Synthesis of Sulfonated Polystyrene-Based Porous Activated Carbon for Organic Dyes Removal from Aqueous Solutions

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Abstract
Nowadays, aquatic pollution is one of the most important global challenges, due to discharges a wide variety of various hazardous materials from different activities which have significant environmental, economic and healthy impacts. Industries and activities related to organic dyes consume large amounts of water and contribute significantly to the growing problem of water pollution. The current study describes the conversion of plant wastes into activated carbon sphere incorporated sulfonated polystyrene (AC/SPS) for adsorptive removal of Rhodamine B (RhB) and Congo Red (CR) dyes in a batch process. The prepared materials were characterized by different techniques such as XRD, FTIR, FESEM, TEM, BET-BJH, and TGA. The AC/SPS in weight ratio of 10% was used as a novel adsorbent for RhB and CR remediation. Batch adsorption experiment have been studied by investigating the effect of contact time, adsorbent dose, initial pH, and temperature. The maximum removal efficiency of RhB and CR onto AC/SPS under optimized conditions was estimated to be 34% and 98%, respectively. In addition, it has been found that Freundlich model provided the most appropriate fit for the adsorption of both RhB and CR dyes. Based on the thermodynamic study, it was proven that the adsorption process for both dyes is endothermic and spontaneous. The change in entropy was estimated to be 190.59 and 65.40 J/mol K for CR and RhB dyes, respectively. The kinetic study revealed that the adsorption of CR and RhB dyes followed the pseudo-second order and pseudo-first order kinetic models, respectively.
Keywords Sulfonated polystyrene · Activated carbon · Water treatment · Bio-waste · Congo red · Rhodamine B

Introduction

Water is one of the most important and effective substance of human life and other organisms. The rapid growth of the urbanization and global population, combined with the increased development of agricultural and industrial activities, have accelerated water pollution through release of various toxic substances in water bodies. The accelerated growth of various industries including textile, leathers, paint, food processing, cosmetic, paper, plastics, printing and pharmaceuticals has led to consumption and pollution of large volumes of water. The widespread use of synthetic dyes in various industries has reflected in series of risks to the human health and aquatic life because of the mutagenic, bio-accumulative, carcinogenic and toxic properties of such substances [1]. In addition, discharging dyes into aquatic environment decreases the transmission of solar light [2], decreases dissolved oxygen (DO) level [3], suppress photosynthesis which means a decrease in plant survival [4]. Dyes-containing wastewater is a significant polluter because most of the dyes are chemically and photolytically stable which make them resistant towards temperature and conventional aerobic and anaerobic biological degradation processes [5]. Due to their color brightness, chemical stability, and strong adhesive properties, the red-synthetic organic dyes, Rhodamine B (RB) and Congo red (CR) were extensively used in textile coloring, cosmetics, printing, paint, leather making and many other industries [6]. Both dyes possess high solubility in water and have various health risks like neurotoxic, mutagenic, and carcinogenic effects [7, 8].

Different technologies have been adopted to remove various pollutants from aqueous solutions and industrial wastewaters, among which enzymatic oxidation, adsorption, sonocatalytic, ozonation, photocatalysis and advanced oxidation processes and are the most efficiently used methods [9–15]. Sometimes some of these methods have been revealed to be limited efficiency and time consuming, in addition, they require high operating costs. In contrast, adsorption as a physico-chemical treatment has been identified to be the most effective, the simplest and cheapest technique and most appropriate method for organic dyes removal [16]. In comparison with former removal and treatment methods, the adsorption technique is considered as preferred over other wastewater treatment processes due to its advantages such as
high activity, effective cost and facile operation [17]. Adsorbents such as activated carbon, zeolite, silica gel, polymers, graphene, carbon nanotubes, clays, metal oxides, chitosan and magnetic composites are extensively used in contaminated water remediation [16, 18–22]. Among these materials, activated carbon is considered as one of the most efficient adsorbents and a stable supporting material because it's large specific surface area and adsorption capacity [23–26]. Activated carbon is a microporous substance applied in many fields as efficient adsorbent for removal of various pollutants from aqueous media. Activated carbon derived from biomass resources was proven to be versatile material due to its capability to remove various water contaminations which relates with its high surface area and more active adsorption sites [27–29]. In addition to hydrothermal carbonization, the most reliable technique to produce activated carbon is chemical activation of carbon sources followed by carbonization under inert atmosphere at temperatures. The chemically activating agents are H2SO4, H3PO4, HCl, HNO3, KOH, NaOH, ZnCl2, FeCl3, Ca2CO3 and K2CO3 [30]. Nanostructured polymers based activated carbon such as AC/poly aniline [31, 32], AC/chitosan [33], AC/polystyrene [34] AC/cellulose [35] and AC- polysulfide rubber [36] were designed for better adsorption properties. Polystyrene (PS), an amorphous, high molecular weight, organic thermoplastic resin, is extensively employed in a wide range of application ranging from food packaging to household applications, thermal insulation and electronic cabinets. PS is a water insoluble material, but with sulfonation process, the insertion of acid sulfonic groups (-SO3H) in the polymer chain produces sulfonated polystyrene (SPS), the hydrophilicity is greatly improved [37]. SPS and its hybrid composites has shown efficient adsorption activity to due to the presence of sulfonic groups that play a major role in increasing of porosity and surface area [38].

The major aim of present study was to synthesis of activated carbon via green route using Hibiscus Sabdarriffa L leaves as natural source. In addition, waste polystyrene was employed to preparation of sulfonated polystyrene and activated carbon/polystyrene composite. Furthermore, the characterization study of synthesized materials was conducted using X-rays diffraction (XRD), Scanning electron microscopy (FESEM), Fourier transform-infrared spectroscopy (FT-IR), Transmission electron microscopy (TEM), Brunauer, Emmett and Teller (BET), Barrett-Joyner-Halenda (BJH),and Thermogravimetric analysis (TGA) The adsorption behavior of as synthesized AC/SPS in removing RhB and CR dyes from aqueous solution was investigated. In addition, the effects of operating parameters such as equilibrium time, adsorbent mass, temperature and initial pH on the dye adsorption efficiency have been examined.

**Experimental**

**Materials**

Waste commercial polystyrene (Fig. 1a) were obtained from local market. Sulphuric acid (H2SO4, 98%), Phosphoric acid (H3PO4, 85%), Diethyl ether (Et2O, 99.5%), Sodium hydroxide (NaOH, 99.5%) and Hydrochloric acid (HCl, 37%) were purchased from Scharlue. N, N-Dimethylformamide (DMF), Rhodamine B (RhB, Fig. 1b) and Congo red (CR, Fig. 1c) were supplied by Merck.

**Preparation of Activated Carbon**

The fresh leaves of the Hibiscus Sabdarriffa L were collected from a local farm et al.-Diwaniyah City (south of Iraq). Afterwards, the wet leaves of plant were thoroughly washed with doubled distilled water (DDW) to remove adhered impurities, and then it dried in an oven at 110°C overnight after being cut into small pieces. Afterwards, the dried product was grinded well for fine powder using an agate mortar. The carbonization process was conducted at high temperature for 4 h under N2 atmosphere adopting phosphoric acid as an activating agent as reported previously [39]. Typically, 10 g of dried powder was impregnated into 100 ml of 18.9% phosphoric acid solution. The suspension is then stirred at 100 rpm for 2 h and left to soak for a week at ambient conditions. When the activation was completed, the liquid phase was removed by centrifugation and the activated

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**Fig. 1** Image of PS waste (a) and structural formula of RhB dye (b) and CR dye (c)
sample was dried at 105°C for 24 h. The dried material was then carbonized at 650°C for 4 h under N₂ atmosphere in a heating at a rate of 5 °C min⁻¹.

Preparation of Sulfonated Polystyrene

Briefly, 25 g of commercial polystyrene waste was dissolved into DMF, followed by stirring and heating at 100°C for 25 min to obtain homogeneous solution. The obtained solution was then submerged into 20% diluted sulfuric acid for 4 h. After the sulfonation ends, excess acid was removed from sulfonated polymer solution by adding an excess of DDW [40]. Finally, the sample was separated, washed and dried.

Preparation of Activated Carbon/ Sulfonated Polystyrene (AC/SPS)

AC/SPS nanocomposite was prepared as reported previously with some modifications [37]. Typically, a certain amount of sulfonated polystyrene was dissolved into 25 ml of DMF. The desired amount of AC in weight ratio of 10% was suspended into DMF and sonicated for 30 min to get well dispersion. Afterwards, the AC suspension was added drop by drop to the SPS solution with steady agitating for 1 h. Then, the AC/SPS sample was precipitated by dripping the suspension into an excess amount of Et₂O with vigorous stirring. Finally, the product was separated, washed and dried.

Characterization

The crystalline structure of the prepared nanocomposites was investigated adopting powder X-ray diffraction patterns recorded by X-rat diffractometer (XRD-6000, Shimadzu-Japan, 20 of 10°–80°, λ = 1.5405 Å). In order to identify the functional groups in as fabricated samples, Fourier transform infrared spectra were recorded using Shimadzu-8400S spectrophotometer. Morphology of the samples was analyzed using field emission-scanning electron microscope (Zeiss Libra 200 FE, Germany). The pore size distribution of the prepared substances were analyzed using N₂ adsorption employing BJH equation. The specific surface area of samples were examined by N₂ absorption using Brunauer–Emmett–Teller method adopting an automatic gas sorption analyzer (Quaderosb SIMP). Transmittance electron microscopy images were recorded employing a Philips model FEI, Quanta 400. Thermogravimetric analysis (TGA, 8065 D1) was investigated under air atmosphere with heating rate of 10 °C min⁻¹. Raman spectra were analyzed by a micro Raman spectrometer (Takram, 785 nm Laser).

Batch Adsorption Experiments

For the comparison of adsorption efficiency, contact time (0–120 min), adsorbent dose (0.001–0.01 g/25 ml), pH (4–11), initial concentration of dyes (10 ppm for RhB and 50 ppm for CR) and temperature (288–303 K) were investigated. In this regard, batch adsorption process was conducted. Firstly, in 50 ml conical flask, certain mass of adsorbent was suspended to 25 ml of dye solution. The pH of solutions was adjusted using 0.1 M HCl or 0.1 M NaOH. The solution was stirred in an orbital shaker at 150 rpm. After a particular time period, the adsorbent particles were removed via centrifugation at 6000 rpm for 15 min and the absorbance was monitored at λ max of dye (498 nm for CR and 553 nm for RhB) [37, 41]. Accordingly, the general approach outlined above was used, with the desired parameter being varied while the others were kept constant. At each interval time, the removal efficiency (%) and/or adsorption capacity (Qₑ) were determined adopting Eqs. (1) and (2), respectively [41]:

\[
\text{Removal efficiency(%) } = \left( \frac{C_i - C_e}{C_i} \right) \times 100 \quad (1)
\]

where \( C_i \) is the initial concentration, \( C_e \) represents the equilibrium concentration of dye (mg/l), the adsorption capacity \( Q_e \) is the amount adsorbed at equilibrium (mg/g) was calculated as in following expression:

\[
Q_e = \frac{(C_i - C_e)V}{m} \quad (2)
\]

where, \( V \) is the volume of dye solution (L) and \( m \) is the weight of adsorbent mass (g).

Results and Discussion

Characterization of the Synthesized Materials

FTIR Analysis

FTIR analysis was employed for identifying surface functional groups of the prepared materials. Figure 2a displays the FT-IR spectra in the wavelengths range of 500 and 4000 cm⁻¹. For AC sample, the noticeable band centered at 2824 cm⁻¹ is associated to the CH₂ symmetric stretching. There was another peak observed at 2930 cm⁻¹ corresponding to the C-H sp³ stretching. The strong band at 1732 cm⁻¹ is assigned to the C=O stretching vibration in carboxylic acid, ketones and aldehydes. The broad band around 3432 cm⁻¹ is assigned to hydroxyl groups
(OH) corresponding to the adsorption of water molecules [42]. The bands observed in wavenumber range between 1000 and 1260 cm\(^{-1}\) include C-O (in carboxylic acid, alcohol and esters) [43]. The weak band noticed at 550 cm\(^{-1}\) assigned to C–C stretching vibration. The broad band appeared at 3440–3450 cm\(^{-1}\) is assigned to the -OH stretching band. The peak identified at 1500 cm\(^{-1}\) confirmed the presence of C–C aromatic ring. The appeared bands at 1040 and 1129 cm\(^{-1}\) corresponding to the stretching vibrations of sulfonic group [37]. The peak at 1550 cm\(^{-1}\) is related to aromatic C=C stretching. For AC/SPS sample, most of the above bands have noticed with a slight shift. These results prove the efficient synthesis of AC sample.

**XRD Patterns**

X-Ray Diffraction patterns have been performed in order to determine crystallinity of fabricated samples. The results of XRD analysis for AC, SPS and AC/SPS nanocomposite are shown in Fig. 2b. In all samples, appearance of broad diffraction peaks and absence of sharp peaks proves the dominant structure is amorphous. In XRD pattern of AC, a broad peak observed at 2\(\theta\) = 24.21\(^{\circ}\) correspond to the 002 plane (JCPDS 41–1487) can be observed which related to the amorphous carbon [44]. The intensity and position of peak indicates the low degree of graphitization. This indicates that the heating of biomass at high temperature causes the pyrolysis of organic substances into volatile products, which means that most of the non-carbon elements such as hydrogen, nitrogen and oxygen are removed in gaseous forms and leave a solid residue enriched in carbon. For SPS material, the relatively strong peak appeared at 2\(\theta\) = 19.61\(^{\circ}\) suggesting that the SPS have certain crystallinity. The X-ray analysis of AC/SPS sample indicates the appearance of peak centered at 2\(\theta\) = 20.94\(^{\circ}\) which may be attributed to overlap between AC and SPS particles. Moreover, the AC/SPS composite shows the characteristic peaks of both AC and SPS without any additional peaks. Furthermore, compared to SPS, the crystallization behavior in AC/SPS is not altered. The disappearance of diffraction peak belong to AC may be due to incorporation small ratio of AC. The appearance of only one diffraction peak in all samples, indicates the high purity of the prepared materials. The crystallite size of the AC, SPS, and AC/SPS particles were estimated from diffraction patterns by using Scherrer equation:

\[
D = \frac{K\lambda}{\beta \cos \theta}
\]

(3)

where D is crystallite size (nm), K is the Scherrer constant (0.96), \(\lambda\) is the wavelength of the X-ray source (CuK\(\alpha\) radiation, 0.15405 nm), \(\beta\) is the integral breadth which means the full width half maximum (FWHM) of the Bragg’s diffraction peak and \(\theta\) is the reflection angle (Å). Based on the data in Table 1, it can show that the diffraction peak width is narrow, then the half peak width value is small. The difference in the crystal size of AC/SPS indicates the effective interaction of the polymer and activated carbon by sulfonated groups in SPS.

| Samples | 2\(\theta\) | FWHM | D(nm) |
|---------|-----------|------|-------|
| AC      | 24.21736  | 2.74552 | 2.95823 |
| SPS     | 19.61717  | 4.28752 | 1.87964 |
| AC/SPS  | 20.94526  | 5.79201 | 1.39428 |

![Fig. 2 FT-IR spectra (a) and XRD patterns (b) of AC, SPS and AC/SPS](image-url)
**BET/BJH Analysis**

BET/BJH analyses were performed to investigate the textural properties of prepared materials. Figure 3 shows the N$_2$ adsorption–desorption isotherms and the pore size distribution curves of AC, SPS and AC/SPS samples. The values of BET surface area and pore size are listed in Table 2. As the BET surface area size was changed from 28.3474 m$^2$/g (AC) to 73.0349 m$^2$/g (AC/SPS), the value of pore volume increased from 0.025993 cm$^3$/g to 0.087205 cm$^3$/g. The SPS material had higher specific surface area (155.3987 m$^2$/g) and porosity (0.233187 cm$^3$/g) due to its advantage of having small particle size as conclude from XRD analysis. The decreasing BET surface area of AC/SPS in comparison with SPS, may be mainly due to the loading of AC particles cover most of the mesopores and micropores. As can be observed, the pore size distribution curves is almost narrow broad. The pore size distribution of the samples further indicates the formation of mesoporous materials (pore size 2 ~ 50 nm) [45]. Additionally, according to the classification of IUPAC, the N$_2$ sorption isotherms of three samples can be classified as type III with H$_3$ hysteresis loops which indicates unrestricted multilayer formation process and existence of slit-like mesoporous formed between aggregated nanoparticles [46].

**TEM Analysis**

Transmission electron microscopy technique (TEM) was used to determine the material morphological and size
properties. The TEM images of AC along with the SPS and AC/SPS are shown in Fig. 4. The TEM image of AC shows that aggregated particles had similar spherical shapes. The morphology of the AC is a porous multilayer texture, which is in agreement with the surface area results. This suggests that activation makes the carbon surface porous. The size of the prepared particles was calculated and the value is 88.45 nm. The SPS morphology is clear and pores with different sizes are formed as uneven surfaces, mostly like collapsed surface. The external edges of SPS exhibit little agglomerates with particle size of 23.58 nm. In TEM image of AC/SPS, it was noticed that the merging of AC with SPS alters particle shapes and sizes where the AC surfaces seem to be completely covered with SPS layers which means encapsulation of the AC with SPS. Accordingly, the mean particle size of AC/SPS composite was estimated to

![TEM images and grain size histograms of AC, SPS and AC/SPS samples](image)

**Fig. 4** TEM images and grain size histograms of AC, SPS and AC/SPS samples
be 71.84 nm. Additionally, the surface of AC/SPS seems to have large amount of interspaces in the order of mesostructured which indicates a highly disorganized porosity.

**SEM Analysis**

Scanning electron microscopy was used to characterize the structure and surface of prepared samples and the results are shown in Fig. 5. The surface of AC shows a uniform and porous surface. The chemical activation with H₃PO₄ and high temperature treatment were effective in creating porous surface. Thermal carbonization of Hibiscus *Sabdariffa L* leaves powder produced active carbon with porous surface. The SEM micrograph of SPS exhibits that sulfonating of PS created rough surface with morphology of irregular layers. Also, it is observed that the sample has a good homogeneity and no agglomerations were observed. The SEM image of AC/SPS shows obviously the rough and homogeneous surface. The sample shape is correlated to hydrophilic behavior results from agglomeration of sulfonic groups (-SO₃H) attached to aromatic ring of PS increase the average particle sizes due to swelling with water [47].

**TGA Analysis**

In order to investigate the thermal characteristics of as synthesized materials, a series of typical thermal capacity and stability curves were performed under air atmosphere by thermal gravimetric analysis and the results are shown in Fig. 6. In TGA profile of AC sample, three stages of weight loss have been noticed, the first one for the vaporization of moisture and ending at 180 °C. The second stage exhibits on increasing the temperature from 200 to 600 °C, there was a slight reduction in the weight of AC which assigned to decarbonation of the carbon structure. The third stage beginning at 650 °C and ending nearly at 900 °C is attributed to the thermal pyrolysis of carbon dioxide gas. It is noteworthy that the weight of the residue remains at 900 °C is almost 76% as AC nanoparticles [48, 49]. Thus, due to the AC residue was burned and activated at 650 °C, it may and have low ash content. For the SPS sample, it can be noted that the observed TGA profile exhibits that the weight loss proceeds in successive stages with raising temperature. The TGA curve of SPS material displays that the sample have three main weight loss stages, the first weight-loss step between 40 °C and 150 °C is mainly due to evaporation of residual moisture and sulfonic groups. The second stage beginning at 400 °C and ending at about 475 °C, which is a major weight loss, associated with 99% sample loss. The third step ending corresponding to the decomposition of polymer fractions [38]. AC/SPSC sample exhibits a total weight equal to 89%. Accordingly, the total weight loss of AC/SPS nanocomposite was found to be less than that of pure SPS material. Adding AC with 10% weigh ratio is significantly improved thermal characteristics of the polymer composite. Thus, the AC/SPS sample has better thermal stability compared to unmodified SPS sample.

**Raman Spectroscopy**

Figure 7 shows the Raman spectra of the, AC and AC/SPS samples. Since Raman spectrum of AC shows characteristic peaks are 1352 cm⁻¹ (D band) referred to as disordered carbon (sp³) and 1561 cm⁻¹ (G band) assigned to as graphitic carbon (sp²), this confirms the successful synthesis of AC. The Raman spectrum of AC/SPS nanocomposite exhibited two peaks at around 1321 cm⁻¹ (D band) and 1559 cm⁻¹ (G band), both peaks are related to the presence of AC. A significant reducing in the intensity of AC peaks was noticed in the prepared AC/SPS due to the low AC content. The intensity ratio (I_D/I_G) can be used to investigate the features of crystalline structures. The I_D/I_G ratios were calculated to be are 1.08, and 1.10 for AC and AC/SPS, respectively. The higher I_D/I_G ratios suggest an increase of disordered carbon (sp³) in comparison with graphitic carbon (sp²).

**Adsorption of RhB and CR onto AC/SPS**

**Effect of Contact Time**

Irrespectively of other operational factors that influence the adsorption efficiency, in any uptake process of water
contaminations, the contact time is a fundamental parameter that plays the major role to control the adsorption process. The removal efficiency of RhB and CR adsorbed by the AC/SPS was thoroughly conducted at an adsorbent dose of 0.01 g/25 ml, temperature of 25°C, pH of 7 and an initial RhB and CR concentration of 10 and 50 ppm, respectively. The contact time was continued for 120 min at a constant shaking velocity of 150 rpm. As shown in Fig. 8a, it can be observed that the adsorption efficiency increases as the process contact time increases. It can be noted that the uptake of dyes increases rapidly in the initial stage and becomes slow in later stage till saturation where there was no more dye molecules being adsorbed by AC/SPS. It can be observed that the equilibrium state was reached after 60 min of RhB and 20 min of CR, respectively. This is attributed to a large number of surface sites were available in the initial time and after progress of adsorption and saturation of active sites by the dye molecules, the remaining vacant surface sites were not allow more adsorption [50].
Effect of Adsorbent Dosage

Adsorbent dose is an effective parameter in adsorption process that can affecting the removal efficiency and adsorption capacity. In order to explore the influence of adsorbent dosage on adsorption efficiency, various adsorbent doses ranging from 0.04 to 0.4 g/l of AC/SPS were investigated for adsorption experiments at fixed initial pH of 7, initial RhB and CR concentration, 10 and 50 ppm, respectively and optimum contact time (30 min for RhB and 60 ppm for CR). The results shown in Fig. 8b exhibit that the increasing the AC/SPS dose of in the range of (0.04–0.4 g/l) caused an increase in removal efficiency. This may be attributed to the increase in the availability adsorption centers owing to the increase in the influent surface area resulting from the raise of Ac/SPS mass [51]. In addition, the decline in adsorption efficiency at the higher doses may be due to agglomeration of adsorbent particles or blocking of its surface active sites [52]. Accordingly, the removal efficiency of CR removal is steadily increases with the adsorbent loading up to 0.4 g/l to reaches its maximum 96.41%. On the other hand, the maximum adsorption efficiency of RhB was reached 25.57% using in 0.4 g/l of AC/SPS.

Effect of pH

The pH of the aqueous solution has been reported as the most important factor that controls the sorption process so it is necessary to be studied. The adsorbent surface acidity and basicity are intensely influenced by solution pH. The pH of contact solutions plays such a major role in the adsorption process mainly because of its contribution towards controlling the charge of the adsorbent surface and the degree of ionization of the adsorbate [53]. The effect of pH on the adsorption efficiency was investigated from pH range 4–11 using AC/SPS nanocomposite and the results are displayed in Fig. 8c. As the figure depicts, the highest Re% values of RhB were observed at higher pH. At high pH, the uptake of RhB is due to the interaction between the anionic surface of AC/SPS and the positively charged RhB molecules. Adsorption efficiency was observed to attain a highest of 31.15% at pH of 11 while a contrasting value of only 11.57% was noted at pH of 4. In contrast, Re% values of CR dye was calculated to be progressively declined as the pH increased towards a more basic media due to Congo red is an anionic dye, therefore it prefers acidic media for adsorption on the AC/SPS surface with higher efficiency. When the pH of the CR solution increases, the negative charges at the interfaces increase.
causing a decline in CR adsorption due to the more significant electrostatic repulsion between negatively charged dye molecules and negatively charged AC/SPS surface. The highest Re% of CR was reached value of 97.45%. As shown in Fig. 8d, the point of zero charge (pH_{zpc}) of the AC/SPS nanoadsorbent was estimated to be 7.1 meaning that the adsorbent’s surface is positively charged at pH below 7.1 and negatively at a pH above 7.1. At pHs below 7.1, a competition between H^+ ions and RhB adsorption causes a reduction of dye adsorption. On the other hand, the adsorption of CR dye will increase below 7.1 due to attractive interactions between negatively charged dye molecules and positively charged adsorbent’s surface. At pHs more than 7.1 where the adsorbent’s surface is predominantly positively charged, the adsorption will increase for RHB and decrease for CR.

**Effect of Temperature and Thermodynamic Study**

It is well known that temperature plays a vital role and can influence several features of dye adsorption. Reality, in most cases, temperature has a positive effect on the adsorption capacity which may be related with swelling effect of the internal surface of an adsorbent that had an effect on the internal structure of adsorbent. In addition, the mobility of dye molecules, the number of active sites and the interaction between the adsorbate and adsorbent, they are all affected by temperature. Effect of temperature on uptake of RhB and CR dyes from solution phase on AC/SPS surface was investigated at temperature range of 288–303 K for 120 min. As shown in Fig. 9a, the adsorption efficiency of both dyes increased with an increase in temperature which means that the adsorption process is an endothermic. Consequently, a gradual increasing of removal efficiency from 25.51 to 34.69% for RhB and from 95.15 to 98.11% for CR was noticed as the temperature was raised from 298 to 303 K. Increasing the adsorption efficiency with increasing temperature may be relates with the strength of the physical bonds between the active sites and dye molecules that makes the interaction between the solvent and dye molecule much weak than with adsorbent, that causes the uptake of dye molecules from aqueous solution. Based on the obtained results, the optimal temperature for adsorption process 303 K.

The thermodynamic functions are of great importance in the estimation of spontaneity and heat of adsorption process. Thermodynamic parameters of adsorption such as the change in Gibbs free energy (ΔG°), enthalpy change (ΔH°) and entropy change (ΔS°) were calculated using the Eqs. 4–7 [41]. The values of thermodynamic parameters are given in Table 3.

$$K_{eq} = \frac{Q_x V}{C_{x_m}} \quad (4)$$

$$G^\circ = -RT \ln K_{eq} \quad (5)$$

![Fig. 9 Effect of temperature on the RhB and CR adsorption onto AC/SPS (a) and Vant Hoof plot of RhB and CR adsorption on AC/SPS (b)](image)

| Table 3 | The thermodynamic parameters of the dye CR and RhB adsorption on AC/SPS |
|---------|--------------------------|
| Adsorbate | Temp. K | −ΔG° (kJ/mol) | ΔH° (kJ/mol) | ΔS° (J/mol k) | LnKeq |
| CR      | 288   | 7.13072      | 47.796       | 190.719        | 2.97804 |
|         | 298   | 8.06385      | 190.649       | 3.31028 |
|         | 308   | 9.12513      | 191.011       | 3.68309 |
|         | 318   | 9.95374      | 190.594       | 3.95124 |
| RhB     | 288   | 2.56509      | 21.430       | 65.5066       | -1.07128 |
|         | 298   | 2.26837      | 65.4014      | -0.93119 |
|         | 308   | 1.90733      | 65.5156      | -0.76984 |
|         | 318   | 1.59380      | 65.4692      | -0.63268 |
\[
\ln K = \frac{\Delta S^\circ}{R} - \frac{\Delta H^\circ}{RT}
\]  

(6)

\[
G^\circ = H^\circ - TS^\circ
\]  

(7)

where \( K_{eq} \) is the thermodynamic equilibrium constant for the adsorption process, \( q_e \) is the amount of dye adsorbed in solution (mg g\(^{-1}\)), \( C_e \) is the equilibrium concentration of dye solution (mg/l), \( T \) is the absolute solution temperature (K) and \( R \) is the universal gas constant (8.314 J/mol k).

At acidic pH media, functional groups presented on AC/SPS surface (phosphate, sulphate, hydroxyl, amine) are strongly protonated leading to the surface getting a positive overpowering charges, hence a strong interactions occurred between the CR-SO\(_3^-\) anions and the positively charged surface. In contrast, the modification of both polystyrene and carbonic source creates acidic functional groups such as the phosphate and sulfate on the on AC/SPS surface, thus this might drive the adsorption of positively charged RhB molecules onto positively charged AC/SPS surface. Due to low value of \( \Delta H^\circ \) for RhB adsorption, electrostatic attraction might possibly occur between the positively charged \( ^{+}\text{NH-CH}_3\text{RhB} \) molecules negatively charged AC/SPS surface [59].

### Adsorption Isotherms

Adsorption isotherms explain the modes of interaction of adsorbate molecules with surface of an adsorbent. In addition, the adsorption isotherms present an understanding of distribution of species in solid and aqueous phase at the equilibrium state. In current study, the data obtained on the adsorption of dyes have been analyzed with respect to Langmuir, Freundlich and Temkin models. The Langmuir, Freundlich, and Temkin models are generally employed in selecting adsorbents wastewater and industrial water remediation systems.

#### Langmuir Adsorption Isotherm

The basic assumption of Langmuir isotherm is based on monolayer, homogeneous and finite adsorption site assumptions. Therefore a saturation value is reached beyond which no further adsorption takes place and only one dye molecules could be adsorbed on site and no interaction between the molecules adsorbed on other sites. The Langmuir adsorption isotherm can be applied as equation below [11].

\[
q_e = \frac{q_m K_L C_e}{1 + K_L C_e}
\]

(8)

The linear formula of equation can be expressed as:

\[
\frac{C_e}{q_e} = \frac{C_e}{q_m} + \frac{1}{K_L q_m}
\]

(9)

where \( q_m \) represents the quantity of dye adsorbed per unit mass of adsorbent (mg g\(^{-1}\)), \( K_L \) is the Langmuir constant related to the free energy of adsorption (L mg\(^{-1}\)), \( C_e \) represents the concentration at equilibrium (mg/l). The \( q_m \) and \( K_L \) can be predicted from the slope and intercept of the plot between \( C_e/q_e \) vs \( C_e \). The essential feature of the Langmuir isotherm represented in term of a dimensionless constant known equilibrium parameter \( R_L \), which is defined by following equation [60]:
$R_L = \frac{1}{1 + K_L C_0}$

where $C_0$ is the initial concentration of adsorbate (mg/l), $K_L$ is the Langmuir constant. Figure 10a and b and Table 4 display the results of Langmuir adsorption isotherm of RhB and CR dyes. As results show, the values of $R_L$ refers to the type of Langmuir isotherm to be favorable, since the value of correlation coefficient ($R^2$) for RhB is more than CR, so the saturation with monolayer coverage of the adsorbate molecule at the outer AC/SPS surface applies more to RhB dye than CR dye. Therefore, it can be said that the adsorption of RhB on AC/SPS is described by Langmuir model. The value of $R_L$ is essentially important and defines the isotherm to be linear ($R_L = 1$), unfavorable ($R_L > 1$) and favorable

![Fig. 10 Langmuir isotherm (a and b), Freundlich isotherm (c and d) and Temkin isotherm (e and d) for the adsorption of RhB and CO onto AC/SPS at $T = 30 \, ^\circ C$](image)

| Isotherm model | Isotherm parameters | RhB     | CR     |
|----------------|---------------------|---------|--------|
| Langmuir      | $q_m$ (mg/g)        | 21.413  | 222.22 |
|                | $b$ (L/mg)          | 0.1358  | 1.4062 |
|                | $R_L$               | 0.4240  | 0.0102 |
|                | $R^2$               | 0.9227  | 0.892  |
| Freundlich     | $K_f$ (mg/g)        | 2.584   | 136.36 |
|                | $n$                 | 1.3614  | 1.5039 |
|                | $R^2$               | 0.9761  | 0.9974 |
| Temkin         | $K_T$ (L/mg)        | 2.1512  | 24.784 |
|                | $B$                 | 3.6763  | 38.181 |
|                | $R^2$               | 0.9361  | 0.8881 |
Adsorption data was also utilized on Freundlich isotherm model. The Freundlich model considers that active sites for adsorption are heterogeneous, and there is also a possibility that multilayers of adsorbate may be formed on the adsorbent surface in non-uniformly distribution. Also, Freundlich model hypothesized an equilibrium relationship between the adsorbate and adsorbent in case of multilayer adsorption. Freundlich isotherm is expressed by Eqs. 11 [61]:

\[ Q_e = K_F C_e^{1/n} \]  

(11)

The linear formula of Freundlich is represented as follows:

\[ \log Q_e = \log K_f + \left( \frac{1}{n} \right) \log C_e \]  

(12)

where \( K_f \) is the Freundlich isotherm constant (L/mg), \( n \) is the adsorption parameter related to the adsorption intensity. The values \( \frac{1}{n} \) and \( K_f \) can be obtained by plot of \( \ln q_e \) versus \( \ln C_e \). The linear plot of Freundlich isotherm of RhB and CR dyes is shown in Fig. 10c and d. By plotting \( \log Q_e \) versus \( \log C_e \), the Freundlich constants \( n \) and \( K \) were calculated from the slope and interception of the Freundlich plots, respectively. The obtained values of Freundlich parameters as well as the correlation coefficient (R²) were summarized in Table 4. The \( 1/n \) value refers to the flatness or steepness of slope. Value of \( 1/n \) near to 1 indicates the steeper slope which associates with greater adsorption capacity at equilibrium concentration. While, \( 1/n < 1 \) confirms the flattens of slope which express the lesser adsorption capacity at low equilibrium conditions [62]. This suggested that the Freundlich model presents a better fit to the experimental data than Langmuir, indicating the adsorption on a heterogeneous surface [63].

**Temkin Adsorption Isotherm**

Temkin isotherm supposes that the adsorption heat of all molecules in the layer decreases linearly with coverage due to adsorbate-adsorbent interactions. The linear formula of Temkin isotherm cab be represented by the following expression [64]:

\[ q_e = BlnK_f + BlnC_e \]  

(13)

where \( B = RT/b \), \( K_f \) represents the equilibrium binding constant (L/mg) corresponding to the maximum and constant binding energy, and \( B \) is related to the adsorption temperature. A plot of \( q_e \) versus \( lnC_e \) (Fig. 10e and f) enables the determination of the isotherm constants, where the isotherm parameters \( B \) and \( K_f \) are obtained from the slope and intercept, respectively. The Temkin isotherm parameters are given in Table 4. In general, on comparing the isotherms applied in the current study, the best fit for CR dye was obtained with the Freundlich isotherm, \( R^2 = 0.997 \) followed by the Langmuir, \( R^2 = 0.892 \), then the least the Temkin isotherm, \( R^2 = 0.888 \). On the other hand, the fitness order of RhB is different, where the best is Freundlich (\( R^2 = 0.976 \)) followed by Temkin (\( R^2 = 0.936 \)) and the least is Langmuir isotherm (\( R^2 = 0.922 \)) indicating that the Freundlich model exhibited the best fit to the experimental data and a multi-monolayer adsorption taking place on the adsorbent surface.

**Kinetic Study**

Adsorption kinetics have been used to investigate the mechanism of adsorption efficiency through full-scale batch process by different application of the kinetic model. The kinetics of adsorption is determined by assessing the removal of RhB and CR dyes from the aqueous solution at various time durations using pseudo-first order and second order equations. These models explain the relationship between the rate of sorption sites of the adsorbents are occupied and the number of unoccupied sites. The linearity of each plotted model indicates whether the model describes the adsorption process properly or not. The adsorption kinetics is very imperative from the viewpoint of process efficiency. The pseudo-first order kinetic model, also known as Lagergren model, assumes that the rate of change of adsorbate uptake with time is directly proportional to the difference in the saturation concentration and the amount of solute uptake with time. The Pseudo-first order model of Lagergren is given by equation [65]:

\[ \ln (q_e - q_t) = \ln q_e = k_1 t \]  

(14)

where \( q_e \) is the amount adsorbed at equilibrium (mg/g), \( q_t \) is the amount adsorbed at at time (mg/g), \( t \) is the time (min), and \( k_1 \) is the rate constant for the pseudo-first order sorption (tim⁻¹). The linear form was obtained by plot of log (\( q_e - q_t \)) against time to calculate the rate constant \( k_1 \) (Fig. 11a and b). The values of \( k_1 \) was obtained from slope and \( q_e \) from intercept are given in Table 5. The data obtained were show poor correlation coefficient indicating that rate of removal of RhB and CR onto adsorbent SPS/AC does not obey Pseudo –first –order model. Pseudo-second- order kinetic model assumes that the rate of adsorption of solute is proportional to the available sites on the adsorbent. The kinetic data of RhB and CR on AC/SPS can be obtained.
from pseudo-second–order model that expressed in its linear
formula equation [66]:

\[
\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e t}
\]  \tag{15}

where \( t \) is the time (min), \( q_t \) is the adsorption capacity (mg g\(^{-1}\)), \( q_e \) is the equilibrium sorption capacity (mg g\(^{-1}\)), and \( h \) (mg g\(^{-1}\) min\(^{-1}\)) is the initial sorption rate at \( t = 0 \). \( k_2 \) is the rate constant. From linear relationship between \( t/q_t \) and \( t \), the equilibrium adsorption capacity and \( k_2 \) can be calculated from slope and intercept, respectively and the results are shown in Fig. 11c and d. The values of the parameters and correlation coefficient are listed in Table 5. The correlation coefficients were found to be very high, that means the sorption of RhB and CR on AC/SPS followed the pseudo second order model. The pseudo second order model as a better model fitting the kinetics of the adsorption means that the adsorption mechanism depended on the adsorbent and adsorbate.

**Comparison of Efficiencies by AC-Polymer Nanoadsorbents**

A huge number of original research studies have been conducted to synthesis of activated carbon-based polymers. Many research studies on the modification of activated carbon by polymer chains have been performed for the preparation of specific adsorbents to improve the removal of organic pollutants. Table 6 give a literature review on AC and other materials as efficient adsorbents for removal of CR dye from aqueous media. As observed from Table 6, the adsorption
efficiency achieved in the current work can be compared with those resulted from other adsorptive removal studies. However, highly efficient and cost effective make AC/SPS promise material for water treatment applications. Furthermore, the facile synthesis route, and enhances the adsorption process make synthesized nanocomposite may be suitable for other applications, such as photocatalysis, air purification and decolorizing studies.

Conclusions

The current study demonstrated the synthesis of activated carbon from Hibiscus Sabdariffa L leaves using phosphoric acid as an activated agent. The study also included the preparation of sulfonated polystyrene employing sulfuric acid via treating of polystyrene waste with dilute sulfuric acid. In addition, activated carbon/sulfonated polystyrene nanocomposite (AC/SPS) was prepared in DMF solution. The prepared materials were analyzed using different characterization techniques where the success of preparation method has been proven. The fabricated nanocomposite was applied in removal of Rhodamine B and Congo red dyes by adsorption from aqueous media which exhibited an excellent adsorption for the Congo red. The highest adsorption efficiency of CR and RhB dyes onto AC/SPS was reached 98% and 34%, respectively. The best fit for both dyes was obtained with the Freundlich isotherm. Additionally, the thermodynamic investigation confirmed that adsorption of both dyes is endothermic and spontaneous. The adsorption enthalpy of CR and RhB dyes was estimated to be 47.79 and 21.43 kJ/mol, respectively. On the other hand, the kinetic results showed that the adsorption of RhB and CR dyes onto AC/SPS follows pseudo-first-order and pseudo-second-order kinetic models, respectively.

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Data Availability The datasets generated during and/or analysed during the current study are available from the corresponding author on reasonable request.

Declarations

Conflict of Interest Author Nahla S. Salman and Hassan A. Alshamsi declares they have no financial interests.

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