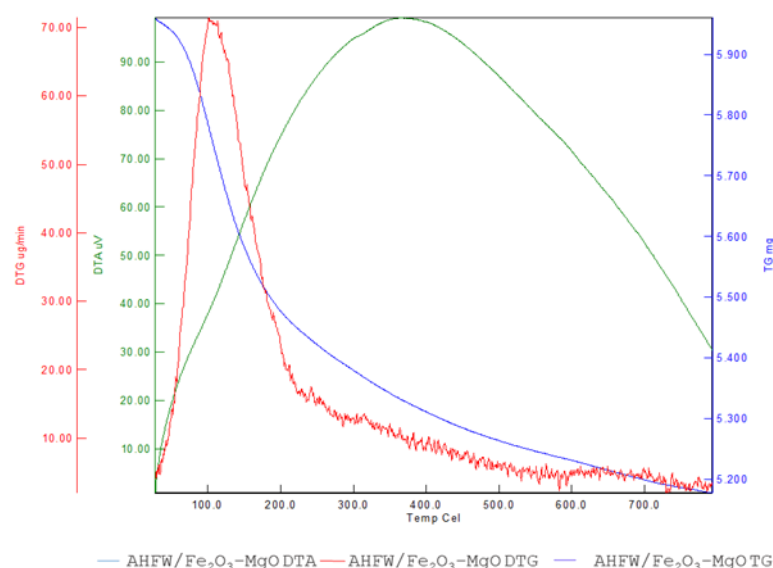


Fig. 4: TGA curve demonstrating excellent thermal stability of AHFW/Fe₂O₃–MgO NC nanocomposite

3.4 Adsorption Performance of AHFW/Fe₂O₃–MgO NC

The adsorption efficiency of acridine orange dye onto the AHFW/Fe₂O₃–MgO NC, produced using co-precipitation, was optimized utilizing the OVAT method. Critical factors such as, contact duration, AHFW/Fe₂O₃–MgO NC adsorbent quantity, pH level, initial ACO dye concentration, and temperature were methodically adjusted to identify optimal removal conditions. These methodologies are extensively employed to assess adsorption efficacy, equilibrium, and kinetics under regulated settings (Nistor *et al.* 2022; Chandrika *et al.* 2021; Rani *et al.* 2024).

3.4.1 Effect of Contact Time

The investigation of agitation time for ACO dye removal utilizing the AHFW/Fe₂O₃–MgO NC adsorbent demonstrated a concentration-dependent pattern (Fig. 6a). At a ACO dye initial concentration of 20 mg/L, the clearance rate escalated swiftly to 53.84% within 20 minutes and attained 71.27% at equilibrium after 40 minutes. Conversely, at 100 mg/L, the removal increased from 10.38% to 47.56%. Intermediate doses (40–80 mg/L) exhibited analogous trends, signifying advancing site saturation and diffusion-limited adsorption behavior (Verma *et al.*, 2021).

3.4.2 Effect of AHFW/Fe₂O₃–MgO NC Dosage

The AHFW/Fe₂O₃–MgO NC dosage on the removal of ACO shown a significant improvement in percentage removal as the dosage increased from 0.1 to 0.3 g/L (Fig. 6b), at agitation time of 40 mins. At a concentration of 20 mg/L, clearance efficiency improved from 42.56% to 78.16%, but at 100 mg/L, it climbed from 18.69% to 54.39%. Beyond 0.3 g/L, the removal reached a plateau due to aggregation and overlap of active areas, diminishing the effective surface area. This tendency indicates increased site availability, subsequently followed by saturation at elevated dosages (Verma *et al.*, 2022; Li *et al.*, 2021).

3.4.3 Effect of ACO solution pH

The pH of the ACO solution strongly regulates the surface charge of AHFW/Fe₂O₃–MgO NC and the ionization degree of ACO dye molecules (Fig. 6c), therefore affecting adsorption performance. The removal efficiency improved from 46.21% (20 mg/L) and 19.44% (100 mg/L) at pH 5 to peak values of 81.45% and 57.68% at pH 9, respectively. Above pH 9, a decrease in removal effectiveness was noted, presumably due to diminished electrostatic interaction between the adsorbent surface and dye molecules. Increased removal at

diminished concentrations indicates enhanced accessibility of active adsorption sites (Gupta et al., 2020; Tran et al., 2021).

3.4.4 Effect of Initial Dye Concentration

Fig. 6d shows the variation of ACO % removal with *initial* dye concentration by the AHFW/Fe₂O₃–MgO NC. The elimination efficiency diminished from 83.28% at 20 mg/L to 57.68% at 100 mg/L, attributable to the saturation of accessible active sites at elevated doses. In contrast, the adsorption capacity increased from 30 mg/g to 169 mg/g with elevated concentration, signifying an augmented driving force for mass transfer. This tendency underscores effective absorption at low concentrations and capacity-limited adsorption at elevated levels (Ahmed et al. 2021; Bhatnagar et al. 2020).

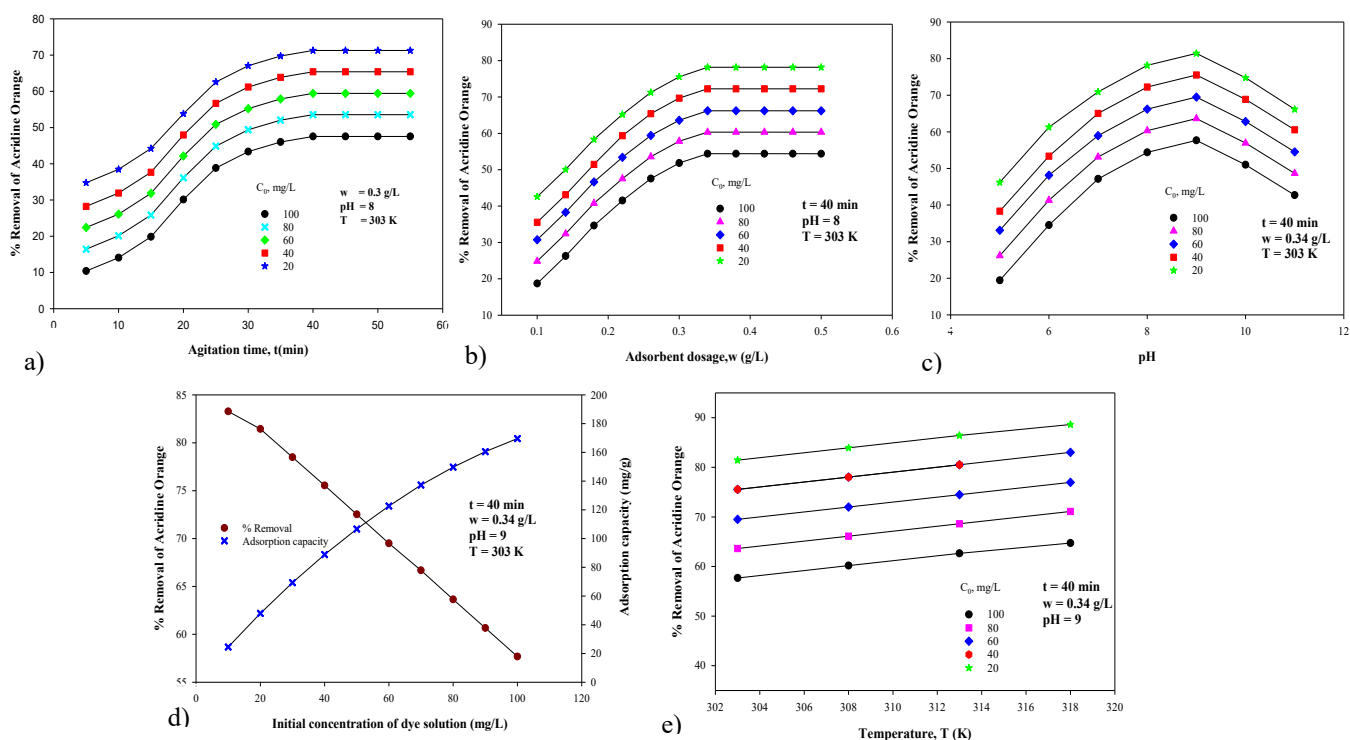


Fig. 6 (a–e): Operational variables effect on the adsorption of ACO dye by AHFW/Fe₂O₃–MgO NC: (a) contact time, (b) AHFW/Fe₂O₃–MgO NC dosage, (c) pH showing optimum removal at pH 9, (d) initial dye concentration, and (e) temperature

3.4.5 Effect of Temperature

The temperature effect on the adsorption of ACO utilizing the AHFW/Fe₂O₃–MgO NC shown a progressive enhancement in percentage removal as the temperature increased from 303 to 318 K (Fig. 6e). At a concentration of 20 mg/L, the removal efficiency enhanced from 81.45% to 88.63%, but at 100 mg/L, it rose from 57.68% to 64.74%. This improvement indicates an endothermic adsorption mechanism, wherein elevated temperatures facilitate dye mobility and contact with active sites. The trend demonstrates enhanced adsorption efficiency at all concentrations with rising temperature (Liu et al., 2021; Zhang et al., 2022).

3.5 Regression model equation development

Using CCD, the four independent variables were coded as — initial dye concentration (A), adsorbent dosage (B), solution pH (C), and temperature (D) are linked to the ACO dye adsorption on AHFW/Fe₂O₃–MgO NC from aqueous solution. The experimental error is calculated using 30 runs at the central point. The experiments are designed to produce a quadratic model with 24 trials, a star configuration ($\pm\alpha$), and 6 replicates at the centre.

Table 2: Results from CCD for ACO % removal efficiency by AHFW/Fe₂O₃–MgO NC in actual factors.

Run	A: Time (min)	B: Dosage (g/L)	C: pH	D: Concentration (mg/L)	ACO % Removal Efficiency	
					Experimental	Predicted
1	20	0.34	9	318	88.63	88.63
2	15	0.26	8	313	82.85	82.65
3	20	0.18	9	318	81.18	81.20
4	20	0.34	9	318	88.63	88.63
5	15	0.42	10	313	83.70	83.66
6	15	0.26	8	323	81.20	81.39
7	25	0.26	10	323	82.48	82.64
8	20	0.34	9	308	84.57	84.77
9	25	0.42	10	313	82.26	82.13
10	25	0.42	10	323	82.32	82.23
11	25	0.42	8	313	84.21	84.07
12	20	0.34	9	318	88.63	88.63
13	15	0.42	8	313	84.50	84.39
14	25	0.42	8	323	83.38	83.37
15	20	0.34	9	318	88.63	88.63
16	20	0.34	9	318	88.63	88.63
17	15	0.26	10	323	83.47	83.31
18	15	0.42	10	323	83.06	83.12
19	20	0.34	11	318	83.80	83.79
20	25	0.26	10	313	82.54	82.46
21	25	0.26	8	313	82.55	82.55
22	20	0.34	9	328	83.57	83.61
23	25	0.26	8	323	82.20	81.94
24	15	0.26	10	313	83.70	83.77
25	20	0.34	9	318	88.63	88.63
26	10	0.34	9	318	82.23	82.31
27	20	0.34	7	318	83.57	83.82
28	20	0.5	9	318	82.32	82.53
29	15	0.42	8	323	83.26	83.05
30	30	0.34	9	318	81.17	81.33

To evaluate the suitability of the proposed model, we conducted a rigorous characteristic test utilizing Fisher's F-test through analysis of variance (ANOVA). This robust method ensures a comprehensive assessment and enhances the credibility of our findings. The regression coefficient (R^2) shows how much of the variance around the mean can be explained by the model. Table 2 displays the experiment's design as well as its actual and predicted results. Regression analysis is used to fit the response function of ACO dye adsorption. The quadratic model is expressed by the following equation:

$$\begin{aligned} \% \text{ ACO dye removal} = & 88.63 + -0.246667 * A + 0.3325 * B + -0.00666667 * C + -0.289167 * D + -0.05625 * \\ & AB + -0.30375 * AC + 0.16125 * AD + -0.4625 * BC + -0.0225 * BD + 0.2 * CD + -1.70271 * A^2 + -1.69021 * \\ & B^2 + -1.20646 * C^2 + -1.11021 * D^2 \end{aligned} \quad \dots(3)$$

3.5.1. Model fitting and statistical validation

The quadratic polynomial regression model was developed to correlate the independent variables with ACO removal efficiency. The analysis of variance (ANOVA) demonstrated that the model was highly significant ($p < 0.0001$) shown in Table 3, with goodness of fit ($R^2 > 0.9972$), along with adjusted R^2 ($= 0.9946$) and predicted R^2 ($= 0.9840$) values in close agreement (Table 5), confirming the robustness of the model. The low value of

Table 3: ANOVA Evaluation of the Quadratic Response Model for ACO Removal.

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	178.03	14	12.72	385.59	< 0.0001
A-Initial concentration	1.46	1	1.46	44.28	< 0.0001
B-Dosage	2.65	1	2.65	80.46	< 0.0001
C-pH	0.0011	1	0.0011	0.0323	0.8597
D-Temperature	2.01	1	2.01	60.85	< 0.0001
AB	0.0506	1	0.0506	1.54	0.2344
AC	1.48	1	1.48	44.76	< 0.0001
AD	0.4160	1	0.4160	12.61	0.0029
BC	3.42	1	3.42	103.78	< 0.0001
BD	0.0081	1	0.0081	0.2456	0.6274
CD	0.6400	1	0.6400	19.41	0.0005
A ²	79.52	1	79.52	2411.28	< 0.0001
B ²	78.36	1	78.36	2376.01	< 0.0001
C ²	39.92	1	39.92	1210.58	< 0.0001
D ²	33.81	1	33.81	1025.12	< 0.0001
Residual	0.4947	15	0.0330		
Lack of Fit	0.4947	10	0.0495		
Pure Error	0.0000	5	0.0000		
Cor Total	178.52	29			
Fit Statistics					
Standard Deviation	0.1816		R²		0.9972
Mean	84.06		Adjusted R²		0.9946
C.V. %	0.2160		Predicted R²		0.9840
			Adequate Precision		57.8285

lack-of-fit ($p > 0.05$) suggested that the model adequately represented the experimental data. Model terms with P-values < 0.0500 are considered significant; accordingly, *A*, *B*, *D*, *AC*, *AD*, *BC*, *CD*, *A²*, *B²*, *C²*, and *D²* show significance. P-values > 0.1000 reflect non-significant effects.

3.5.2. Response Surface Plots and Interaction between the Parameters for ACO Adsorption

The 3D response surface analysis clarifies the accompanying effects of temperature (*D*), pH (*C*), adsorbent dosage (*B*), and initial dye concentration (*A*) on ACO removal by AHFW–Fe/Al NC. The concentration–pH interaction (Fig.-7a) indicates peak efficiency (88.63%) at 20 mg/L and pH 9, whereas deviations diminish up-take due to surface charge alteration and competitive ions. Although larger concentrations induce site saturation, the concentration–temperature profile (Fig.-7b) confirms an endothermic process by confirming better adsorption at hotter temperatures (313–318 K). A higher dosage (0.18–0.5 g/L) improves elimination through more

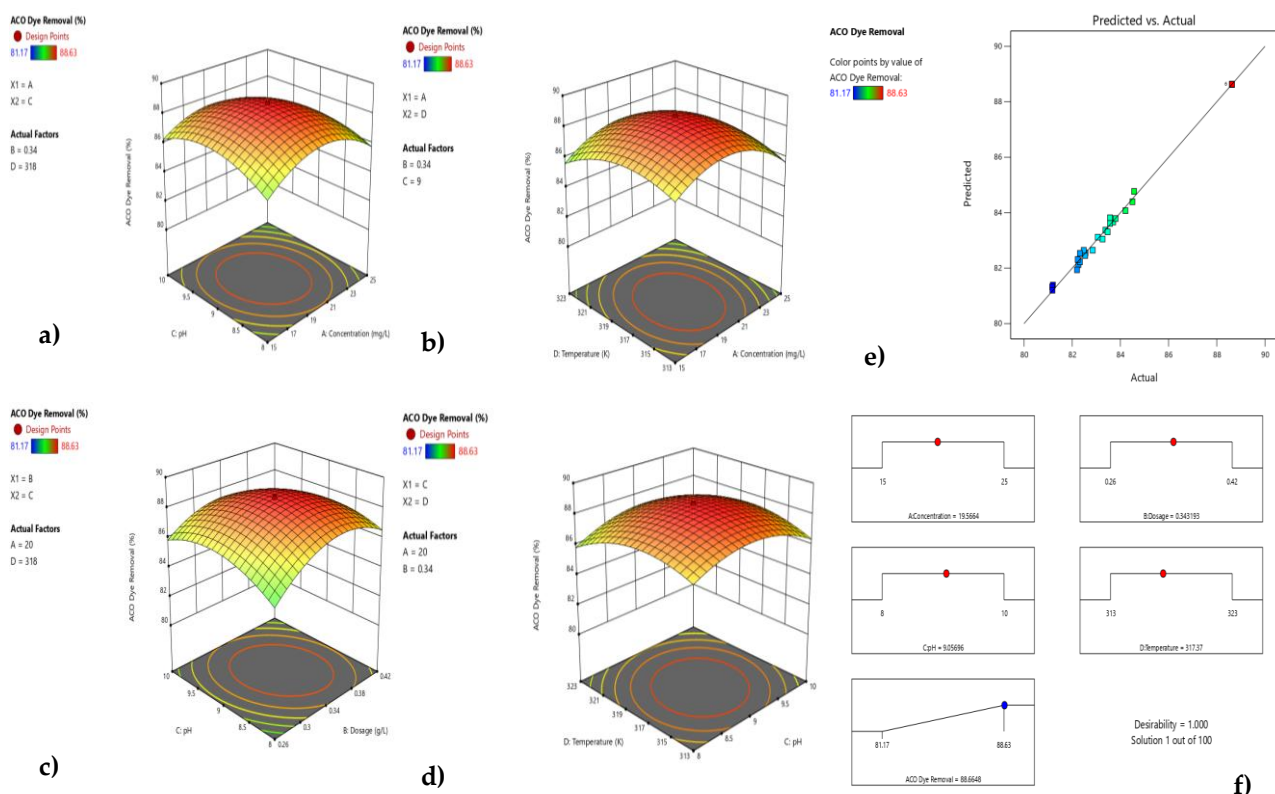


Fig. 7 (a-f): (a-d) Three-dimensional surface plots showing the combined effects of key inputs on ACO dye onto AHFW/Fe₂O₃–MgO NC; (e) AHFW/Fe₂O₃–MgO NC's Actual and predicted efficiencies; (d) Ramp plot depicting optimal factor settings with desirability for adsorption of ACO removal efficiency

active sites (Fig.-7c). About 88.73% effectiveness is produced by a synergistic pH–temperature interaction (Fig.-7d). Significant quadratic interactions and ideal operating conditions were validated by elliptical contours and dome-shaped surfaces.

3.5.3. Optimization

Fig. 7 (e-f) illustrating the agreement between experimental and model-predicted values and the optimization of process parameters for maximum removal efficiency. The design expert program selects the variables that provide the optimal results. As it is given in Fig 8b, the optimal conditions of the concentration of ACO is 19.5 mg/L, dose of AHFW/Fe₂O₃–MgO NC is 0.34 g/L, pH of the ACO dye solution is 9.05, and the temperature of 317 K, which yielded a maximum ACO adsorption of 88.70% with a desirability of 1.00, at the equilibrium agitation time of 40 min. Experimental validation under these optimized conditions showed close agreement with predicted values (Fig. 7e), confirming the reliability of the model.

3.6. Kinetic analysis

The order of acridine orange dye uptake capacity by AHFW/Fe₂O₃–MgO NC was explored using the pseudo-first order (PFS) and pseudo-second order models (PSO) (Fig. 8(a) and 8(b)). Equations (4) and (5) present the linear forms of these models, offering valuable insights into the adsorption process.

$$\ln(q_e - q_t) = -k_1 t + \ln q_e \quad \dots (4)$$

$$\frac{t}{q_t} = \frac{t}{q_e} + \frac{1}{k_2 q_e^2} \quad \dots (5)$$

where, t is the time of adsorption, k_1 and k_2 are the pseudo-first order and the pseudo-second order rate constants respectively. To probe whether internal diffusion within the nanocomposite pores contributes significantly, we employed the Weber–Morris intraparticle diffusion model (IPD). The linear form is represented by Equation (6).

$$q_t = k_{diff} t^{1/2} + C \quad \dots (6)$$

where, k_{diff} is the rate constant for intraparticle diffusion, and C serves as the intercept reflecting boundary-layer thickness. Table 4 shows the parameter estimates, among these, the PSO model provided the best correlation ($R^2 = 0.9918$), suggesting that the adsorption process is chemisorption-controlled (Ho and McKay, 2020; Liu et al., 2023; Subhashita et al. 2022). The electrostatic interactions, the surface complexation and π - π interactions between the dye molecules and carbonaceous matrix also facilitated rapid dye-adsorbent interaction, leading to a rate constant ($k_2 = 0.000344 \text{ min}^{-1} \cdot (\text{mg/g})^{-1}$) and equilibrium loading capacity ($q_e = 238.0952 \text{ mg/g}$).

Table 4: Kinetic adsorption parameters of ACO dye by AHFW/Fe₂O₃-MgO NC at 100 mg/L

Model	Parameters	estimates	R ²
Pseudo-I-order	q_e (mg/g)	235.8038	0.9721
	k_1 (min ⁻¹)	0.0922	
Pseudo-II-order	q_e (mg/g)	238.0952	0.9918
	k_2 (min ⁻¹ · (mg/g) ⁻¹)	0.000344	
IPD	C (mg/g)	36.2646	0.9025
	K_{diff} (min ^{-1/2} · (g/mg) ⁻¹)	22.5589	

The multi-stage diffusion profile indicated by the Interparticular diffusion (IPD) suggests that the adsorption proceeds via external film diffusion, followed by intraparticle diffusion into mesoporous Fe₂O₃-MgO with bioextract functional structure created during precipitation and calcination process.

3.7 Adsorption Isotherm Studies

Adsorption capacity of ACO onto AHFW/Fe₂O₃-MgO NC at equilibrium initial concentration of 100 mg/L and 318 K was evaluated by Langmuir, Freundlich (Fig.8(c-d)) and Temkin isotherms, with the Langmuir model assuming monolayer adsorption on homogeneous surfaces and is represented by:

$$C_e/q_e = C_e/q_{max} + 1/(q_{max}K_L) \quad \dots (6)$$

where q_{max} is monolayer capacity (mg/g), and K_L is Langmuir constant (L/mg). The Freundlich isotherm describes heterogeneous adsorption and is expressed as:

$$\ln q_e = \ln K_F + \left(\frac{1}{n}\right) \ln C_e \quad \dots (7)$$

where K_F is Freundlich constant and n is the heterogeneity factor. The Temkin model ($q_e = B \ln(K_T C_e)$) further supported the interaction between adsorbate and adsorbent, showing moderate linearity and implying a gradual decrease in adsorption heat with increasing surface coverage (Nguyen 2024). Table 5 shows the parameter estimates of all isotherms. The better fit of the Langmuir model ($R^2 = 0.9949$) shows that chemisorption is the main mechanism, involving strong electrostatic attraction and surface complexation between cationic Acridine Orange molecules and AHFW/Fe₂O₃-MgO active sites. The Freundlich model indicates adsorption also occurs on heterogeneous sites such as hydroxyl and carboxyl groups in the extract (Ho and McKay, 2020; Ratnam et al. 2022). The Temkin model suggests that adsorption energy decreases as surface coverage increases (Temkin and Pyzhev, 2020; Liu et al., 2023). Overall, adsorption mainly follows monolayer chemisorption with some multi-layer formation, enhanced by Fe₂O₃-MgO derived with artocarpus heterophyllum extract which increases active sites and adsorption efficiency.

Table 6: Adsorption isotherm parameters for ACO dye adsorption on AHFW/Fe₂O₃-MgO NC at 318K

Model	Parameters	values	R ²
Langmuir	q _{max} (mg/g) K _L (L/mg) R _L	227.27 0.1152 0.7927	0.9949
Freundlich	1/n K _F (mg/g. (L/mg) ^{1/n})	0.6949 31.8998	0.9785
Temkin	B (J/mol) K _T (L/mg)	55.870 1.3769	0.9896

3.8 Thermodynamic Analysis at Different Temperatures

Adsorption thermodynamic properties for the ACO removal by AHFW/Fe₂O₃-MgO NC, such as the standard change in potential, the free energy (ΔG°), the enthalpy (ΔH°), and the entropy (ΔS°) are depicted as, the distribution coefficient K_d , assuming the adsorption procedure, stated by the equations (8)-(10):

$$\Delta G_{ad}^{\circ}/RT = -\ln K_d \quad \text{.. (8)}$$

$$K_d = \frac{C_{ad,e}}{C_e} \quad \text{.. (9)}$$

$$\ln K_d = -\frac{\Delta H_{ad}^{\circ}}{RT} + \frac{\Delta S_{ad}^{\circ}}{R} \quad \text{.. (10)}$$

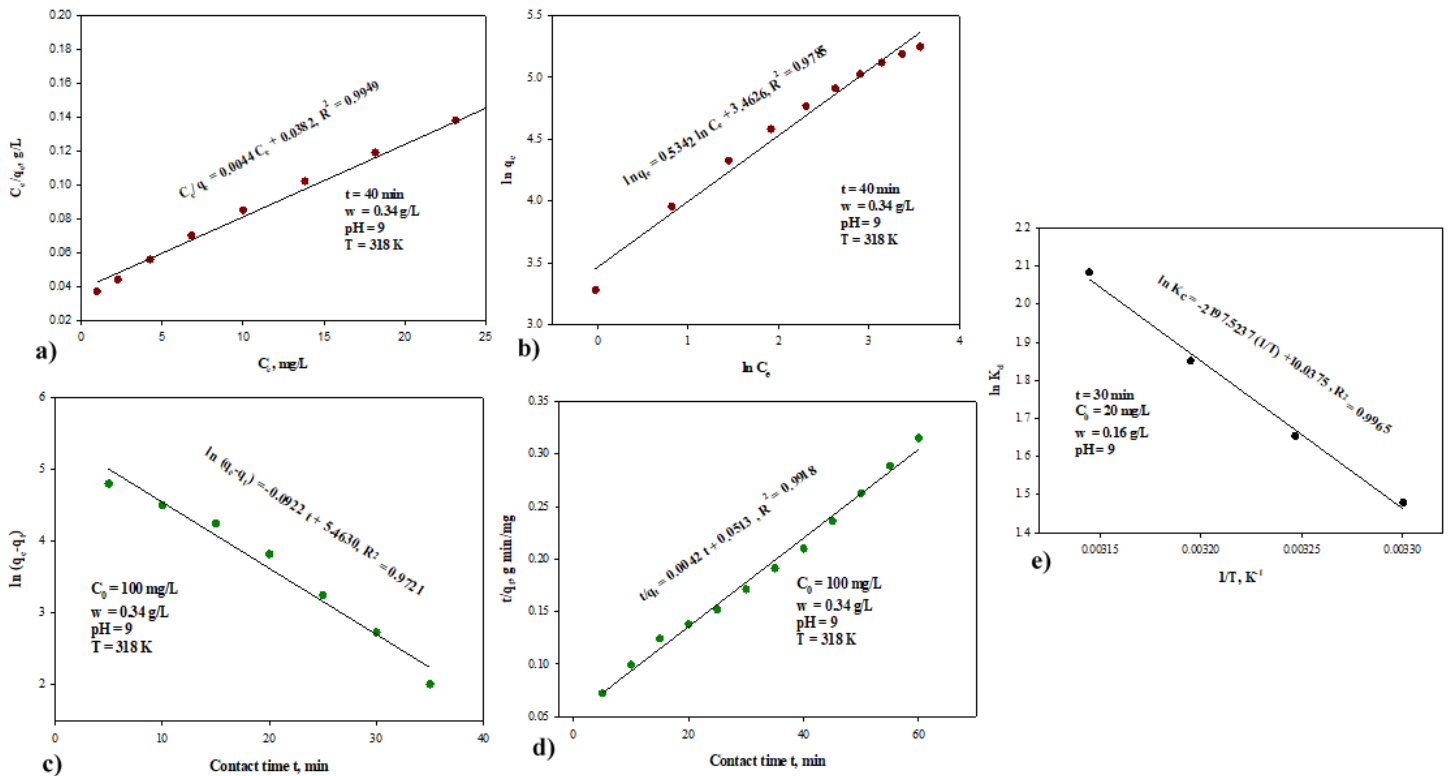


Fig. 8: (a) Langmuir Isotherm, (b) Freundlich Isotherm, (c) Pseudo-I-Order Model, (d) Pseudo-II-Order Model, and (e) Van't Hoff Plot for ACO dye adsorption on to AHFW/Fe₂O₃-MgO NC

where, $\Delta G_{ad}^{\circ} = \Delta H_{ad}^{\circ} - T\Delta S_{ad}^{\circ}$ corresponds to the standard Gibbs free energy, enthalpy, and entropy of adsorption expressed in kJ/mol and kJ/mol·K; T is the absolute temperature, R the gas constant (8.314 J/mol·K), and $C_{ad,e}$ (mg/L). Thermodynamic studies at 303, 308, 313, and 318 K provided further insight into the energetics of adsorption (Fig. 8(e)). The negative ΔG° values (-3.6760, -4.2675, -4.8590, and -5.4505) at respective temperatures confirm that the process is spontaneous and thermodynamically favorable (Ho &

McKay, 2020). The increasing magnitude of ΔG° with temperature and the positive ΔH° (32.1689 kJ/mol) indicate an endothermic adsorption mechanism, while the positive ΔS° (0.1183 kJ/mol.K) suggests increased randomness at the solid–solution interface (Sharma et al., 2022).

5. CONCLUSIONS

This study employed a green co-precipitation method to synthesis an iron oxide and magnesium oxide dually synthesized assisted Artocarpus heterophyllus fiber waste nanocomposite (AHFW/Fe₂O₃–MgO NC) for the effective removal of acridine orange (ACO). Characterization analysis utilizing SEM, XRD, FTIR, TGA, and BET demonstrated a well-defined semi-crystalline structure, heterogeneous surface morphology, exceptional thermal stability, and a significant surface area of 117.8 m²/g. the FT-IR spectra emphasized the functional groups engaged in dye interaction. Optimization of process inputs was conducted utilizing Response Surface Methodology (RSM) with a Central Composite Design (CCD), determining optimal conditions of ACO concentration at 19.5 mg/L, a dosage of 0.34 g/L, pH 9, and a temperature of 317 K, achieving an 87% removal rate under the optimized parameters. The Langmuir isotherm predicted a maximum absorption capacity of 227.3 mg/g, which closely aligned with the actual findings. Kinetic analyses indicated that adsorption adhered to a pseudo-second-order model, whilst thermodynamic evaluations verified that the process was spontaneous, endothermic, and predominantly governed by physisorption. These findings underscore AHFW/Fe₂O₃–MgO NC as an effective, sustainable, and cost-efficient material for water purification.

Author Contributions: Alpitha Suhasini Juttuka handled data preparation, assessment, experimentation and computation. Pulipati King and Meena Vangalapati contributed to research planning, research approach, project administration and mentorship.

Funding: The authors did not receive any specific funding for this work.

Acknowledgments: The authors sincerely thank the Department of Chemical Engg., AU College of Engg, for providing the necessary research facilities and academic support throughout the study.

Conflicts of Interest: No competing financial interests have been declared by the authors

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