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The Removal of Azo Dyes from an Aqueous Solution Using NaOH-Activated Carbon from Phenolic Resin

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Liquid dyes are discharged usually from different industries, such as textile factories, into water bodies. They are toxic compounds that can cause significant harm to humans, animals, and aquatic life due to increased water pollution. For this reason, removing dyes from wastewater is of great importance to control their negative impact and meet the standards of water quality. In this study, activated carbons were produced from phenolic resin through a chemical activation process with NaOH at high temperatures. The sample that had highest porosity and pore volume was used to adsorb two types of azo dyes Remazol Brilliant Blue R (RBBR) and Remazol Yellow dye (RY) from an aqueous solution under different conditions, i.e., changing contact time, pH, dosage, initial concentration, and temperature. The maximum removals of RBBR and RY were recorded separately at low concentration (10 ppm), acidic condition ($pH < 7$), adsorbent dose of 0.3 g, temperature of aqueous solution 20°C and contact time of 120 min. The Freundlich adsorption isotherm model, with its higher correlation coefficient, provided a better fit for the equilibrium data compared with the Langmuir isotherm model. The activated carbon derived from phenolic resin, which is chemically activated with NaOH at 850°C, is a promising adsorbent for the removal of azo dyes from aqueous solutions. The optimum conditions can increase the efficiency of the adsorption process.

Keywords: wastewater treatment, azo dyes, adsorption, activated carbon.

Introduction

With the dawn of civilization, people have painted and coloured a variety of items, including clothing and some types of pottery, using dyes. All colorants that were utilized came from naturally occurring sources, including lichens, fungi, insects, and plants (Ardila et al., 2021). The quantity and quality of artificial dyes increased, and they became commercially produced at the beginning of the 20th century. This led to an expansion in the use of dyes for colouring textiles, paints, leather, cosmetics, pharmaceuticals, and foodstuffs (Deka et al., 2022). Because of their simplicity of use, colour stability, and washing resistance, azo dyes are used in textile treatment. However, the release of dye-laden wastewater into surface waters increases water pollution because it contains toxins that harm aquatic life and human health (Mohammed et al.,2013; Naseer et al., 2011). For instance, (Mohan) et al. 2007 referred that 50 % of the dye will be lost during the operation due to inadequate fixing rate. Thus, the use of aerobic and anaerobic microbial decomposition, coagulation, chemical oxidation, membrane separation processes, electrochemical dilution, filtration, flotation, softening, reverse osmosis, and other different methods for the removal of dye and pigment from wastewater are crucial for safeguarding the aquatic environment (Oribayo et al., 2020). Adsorption is one of those techniques that are attractive and promising because of how easy and inexpensive it is to use. Various substances, including zeolites, silica, MOFs, commercially activated carbons, and others, have previously been utilized as adsorbents for wastewater dyes. A variety of natural materials, including peat, bagasse pitch, lignite, and coal, as well as manmade ones like plastic, have been utilized as precursors for adsorbents (Azam et al., 2022). In general, physical activation and chemical activation are the two essential processes for the manufacture of activated carbon. Compared with physical activation, chemical activation has more advantages as the final carbon yields are higher, the process can be completed in one step at low tempertures and the porosity can be easily modified (Bergna et al., 2018). In this study, activated carbon with micro and mesoporous structures was produced from phenolic resin via activation with NaOH, and subsequently utilized for the adsorption of Remazol Brilliant Blue R (RBBR) and Remazol Yellow (RY) dyes from aqueous solutions. Experimental investigations were carried out under various conditions, including variations in contact time, pH, dosage, initial dye concentration, and temperature to determine the optimum conditions for the adsorption process. The objective is to examine the efficiency of a new adsorbent (NaOH-activated carbon from phenolic resin) in the removing azo dyes from an aqueous medium. The contribution of this study is focused on giving more understanding about the performance of activated carbons in the adsorption processes especially in removing azo dyes. Furthermore, the new synthesized adsorbent is applicable to examine the removing any other type of dyes from wastewater.

Materials and Methods

Reagents and materials required

The analytical-grade azo dyes, Remazol Brilliant Blue-R (RBBR) (molecular formula = $C_{22}H_{16}N_2Na_2O_{11}S_3$, M.W = 0.62654 kg/mol, λmax = 5.9×10^{-7} m) and Remazol Yellow (RY) (chemical formula of $C_{19}H_{19}N_{4-}S_3O_{11}Na_2$, M.W = 0.621 kg/mol, λmax = 4.2×10^{-7} m), both with 95% purity, were purchased from Basra Legend Company in Basra, Iraq. Additionally, all other chemicals, including sodium hydroxide (NaOH), hydrochloric acid (HCl), hexamethylenetetramine ($C_6H1_2N_4$), formaldehyde (CH_2O), and phenol (C_6H_6O), were sourced from the same company.

Analytical measurements

The surface morphology changes of selected samples were examined using a Quanta 600 Scanning Electron Microscope (SEM) in µm scale. The surface area and pore volume of the prepared adsorbent were determined using a Micromeritics Accelerated Surface Area and Porosimetry analyzer (ASAP 2420, Micromeritics Corporation). The pH of the dye solution was measured at room temperature by a digital pH meter (InoLab 7110, Wellheim, Germany). The unknown concentrations of dyes were estimated by measuring the absorbance using an UV/vis spectrophotometer (Ubi-600, MicroDigital Co., Ltd., Republic of Korea).

Preparation of dye solutions

1000 mL of RBBR stock solution was prepared by adding 1000 mg of RBBR dye powder in 1000 mL of distilled water and subsequently filtering the solution three times through filter papers to ensure purity. For further experimental work, standard solutions of RBBR dye with concentrations ranging from 10 to 450 mg/L were prepared using the dilution method. Using the same methods, RY dye solutions with concentrations ranging from 10 to 450 mg/L and pH levels between 2 and 13 were prepared.

Preparation of phenolic resins

Novolac-type phenol-formaldehyde resins, the precursors used in this work, were synthesized using a particular technique previously documented: "Formaldehyde and phenol were mixed to form a solution with a molar ratio of 0.3. A small quantity of 1 N HCl, constituting 1 wt% of the mixture, was subsequently added. The resulting mixture was stirred and placed in an Erlenmeyer flask equipped with a reflux condenser, where it was heated to 100°C for a duration of 4 hours. Following this, a crosslinking agent (hexamethylenetetramine) was incorporated at a concentration of 5 wt% of the mixture. The flask was then reheated to 150°C for approximately 8 hours in the presence of air. The resulting resin was then processed in an electric blender to produce a fine powder with particle sizes ranging from 1.2 to 1.4 mm" (Nakagawa et al., 2007). Finally, phenol formaldehyde resin (PFR) was obtained.

Activation process

The preparation of NaOH-activated carbons was conducted following a specific procedure detailed in previous literature: phenolic resin was dissolved in a 2:1 aqueous NaOH solution. The resulting solution was subjected to drying at 150°C for 1 hour in an air convection oven to remove moisture. Subsequently, activation occurred in a horizontal tube furnace (Elite Thermal Systems Ltd., UK), where the material was heated at a rate of 30°C/min to temperature ranging from 750 to 900°C under nitrogen. To neutralize the pH, the activated sample was washed thoroughly with distilled water and then with 0.5 M HCl. The final product was dried for a total of 13 hours at 150°C, consisting of one hour in an air convection oven and 12 hours in a vacuum oven (Yue et al., 2006). The phenol formaldehyde resin samples with NaOH were labelled $PFRN_{750}$, $PFRN_{800}$, PFR_{850} and $PFRN_{900}$.

Batch Experiment

To identify the ideal operating conditions for dye removal, adsorption tests were conducted in batch mode with a range of operational variables in the current study. The experimental procedure was carried out in three step process. Initially, solutions with concentration ranging from 10 to 450 ppm were prepared and stored in 50-mL Erlenmeyer flasks. Second, a 0.1 M NaOH/HCl solution was used to adjust the pH of the dye solution (250 ppm) to a range from 2 to 13. In the final step, 50 mL volumetric flasks were used to mix 25 ml of dye solutions to six amounts of the adsorbent (ranging from 0.1–0.6 g) which was heat treated at 850°C. Shaker incubator was used to shake the flasks at 150 rpm of speed. This shaking was carried out for seven durations ranging within 30–210 min and temperatures within25–70°C. Each solution was then filtered using filter paper to determine the dye's absorbance using a spectrophotometry. Each experiment was implemented three times to check the results, and the average values were recorded. The following formula was used to calculate the percentage of dye removal:

$$\text{Removal }\% = \frac{C_O - C_e}{C_O} \times 100 \tag{1}$$

where C_o is the starting concentrations of dye in solution (mg/L); and C_e is the dye concentrations at equilibrium (mg/L).

The amount of dye removal on the material at equilibrium, qe (mg/g), was calculated according to this equation:

$$\text{qe} = \frac{C_O - C_e}{\text{m}} \times \text{V} \tag{2}$$

where m adsorbent amount in g; and V solution volume in L.

Determination of pH_{PZC}

The point of zero charge (PZC) of the $PFRN_{850}$ was established. To adjust the pH levels to a range of 2 to 12, few drops of 0.1M HCl or 0.1M NaOH were added to 40 mL of deionized water contained in each of the 11 volumetric flasks. Subsequently, 0.1 g of phenolic resin at 850 $PFRN_{850}$ was added to each flask and agitated at 125 rpm for a duration of 24 hours at room temperature. After that, the solutions were filtered using Whatman papers. The differences between pH values were used to calculate PZC.

Results and Discussion

The textural characteristics of the modified activated carbons were investigated using ASAP 2420. *Table 1* displays the surface areas and pore volumes measured for the prepared activated carbon at various activation temperatures ranging from 750 to 900°C. The table presents the physical properties of $PFRN_{750}$, $PFRN_{800}$, $PFRN_{850}$, and $PFRN_{900}$.

In general, surface area and pore volume are crucial properties of adsorbents such as activated carbons, as they directly influence the material’s adsorptive capacity and its ability to store gases and liquids. According

Table 1. *Physical properties of $PFRN_{750}$, $PFRN_{800}$, $PFRN_{850}$ and $PFRN_{900}$*

Samples	S_{BET} (m^2/g)	V_{Micro} (cm^3/g)	V_{Meso} (cm^3/g)	V_{Total} (cm^3/g)
$PFRN_{750}$	1694	0.79	0.72	1.51
$PFRN_{800}$	1726	0.86	0.78	1.64
$PFRN_{850}$	1752	0.91	0.83	1.74
$PFRN_{900}$	1436	0.69	0.40	1.09

to *Table 1*, with an increase in activation temperature from 750 to 850°C, the BET surface area and total pore volumes for $PFRN_{750}$, $PFRN_{800}$, and $PFRN_{850}$ increased from 1694 to 1752 m^2/g and 1.51 to 1.74 cm^3/g, respectively. However, upon increasing the activation temperature to 900°C, a significant decrease was observed in the surface area and pore volume of $PFRN_{900}$, reaching 1436 m^2/g and 1.09 cm^3/g, respectively. This decrease may be attributed to pore wall collapse and shrinkage of the structural carbon matrix as a result of elevated temperature to 900°C. Previous studies have reported similar effects of activation temperature on the BET surface area and total pore volume of NaOH-activated carbon derived from different precursors (Cho et al., 1995; Khalili et al., 2000; Ohba et al., 2004; Slasli et al., 2004). Although $PFRN_{800}$ exhibited comparable BET surface area and pore volume to $PFRN_{850}$, the latter demonstrated a slight advantage. Given that the structural characteristics of materials significantly influence their ability to adsorb and store gases and liquids, the $PFRN_{850}$ sample was selected for further experiments.

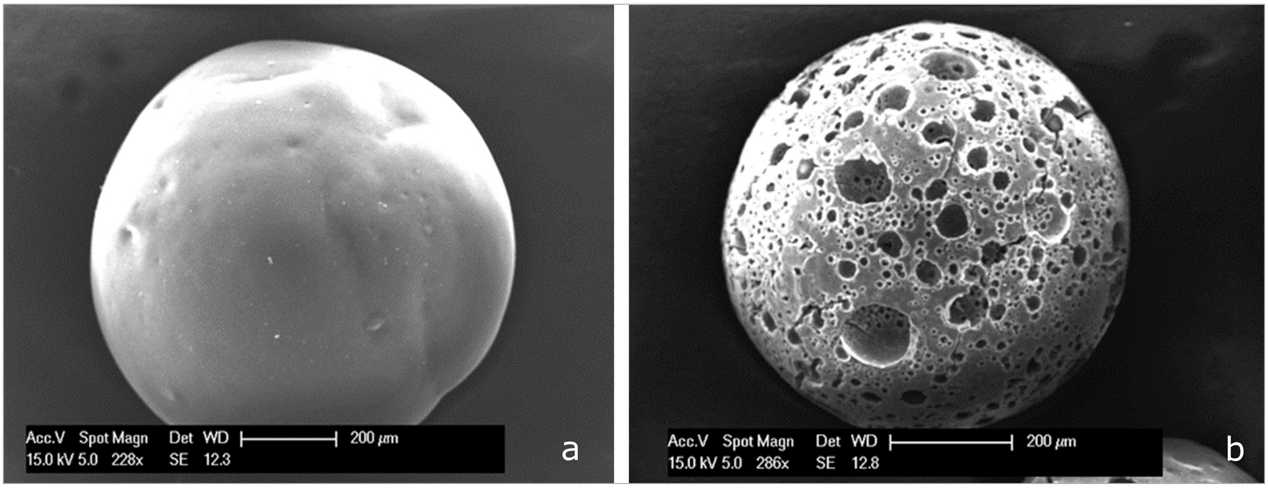
The SEMs images in *Fig. 1a* and *b* illustrate the evolution of the porosity of activated carbon (the sample $PFRN_{850}$ which showed a larger specific surface area and a larger total pore volume) after NaOH intercalation. As depicted in *Fig. 1*, the outer surface of the sample is characterized by numerous voids and cracks, indicating the effectiveness of the activation process in developing well-defined pore structures on the precursor's surface. Souza et al. (2009) referred that many small pores on the surface of activated carbon transform into mesopores after NaOH intercalation. NaOH was chosen as the chemical activation agent for several reasons:

1. Phenolic resin exhibits high solubility in aqueous NaOH solutions, facilitating the activation process (Yue et al., 2006).
2. NaOH activation results in a significant increase in mesoporous structure within the activated carbon (Liew et al., 2018).
3. NaOH is a more cost-effective option compared with KOH (Lillo-Ródenas et al., 2001).

It is worthy of note that the mechanism of reaction for NaOH and activated carbon exhibits similarities to that of KOH (Lillo-Ródenas et al., 2003).(Lillo-Ródenas et al., 2003).

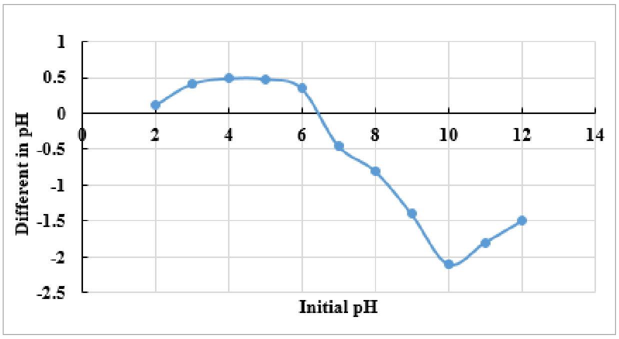
The pH values at zero charge were evaluated to ascertain whether the surface of $PFRN_{850}$ activated carbon is charged predominantly positively or negatively. Generally, the interaction between hydroxyl groups (OH^-) or protons (H^+) present in the solution with the adsorbent surface greatly affects the charge of that surface.

Fig. 1. *The results of SEM images at 200 x magnification*



The ion concentration on the activated carbon surface is crucial in determine its charge. The hydroxyl group represents the counter ion layer on the surface of the absorbent, resulting in a strong electrostatic attraction with the protons. This interaction results in a decrease in pH values. Under these conditions, the adsorption of dye onto the sample surface is enhanced. As shown in *Fig. 2*, the value of pHzpc for the sample is 6.51, indicating that the surface of PFRN$_{850}$ is slightly positively charged. Several studies (Xu et al., 2005; Mohammed et al., 2014; Zafar et al., 2009; Zafar et al., 2015) have reported that the pHpzc values for commercial activated carbons are in the range from 6.50 to 7.33.

Fig. 2. *The point zero charge of the PFRN$_{850}$ at 25°C*



By separately shaking 1.25 g of PFRN$_{850}$ with varying initial concentrations of RBBR dye and RY dye, the effect of the initial dye concentration was investigated. The results, depicted in *Fig. 3*, revealed that dye removal efficiency decreased with increasing initial dye concentration, eventually reaching equilibrium adsorption capacity for both dyes on the adsorbent. Notably, RBBR dye exhibited higher removal percentage at low concentrations (10–140 ppm) compared with RY dye. On the contrary, between 140 and 350 ppm, it was somewhat lower for RBBR and higher for RY, and the difference increased after that. At low concentration, although there are few dye molecules in the solution relative to the number of available active sites on the surface of PFRN$_{850}$, the adsorption is more frequent and intense. This means that the dye molecules have more opportunities to interact with the available surface sites.

More free dye molecules become available in the solution as the dye concentration rises, and quickly occupy the majority of active sites on the activated carbon surface. The differing reactions of the both dyes with PFRN$_{850}$ can be attributed to their distinct structures.

The pH of a solution plays a crucial role in every adsorption process as it influences both the charge density on the adsorbent surface and the ionization state of the adsorbate. In this study, we investigated the impact of altering the solution of pH from 2 to 13 on the adsorption behaviour of RBBR and RY dyes onto PFRN$_{850}$ using a pH meter for accurate measurement. The experimental parameters such as adsorbent dosage, contact time, initial dye concentration of the solution, and temperature were fixed at 1.25 g, 120 min, 250 ppm, and 25°C, respectively. The experimental results, as shown in *Fig. 4*, illustrate a significant reduction in the ability of both azo dyes to adsorb onto activated carbon as the pH increases. Optimal adsorption, 94.5% for RBBR and 91.2% for RY, occurred under acidic conditions at pH 2. As the pH increased to 7, the removal percentages slightly decreased to 87.6% for RBBR and 80.5% for RY. Further declines were observed as the pH increased from 7 to 12, with the minimum removal efficiencies of RBBR and RY occurring at pH 12. Importantly, a similar effect of pH was observed for both azo dyes. The strong electrostatic attraction between oppositely charged ions indicates that positively charged adsorbent surfaces are more effective at adsorbing

Fig. 3. *The effect of the initial concentration*

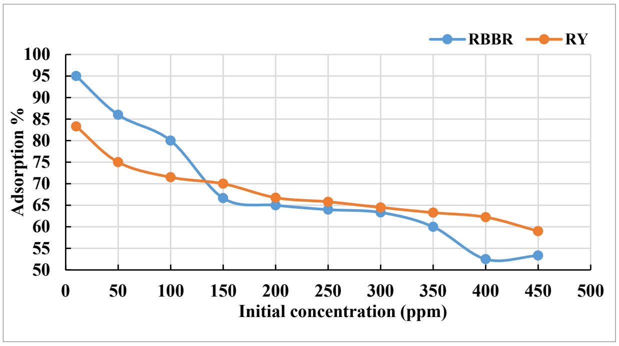
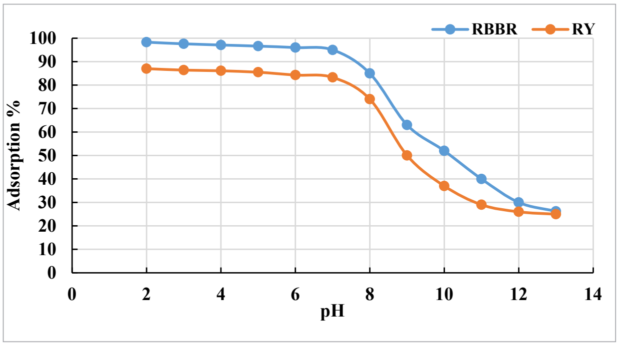


Fig. 4. *The effect of the pH*



and removing negatively charged azo dye molecules in acidic media (pH < 7). Conversely, as the pH transition into the basic range, the surface of the sorbent acquires a negative charge due to the deprotonation of its active sites by OH^- ions.

On the other hand, the adsorption efficiency is significantly influenced by the amount of the adsorbent used, showing a direct proportional relationship (Nazir et al., 2020). To evaluate the impact of changing the adsorbent dosage on the removing of dye from aqueous solutions, the amount of $PFRN_{850}$ was varied between 0.1 and 0.6 g. Each dose was shaken individually with 100 ml of a dye solution (RBBR and RY) at a fixed pH, temperature of 25°C, rotation speed of 120 rpm, and initial dye concentration of 250 ppm for 1 hour. As illustrated in *Fig. 5*, the percentage of dye removal for both RBBR and RY dyes increased significantly, reached 90.1% and 83%, respectively, as the adsorbent was raised from 0.1 to 0.3 g. This can be attributed to the availability of additional active sites on the adsorbent surface for adsorption as the dosage increases, leading to improved adsorption efficiency by accommodating more dye molecules. However, it is significant to note that after increasing the adsorbent dose from 0.3 to 0.7 g, the removal efficiency remained relatively constant. Therefore, the optimum initial adsorbent dosage is determined to be 0.3 g.

Fig. 6 indicates the adsorption efficiency of RBBR and RY dyes on $PFRN_{850}$ over different contact times at the optimum initial concentration. At t = 0, a large number of active sites were available for adsorption on the adsorbent surface. With increasing contact time, the adsorption efficiency increases as more of the available active sites are occupied by dye molecules, and are almost filled at equilibrium. The adsorption almost stop increasing at t = 120 min (*Fig. 6*).

Fig. 5. *The effect of adsorbent dosage (g)*

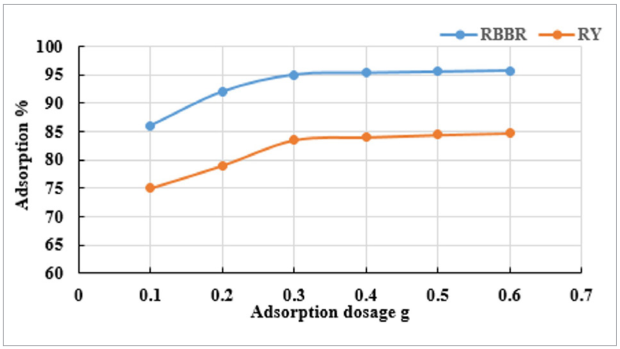


Fig. 6. *The effect of the contact time (min)*

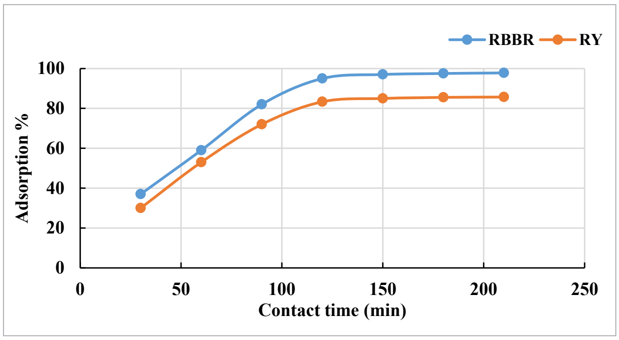
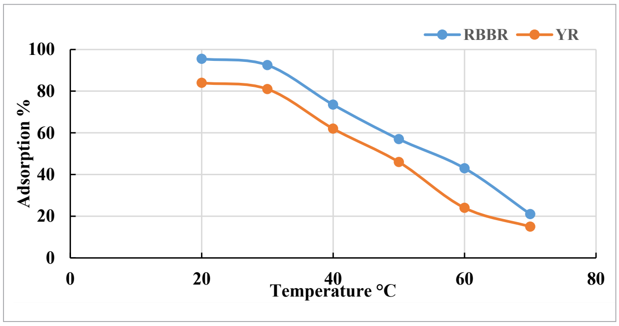


Fig. 7. *The effect of the temperature (°C)*



The relationship between the temperature of aqueous solutions and the efficacy of $PFRN_{850}$ in azo dye removal reveals a decrease in dye adsorption with increasing temperature, particularly evident between 30 and 70°C, as depicted in *Fig. 7*. This phenomenon can be attributed to the inverse proportionality between the kinetic energy of dye molecules and the intermolecular interactions between the adsorbate and the adsorbent (Khalili et al., 2000). Low temperatures negatively affect the kinetic energy of the dye molecules, allowing strong intermolecular attractive with the adsorbents, resulting in a higher retention of dye molecules on the adsorbent surface. With increasing temperature, the dye molecules gain greater kinetic energy, leading to a decrease in adsorption on the adsorbent surfaces. Therefore, the dye adsorption on the $PFRN_{850}$ sample decreases as the temperature increases.

Adsorption isotherms

The dye removal efficiency of the sample was evaluated under various conditions. In general, different adsorption isotherm models are used to describe the adsorption process between two phases, liquid and sold, at equilibrium and constant temperature. In this study, the obtained data were analysed using the Langmuir and Freundlich models. The Langmuir model describes monolayer adsorption, wherein the outer surface of the adsorbent (activated carbon in this study) can adsorb only one molecule of the adsorbate (dye), with no further adsorption occurring. The Langmuir equation is expressed as follows:

$$\frac{C_e}{q_e} = \frac{1}{q_e}KL + \frac{C_e}{q_m} \qquad (3)$$

where C_e and qe have the same meaning; q_m is the maximum adsorption capacity (mg/g); and KL is the sorption constant (L/mg) at a given temperature.

On the other hand, the Freundlich isotherm assumes multilayer adsorption on the surface of the adsorbent. Its linear form is expressed as:

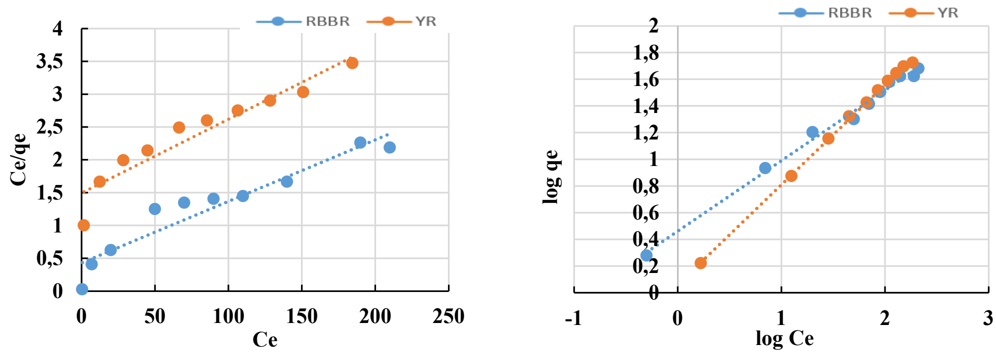
$$\log q_e = \frac{\log K_f}{2a} + \frac{1}{\mathrm{n}} \log C_e \qquad (4)$$

where K_f is the first Freundlich constant, which represents the adsorption capacity; and n is the second Freundlich constant, representing the adsorption intensity.

The parameter values were found from *Fig. 8*.

Experimental data from tests of RBBR and RY adsorption over $PFRN_{850}$ were analysed using the linear forms of isotherms (Eqs. (3) and (4)), as illustrated in Fig. 8. *Table 2* reveals that $PFRN_{850}$ exhibits a high adsorption capacity (q_m) for RBBR and RY dyes, with values of 106.4 and 90.11 mg/g, respectively, indicating its significant capability to remove azo dyes from aqueous media. The Langmuir isotherm constants (KL) for the two dyes were found to be 0.321 and 0.214 m^3/mg, respectively, as determined from the evaluation of the Langmuir isotherm plot (*Fig. 8a*) at 25°C. These values are comparable with those reported by Tilková et al. (2013). Furthermore, the slope and intercept from the log qe versus log C_e in *Fig. 8(b)* yielded the K_F and 1/n values, as detailed in *Table 2*. The elevated K_f values (2.898 and 1.153 mg/g) and n values (1.89 and 1.334) for RBBR and RY suggest that $PFRN_{850}$ possesses a strong affinity for azo dye molecules, rendering the Freundlich isotherm favourable for this type of adsorption process. Moreover, both isotherms gave high correlation coefficient (R^2), with the values of qe or log q_e showing a linear increase with C_e or log C_e, indicating that both models are suitable for describing the adsorption behavior of the dyes.

Fig. 8. *Experimental data of RBBR and RY over PFN_{850} at 25°C fitted to linearized forms of (a) Langmuir and (b) Freundlich isotherms*



(a) (b)

Table 2. *The adsorption coefficients of Langmuir and Freundlich models for the two dyes on the $PFRN_{850}$*

	Langmuir			Freundlich		
Dye	qm (mg/g)	KL (mg/l)	R^2	Kf(mg/g)	n	R^2
RBBR	106.4	0.321	0.9081	2.898	1.89	0.9925
RY	90.11	0.214	0.9073	1.153	1.334	0.999

Conclusion

This study described the synthesis of NaOH-activated carbons derived from phenolic resin using sodium hydroxide chemical activation in the 750–900°C range, and evaluated its effectiveness in removing azo dyes (RBBR and RY) from aqueous solutions. Increasing the temperature up to 850°C. Increasing the temperature up to 850°C enhanced the specific surface area and pore volume of the samples due to pore development. The findings indicate effective adsorption of both types of azo dyes onto the $PFRN_{850}$ surface. The study evaluated the impact of various system variables on azo dye removal and observed that the percentage of removal increased with longer contact times and higher amounts of adsorbent, but decreased with higher pH levels, aqueous solution temperatures and initial dye concentrations. Additionally, the pHpzc of the $PFRN_{850}$ surface was determined to be 6.51. Notably, when the solution pH was below the pHpzc, removal rates of RBBR and RY dyes were relatively high, possibly indicating an abundance of positive charges on the carbon surface.

Although both Langmuir and Freundlich adsorption isotherm models were applicable for fitting the data and estimating model parameters. However, the Freundlich model demonstrated superior performance in describing the equilibrium data, as indicated by a high correlation coefficient ($R^2 = 0.99$). This suggest that the heterogeneous adsorption occurs on the surface of $PFRN_{850}$. Consequently, $PFRN_{850}$ proves to be a promising adsorbent for the removal of azo dyes from aqueous solutions.

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