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Application of native *Parthenium hysterophorus* for dye sequestration: Kinetic, isotherm, and thermodynamic analysis

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The present study demonstrated native *Parthenium hysterophorous* (NPH) in its raw form as an eco-friendly biosorbent for the remediation of Acid Green 25 (AG25) dye from aqueous stream. Surface characteristics of native biomass were determined using BET analysis and scanning electron microscopy. Effect of experimental parameters as pH (2-10), exposure time (15-390 min), AG25 concentration (50 – 200 mgL^{-1}), NPH dosage (1-5 g L^{-1}) and temperature (288–318 K) were studied in batch process. The adsorption of AG25 on NPH followed pseudo-second-order kinetics, and the equilibrium data of isotherm followed Langmuir model, ensuring monolayer adsorption. The highest NPH adsorption capacity recorded was 121.95 mg g^{-1}, achieved under conditions of 318 K, sorbent dosage of 3 g L^{-1}, and at a lower pH of 2. Thermodynamic data confirmed that the adsorption of AG25 on NPH is an endothermic process and occurs spontaneously. Present work highlighted the potential of NPH as an eco-friendly, inexpensive, and efficient substrate for AG25 remediation from water streams.

Keywords: Acid Green 25, Remediation, *Parthenium hysterophorous*, Thermodynamics, Isotherm

Introduction

The discharged effluent of various industries like food, leather, textile, paper, etc. contain dyes in excessive quantity, which can be lethal for living beings[1].The presence of textile dyes even in little amount in water streams is indeed a major concern, owing to their stable nature and thus offering resistance to degradative atmosphere[2]. Even a little amount of dye discharged directly into the water streams is a matter of concern as it changes colour of the water bodies and can cause damaging effects on the organisms for a longer period. According to the World Bank reports, 17-20% of the total aquatic pollution is contributed by the textile dyeing sector[3]. The accumulation of dyes and their derivatives upon destruction in aquatic organisms and sediments can also reduce BOD and COD levels of water bodies[4], impair visibility, obstruct photosynthesis and subsequently it can harm ecosystem[5].

Toxic wastewater generated by various processes negatively affects integrity of ecosystem, aquatic health, soil characteristics, and water resources[6]. Hence, it is of great need to develop eco-friendly, robust and cost efficient technology for the treatment of dye contaminated water streams[7]. Numerous techniques have been developed by the research community for the treatment of wastewater containing pollutants. However, many of these methods fall short in terms of dye removal efficiency and often generate intermediate compounds that act as secondary pollutants. Among the various developed and applied technologies, adsorption has emerged as the most effective treatment method for dye-laden wastewater due to its simplicity, high efficiency, and the absence of secondary pollutant generation[8–10]. Varieties of agricultural residues as adsorbents have been tried by different researchers across the world[11–13].

The adsorbent in the present work is synthesized from *Parthenium hysterophorous*, a solid waste. Native *Parthenium hysterophorous* (NPH) has been applied in the present work. NPH is an invasive weed, originally from the Americas, now widespread in Asia, Africa, and Australia. It thrives in disturbed areas and various climates, spreading rapidly. The plant releases chemicals that suppress nearby crops and vegetation. It triggers allergic reactions in humans, including skin and respiratory issues. It reduces agricultural productivity and degrades grazing lands. It poses a threat to biodiversity by outcompeting native species. The NPH biomass used in the present work is a solid waste with no practical use and is typically disposed of along roadsides after uprooting. Managing its spread is difficult and requires chemical, mechanical, or biological control[14]. Due to abundant availability of NPH and associated drawbacks, NPH have been chosen as a substrate in the present research for the treatment of Acid Green 25 (AG25) dye-bearing wastewater. The presented approach will serve a dual purpose of solid waste and liquid waste management.

Experimental Section

Materials

The experiments were conducted in the laboratory to investigate the adsorptive separation of AG25 using NPH. AG25 dye sample was obtained from Lotus Enterprises, India. Solutions were prepared using deionised water. Stock solution of 1000 mg L^{-1} concentration of AG25 was prepared initially, which was then diluted to get the desired concentration solutions. The adsorbent was prepared using NPH. Image of NPH is shown in Fig. 1. The biomass was washed to remove adhering dirt and then dried in sunlight for 24 h. It was subsequently crushed into a fine powder, screened, and used in its native form for the removal of dye.

Experimentation and instrumentation

Experiments were conducted in orbital shaker (Bio Technics, India) using 50 mL of AG25 solution. The effects of operational pH, NPH dose, AG25 concentration, temperature and time were investigated on AG25 removal. pH was adjusted using HCL and NaOH solutions. The speed of shaking was maintained at 150 rpm. Temperature was adjusted between 288 to 318 K for the isotherm experiments. Centrifuge **(**Remi scientific works, Mumbai) was used to separate supernatant from the adsorbent-dye solution. AG25 concentration was analyzed using UV-visible spectrophotometer at wavelength (λ_{max}) of 642 nm. Surface area analysis of NPH was performed using a NOVA Touch 4LX surface area and pore size analyzer. The surface characteristics of NPH were examined with a scanning electron microscope (SEM; Model Supra 55, Germany).

Point of zero charge

Point of zero charge (PZC) signifies the pH at which surface of the adsorbent has net charge as zero. It is necessarily important to determine PZC to adjust pH of the solution either above or below the PZC to improve rate of adsorption. pH of NaCl solution of concentration 0.01 N was adjusted between 2 to 10 in different flasks, which was then taken as starting pH. In each flask, 0.1 g of NPH was added and solutions were then placed in shaker for 24 h period. After shaking, pH was measured for each solution and recorded as ultimate pH. Graph of ultimate pH was plotted against starting pH and converge is taken as pH of PZC[15].


Fig. 1 — Image of *Parthenium hysterophorous*

Results and Discussion

Characterization

The surface structure of the NPH, imaged pre and post adsorption, is presented in Fig. 2. Prior to adsorption (Fig. 2a), the surface of the NPH appears porous, and fibrous, with a high population of binding sites, characteristics that favour the adsorption of AG25 dye molecules. These pores facilitate the diffusion of dye molecules into the lignocellulosic matrix, thereby enhancing adsorption efficiency. Post-adsorption (Fig. 2b), the SEM image reveals that the surface cavities are filled, indicating the successful adherence and penetration of AG25 dye onto and within the NPH structure. Surface area of NPH based on BET analysis was reported as 179.4 m^2 g^{-1}, with pore volume of 0.419 cm^3g^{-1} and pore diameter of 9.34 nm. The high surface area (179.4 m^2 g^{-1}) suggests a significant number of adsorption sites, which contribute to the high adsorption capacity.

Influence of pH

The pH of the AG25 was changed from 2 to 10, and the trend of removal is depicted in Fig. 3. The removal of AG25 was 95.58 ± 0.89% at a pH of 2,

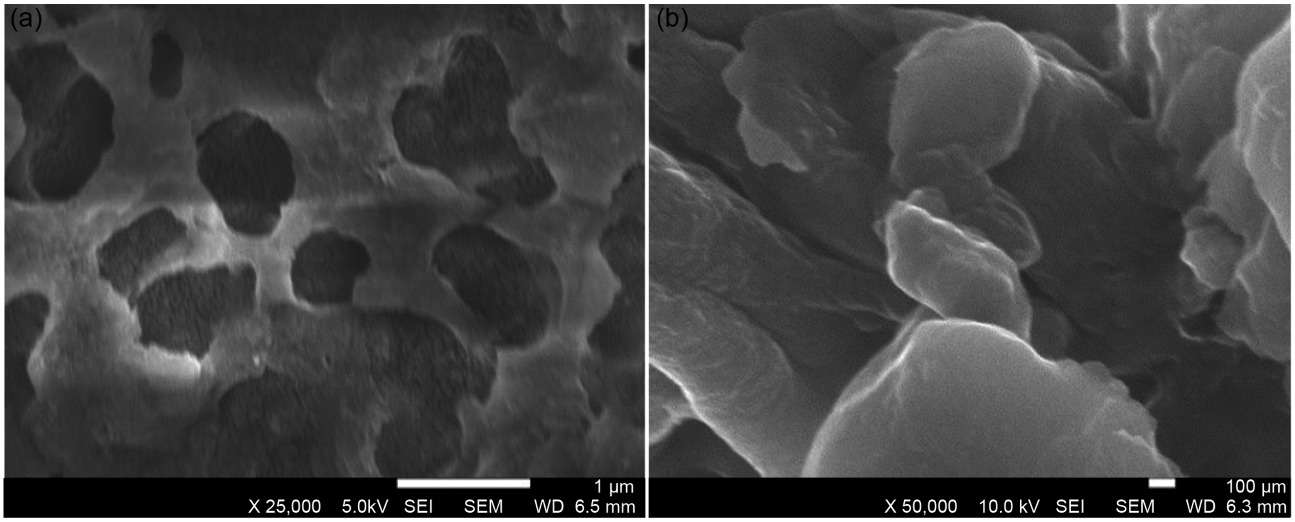

Fig. 2 — SEM images NPH (a) before and (b) after adsorption

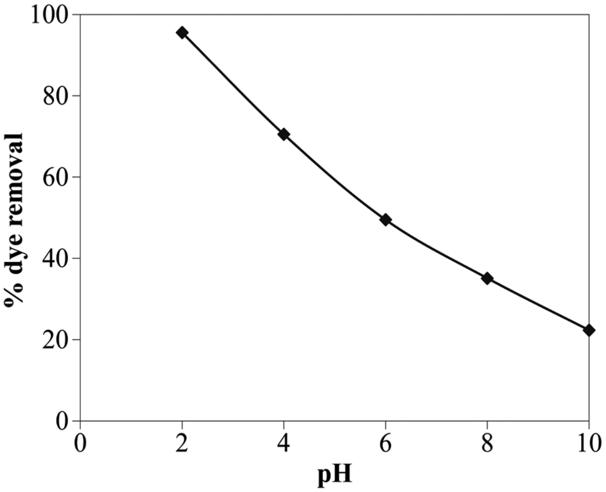

Fig. 3 — Effect of pH on the removal of AG25

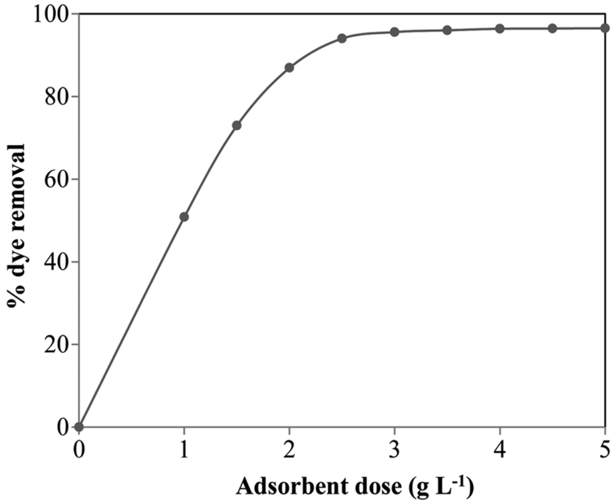

Fig. 4 — Effect of NPH dose on the removal of AG25

which decreased significantly to 22.36 ± 1.04% at a pH of 10. This trend was further supported by the pH_{pzc} value of the NPH adsorbent. The pH_{pzc} value was found to be 5.87 for the NPH adsorbent, which indicates that the adsorbent is negatively charged when the pH is above 5.87. AG25 dye is an anionic dye. Therefore, at pH values above 5.87, the NPH adsorbent acquires a negative charge, which causes it to repel the negatively charged AG25 dye, resulting in a lower removal rate at enhanced pH values. Conversely, maximum removal was observed at the lower pH value of 2, where the adsorbent remains positively charged and effectively attracts the anionic dye[16]. Similar trend of maximum removal at acidic pH values was also reported for the Acid Blue 25 removal using chitosan based adsorbent[17].

Influence of adsorbent dose

One of the essential aspects of the adsorption experiments is to optimize the dose of the adsorbent. Dose of NPH adsorbent was varied from 1 to 5 g L^{-1}, and the trend of removal is depicted in Fig. 4. AG25 removal was noted as 50.79± 0.93% at lesser dose of 1 gL^{-1}, which was significantly increased to 95.58 ± 0.89% at the dose of 3 g L^{-1}. The increase in the removal with increase in the dose is attributed to significant quantum of adsorbent available at higher dose values. Removal increased slightly from 95.58± 0.89% at the dose of 3 g L^{-1} to 96.49± 0.83% at the dose of 5 g L^{-1} which can be attributed to aggregation of sites available for adsorption[18]. Adsorption capacity was observed to decrease significantly from 50.79 mg g^{-1} at a lower dose of 1 g L^{-1} to 31.86 mg g^{-1} at a higher dose of 3 g L^{-1}. This trend of reduced adsorption capacity with increasing dose is attributed to the enhanced surface area of NPH at the lower dose, which results in more frequent collisions between adsorbent sites and dye molecules. A similar trend of increased uptake at lower doses was also reported for the removal of Reactive Red 195 using *Litchi chinensis* peels[19].

Influence of exposure time and dye concentration

The effect of the time (15-390 min) on AG25 removal was studied by varying AG25 concentration in the range of 50-200 mg L^{-1} and the obtained trend is depicted in Fig. 5. AG25 removal for 100 mg L^{-1} concentration was noted to rise significantly from 29.85 ± 0.74% at 15 min to 95.58 ± 0.89% at 210 min. However, marginal increase in the removal was noted with further enhancement in the exposure time from 210 to 390 min. The established trend of significant removal with increase in the agitation time up to certain time value and then marginal increase with further in the time is pertaining to large quantum of empty sites available for adsorption during the earlier period and subsequently saturation of sites in the later period. Established trend is also confirmed from Fig. 5 by noting the steepening of the curves in the initial period and flattening in the later periods[20].

The effect of the dye concentration on the adsorption capacity was studied by changing the AG25 concentration in the range of 50-200 mg L^{-1} and the established trend is depicted in the Fig. 6. NPH capacity of adsorption was increased significantly from 16.35 mg g^{-1} at 50 mg L^{-1} to 58.81 mg g^{-1} at 200 mg L^{-1} of AG25 concentration. Higher values of capacity at higher AG25 concentration could be due to enhanced mass transfer due to decline in resistance[21]. Similar trend of enhanced adsorption at higher concentration was reported for acidic dyes remediation using graphene oxide modified with polystyrene[22]. Removal was decreased significantly from 97.84± 0.78% for 50 mg L^{-1} to 87.34± 1.13% for 200 mg L^{-1} of AG25 concentration. The observed trend of maximum removal at lower AG25 concentration is attributed to the lower amount of AG25 present at these concentrations for a fixed adsorbent dose. At higher AG25 concentrations, competition occurs among AG25 molecules for adsorption onto the NPH adsorbent, since the adsorbent quantity remains the same at both lower and higher concentration levels.

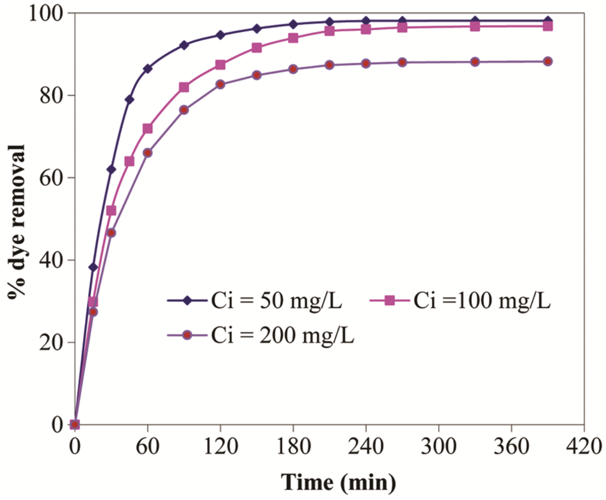

Fig. 5 — Effect of contact time and initial concentration on the removal of AG25

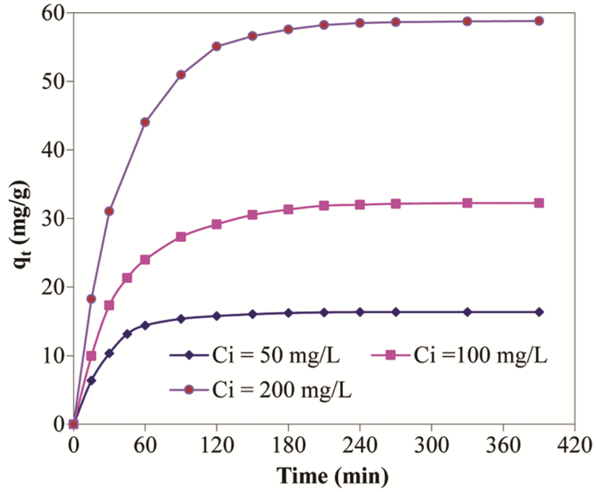

Fig. 6 — Effect of contact time and initial concentration on the adsorption capacity of NPH

Influence of temperature

The influence of temperature on AG25 removal was studied by varying the temperature in the range of 288–318 K, and the resulting trend in equilibrium adsorption capacity is depicted in Fig. 7. A significant improvement in sorption capacity was observed, increasing from 102.04 to 121.95 mg g^{-1} as the temperature increased from 288 to 318 K. This trend could be attributed to the decrease in solution viscosity and hence more solubility of dye with increasing temperature[23]. The observed trend also indicates an endothermic nature for the studied adsorption process. A similar trend was reported earlier for the removal of methylene blue using an adsorbent based on parsley stalks [24].

Kinetic and isotherm fitting

In the presented work, kinetic terms for AG25 removal using NPH are determined by subjecting the obtained experimental data to pseudo-second-order (PSO)[25] and pseudo-first-order (PFO)[26] equations as shown below:

PFO model:

$$\ln(q_e - q_t) = -k_1 t + \ln q_e \qquad \ldots (1)$$

PSO model:

$$\frac{t}{q_t} = \frac{t}{q_e} + \frac{1}{k_2 q_e^2} \qquad \ldots (2)$$

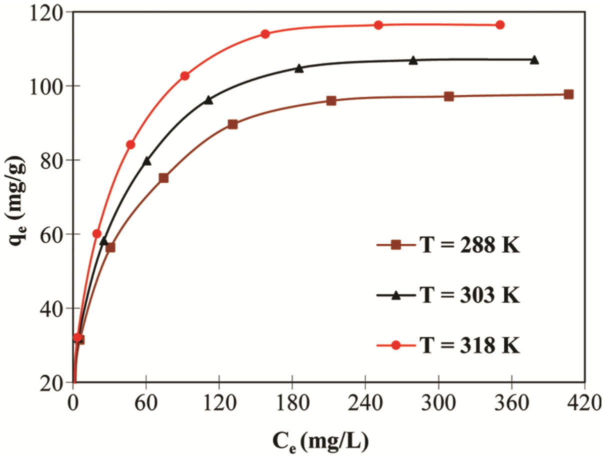

Fig. 7 — Equilibrium isotherms for removal AG25 using NPH

k_2 (g mg $^{-1}$ min^{-1}) and k_1 (min^{-1}) are PSO and PFO constants, q_t (mgg^{-1}) and q_e are sorption capacity at any time t and equilibrium, respectively.

The kinetic terms, as determined by fitting, are shown in Table 1. The values of the second-order rate constant were found to decrease significantly from 0.0045 to 0.0005 g mg^{-1} min^{-1} when the AG25 concentration enhanced from 50 to 200 mg L^{-1}. This trend could be attributed to increased competition for adsorption sites on the NPH adsorbent at higher AG25 concentrations, leading to lower rate constant values. The experimentally determined sorption capacities for all studied AG25 concentrations were found to be closer with the values obtained by fitting the PSO model, while the trend for the PFO model showed deviations. The average value of the correlation coefficient (R^2) for the PSO model was 0.9981, which is closer to 1 compared to the values obtained from fitting the PFO model. These trends confirmed that the PSO model fitted the experimental adsorption data of AG25 on NPH. A similar PSO fitting trend has also been reported in the literature for the adsorption of azo dyes onto naturally occurring adsorbents[27].

Equilibrium sorption capacity of NPH at various temperatures was determined by fitting the experimental isotherm data to various isotherms as mentioned below:

Table 1 — Kinetic parameters for AG25 removal using NPH(pH = 2, m = 3g L^{-1})

Pseudo-first-order model				
C_i (mg L^{-1})	$q_{e,exp}$ (mg g^{-1})	$q_{e,cal}$ (mg g^{-1})	k_1 (min^{-1})	R^2
50	16.35	14.90	0.0308	0.9542
100	32.26	35.78	0.0234	0.9607
200	58.81	53.83	0.0213	0.9653
Pseudo-second-order model				
C_i (mg L^{-1})	$q_{e,exp}$ (mg g^{-1})	$q_{e,cal}$ (mg g^{-1})	k_2 (g mg^{-1} min^{-1})	R^2
50	16.35	16.98	0.0045	0.9984
100	32.26	33.11	0.0013	0.9979
200	58.81	60.24	0.0005	0.9981

Table 2 — Isotherm parameters for AG25 removal using NPH (pH = 2, m = 3g L^{-1})

Isotherm	Parameter and Values			
Langmuir	T (K)	288	303	318
	q_m(mg g^{-1})	102.04	112.36	121.95
	K_L (L mg^{-1})	0.053	0.062	0.081
	R^2	0.9979	0.9974	0.9977
Freundlich	T (K)	288	303	318
	K_F (mg g^{-1}) (L mg^{-1})$^{1/n}$	16.72	18.33	19.78
	$1/n$	0.3219	0.3276	0.3366
	R^2	0.9607	0.9710	0.9593

Table 3 — Thermodynamic parameters for AG25 removal using NPH

ΔG^0 (kJ mol^{-1})			ΔH^0 (kJ mol^{-1})	ΔS^0 (kJ mol^{-1} K^{-1})
288 K	303 K	318 K		
-24.90	-26.73	-28.64	11.08	0.1247

Table 4 — Maximum adsorption capacity (q_{max}) for removal of AG25 from aqueous solution by native/modified adsorbents

Adsorbents	q_{max} (mg g^{-1})	Reference
Native *Parthenium hysterophorous* (NPH)	121.95	Present study
Walnut shells (*Juglansregia*)	21	34
Polyaniline nanotubes salt/silica composite	6.9	35
Shells of bittim	16	36
Alkali activated *Prunusdulcis*	50.79	37
Ananas comosus (L) activated carbon	182.6	38

Freundlich model[28]:

$$\ln q_e = \frac{1}{n}\ln c_e + \ln K_F \qquad \dots (3)$$

Langmuir model[29]:

$$\frac{c_e}{q_e} = \frac{c_e}{q_m} + \frac{1}{q_m K_L} \qquad \dots (4)$$

K_F (mg g^{-1}) (L mg^{-1})$^{1/n}$, K_L (L mg^{-1}) and K_T (L mg^{-1}) are Freundlich, Langmuir and Temkin constants respectively.

C_e is AG25 concentration at equilibrium (mg/L) and q_m indicates maximum capacity of AG25 adsorption (mg g^{-1}).

The isotherm terms, as determined by fitting the experimental data to various isotherms, are tabulated in Table 2. As depicted in the table, the R^2 values are found to be closer to 1 for the Langmuir isotherm in comparison to the Freundlich equation. The values of K_L are also observed to increase with temperature, confirming the favorable adsorption of AG25 on NPH at higher temperatures. A similar Langmuir fitting to the isotherm data has also been reported in the literature for rhodamine removal using biochar[30].

Thermodynamic determination

The feasibility, spontaneity, and nature of AG25 adsorption on NPH were predicted based on the determined values of thermodynamic terms, as tabulated in Table 3. As shown in Table 3, the ΔH^o value was found to be positive as 11.08 kJ mol^{-1}. Low enthalpy values as obtained in the present study typically suggest physisorption dominated by weak interactions indicating physical adsorption of AG25 on NPH[31]. The ΔG^o values were negative at all studied temperatures, confirming the feasibility and spontaneity of AG25 adsorption on NPH[32]. Positive values of ΔS^o indicated an increased disorder of AG25 on NPH[33]. The established values of ΔH^o, ΔG^o, and ΔS^o confirmed that the AG25 adsorption on NPH is endothermic, spontaneous, and feasible.

Adsorption capacities of native/modified adsorbents

Table 4 depicts adsorption capacities of native and modified adsorbents for the removal of AG25 as reported in the literature. In the present work, maximum capacity for AG25 removal using NPH is reported as 121.95 mg g^{-1}, which is comparable with other native and modified adsorbents as reported by various researchers for remediation of AG25 from water streams. The comparable and significant value of maximum uptake capacity of 121.95 mg g^{-1}at elevated temperature of 318 K in the present work proved the potential applicability of native adsorbent as applied in the present work.

Conclusion

Presented investigation successfully establishes native *Parthenium hysterophorous* (NPH) in its raw form as a eco-friendly biosorbent for the remediation of AG25 dye from aqueous stream The operating parameters including solution pH, NPH loading, exposure time, influence of starting concentration, and influence of temperature were found to significantly influence the removal efficiency. The adsorption process was noted to be endothermic, exhibiting increased favourability at higher temperatures. Kinetic analysis indicated that the adsorption followed pseudo-second-order model.

Furthermore, the equilibrium data were most accurately fitted by the Langmuir isotherm, indicating monolayer coverage yielding a maximum adsorption capacity of 121.95 mg g^{-1}. In conclusion, native *Parthenium hysterophorous* demonstrates strong potential as a sustainable and efficient biosorbent for the removal of AG25 dye from aqueous solutions.

Conflict of Interest

The authors declare that they have no conflict of interest.

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