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# Characterization of protonated amine modified lotus (*Nelumbo nucifera*) stem powder and its application in the removal of textile (Reactive Red 120) dye from liquid phase



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# ABSTRACT

The protonated amine modified-lotus stem powder (PAM-LSP) can be used to eliminate Reactive Red 120 (RR-120) from an aqueous solution as a potential adsorbent. Different operating variables like pH, agitation speed, adsorbent dosage, contact time, temperature, and initial RR-120 concentration were highly dependent on the adsorption process. FE-SEM (morphological), EDX (elemental), FTIR (functional), and N<sub>2</sub> adsorption/desorption isotherms (texture properties) have been conducted to detect the adsorptive characteristics of LSP and PAM-LSP. To analyze the equilibrium data, non-linear isothermal models of Langmuir, Temkin, D-R (Dubinin-Radushkevich), and Freundlich were used. Among the four isothermal models, as shown by lower  $\chi^2$  value (19.85), higher  $R^2$  (0.9956), the Langmuir matches the experimental data better than others. The experimental data were examined using non-linearized kinetic models of PFO (pseudo-first-order) and PSO (pseudo-second-order) and were better represented in the PSO model  $(R^2 > 0.9902)$ . It is noticeable that the adsorption of the RR-120 on PAM-LSP was a feasible, spontaneous, and endothermic physisorption process based on the calculated thermodynamic parameters like  $\Delta H^{a}$  $(10.07 \text{ kJ/mol}), \Delta S^{\circ}$  (33.8 J/mol K),  $\Delta G^{\circ}$  (-0.0539 to -1.3816 kJ/mol),  $E_{a}$  (5.9 kJ/mol), and S<sup>\*</sup> (0.0043). Desorption experiments were performed with some selected eluents and the best desorbing eluent was found between NaOH, NaHCO<sub>3</sub>, NaCl, HCl, HNO<sub>3</sub>, CH<sub>3</sub>COOH, and DIW. The PAM-LSP was used without significant adsorptive performance loss for five adsorption/desorption cycles. Electrostatic interactions between the RR-120 dye molecules and the PAM-LSP are responsible for the primary adsorption mechanism. To remove RR-120 from wastewater, the eco-friendly prepared PAM-LSP was considered a highly recyclable and efficient adsorbent.

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#### 1. Introduction

In textiles, plastics, tanneries, pharmaceuticals, carpets, paper, fruit, cosmetics, etc., synthetic dyes are widely used. Around 700,000 tons of dyes are manufactured annually, and more than 100,000 varieties of dye are available on the market. One of the most critical water contamination sources is the textile industry; it uses a significant amount of water and generates vast amounts of colored wastewater. Textile dyes are categorized as nonionic, anionic, and cationic types, which are mostly reactive dyes, acid, and direct [1]. The chemical structure of bright colored dyes possesses a chromophore group (anthraquinone, triarylmethane, azo, and phthalocyanine) and a functional group. These groups are involved in the formation of covalent bonds with the cellulosic fiber [2]. Among these dyes, the azo group and anthraquinone containing dyes comprise approximately 70% and 15%, respectively [3]. These groups are lowly fixed due to the highly water-soluble reactive dyes of electrophilic vinyl sulfones and cause highly colored wastewater formation. These days are not aerobic, but azo bonds can transform into colourless, toxic, and cancer-related

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Nomenclature

itomene								
$A_T$ $b_T$ $C_e$ $C_o$ $C_{Ae}$ $E_a$ $k_1$ $k_2$ K $K_c$ $K_f$ $K_L$ M	equilibrium binding constant (L/mg) Temkin constant (J/mol) equilibrium RR-120 concentration (mg/L) initial concentration of RR-120 (mg/L) solid phase concentration at equilibrium (mg/L) activation energy PFO rate constant (1/min) PSO rate constant (g/mg min) constant related to sorption energy (mol <sup>2</sup> /J <sup>2</sup> ) distribution constant Freundlich constant (mg/g) equilibrium constant of Langmuir model (L/mg) mass of the adsorbent (g)	qe qe,cal qe,exp qe1, qe2 qt qmax qs R S* T V ΔH <sup>o</sup> ΔS <sup>o</sup>	amount of RR-120 adsorbed (mg/g) model computed equilibrium sorption uptake (mg/g) experimental equilibrium sorption uptake (mg/g) amount of dye adsorbed at equilibrium (mg/g) adsorption uptake at time t (mg/g) adsorption uptake (mg/g) theoretical isotherm saturation uptake (mg/g) universal gas constant (8.314 kJ/mol K) sticking probability temperature in Kelvin volume of RR-120 solution (L) enthalpy change entropy change					
п	number of performed experiments ( $\gamma^2$ analysis)	$\Delta G^{o}$	Gibbs free energy change					
n	Freundlich exponent	Greek let	ters					
 1/n	heterogeneity factor	£	Polanvi potential					
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aromatic amines under anaerobic conditions [4–6]. Colored wastewater discharged without processing or insufficient processing into the receiving environment also prevents sunlight passage, thereby slowing the photosynthesis. This decreases the oxygen dissolved, which affects the ecosystem negatively [7,8]. Therefore, eliminating the color and reducing its effect on the industrial effluents before they are mixed up with natural water bodies is essential.

One of the notable and widely used azo-based dyes under frequent use in the textile industry is the RR-120 dye. Due to the azo moiety (-N=N-), this RR-120 dye has a low biodegradation rate and poses a risk to humans and the eco-system [9]. A range of biological, chemical, and physical methods was applied to remove various textile reactive dyes in recent years. Several methods like Fenton oxidation [10], catalytic ozonation [11], nanofiltration [12], coagulation [13], liquid membrane separation [14], incineration [15], photodegradation [16], ion exchange [17], and adsorption [18] have been applied to remove these pollutants prior to discharge into water. However, several drawbacks, such as high cost, complicated operation, consumption of energy and chemicals. and regeneration of toxic materials, limit the application of these methods in wastewater treatment. Due to its simple operation design, high efficiency, relatively low initial cost, reusability, nonsusceptibility to pollutants, and low waste generation, adsorption is an effective method among all the preceding methods. It has been widely reported that commercial activated carbon is one of the most efficient biosorbents for many pollutants [19]. While high removal rates are given by these commercial adsorbents, they also have a major disadvantage; their high cost restricts their use in large-scale systems. Therefore, the focus has been diverted to the preparation of biosorbents based on agricultural, natural materials, household, and industrial waste.

The Lotus scientific name is *Nelumbonucifera*, an appetizing aquatic herb with good nutritional value in the monogeneric world of the Nelumbonaceae family. Different herbal medicines for many diseases have been utilized, including cancer, depression, diarrhea, heart problems, high blood pressure, and insomnia [20]. The Lotus stem is a reproducible and abundant ingredient. The lotus stem is inherently natural and consists longitudinally of macroporous vascular bundles, transporting water and mineral nutrients upwards. Lotus stem is a great vitamin C source, which boosts a person's immunity and fights viral infections. Lotus stems involves protein 8.48%, total sugars 19.08%, ash 1–10%, nitrogen 1.36%, and free

amino acids 0.78%. Most lotuses are wasted directly because of their lower value, but some of the roots or leaves of the lotuses have been used as food or tea. Most of the lotus stalk was dropped in the pond every autumn, resulting in low or no use of solid waste and pollution of the environment. A selection of biosorbents based on natural waste, such as pine cones, leaves, barks, fruit peels, nuts, seeds, stems and other related low-cost materials released from agricultural and domestic activities, have been used to remove various contaminants from the aqueous process successfully. However, adsorbents obtained from a single source have a specific nature that minimizes their usage. Therefore, chemically modified adsorbents are replacing the traditional natural biosorbents with an extensive scope and increased efficacy. The factors affecting selecting a chemical agent include the nature of starting material, adsorption efficacy, adsorption conditions, the overall cost of the procedure, and the nature of polluting adsorbate. A variety of chemically modified agricultural and plant-based adsorbents have been successfully used for the elimination of dyes and herbicides such as base-modified Artocarpus odoratissimus leaves [21]. cationic modified orange peel powder [22], modified *Glossogyne* tenuifolia leaves [23], spent tea leaves modified with polyethyleneimine [24], pine cone powder modified with  $\beta$ -cyclodextrin [25], NaOH modified areca nut husk [26], quaternary aminemodified orange peel powder [27], aminated pumpkin seed powder [18], acid-treated lathyrus sativus husk [28], surface-modified delonix regia seed [29], powdered mandacaru cactus leaves [30], treated pecan pericarp [31], activated carbon from peanut shell [32], *physalis peruvian* chalice treated with sulfuric acid [33], and activated carbon derived from queen palm endocarp [34]. A protonated amine modified lotus stem powder was chosen as a biosorbent to remove RR-120 from an aqueous medium in the current study.

None of the earlier studies has reported literature about the PAM-LSP as an adsorbent for removing RR-120 from aqueous media to the best of our information. The objectives of this analysis are to; (1) synthesis of PAM-LSP from lotus stem, which is a unique approach; (2) investigate the factors affecting the adsorption process; (3) LSP and prepared PAM-LSP was characterized by FTIR, FE-SEM, EDX, and BET; (4) investigate the adsorption isothermal models, kinetic models, and thermodynamic parameters of PAM-LSP; (5) evaluate the regeneration of the PAM-LSP; (6) explore the possible mechanism for the removal of RR-120 onto PAM-LSP.

# 2. Materials and methods

# 2.1. Chemicals

RR-120 (Linear formula:  $C_{44}H_{24}Cl_2N_{14}O_{20}S_6Na_6$ ; Molecular weight: 1469.98; Dye content  $\geq$  50%;  $\lambda_{max}$ : 515 nm) was purchased from Sigma Chemical Company (Taiwan) and used without further pre-treatment as an adsorbate. In this scenario, all other reagents were of grade AR and were directly used without any further purification. In these experiments, DIW (de-ionized water) was used to prepare all solutions. By dissolving an accurate amount of dye in DIW, a stock solution of RR-120 was prepared. To get the desired concentration ranging from 100 to 1000 mg/L, the stock solution was diluted with DIW. The RR-120 chemical structure is shown in Fig. 1.

#### 2.2. PAM-LSP preparation

Lotus stems were locally collected from the YUNTECH campus, Douliu, Taiwan. In order to clean away the dust and other impurities from their surfaces, the collected stems were thoroughly washed many times with DIW. The stems were allowed to dry for 3–4 days in sunlight after cleaning and then oven-dried for 24 h at 343 K. To obtain particles of an average size of 150 mm, the dried stems were crushed and sieved. This powder was named lotus stem powder (LSP).

The chemical modification process involves three steps under which the LSP is cross-linked to several chemical agents in order to improve the stability and adsorption capability. Prior to the chemical treatment, 10 g of LSP was pretreated with 150 mL of 1 M alkali solution (NaOH) for a duration of 3 h in a magnetic stirrer maintained at 250 rpm. The slurry was thoroughly rinsed with DIW to neutralize the pH and finally dried in an oven at 323 K for 8 h. The alkali-treated LSP was immersed in dichloroethane followed by heating at 343 K for 2 h under stirring conditions. This was followed by the filtration, DI washing, and oven drying at 343 K for 8 h. the second step involves modification of the crosslinked LSP powder with 30 mL of diethylenetriamine and 50 mL of water under constant stirring conditions at 333 K for 4 h. The reaction mixture was then filtered, washed, dried at 333 K. Finally, the third step involves the mixing of as-synthesized powder to 150 mL 1 M HCl for a protonated reaction for 2 h at 298 K, filtered, rinsed with DIW and dried, and the product was named as PAM-LSP. The entire preparation procedure was demonstrated in Fig. 2.

#### 2.3. Characterization

Fourier Transform Infrared (FTIR) spectroscopy has been used to record the surface functional groups (Nicolet iS10, Thermo Scientific, USA), LSP, PAM-LSP, and PAM-LSP after RR-120 adsorption in the 400–4000 cm<sup>-1</sup> range (KBr pellet procedure). Using Field Emission-Scanning Electron Microscopy (FE-SEM/EDX) (JEOL, JSM-7610F plus, Japan) at an accelerated voltage of 25 kV, the surface morphology and elemental composition of the LSP, PAM-LSP, and PAM-LSP after RR-120 adsorption were analyzed. The samples were first sputter-coated and then loaded onto a copper substrate with a homogeneous gold layer. The LSP and PAM-LSP surface area and porosity measurement were performed with N<sub>2</sub> adsorption at 77 K using the Autosorb-I (Quantachrome Instruments, USA). The sample was degasified at 473 K for 2 h prior to each measurement of N<sub>2</sub> adsorption. Using the BET (Brunauer-Emmett-Teller) equation, the N<sub>2</sub> adsorption of the sample was used to define the specific surface area. At a relative pressure of around 0.99, the total pore volume was determined to be the volume of the liquid N<sub>2</sub>. The distribution of the pore size was determined by the BJH (Barrett-Joyner-Halenda) method from the isotherm desorption branch. Using a digital pH meter (Mettler Toledo, USA), which was calibrated using a buffer solution, the pH of the solutions was measured. Based on the reported method, the  $pH_{PZC}$  (point of zero charge) for the PAM-LSP was calculated. 30 mL of (0.01 M) NaCl solution was taken to a series of 50 mL polypropylene tubes in this process. Each tube's initial pH solution (pH<sub>i</sub>) was adjusted from 2.0 to 12.0 by either adding NaOH/HCl (0.1 M). Then 0.04 g of PAM-LSP was added to each tube, and the tubes were put in a rotary incubator shaker at a constant temperature for 24 h. The suspensions were filtered, and the solution's final pH (pH<sub>f</sub>) was noted. The intersection of the curve with the axis of zero gives the isoelectric point on a graph of  $\Delta pH = pH_i - pH_f$  against  $pH_i$ .

#### 2.4. Batch adsorption analysis

Batch sorption tests were conducted on a rotary incubator shaker with a steady agitation speed of 200 rpm to examine the adsorption activity of PAM-LSP as a biosorbent for the elimination of RR-120 from an aqueous medium. The kinetic study was performed by adding 0.04 g PAM-LSP with 30 mL of RR-120 solution (100, 300, and 500 mg/L, pH 2.0), followed the adsorption process at a 298 K time interval of 0–600 min. The effect of pH, 0.04 g of PAM-LSP, was added with 30 mL of RR-120 (300 mg/L) solution at 298 K and specified pH range (2.0–10.0) for 120 min. Adjusted



Fig. 1. Chemical structure of the RR-120.



Fig. 2. The preparation procedure of PAM-LSP.

the pH of the solutions from 2.0 to 10.0 and controlled by adding NaOH/HCl (0.1 M). The effect of adsorbent dosage over adsorbent dosage between 0.01 and 0.08 g/30 mL at a known concentration of 300 mg/L was examined. The evaluation of isotherms included an adsorption experiment at pH 2.0 with a different initial concentration of RR-120 (100-1000 mg/L) solutions and the state of adsorption was identical to the effect of the pH experiment. Similar experimental conditions were used for the thermodynamics study to perform adsorption at different temperatures (298–323 K). After completing the adsorption process, the biosorbent was separated from the solution by using a centrifuge. The residual amount of RR-120 in the solution was determined at a maximum  $\lambda_{max}$  of 515 nm using a UV–Vis spectrophotometer (JASCO V-750, Japan). All the adsorption experiments were duplicated and the analysis used only the average values. According to the following equations, the equilibrium adsorption capacity and dye removal efficiency were calculated.

$$q_e(mg/g) = \frac{[C_o(mg/L) - C_e(mg/L)]V(L)}{M(g)}$$
(1)

$$Removal(\%) = \frac{(C_o - C_e)}{C_o} \times 100$$
<sup>(2)</sup>

#### 2.5. Desorption and recyclability

The desorption studies were conducted with various desorbing eluents in the batch method. The experiments were carried out with the treatment of 0.04 g PAM-LSP with 30 mL of 300 mg/L RR-120 solutions at 298 K for 120 min. After separation by centrifugation, the RR-120 loaded adsorbent was carefully washed with DIW a few times to eliminate any potential presence of unadsorbed RR-120. After adsorption, the RR-120 loaded PAM-LSP was treated with 30 mL of the various desorbing eluents. After the desorption tests, the supernatant was quantified spectrophotometrically, and the desorption efficiency was measured using the following Eq. (3):

Desorption (%) = 
$$\frac{Desorption}{Adsorption} X \ 100$$
 (3)

Consecutive adsorption-desorption studies have been conducted over five repeated cycles to examine the reusability potential of the PAM-LSP biosorbent.

#### 3. Results and discussion

# 3.1. Characterization of the LSP and PAM-LSP

Various functional groups present on the surface of the biosorbent material are basically defined by the FTIR analysis. The FTIR spectra for LSP and prepared PAM-LSP before and after adsorption of RR-120 are presented in Fig. 3. The spectrum (Fig. 3a) shows a wide peak appearing at 3273 cm<sup>-1</sup> is ascribed to –OH stretching, which belongs to the hydroxyl group [35]. The presence of two small peaks at 2922 and 2845 cm<sup>-1</sup> can be attributed to the C-H stretching vibrations of the CH<sub>2</sub> groups [36]. The band at 1622 cm<sup>-1</sup> is assigned to asymmetrical stretching vibrations of the carbonyl group [37]. The peak at  $1520 \text{ cm}^{-1}$  is due to the C=C or -C-O stretching vibrations [38]. The bands at 1159, 1081, and 1011 cm<sup>-1</sup> corresponded to C=C stretching vibrations and C–O–H bending vibrations [39]. The PAM-LSP spectrum Fig. 3b, a broad band at  $3287 \text{ cm}^{-1}$  can be observed, which is related to an O-H group and asymmetric stretching vibration of N-H in amines on the PAM-LSP surface [27]. The two intense bands at 2918 and 2843 cm<sup>-1</sup> were assigned for C-H stretching of the  $-CH_3$  and  $-CH_2$  groups [40]. The peak appearing at 1730 cm<sup>-1</sup> was attributed to the C=O stretching vibration of the carbonyl group [41]. The peaks at 1631 and 1543 cm<sup>-1</sup> were attributed to the C=O and N-H bonds of amine groups, respectively [42]. The band at 1446 cm<sup>-1</sup> was assigned to the bending vibrations of hydroxyl groups [43]. The band at 1369 cm<sup>-1</sup> was assigned to C-N stretching vibrations of the amine group [18]. The band at 1317 cm<sup>-1</sup> may be attributed to the O–H bending of alcohol and the alkyl group's C–H bending [41]. The presence of a peak at  $1242 \text{ cm}^{-1}$  was assigned for the stretching vibration of C–O [42]. The peak at 1030 cm<sup>-1</sup> can be attributed to the stretching vibrations of C—N and C=O groups [36]. After the chemical treatment (PAM-LSP), some new peaks (1730, 1446, 1369, 1317, and 1242 cm<sup>-1</sup>) have appeared. FTIR spectrum for PAM-LSP after adsorption of RR-120 (Fig. 3c) clearly indicates that there is shift in band positions from 3287, 2918, 2843, 1730, 1631, 1543, 1446, 1369, 1317,1242, and 1030 cm<sup>-1</sup> to 3334, 2922, 2848, 1737, 1618, 1512, 1468, 1373, 1319, 1229 and 1027 cm<sup>-1</sup>, respectively. A few small shifts in other frequencies have also been observed. The change in band position indicates the respective group's involvement during the adsorption of RR-120 by PAM-LSP.

For the larger adsorption capacity of an adsorbent, the surface area and pore size play critical roles.  $N_2$  adsorption/desorption



Fig. 3. FTIR spectrum: (a) LSP, (b) PAM-LSP, and (b) RR-120 loaded PAM-LSP.



Fig. 4. (a) N<sub>2</sub> adsorption-desorption isotherms, and (b) BJH pore size distribution plots of the LSP and PAM-LSP.

analysis using BET and BJH methods has assessed the surface area and pore size distribution of the LSP and PAM-LSP. The curves of the isotherms of  $N_2$  adsorption-desorption are shown in Fig. 4a. At high relative pressure, the adsorption–desorption isotherm curves conform to IV-type with an H3 hysteresis loop,  $P/P_o = 0.40-0.99$ , suggesting a mesoporous structure of both

materials [44]. The obtained BET and BJH values for the LSP and PAMLSP are presented in Table 1. It was observed from Table 1 that there is a significant increase in the BET surface area, pore volume, and pore diameter values for PAM-LSP material compared to LSP after chemical treatment, which was expected to benefit the dye adsorption. Also, Fig. 4b illustrates the pore size distribution of LSP and PAM-LSP. The pore size distribution peaks were located at 2.11 and 2.53 nm for LSP and PAM-LSP, respectively. The mesoporous material contains pores with diameters in the 2.0–50 nm range, according to the IUPAC nomenclature [45]. These results confirm that the PAM-LSP has a high surface area and mesoporous and can be considered a potential adsorbent for environmental remediation.

The morphology and elemental composition of the LSP, PAM-LSP, and RR-120 loaded PAM-LSP was observed by FE-SEM with EDX analysis (Fig. 5). It was observed from Fig. 5a. the outer surface of LSP was rough, irregular, and heterogeneous. After chemical modification (Fig. 5b), the PAM-LSP surface was a porous structure with an irregular rough surface full of large pores with cavities. These morphologic characteristics are typical of adsorbent materials and are favourable for the adsorption process. After adsorption (Fig. 5(c)), the PAM-LSP surface was entirely covered by RR-120 dye, confirming the successful adsorption of RR-120 onto PAM-LSP. The EDX spectrum of raw LSP (before chemical treatment) contains the following elemental composition (C, O, Na, S, Cl, and Ca), and the respective weight % values were presented in Fig. 5d. Upon chemical modification (before adsorption) of the adsorbent, the weight % of the existing elements were changed with the appearance of new components (K and Mg) reported in Fig. 5e. After the dye adsorption (Fig. 5f), the surface of the adsorbent reported the presence of the 'S' element due to the binding of -SO<sub>3</sub> (sulfonate) groups of RR-120 confirms the successful adsorption of RR-120 onto the PAM-LSP surface.

# 3.2. Effect of pH

Solution pH dramatically influences the physical and chemical properties of adsorbate and adsorbent and plays a critical role in the aqueous medium pollutant adsorption process. In the pH range from 2.0 to 10.0, the amount of RR-120 adsorbed on PAM-LSP at 298 K is displayed in Fig. 6a. The sorption capacity of RR-120 was progressively reduced from 94 to 6 mg/g, with the pH solution increasing from 2.0 to 10.0. The RR-120 adsorption was extremely pH-dependent, and the overall uptake was found at 2.0. It is also possible to use the pH<sub>PZC</sub> to elucidate the effect of pH on RR-120 adsorption. Out of Fig. 6b, the PAM-LSP's pH<sub>PZC</sub> was found to be 6.5. The RR-120 molecules carry a net negative charge because of the presence of sulfonate  $(-SO_3^-)$  groups ionized in an aqueous solution (Eq. (4)). Owing to the availability of a considerable number of protons for the protonation of the amine groups of PAM-LSP, the surface of PAM-LSP was possibly positively charged at lower pH (pH < pH<sub>PZC</sub>); thus, a higher number of-NH $_3^+$  ions result in increased adsorption sites (Eq. (5)). As a result, the electrostatic interaction between negatively charged sulfonate ions  $(-SO_3^-)$  of RR-120 and positively charged protonated amine increases the adsorption capacity at low pH. As the pH of the solution raises (pH > pH<sub>PZC</sub>), the dissociation equilibrium of the amine groups on

Table 1			
Characteristics	of LSP	and	PAM-LSP.

Adsorbent	BET surface area (m²/g)	Total pore volume (cm <sup>3</sup> /g)	Average pore diameter (nm)
LSP	8.08	0.0036	2.48
PAM-LSP	23.4	0.0531	7.963

the molecules PAM-LSP and  $H_2O$  results in the formation of  $OH^$ ions (Eq. (6)). The anionic RR120 dye will not be adsorbed on the adsorbent's negatively charged surface. The abundant concentration of  $OH^-$  ions competing with dye molecules for the available positively charged adsorption sites on the biosorbent surface would also be required at higher pH values, which will reduce the adsorption uptake of RR120. The pH of solution 2.0 was, therefore, selected for further experiments. Similar results were found for the pH solution's effect using different adsorbents on the adsorption of RR-120 [27,46–48].

$$RR120 - SO_3Na \rightleftharpoons RR120 - SO_3^- + Na^+$$
(4)

$$PAMLSP - NH_2 + H^+ \rightleftharpoons PAMLSP - NH_3^+ + O_3^-S$$
$$- RR120 \rightleftharpoons PAMLSP - NH_3^+ \cdots \cdots O_3^-S - RR120$$
(5)

$$PAMLSP - NH_2 + H_2O \rightleftharpoons OH^- + PAMLSP - NH_3^+ + O_3^-S$$
$$- RR120 \rightleftharpoons PAMLSP - NH_3^+ \cdots \cdots O_3^-S - RR120$$
(6)

#### 3.3. Agitation speed

In the adsorption process, agitation is an essential parameter that affects the distribution of solute and adsorbent in the bulk solution. Fig. 6c displays the effect of agitation speed on PAM-LSP's adsorption of RR-120 dye in the range of 50–400 rpm, whereas all other conditions were kept constant. PAM-LSP's adsorption of RR-120 has been found to increase by increasing the agitation rate from 0 to 200 rpm, then decreasing slightly. However, the increased speed of agitation results in a greater degree of a collision between the dye molecules and the active sites, the adsorption of the dye decreases after 200 rpm. It can be said that this speed will be so fast after changing the agitation speed after 200 rpm that it hinders and disrupts the absorption of dye molecules on the active sites. For subsequent experiments, the optimum agitation speed of 200 rpm was therefore chosen.

# 3.4. Effect of adsorbent dosage

Experiments have been performed at an initial dye concentration of 300 mg/L to study the effect of the adsorbent dosage (range 0.01-0.08 g/30 mL) on dye adsorption, and other parameters are constant. As illustrated in Fig. 6d, with a rise in the adsorbent dosage from 0.01 to 0.04 g/30 mL, the removal efficiency of RR-120 significantly increased from 13 to 89%. The increase in the number of available active sites required for RR-120 sorption can be assigned to this by increasing the amount of PAM-LSP. Above the dosage of 0.04 g/30 mL, the RR-120 removal efficiency does not change with further increases in the biosorbent dosage due to the limited availability of RR-120 molecules in the solution. When the dose grew to 0.08 g/30 mL, the optimum removal efficiency of RR-120 was achieved. On the other hand, as the PAM-LSP dosage increased from 0.01 to 0.08 g/30 mL, the adsorption uptake of RR-120 was found to decrease significantly (Fig. 6d). The decrease in adsorption uptake by raising the dosage of the adsorbent was due to a higher surface area at the lower dosage of the adsorbent and, therefore, a more significant collision between the adsorbent site particles and the dye molecules. Secondly, an increase in adsorbent dosage at constant dye concentration and volume leads to saturation of active sites. It can decrease adsorption capacity due to particulate interaction such as aggregate formation at higher adsorbent dosage since aggregation may lead to a decrease in the total adsorbent surface area and a decrease in the length of the diffusion pathway [49]. According to these findings, 0.04 g/30 mL was used in further experiments as the optimal adsorbent dosage for RR-120.



Fig. 5. FE-SEM images of (a) LSP, (b) PAM-LSP, and (c) RR-120 loaded PAM-LSP (d-f); EDX images of (e) LSP, (f) PAM-LSP, and (g) RR-120 loaded PAM-LSP.

#### 3.5. Effect of contact time & initial dye concentration

In order to understand the efficiency of the adsorbent, it is essential to analyze the effect of contact time and the initial concentration of the dye. A different concentration of 100, 300, and 500 mg/L of RR-120 from 0 to 600 min were used to research the effect of contact time and initial concentration of RR-120 on adsorption. As illustrated in Fig. 6e, it was noticed that the adsorption uptake increased with the contact time, which was subsequently balanced at 120 min. Despite an increase in contact time, adsorption efficacy was more or less the same beyond this point. Initial adsorption rises were attributed to the availability of more empty active sites on the adsorbent surface. However, as time progressed, owing to the repulsion between the liquid phase and the solvent molecules on the adsorbent, the residual empty sites had not been filled. Once the equilibrium is reached in 120 min, all the active sites on the adsorbent surface will be saturated, and no further adsorption will occur. For all the dye concentrations under analysis, a similar adsorption trend was observed. As the initial concentration of dye raised from 100 to 500 mg/L, the RR-120 equilibrium adsorption was found to rise from 39.75 to 145.85 mg/g. For further experiments, the optimum equilibrium time of 120 min was considered from the results.

# 3.6. Adsorption kinetic studies

The rate of adsorbate uptake that governs the contact time of the adsorption reaction is described by adsorption kinetics, which is an important aspect that defines the efficacy of adsorption. In order to provide an understanding of the adsorption mechanism, information on adsorption kinetics is also crucial. In order to determine the effect of the initial dye concentrations on the kinetic behaviour, kinetic experiments were carried out using three separate initial concentrations of 100, 300, and 500 mg/L. The following



**Fig. 6.** Effect of (a) solution pH [dosage: 0.04 g, initial concentration: 300 mg/L, agitation: 200 rpm, volume: 30 mL, time: 120 min, temperature: 298 K], (b) point of zero charge [pH range 2.0–10.0, agitation: 200 rpm, temperature: 298 K, electrolyte volume: 30 mL], (c) agitation speed [dosage: 0.04 g, pH: 2.0, initial concentration: 300 mg/L, volume: 30 mL, time: 120 min, temperature: 298 K], (d) adsorbent dosage [initial concentration: 300 mg/L, agitation: 200 rpm, volume: 30 mL, time: 120 min, temperature: 298 K], (e) contact time and initial dye concentration [initial concentration: 100–500 mg/L, pH: 2.0, dosage: 0.04 g, agitation: 200 rpm, volume: 30 mL, temperature: 298 K], add (f) temperature on RR-120 removal [initial concentration: 300 mg/L, agitation: 200 rpm, volume: 30 mL, time: 120 min, pH: 2.0, dosage: 0.04 g, temperature range: 298–338 K].

equations can be described as the non-linearized forms of the PFO and PSO kinetic equations [50,51]:

$$q_t = q_{e1}(1 - \exp(-k_1 t))$$
(7)

$$q_t = \frac{q_{e2}^2 k_2 t}{1 + q_{e2} k_2 t} \tag{8}$$

The results of the kinetic model of PFO and PSO for adsorption of RR-120 on PAM-LSP are presented in Table 2. The result of the table shows that, with increased dye concentration, the experimental and calculated adsorption capacity increases. The correlation coefficients ( $R^2$ ) of the PSO model can be seen to be greater than those of the PFO model.  $R^2$  values range from 0.978 to 0.999 for the PSO model and from 0.799 to 0.971 for the PFO model. For the PSO model, the calculated value ( $q_{e, cal}$ ) is similar to the experimental value ( $q_{e, exp}$ ) than for the PFO model ( $q_{e, cal}$ ). These findings show that the PSO model better describes the kinetic adsorption of RR-120 on PAM-LSP than the PFO model, which was similar to the earlier reports [22,27]. Fig. 7 displays the nonlinear plots of kinetic models of PFO and PSO for RR-120 adsorption onto PAM-LSP.

#### 3.7. Modeling of adsorption isotherms

As the adsorption equilibrium state is achieved, the sorption isotherm displays the distribution of adsorption molecules between the solid and liquid phases. In order to find an appropriate model that can be used for design purposes, it is vital to examine isothermal data by fitting several isotherm models. In order to describe how solutes interact with biosorbents, adsorption isotherm is essential and crucial for optimizing the use of biosorbents. In this analysis, two-parameter non-linearized isothermal models such as D-R, Langmuir, Temkin, and Freundlich were used to evaluate experimental equilibrium data. The Chi-square ( $\chi^2$ ) analysis was also performed to determine the best-fit isothermal adsorption model. The equation for assessing the best-fit model should be proposed as follows:

$$\chi^{2} = \sum_{i=1}^{n} \left( \frac{(q_{e,\exp} - q_{e,cal})^{2}}{q_{e,cal}} \right)$$
(9)

The Langmuir model estimates a monolayer cover on the external surface of the biosorbent by the assumption of monolayer sorption at structurally and energetically homogeneous active sites. The non-linear form of the equation of the Langmuir isotherm is given as [52]:

$$q_e = \frac{q_{\max}K_L C_e}{1 + K_L C_e} \tag{10}$$

The separation factor  $(R_L)$  can be calculated using this data as a dimensionless constant that refers to the nature of adsorption by using the Eq. (11):

Table 2

Kinetic model parameters for the adsorption of RR-120 onto PAM-LSP at various initial concentrations of the dye.

Adsorbent	Conc. (mg/L)	$q_{e, \exp} (mg/g)$	Pseudo-first-order			Pseudo-second-order			
			$q_{e1, cal} (\mathrm{mg/g})$	<i>k</i> <sup>1</sup> (1/min)	$R^2$	$q_{e2, cal} (mg/g)$	$k_2$ (g/mg min)	$R^2$	
PAM-LSP	100 300 500	39.75 94.39 145.85	32.72 87.69 131.85	0.1348 0.1683 0.1648	0.9254 0.8994 0.9136	40.37 95.79 144.98	0.0060 0.0035 0.0022	0.9979 0.9925 0.9902	



Fig. 7. Non-linear kinetic plots for the adsorption of RR-120 onto PAM-LSP: (a) 100 mg/L, (b) 300 mg/L, and (c) 500 mg/L [initial concentration: 100-500 mg/L, volume: 30 mL, time: 120 min, agitation: 200 rpm, dosage: 0.04 g, pH: 2.0, temperature: 298 K].

$$R_L = \frac{1}{(1+K_L C_o)} \tag{13}$$

Adsorption isotherms can be classified from the  $R_L$  value as unfavorable if  $R_L > 1$ , linear if  $R_L = 1$ , irreversible if  $R_L = 0$ , and favorable if  $0 < R_L < 1$ .  $q_{max} = 183.62 \text{ mg/g}$ ,  $K_L = 0.03498 \text{ L/mg}$  and  $R_I = 0.562$  with  $R^2 = 0.9956$  are the Langmuir parameters obtained in this work. The  $R_L$  value confirmed that the RR-120 adsorption to PAM-LSP was favorable.

The Freundlich isothermal model is generally used without saturation of adsorption sites to presume a heterogeneous surface and a non-uniform distribution of adsorption heat over the surface. The Freundlich isothermal model non-linearized form is defined by [53]:

$$q_e = K_f C_e^{1/n} \tag{12}$$

The value of *n* signifies the degree of nonlinearity between the concentration of the solution and the adsorption as follows: if n < 1, chemisorption; if n > 1, physisorption; if n = 1, linear. For PAM-LSP, the value of *n* was found to be 3.863. This indicates the physisorption of RR-120 onto PAM-LSP since n is between 1 and 10.

Based on the potential principle that assumes a heterogeneous surface, the D-R isotherm is applied to determine the adsorption mechanism. The D-R isotherms non-linearized form is expressed as follows [54]:

$$q_e = q_s \exp(-K\varepsilon^2) \tag{13}$$

$$\varepsilon = RT \ln \left( 1 + \frac{1}{C_e} \right) \tag{14}$$

The mean free adsorption energy, E is derived from the following Eq. (15):

$$E = \frac{1}{\sqrt{2K}} \tag{15}$$

The adsorption mechanism is further defined by the magnitude of the E, i.e., transferring one mole of ions onto the biosorbent surface, possessing a value of < 8.0 kJ/mol representing physisorption behaviour. Further, the range of E between 8.0 and 16.0 kJ/mol depicts the adsorption process is solely due to the ion-exchange, whereas the E value in the range of 20-40 kJ/mol denotes the chemisorption [55]. In the current study, the measured *E* value is below 8.0 kJ/mol, implying that the adsorption of RR-120 to the PAM-LSP surface follows the physisorption type.

Temkin and Pyzhev considered the effects of adsorption of indirect adsorbate/adsorbent interplay. By ignoring extremely low and high concentration values, the model assumes that the adsorption heat (temperature function) of all the molecules in the layer would decrease linearly due to adsorbate/adsorbent interplay rather than logarithmic coverage. The non-linearized isotherms of Temkin are represented by [56]:

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$$q_e = B_T \ln(A_T C_e) \tag{16}$$

$$q_e = B_T \ln(A_T) + B_T \ln(C_e) \tag{17}$$

$$B_T = \frac{RT}{b_T} \tag{18}$$

The  $B_T$  value shows whether physisorption (<8.0 kJ/mol) or chemisorption (between 8.0 and 16.0 kJ/mol) is involved. The  $B_T$  value obtained was < 8.0 kJ/mol, indicating that the physisorption process was the adsorption of RR-120 to PAM-LSP.

Fig. 8 shows the plots of the experimental and the predicted two-parameter isotherms for the adsorption of RR-120 onto PAM-LSP by the non-linear method. Table 3 presents all four isothermal model results, together with the correlation coefficients ( $R^2$ ) and  $\chi^2$ . If these non-linearized isotherms are compared, the best-fit adsorption isotherms can be identified instead of the current study; the order of precision is as follows: Langmuir > D-R > Temkin > Freundlich, based on the association of higher  $R^2$  and lower  $\chi^2$  values.

# 3.8. Comparison of PAM-LSP with other adsorbents

As stated in the literature [46,47,57–67], the adsorption ability of different adsorbents towards RR-120 dye was presented in Table 4. A comparison between this work and other reported literature data shows that compared to other adsorbents, PAM-LSP is a better adsorbent for RR-120. It can therefore be safely concluded that the PAM-LSP has considerable potential for the treatment of contaminated water with RR-120 dye.

#### 3.9. Effect of temperature and thermodynamic studies

Temperature is one of the key parameters governing the efficiency of the adsorption process. The influence of temperature on adsorption of RR-120 on PAM-LSP was explored by adsorption experiments at various temperatures and shown in Fig. 6f. The results showed that RR-120 removal increased from 45 to 110 mg/g by raising the temperature from 298 to 323 K, suggesting that RR-120 adsorption to PAM-LSP was endothermic. The viscosity of the solution is reduced with an increase in temperature, which increases the rate of transfer of adsorbate molecules across



**Fig. 8.** Non-linear isotherm plots for adsorption of RR-120 onto PAM-LSP [initial concentration range: 100–1000 mg/L, volume: 30 mL, dosage: 0.04 g, agitation: 200 rpm, time: 120 min, pH: 2.0, temperature: 298 K].

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#### Table 3

Isotherm parameters for adsorption of RR-120 onto PAM-LSP.

Isotherm model	Parameter	
Langmuir	$q_m (mg/g)$	183.62
	$R_L(L/IIIg)$ $R^2$	0.03498
	$\gamma^2$	19.85
Freundlich	$\tilde{K}_{f}$ (mg/g)	37.75
	n	3.863
	$R^2$	0.9396
	$\chi^2$	273.15
Temkin	$B_T$ (J/mol)	77.24
	$A_T$ (L/mg)	0.8211
	$R^2$	0.9701
	$\chi^2$	94.73
Dubinin-Radushkevich	$q_s (mg/g)$	153.93
	$K (\mathrm{mol}^2/\mathrm{J}^2)$	0.0211
	E (kJ/mol)	4.868
	$R^2$	0.8176
	$\chi^2$	577.62

Table 4					
Monolayer adsorption	capacity	comparisons	of various	adsorbents	for RR-120.

	( )		D.C.
Adsorbent	q <sub>max</sub> (mg/g)	рн	Reference
Cross-linked chitosan-GLA/TiO <sub>2</sub>	103.1	3.0	[46]
nanocomposite			
Hydrilla verticillata	120.85	2.0	[47]
Nanoparticles of Fe <sub>3</sub> O <sub>4</sub>	166.67	2.5	[57]
Functionalized sludge	46.8	-	[58]
Sisal fiber activated carbon	110	2.0	[59]
Chitosan/zeiolite	19.14	5.0	[60]
Chitosan/modified montmorillonite beads	5.6	5.0	[61]
Jatropha curcas shells	40.94	2.0	[62]
Jatropha curcas shell treated by	65.63	2.0	[62]
non-thermal plasma			
Chara contraria	112.83	1.0	[63]
Pumpkin husk	98.61	1.0	[64]
CS-TPP/FA-50 composite	165.8	4.0	[65]
Cetylpyridinium-bentonite	81.97	2.0	[66]
Chitosan beads	114.9	4.0	[67]
Protonated amine modified lotus stem	183.62	2.0	Present
powder			study

the phase boundary and into the internal pores of the solid biosorbent, thereby improving the adsorbent's ability to adsorb [68].

In order to measure the thermodynamic parameters, the temperature-dependent sorption data were further used to obtain detailed information on the inherent energy changes involved in the adsorption process. By using the following equations, thermo-dynamic parameters like  $\Delta G^o$ ,  $\Delta S^o$ , and  $\Delta H^o$  are derived.

$$K_c = \frac{C_{Ae}}{C_e} \tag{19}$$

$$\Delta G^{o} = -RT \ln K_{c} \tag{20}$$

$$\ln K_c = -\frac{\Delta G^o}{RT} = -\frac{\Delta H^o}{RT} + \frac{\Delta S^o}{R}$$
(21)

$$\Delta G^o = \Delta H^o - T \Delta S^o \tag{22}$$

The values  $\Delta S^{\circ}$  and  $\Delta H^{\circ}$  can be estimated from the intercept and slope, respectively, by plotting a graph of ln  $K_c$  against 1/T, as seen in Fig. 9a. The thermodynamic parameters of PAM-LSP for removing RR-120 were compared with those of various previously reported adsorbents, as shown in Table 5. At varying temperatures, the negative values of  $\Delta G^{\circ}$  indicate that the adsorption is thermodynamically feasible and spontaneous. With a rise in temperature, the decrease in  $\Delta G^{\circ}$  values implies a reduction in the feasibility of adsorption at upper temperatures. In general, Gibbs free energy ranges from -20 and 0 kJ/mol for physisorption, compared to chemisorption ranging from -80 to -400 kJ/mol [49]. The data in Table 5 showed that the  $\Delta G^o$  values in the -20 and 0 kJ/mol ranges indicated that physisorption was the adsorption of RR-120 to PAM-LSP. During the adsorption of RR-120 to PAM-LSP, the positive value of  $\Delta S^o$  suggested an increase in the randomness at the sorbent/liquid interface. The positive value of  $\Delta H^o$  suggests that the process of adsorption was endothermic. In general, the physisorption and chemisorption  $\Delta H^o$  values are between 2.1 and 20.9 and 20.9–418.4 kJ/mol, respectively [69]. Table 5  $\Delta H^o$  value shows that the adsorption of RR-120 onto PAM-LSP is physisorption. This finding suggests that the RR-120 adsorption process was feasible, spontaneous, and endothermic.

Activation energy ( $E_a$ ) was evaluated using the modified Arrhenius equation that relates the sticking probability ( $S^*$ ) to the surface coverage ( $\theta$ ) via the following equations to further support the assertion that physical adsorption is the predominant mechanism [70]:

$$S^* = (1 - \theta)e^{-E_a/RT} \tag{23}$$

$$\theta = [1 - C_e/C_o] \tag{24}$$

A function of the adsorbate/adsorbent system under consideration is the sticking probability ( $S^*$ ). Its value is within the  $0 < S^* < 1$ range and depends on the system temperature. The  $S^*$  shows the measurement of the ability of an adsorbate to remain on the indefinite adsorbent. The  $E_a$  and  $S^*$  values were determined from the



Fig. 9. (a) Van't Hoff plot, and (b) activation energy of RR-120 onto PAM-LSP.

slope of the linear plot of  $\ln(1-\theta)$  vs. 1/T at various temperatures (Fig. 9b). Based on S<sup>\*</sup> values, four types have been classified: (a) If  $S^* > 1$ , it means that there is no interplay between adsorbate and adsorbent molecule; (b) if  $S^* = 1$ , it is attributed to the probability of physicochemical sorption; (c) if  $S^* = 0$ , it corresponds to the control of chemisorption process; and (d) if  $0 < S^* > 1$ , it correlated to the superiority of the physisorption process as well as implies favourable sticking of adsorbate to the biosorbent [71]. The value of  $S^*$  was 0.0043, which lies in the range  $0 < S^* < 1$ , indicating that the adsorption follows physisorption. An idea of the type of adsorption, which is primarily chemical or physical, is given by the magnitude of  $E_a$ . Physisorption processes typically have energies in the 5.0-40 kJ/mol range, whereas chemisorption is indicated by higher activation energies (40-800 kJ/mol) [72]. The value of  $E_a$  for adsorption of RR-120 into PAM-LSP was found to be 5.9 kI/mol, indicating that the predominant mechanism involved was physisorption.

#### 3.10. Desorption and regeneration of PAM-LSP

The most critical aspect of the study of adsorption is regeneration. It is important to reuse the adsorbent to make the adsorption process more economical. It is preferable to desorb the adsorbed RR-120 dye first, for this reason, then to apply consecutive cycles of adsorption/desorption repeated over five cycles, using the adsorbent PAM-LSP. Batch desorption tests were performed under the same conditions using a 0.1 M solution of the various eluents NaOH, NaHCO<sub>3</sub>, NaCl, HCl, HNO<sub>3</sub>, CH<sub>3</sub>COOH, DIW. The comparison between the different desorption efficiencies is shown in Fig. 10a. The use of CH<sub>3</sub>COOH and DIW resulted in very low desorption performance, 38% and 19%, respectively, while NaOH showed the highest desorption efficiency of 94%. NaOH (0.1 M) was then used as the most effective desorbing eluent. The recovery test was repeated for five cycles, and the findings are illustrated in Fig. 10b. As depicted in Fig. 10b, PAM-LSP's adsorption efficiency decreased after five cycles from 89% to 77%. This decrease in removal efficiency may be due to the inability to accept more dye molecules at new binding sites, changes in morphological properties, persistent adsorbent denaturation, and weak electrostatic interactions between the adsorbent and the adsorbent. The desorption efficiency of PAM-LSP also decreased from 94% in the first cycle to 83% in the fifth cycle, according to the desorption process. These promising results showed that PAM-LSP could be regenerated and used repeatedly in dye removal without much loss.

#### 3.11. Adsorption mechanism

The adsorption performance generally depends on several factors, such as surface morphology, specific surface area, active sites of adsorption, adsorbent-adsorbate interaction, and adsorbent surface charge. Table 4 demonstrates the adsorption capacity of PAM-LSP with various adsorbents seen in previous literature to remove RR-120. It can be observed that when compared to other adsorbents described in Table 4, the adsorption uptake of RR-120 on PAM-LSP was very high. The adsorption of RR-120 onto PAM-LSP can presumably be considered a monolaver adsorption process based on equilibrium and thermodynamics studies, and the adsorption process is physisorption. The intermolecular electrostatic interaction plays a crucial role in the adsorption process for physisorption. Because of the positively charged PAM-LSP surface interactions with negatively charged RR-120 dye molecules, electrostatic interactions arise. The possible adsorption mechanism of the PAM-LSP adsorbent with RR-120 dye was shown in Fig. 11.

#### Table 5

Thermodynamic parameters of RR-120 adsorption with various adsorbents.

Adsorbent	Temp. (K)	Thermodynamic properties					Reference
		$\Delta G^{o}$ (kJ/mol)	$\Delta H^o$ (kJ/mol)	$\Delta S^{o}$ (J/mol K)	<i>S</i> *	E <sub>a</sub> (kJ/mol)	
Nanoparticles of Fe <sub>3</sub> O <sub>4</sub>	278	-1.7					
	288	-5.8	113.6	414.6	-	-	[57]
	298	-9.9					
	308	-14.1					
Jatropha curcas shells	298	-26.58					
	303	-27.37					
	308	-28.25	20.36	157.6			[62]
	313	-29.02					
	318	-29.76					
	323	-30.50					
Chara contraria	298	-26.20					
	303	-27.39	36.61	211	-	-	[63]
	308	-28.31					
Cetylpyridinium-bentonite	298.15	-2.92					
	308.15	-4.04					
	318.15	-5.16	30.38	111.69	-	-	[66]
	328.15	-6.27					
	338.15	-7.39					
Chitosan beads	303	-9.39					
	313	-11.15	51.68	201.7	-	-	[67]
	323	-12.59					
Protonated amine modified lotus stem powder	298	-0.0539					
	308	-0.3016					
	318	-0.6753	10.07	33.8	0.0043	5.9	Present study
	328	-1.0432					
	338	-1.3816					



Fig. 10. Desorption of RR-120 using (a) different eluents, and (b) 0.1 M NaOH after five regeneration cycles.

# 4. Conclusions

The adsorption of RR-120 from the aqueous solution on PAM-LSP used as an adsorbent was examined in this study, and the results can be given as follows:

- RR-120 adsorption was dependent on the agitation speed, initial dye concentration, adsorbent dosage, solution's pH, temperature, and contact time.
- $\bullet$  For the PAM-LSP, the  $pH_{PZC}$  is determined, and its value has been found to be 6.5.
- Among all the initial concentrations studied, the kinetic data of the RR-120 adsorption fitted well with the PSO model ( $R^2 > 0.9902$ ).
- For relevance to the Langmuir, D-R, Temkin, and Freundlich isotherms, experimental data were analyzed and Langmuir was found to be the best-suited isotherm (based on high  $R^2$  and low  $\chi^2$  values).
- In optimal experimental conditions, the maximum adsorption uptake of the PAM-LSP for RR-120 dye was found to be 183.62 mg/g.
- The effect of temperature is used to calculate thermodynamic parameters (ΔS<sup>o</sup>, ΔG<sup>o</sup>, and ΔH<sup>o</sup>), indicating that the adsorption of RR-120 was spontaneous and endothermic.
- The value of *E<sub>a</sub>* for adsorption of RR-120 on PAM-LSP was found to be 5.9 kJ/mol, suggesting that the predominant mechanism involved was physisorption.
- Desorption experiments with NaOH (0.1 M) solution revealed a 94% recovery of RR-120.



Fig. 11. The possible interaction mechanism between the PAM-LSP and RR-120.

- The reusability study showed that, as very little efficiency loss was observed, the PAM-LSP could be effectively used for up to five cycles without further regeneration.
- The adsorption mechanism consisted mainly of electrostatic attractions between + ve charged PAM-LSP and -ve charged RR-120.

Overall this study showed that PAM-LSP could be an alternative to many expensive adsorbents used in wastewater to remove RR-120 dye.

## **CRediT authorship contribution statement**

Venkata Subbaiah Munagapati: Conceptualization, Methodology, Visualization, Formal analysis, Writing - original draft, Writing - review & editing. Hsin-Yu Wen: Conceptualization, Validation, Visualization. Jet-Chau Wen: Conceptualization, Methodology, Resources, Writing - review & editing, Supervision, Funding acquisition. Anjani R.K. Gollakota: Conceptualization, Visualization, Validation. Chi-Min Shu: Conceptualization, Validation. Guda Mallikarjuna Reddy: Editing.

#### **Declaration of Competing Interest**

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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