



Valorization of Municipal Sewage Sludge into Activated Carbon for Cationic Dye Removal

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Abstract

This study demonstrates the potential of municipal sewage sludge collected from the wastewater treatment plant of Sidi Bel Abbès, Algeria, as a low-cost precursor for the production of activated carbon for the removal of Bezacryl Red GRL 180, a cationic textile dye, from aqueous solutions. The prepared material exhibited a synthesis yield of 69.3%, a low residual moisture content of 0.98%, an ash content of 1.9%, and an iodine number of 1129.5 mg g⁻¹, indicating promising characteristics for adsorption applications. FTIR and EDS analyses further revealed modifications in the surface functional groups following carbonization and phosphoric acid activation, while confirming the persistence of certain inorganic constituents in the resulting material. The adsorption experiments demonstrated that the removal efficiency depended on the adsorbent dosage, contact time, and solution pH. Under the investigated conditions, a dosage of 0.30 g of activated carbon achieved approximately 98% dye removal from a solution containing 0.05 g L⁻¹ Bezacryl Red, with the adsorption process reaching an apparent equilibrium after approximately 30 min. High removal efficiencies were also observed under acidic conditions, highlighting the importance of solution pH in the adsorption process. Kinetic modeling showed that the pseudo-second-order model provided the best fit among the investigated kinetic models, with coefficients of determination ranging from 0.97 to 0.98. Regarding adsorption equilibrium, the Langmuir and Freundlich models both described the experimental data well, with reported coefficients of determination of approximately 0.98, while the Temkin model showed a slightly lower value. The thermodynamic results reported in this study were consistent with a spontaneous and exothermic adsorption process over the investigated temperature range, although the thermodynamic parameters should be verified for internal consistency before final publication. Taken together, the findings highlight the potential of sewage-sludge-derived activated carbon as an alternative adsorbent for cationic dye removal and demonstrate the environmental value of converting a wastewater-treatment residue into a useful material. This approach could contribute to more sustainable sludge management while supporting the development of accessible treatment materials for dye-contaminated water. Further work should include detailed textural characterization, assessment of the influence of coexisting pollutants, regeneration and reuse tests, and evaluation of potential metal leaching from the sludge-derived material before its application in real textile wastewater treatment.

Keywords: sewage sludge; Activated carbon; Cationic dye; Bezacryl Red GRL 180; Adsorption; Waste valorization; Wastewater treatment.

Introduction

The valorization of sewage sludge generated from wastewater treatment plants represents a major environmental and economic challenge. Although generally regarded as a waste product, sewage sludge can be transformed into value-added materials, such as activated carbon. In the present study, the sludge used was collected from a municipal wastewater treatment, where it was generated during the biological treatment of wastewater. (H. Martínez-Alvarenga, 2026)

According to the National Sanitation Office (ONA) in Algeria, 60% of sewage sludge from a municipal wastewater treatment of Sidi Bel-Abbes (Algeria) is disposed of in landfills, 25% was used in agriculture, and 15% is stored. According to statistics from the Ministry of Water Resources, the 165 wastewater treatment plants currently generate approximately 250,000 tons of

sludge per year, with a projected production of 400,000 tons by 2020. These figures highlight the urgent need to consider appropriate and sustainable sludge valorization strategies (Mhamed, 2017).

The objective of this experimental work was to prepare activated carbon from sewage sludge through a multistep process involving sludge collection and drying, followed by carbonization and subsequent activation, either by physical or chemical means, to develop a porous structure suitable for adsorption. This transformation aims to convert an abundant waste material into a functional adsorbent, particularly for water treatment applications.

Within the framework of this study, several experimental parameters were investigated to optimize the preparation conditions of activated carbon from sewage sludge. The effects of the adsorbent dosage, solution pH, and contact time between the activated carbon and the adsorbate solution were examined to evaluate their influence on adsorption performance.

In parallel, various physicochemical characterizations were carried out on the resulting activated carbon. These included the determination of the production yield, burn-off (the percentage of material lost through combustion), and residual moisture content of the material. Additional instrumental analyses were also performed to identify the surface functional groups using Fourier-transform infrared spectroscopy (FTIR) and to determine the elemental composition by Energy-dispersive X-ray spectroscopy (EDS). These analyses provide a better understanding of the chemical and structural properties of the prepared activated carbon and contribute to evaluating its potential for adsorption applications.

The investigation of the operating parameters affecting the adsorption process highlighted the crucial role of several factors, particularly adsorbent dosage, contact time, and solution pH, in the removal of the cationic dye Bezacryl Red from aqueous solutions. Adsorbent dosage determines the availability of active sites capable of retaining dye molecules, while contact time makes it possible to monitor the evolution of the adsorption process until equilibrium is reached. Solution pH is also an important parameter, as it can influence the surface properties of the adsorbent and the interactions between the adsorbent and the species present in the solution. Furthermore, modeling the experimental data using the Langmuir, Freundlich, and Temkin adsorption isotherms provides further insight into the equilibrium behavior of the adsorbent–adsorbate system. The Langmuir model assumes monolayer adsorption on a homogeneous surface with equivalent adsorption sites, whereas the Freundlich model is an empirical approach that describes adsorption on heterogeneous surfaces with sites exhibiting different affinities. The Temkin model, in turn, considers adsorbate–adsorbent interactions and assumes that the heat of adsorption decreases linearly with increasing surface coverage. Comparing these three models through their characteristic parameters and coefficients of determination makes it possible to identify the model that best describes the experimental data and to gain a better understanding of the adsorption characteristics of the dye onto activated carbon derived from municipal sewage sludge. Therefore, the combined investigation of operating parameters and adsorption isotherm models contributes to a better understanding of the dye removal process and helps assess the potential of this material for treating contaminated water.

Experimental:

Pre-treatment of the raw material:

The material used for the preparation of the activated carbon was dried sewage sludge collected from the municipal wastewater treatment of Sidi Bel Abbès, Algeria. The sludge samples were collected in January 2025 (Fig.1).



Figure 1. Dried sewage sludge collected from the wastewater treatment plant of Sidi Bel Abbès, Algeria.

First, the sewage sludge collected from the drying beds of the wastewater treatment plant was manually ground using a mortar and pestle. The ground material was then sieved to obtain a uniform particle size suitable for the subsequent treatment steps.

Carbonization was carried out in a Nabertherm muffle furnace at 600 °C for 3 h. After the carbonization process, the material was allowed to cool under an inert atmosphere to prevent oxidation, typically under nitrogen or another inert gas. The sewage sludge (SSB) was chemically activated using phosphoric acid (H_3PO_4) as the activating agent. The selection of the activating

agent is generally determined by the nature of the precursor material, as certain activating agents are better suited to specific precursors, as well as by the properties required for the final product.

The sewage sludge powder (SSB) was mixed with 50% H_3PO_4 (water–acid solution) and stirred for 1 h at 100 °C. The resulting mixture was then subjected to vacuum filtration, and the activated material was stored in hermetically sealed containers until further use (Boudia, Mimanne, Benhabib, & Pirault-Roy, 2019).

The residues resulting from the carbonization process were removed by washing the material with 10% hydrochloric acid (HCl) using a vacuum filtration setup. The sample was subsequently washed thoroughly with distilled water, while the pH of the filtrate was monitored after each washing step until neutral pH was reached. Finally, the treated carbon was dried in an oven at 105 °C for 24 h. The resulting material was designated as activated carbon (SSA).

Characterization of Sewage Sludge-Derived Activated Carbon

Synthesis Yield

The synthesis yield is an important parameter used to determine the amount of activated carbon obtained from a given quantity of raw material (raw sewage sludge).

The synthesis yield is defined as the ratio of the mass of the obtained activated carbon, (m_1), to the initial mass of raw sewage sludge, (m_0), expressed as a percentage:

$$Y = \frac{m_1}{m_0} \cdot 100 \quad (1)$$

- **Y:** synthesis yield (%)
- **m_1 :** mass of the obtained activated carbon (g)
- **m_0 :** initial mass of raw sewage sludge (g)

Burn-off

The burn-off, also referred, is an important parameter used to quantify the mass loss of the precursor material resulting from the activation process. It represents the mass loss between the raw sewage sludge subjected to carbonization without chemical activation and the sludge-derived carbon impregnated with phosphoric acid and subsequently subjected to chemical and thermal activation.

The burn-off can be expressed as follows:

The burn-off, also referred to as the degree of activation, represents the mass loss of the carbonized material resulting from the chemical and thermal activation processes.

Ash content

The ash content was determined by standard method (CEFIC 1986). 0.5 g of activated carbon, with an average particle size of 250 μm , was dried at 80 °C for 24 h and placed in previously weighed ceramic crucibles. The samples were heated in a muffle furnace at 650 °C for 3 h.

The crucibles were then cooled to room temperature and weighed. The ashing rate was calculated using the following equation:

$$\text{Ash (\%)} = \frac{Wm_3 - Wm_2}{Wm_1} \times 100 \quad (2)$$

Where:

- Wm_1 : weight of the original sample used (g),
- Wm_2 : weight of crucible containing a dried sample (g),
- Wm_3 : weight of crucible containing an original sample (g)

Moisture content

The moisture content was determined using oven drying method (Adekola & Adegoke 2005). 0.5 g of activated carbon was placed in a previously weighed ceramic crucible. The sample was dried at 110 °C until the weight was stable.

Thereafter, the sample was cooled to room temperature and weighed. The moisture content was calculated using the following equation:

$$\text{Moisture (\%)} = \frac{Wm_3 - Wm_2}{Wm_1} \times 100 \quad (3)$$

Iodine adsorption capacity

The iodine adsorption capacity of activated carbon produced from sewage sludge (was determined 10 mL of 0.01 N iodine solution was titrated with 0.1 N sodium thiosulfate solution in the presence of a starch solution as an indicator until it became colourless. The reading volume corresponds to V_b . 0.05 g of activated carbon produced from sewage sludge are mixed with 15 mL of an aqueous solution of iodine (0.1 N) and stirred vigorously for 5 min. (Boudia, Mimanne, Benhabib, & Pirault-Roy, 2019)

The mixture is titrated with a standard sodium thiosulfate solution using a starch solution as an indicator, keeping the burette reading corresponding to V_s . The iodine number was then calculated by using the following equation:

$$\text{I.N} = \frac{(V_b - V_s) \cdot N \cdot (126,9) \left(\frac{15}{10}\right)}{M} \quad (4)$$

I.N: iodine number (mg.g^{-1})

V_b and V_s : volume of sodium thiosulfate solution required for blank and sample titrations respectively (mL).

N: normality of sodium thiosulfate solution (mol.L^{-1})

126,9: atomic weight of iodine (g.mol^{-1}).

M: mass of activated carbon produced from sewage sludge (SSA) used (g).

FTIR analysis

The surface functional groups were studied by FTIR spectroscopy (FTIR Perkin Elmer Universal ATR Sampling Accessory). The FTIR spectra of the raw material and the resulting activated carbon were recorded between 650 and 4,000 cm^{-1} in PerkinElmer spectrometer.

Fourier-transform infrared (FTIR) spectroscopy was used to characterize the surface functional groups of the untreated sewage sludge and the activated carbon produced from the same precursor. Two samples were analyzed: SSB before activation, corresponding to the raw sewage sludge without any treatment, and SSA, corresponding to the sewage sludge-derived activated carbon obtained after carbonization and chemical activation. The objective of this comparative analysis was to identify the main functional groups present in the raw and treated materials and to evaluate the structural and chemical modifications induced by the carbonization and activation processes.

spectroscopy (EDS)

The elemental composition of the prepared sewage sludge-derived activated carbon was determined by energy-dispersive X-ray spectroscopy (EDS) coupled with scanning electron microscopy (SEM). A small amount of the dried activated carbon sample was mounted on a suitable sample holder and introduced into the SEM chamber for analysis. The EDS detector was used to collect the characteristic X-rays emitted by the sample under electron-beam irradiation, allowing the qualitative identification of the elements present in the material. The resulting EDS spectrum was recorded as a function of X-ray energy (keV), and the characteristic peaks were used to identify the corresponding elements. The analysis was performed on the activated carbon obtained after carbonization and subsequent chemical activation with phosphoric acid.

Adsorbate:

Bezacrly Red GRL 180 is a cationic textile dye used in the coloration of synthetic and other textile materials. It is a water-soluble dye with the molecular formula $\text{C}_{18}\text{H}_{23}\text{Cl}_3\text{N}_6\text{Zn}$ and a molecular weight of 495.16 g mol^{-1} . The dye is identified by CAS No. 38845-47-5, EC No. 254-149-3, and C.I. 110825. Bezacrly Red GRL 180 contains approximately 3.7% zinc and exhibits a characteristic maximum absorption wavelength (λ_{max}) at approximately 530 nm, which can be used for its quantitative determination by UV–visible spectrophotometry.

Therefore, the removal of Bezacrly Red GRL 180 from aqueous solutions is of particular interest in wastewater treatment studies, especially through adsorption processes using low-cost and environmentally sustainable adsorbents.

Adsorption study

A solution of 0.05 g.L^{-1} Bezacrly red was prepared by dissolving an appropriate amount of dye, which was diluted to the required concentration. Various parameters that influence the adsorption process have been optimized. The optimal contact time is determined by stirring 0.5g of SSA with 50 ml of a solution of Bezacrly Red 0.05 g.L^{-1} at pH 5,6 at times ranging from 5 to 60 min.

The effect of the adsorbent dose is investigated by varying the amount of SSA from 0.05 to 0.9 g in 50 ml of 0.05 g.L^{-1} of dye solution at pH 6,8 and 0.5 g of SSA was mixed with 50 ml of Bezacrly red solution 0.05 g.L^{-1} at different pH values between 2 and 12 (the pH of the solution was adjusted using NaOH and HCl 0.1N) in order to studied effect of pH. To study the effect of the initial concentration of this dye, a mass of 0.5 g of

SSA is mixed with 50 ml of the dye solution at different concentrations (0.01 to 0.07 g.L^{-1}).

The effect of temperature on the adsorption phenomenon was studied by varying the temperature from 8 to 75 °C maintaining the dye concentration at 0.05 g.L^{-1} , the time at 60 min, the mass of the adsorbent at 0.5 g and the pH of the solution at 2.

The dye concentration in supernatant solution was determined at characteristic wavelength BR λ_{max} ¼ 500 nm; by UV–visible spectrophotometer (Perkin Elmer UV-vis Lambda 45).

The percentage removal of the dye and the amount of dye adsorbed on the adsorbents (q_e) was calculated using this Equations (4)and (5), respectively:

$$\text{Bezacrly Red Removal (\%)} = \frac{C_0 - C_e}{C_0} \cdot 100 \quad (5)$$

$$q_e = \frac{(C_0 - C_e) \cdot V}{m} \quad (6)$$

where:

- q_e : amount of dye adsorbed on adsorbent at equilibrium(mg.g^{-1});
- C_0 : initial equilibrium concentration of dye in solution(mg.L^{-1});
- C_e : equilibrium concentration of dye in solution (mg.L^{-1});
- V : volume of the solution (L);
- m : mass of adsorbent (g).

Kinetics studies

Pseudo-first-order Equation (6), pseudo-second-order Equation (7) and intraparticle diffusion model Equation (8) were tested (Mckay1984; Ho & McKay 2000; Li et al. 2009).

$$\ln(q_e - q_t) = \ln q_e - k_1 t \quad (7)$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} \quad (8)$$

$$q_t = k_i t^{0.5} + C \quad (9)$$

where:

- q_e and q_t : adsorption capacity of the adsorbate (mg.g^{-1}) at equilibrium and at time t (min);
- k_1 and k_2 : pseudo-first-order and pseudo-second-order rate constant;
- k_i : intraparticle diffusion rate constant;
- C : the intercept.

Studies of adsorption isotherms models

The adsorbed amount of Bezacryl Red on SSA as a function of equilibrium concentration was determined as mixed 0.5 g of SSA with 50 ml of Bezacryl Red solution with various initial concentration 0,01-0,02-0,03-0,04-0,05-0,07 (g/l) at pH =2 and room temperature.

To determine the maximum BR capacity of SSA, the equilibrium isotherm data were correlated by the Langmuir (Langmuir, 1918);Freundlich (Freundlich, 1906); Temkin (Temkin, 1940). These equations can be written as

Equations:

$$q = \frac{q_m K_L C_e}{1 + K_L C_e} \quad (10)$$

$$q = K_F C_e^{1/n} \quad (11)$$

$$q = \frac{q_m R T}{\Delta Q} \ln(K_T C_e) \quad (12)$$

where:

- q_m : saturated adsorption capacity of the adsorbate (mg.g^{-1});
- K_L : constant of Langmuir isotherm (L.mg^{-1});
- K_F and n : parameters Freundlich isotherm;
- K_T : Temkin constant (L.mol^{-1});
- ΔQ : variation of adsorption energy (J.mol^{-1}).

Thermodynamic parameters

Gibbs free energy ΔG° (KJ.mol^{-1}), standard enthalpy ΔH° (KJ.mol^{-1}) and standard entropy ΔS° ($\text{J.mol}^{-1}.\text{K}^{-1}$) could provide information about the mechanism of adsorption and were determined using the Van't Hoff equation (Naim Hasani, 2022):

$$\Delta G^\circ = -RT \ln k_d \quad (13)$$

$$\Delta G^\circ = \Delta H^\circ - T \Delta S^\circ \quad (14)$$

$$\ln k_d = -\frac{\Delta H^\circ}{RT} + \frac{\Delta S^\circ}{R} \quad (15)$$

where:

- R : universal gas constant ($8.314 \text{ J.mol}^{-1}.\text{K}^{-1}$);
- T : absolute temperature ($^\circ\text{K}$);
- K_d : thermodynamic distribution coefficient.

Results And Discussion

Table 1 | Chemical and textural properties of SSA

Characteristics	Values
Synthesis Yield %	69,3
Burn-off %	68,4
Ash content %	1,9
Moisture content (%)	0,98
Iodine number (mg.g^{-1})	1129.5

- The yield indicates that 69.3% of the initial dry mass of the sewage sludge was retained after the thermal or chemical activation step. (Fidel Vallejo, 2025)
- This represents a relatively high yield, indicating that the activation process, whether chemical or physical, did not result in excessive material loss; and suggests good retention of the carbonaceous framework, which is favorable from both an economic and structural perspective, as it indicates that a substantial proportion of the initial material was retained after treatment (John Chagu, 2026).
- A burn-off value of 68% indicates a substantial conversion of the initial carbonaceous material during activation, with a large fraction of the char mass being removed. This relatively high burn-off reflects the strong extent of the activation process and the resulting transformation of the original carbon matrix. Similar studies have shown that increasing burn-off is associated with greater carbon conversion during physical activation. (K. Phothong, 2021); (J. Li, 2024)
- The ash content of the activated carbon obtained in this study was 1.9%. This value is considerably lower than the 29.96% reported for pure sewage-sludge pellets, which is attributed to the high inorganic fraction of sewage sludge, including minerals, salts, and residual metals (Zanini, (2026)). The relatively low ash content obtained in the present study may be associated with the treatment and acid-washing steps applied during the preparation of the activated carbon. (Sang Youp Hwang, 2020)

- The activated carbon exhibited a low moisture content of 0.98%, indicating that only a small amount of water remained in the material after the drying process. This low moisture level is consistent with previous studies on sludge-derived activated carbons, where low residual moisture contents have been reported after drying and thermal treatment. For example, activated carbon prepared from dried sewage sludge showed a moisture content of 3.2% (Min et al., 2018).
- The iodine number of the prepared activated carbon was 1295 mg I₂·g⁻¹, indicating a high iodine adsorption capacity and suggesting a well-developed microporous structure. This value is relatively high compared with those reported for several sewage-sludge-derived activated carbons. For example, (Carreño Hernández, Synthesis of activated carbon from sewage sludge using phosphoric acid as activating agent. , (2025).)reported iodine numbers of approximately 571–963 mg·g⁻¹ for sewage-sludge-based activated carbons prepared using different chemical activating agents. The higher value obtained in the present study therefore suggests an effective development of iodine-accessible microporous domains during the activation process.

FTIR analysis

Comparison of the FTIR spectra of SSB and SSA provides information on the evolution or disappearance of characteristic absorption bands associated with organic matter, mineral components, and oxygen-containing surface functional groups. Changes in band intensity and position after treatment can be attributed to the decomposition of thermally unstable organic constituents during carbonization and to the chemical modifications occurring during the subsequent activation and washing steps. The FTIR results therefore contribute to assessing the transformation of the raw sewage sludge into a more carbonaceous and chemically modified activated material.

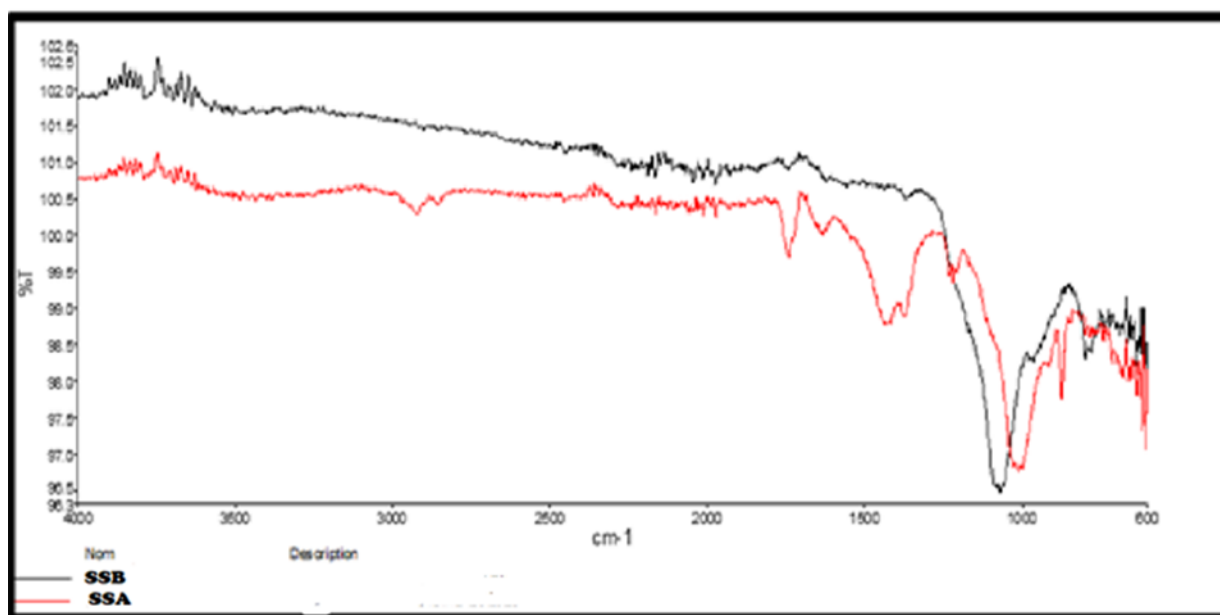


Figure 2: the FTIR spectra of SSB and SSA

- The broad absorption band observed in the 3200–3600 cm⁻¹ region is mainly attributed to O–H stretching vibrations associated with hydroxyl groups and adsorbed water. The decrease in this band after carbonization (SSA) and activation indicates a modification of the hydroxyl-containing surface functionalities and a reduction in moisture-related contributions. Similar changes in O–H-related FTIR bands have been reported for sewage-sludge-derived carbonaceous materials following thermal treatment (Bello, (2025).)
- The weak bands observed around 2920–2850 cm⁻¹ are attributed to the stretching vibrations of aliphatic C–H groups, mainly –CH₂ and –CH₃. Their attenuation after carbonization and activation for (SSA) indicates the degradation of thermally unstable aliphatic structures during the thermal treatment. Similar behavior has been reported for sewage sludge (SSB), where the bands near 2930 and 2853 cm⁻¹ progressively disappeared during pyrolysis as a result of the decomposition of unstable aliphatic structures (Hu, Zhang, & Liu, 2026)
- The intense absorption in the 1000–1100 cm⁻¹ region is particularly relevant and may result from overlapping vibrations associated with C–O groups and inorganic Si–O and P–O bonds, reflecting the complex organic and mineral composition of the sewage-sludge-derived activated carbon (SSA). (Tsvetkov, 2020) (Peng Cheng, 2023)
- The intense absorption in the 1000–1100 cm⁻¹ region is particularly relevant and may arise from overlapping vibrations associated with C–O groups and inorganic Si–O and P–O bonds. This complex spectral region reflects the coexistence of organic and mineral constituents in sewage-sludge-derived activated carbon. In particular, (Bello, (2025).) reported a band at approximately 1043 cm⁻¹ associated with phosphate species in sewage-sludge-derived activated carbon, supporting the contribution of phosphorus-containing groups following chemical activation.
- Overall, the observed spectral changes indicate a substantial transformation of the organic fraction during carbonization and H₃PO₄ activation, while mineral and phosphorus-containing species remain detectable in the resulting activated carbon (SSA).

- These spectral changes are consistent with the thermal conversion of the raw sludge into a more carbonized and chemically modified material. Nevertheless, FTIR characterization alone does not establish the adsorption performance of (SSA) ; its suitability as an adsorbent should be confirmed through adsorption experiments and complementary textural characterization.

EDS analysis:

Energy-dispersive X-ray spectroscopy (EDS) was used to investigate the elemental composition of the raw sewage sludge (SSB) and the activated material (SSA). This analysis provides qualitative and semi-quantitative information on the elemental constituents detected in the samples and allows comparison of their elemental profiles before and after carbonization and chemical activation.

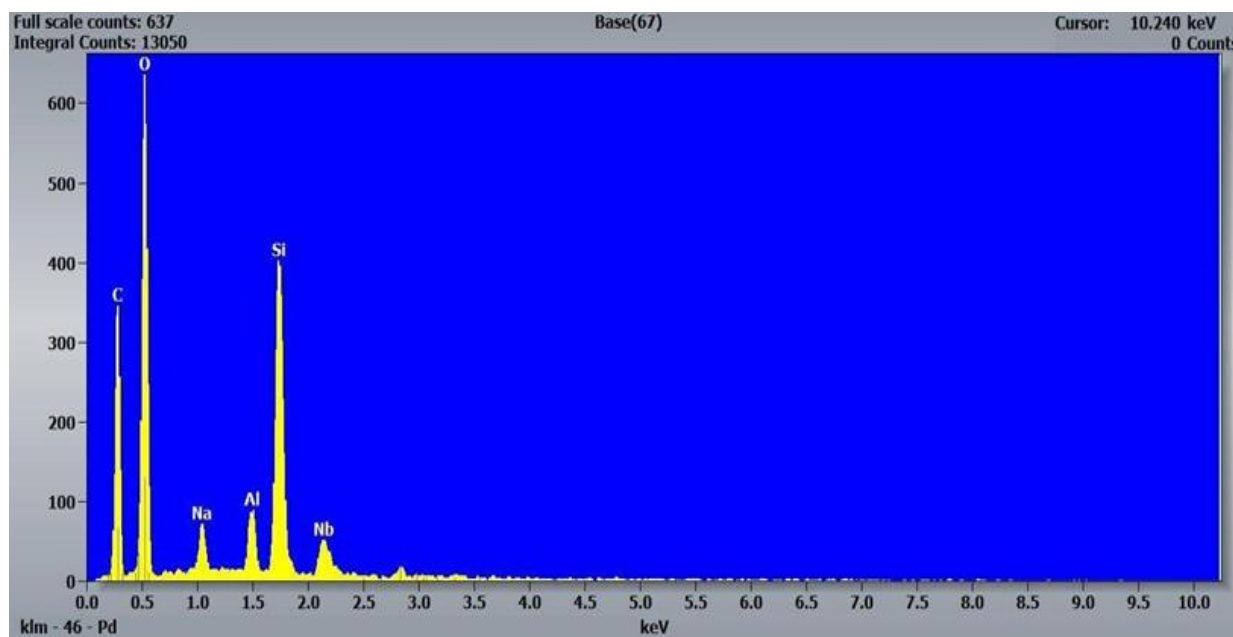


Figure 3: The EDS spectrum of the sewage sludge before activation (SSB)

- The EDS spectrum of the raw sewage sludge (SSB) is dominated by carbon (C) and oxygen (O), indicating the coexistence of organic/carbonaceous matter and oxygen-containing inorganic constituents. Lower-intensity signals corresponding to Si, Al and Na are also observed, confirming the presence of an inorganic mineral fraction in the untreated sludge.

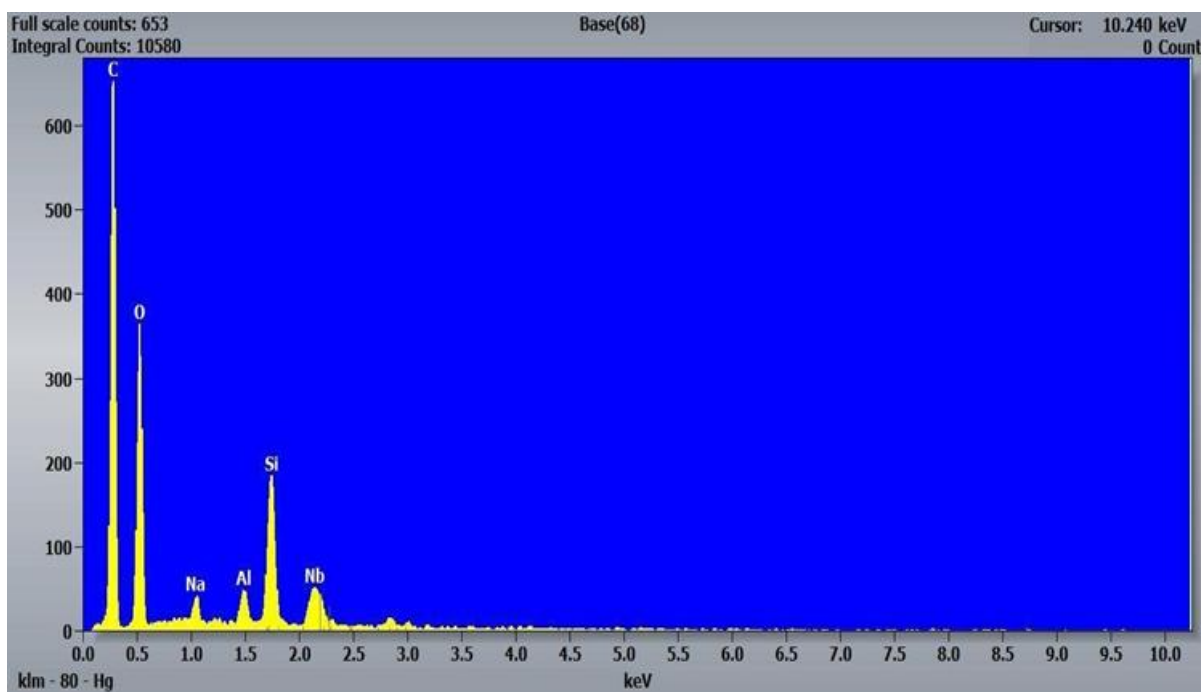


Figure 4: The EDS spectrum of the sewage sludge activated (SSA)

- After carbonization and chemical activation, the spectrum of (SSA) remains dominated by C and O, while Si and Al are still detected, indicating that part of the mineral fraction initially present in the sludge persists after treatment.
- The presence of Si and Al is consistent with the occurrence of silicate- and aluminosilicate-containing mineral phases commonly reported in sewage sludge-derived carbonaceous materials. (Carreño Hernández, 2025) (Masengo, 2022)
- The persistence of these elements after activation is also consistent with recent studies showing that Si-containing mineral phases can remain in phosphoric-acid-activated sewage-sludge carbons, even after washing, whereas several more soluble inorganic constituents may be substantially reduced (Carreño Hernández, 2025).
- Overall, the EDS results indicate that carbonization and chemical activation substantially modify the organic/carbonaceous fraction while retaining part of the original inorganic mineral fraction.

Effect of adsorbent amount

The effect of the adsorbent dose is investigated by varying the amount of SSA from 0.1 to 0.6 g in 50 mL of 0.05 g.L⁻¹ of Bezacryl red dye solution.

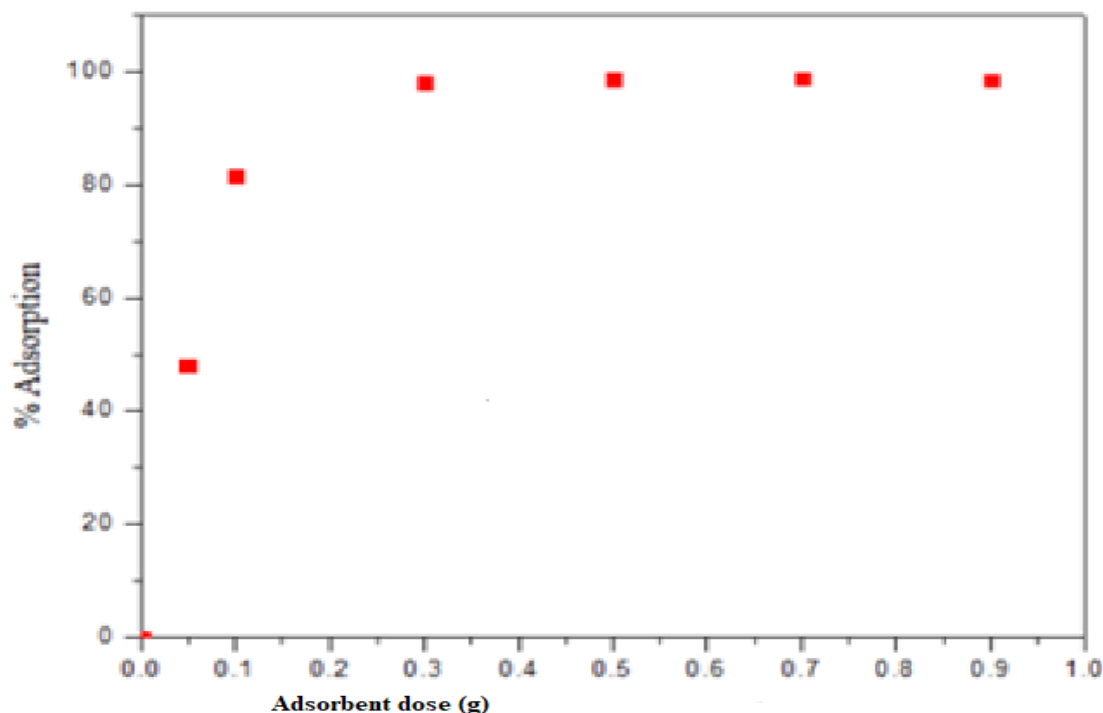
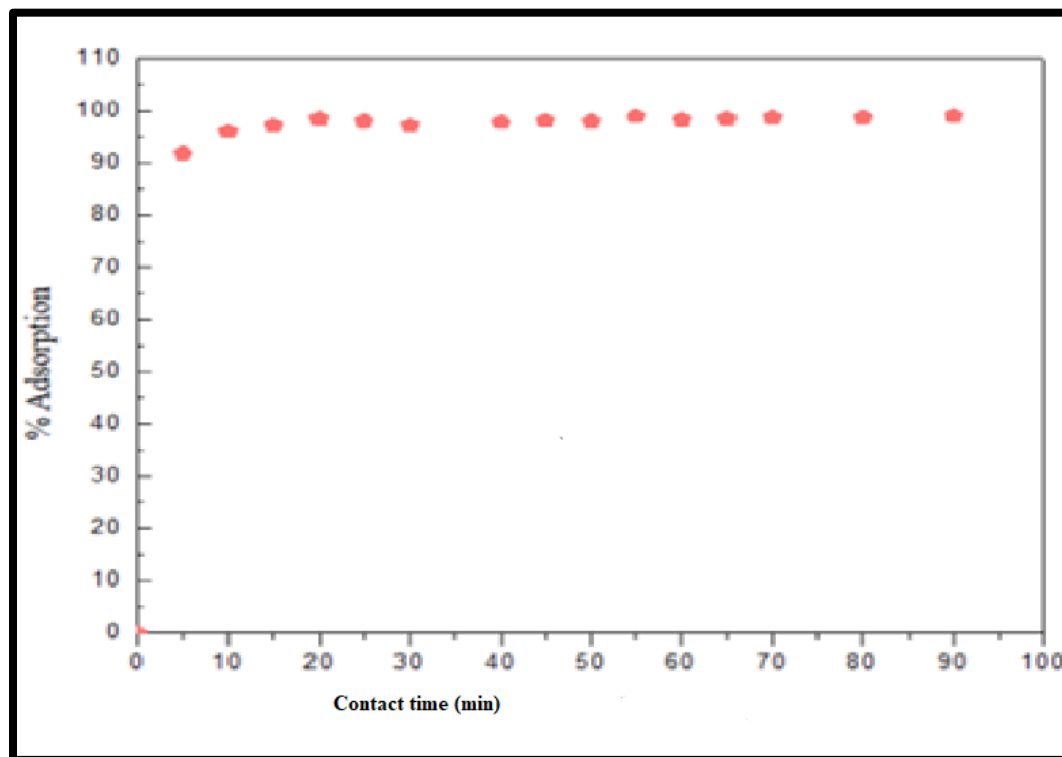


Figure 5: Effect of adsorbent dose

The effect of the activated sludge (SSA) dose on the removal of Bezacryl Rouge GRL 180 was investigated by varying the adsorbent mass from 0.05 to 0.90 g. As shown in Fig. X, the removal efficiency increased rapidly with increasing adsorbent mass, from approximately 48% at 0.05 g to more than 80% at 0.10 g, reaching approximately 98% at 0.30 g. Beyond 0.30 g, the removal efficiency remained almost constant, with values close to 98–99%.

This behavior can be attributed to the progressive increase in the number of available adsorption sites as the adsorbent dose increases. Once a sufficient amount of adsorbent is present, most of the Bezacryl Rouge GRL 180 molecules available in the solution can be retained, resulting in a plateau in the removal efficiency. Similar behavior has been widely reported for dye adsorption systems, where increasing the adsorbent dose initially enhances dye removal because of the greater availability of active sites, followed by a plateau when the adsorbent becomes present in excess relative to the amount of dye in solution. (Nandiyanto, 2021) (Bhatia, 2023)

Therefore, 0.30 g was selected as the optimum adsorbent mass under the experimental conditions used, since higher doses did not produce a significant improvement in dye removal.

Effect of contact time**Figure 6 : Effect of contact time**

The effect of contact time on the adsorption of Bezacryl Rouge GRL 180 onto the activated sludge was investigated. The adsorption efficiency increased rapidly during the initial stage of the process and reached approximately 100% after 30 min. Beyond this contact time, the removal percentage remained almost constant, indicating that the adsorption process had approached equilibrium.

The rapid initial uptake can be attributed to the high availability of accessible adsorption sites on the activated material at the beginning of the process, which facilitates the transfer and retention of dye molecules from the solution onto the adsorbent surface.

As contact time increases, these accessible sites become progressively occupied, leading to a decrease in the adsorption rate and ultimately to a plateau. Similar behavior has been reported for cationic dye adsorption, where a rapid initial uptake is followed by an equilibrium stage as the available adsorption sites become progressively occupied (Bhatia, 2023) ; (Nandiyanto A. B., 2021). Therefore, a contact time of 30 min was selected as the equilibrium contact time under the experimental conditions used.

Effect of pH

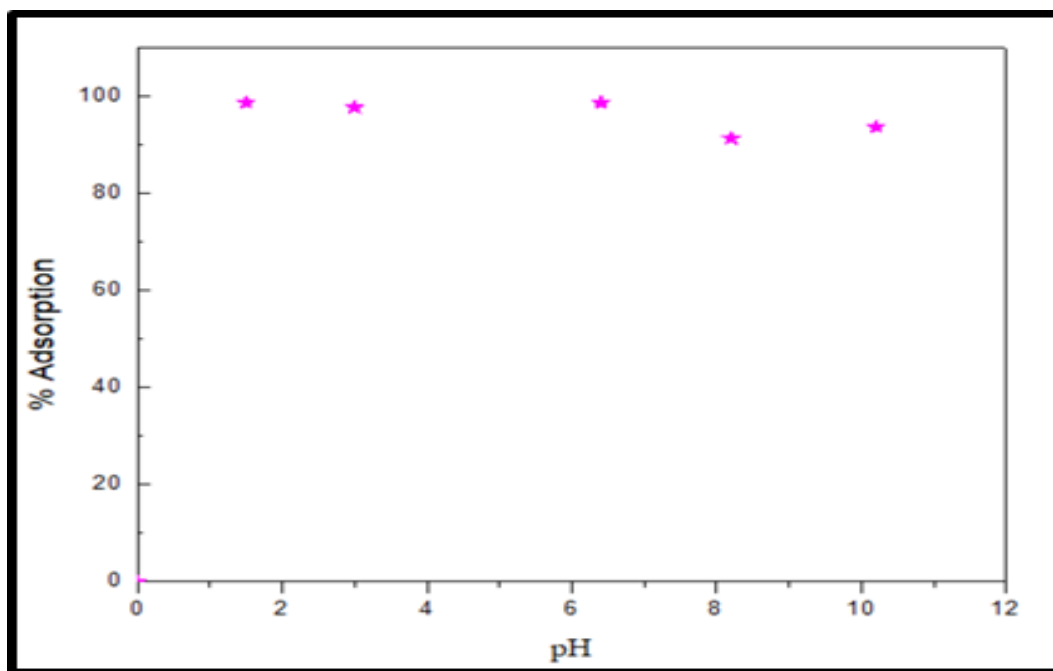


Figure 7: Effect of pH

Effect of pH. The adsorption efficiency was very high under acidic conditions, reaching approximately 98% at pH 2, and remained relatively high as the pH increased. However, a slight decrease in adsorption efficiency was observed at pH values around 7–9.

The effect of pH can be related to changes in the protonation state of the functional groups present on the adsorbent surface and to the resulting interactions between the adsorbent and dye molecules. In acidic media, protonation of surface functional groups may modify the availability and nature of adsorption sites, while changes in the aqueous ionic environment can also influence the interaction between the dye and the adsorbent surface. As the pH increases toward neutral and alkaline conditions, the progressive deprotonation of surface groups may alter these interactions and slightly reduce dye uptake. In addition, hydroxyl ions present at higher pH may compete with dye molecules for accessible adsorption sites and modify the surface chemistry of the adsorbent. (Wasilewska, 2024)

Nevertheless, the relatively high removal efficiency over the investigated pH range indicates that the adsorption process is not governed by a single interaction mechanism. Hydrogen bonding, van der Waals interactions, electrostatic interactions, and interactions involving oxygen-containing surface groups may simultaneously contribute to dye retention. Recent studies have similarly demonstrated that pH can substantially influence dye adsorption through changes in surface chemistry and adsorbate–adsorbent interactions. (El Meziani, 2025)

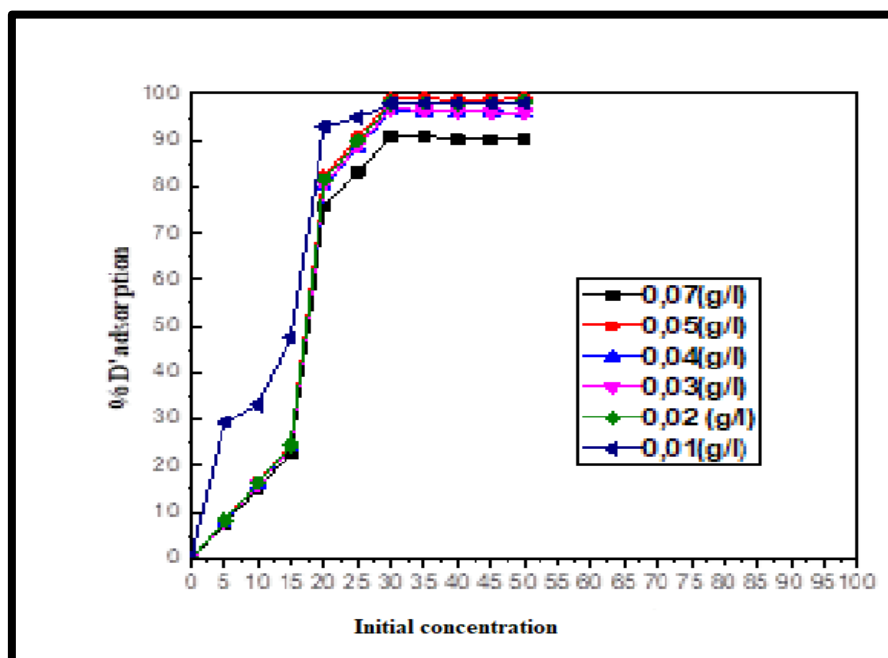


Figure 8: Effect of initial concentration

Effect of initial concentration

For all initial dye concentrations studied, adsorption was rapid during the first minutes and gradually approached equilibrium. The adsorption equilibrium was practically reached after 30 min of contact time, beyond which the removal efficiency changed only slightly. This behavior can be attributed to the initial availability of active adsorption sites, which were progressively occupied by dye molecules until equilibrium was established. (Fig.8).

The adsorption curves show a progressive increase in the removal efficiency during the first 30 min, followed by the development of a plateau, indicating the establishment of a dynamic equilibrium between the activated carbon adsorbent and the Bezacryl Red 180 adsorbate.

The prepared activated carbon exhibited excellent adsorption performance, with removal efficiencies exceeding 90%, even at relatively high initial dye concentrations. The maximum removal efficiency of approximately 99% was obtained at an initial dye concentration of 0.05 g/L using 0.3g of activated carbon, suggesting that these conditions provide highly favorable adsorption performance before the progressive saturation of the available active sites.

Kinetic study

The adsorption kinetics of Bezacryl Red onto activated carbon derived from municipal sewage sludge were investigated using the pseudo-first-order, pseudo-second-order, and intraparticle diffusion models at different initial dye concentrations (0.03, 0.05, and 0.06 g/L).

The results show that the pseudo-second-order model provides the best overall fit to the experimental data, with high coefficients of determination ($R^2 = 0.97-0.98$), compared with those obtained using the pseudo-first-order model ($R^2 = 0.82-0.88$) and the intraparticle diffusion model ($R^2 = 0.87-0.89$). Furthermore, at an initial dye concentration of 0.05 g/L, the adsorption capacity calculated using the pseudo-second-order model (16.117 mg/g) is close to the experimental value (16 mg/g), indicating good agreement between the calculated and experimental adsorption capacities under these conditions.

This agreement supports the suitability of the pseudo-second-order model for describing the adsorption kinetics at this concentration. However, the calculated adsorption capacities should be compared with the experimentally determined values at all initial concentrations before drawing a definitive conclusion. The intraparticle diffusion model also provides useful information about the possible contribution of diffusion to the overall adsorption process; nevertheless, the available results do not establish whether intraparticle diffusion is the rate-controlling step. (Vareda, (2023))

Collectively, the kinetic results suggest that the pseudo-second-order model describes the experimental data more accurately than the other models investigated, although further assessment based on experimental adsorption capacities and residual analysis would strengthen this conclusion.

Table 2 | Kinetic parameters for adsorption of Bezacryl Red on SSA

		Results		
Concentrations		Ci= 0,03g/l	Ci= 0,05g/l	Ci=0,06g/l
Pseudo first order	$q_{e,cal} (mg.g^{-1})$	14,7811	15,35	17,70
	$k_1 (mn^{-1})$	$15,08.10^{-3}$	$15,36.10^{-3}$	$15,03.10^{-3}$
	R^2	0,82	0,88	0,84
Pseudo second Order	$q_{e,cal} (mg.g^{-1})$	16,034	16,117	17,0978
	$K_2(mn^{-1})$	$1,53.10^{-3}$	$2,83.10^{-3}$	$2,457.10^{-3}$
	R^2	0,98	0,97	0,98
Intraparticle Diffusion	$q_{e,cal} (mg.g^{-1})$	17,239	18,5512	19,24
	C	1,98	2,02	2,11
	$K_i(mn^{-1})$	1.58	1,79	1,93
	R^2	0,87	0,89	0,87

Table 3 | R² and constants values for the different isotherm models

Models	Parameters	Values
Freundlich	R ²	0,97
	1/n	0.42
	K _F (mg.g ⁻¹)(L.mg ⁻¹) ^{1/n}	0,11
Langmuir	R ²	0,98
	q _m (mg.g ⁻¹)	18.26
	K _L (L.mg ⁻¹)	0.28
Temkin	R ²	0,97
	B _T (kJ.mol ⁻¹)	535.65
	KT (L.mol ⁻¹)	536.33
	ΔQ (J.mol ⁻¹)	-15.567

Adsorption isotherm study

The equilibrium adsorption data of Bezacryl Red onto activated carbon derived from municipal sewage sludge were analyzed using the Langmuir, Freundlich, and Temkin isotherm models. The Langmuir model exhibited a coefficient of determination of 0.98, with a theoretical maximum adsorption capacity q_m of 18.26 mg.g⁻¹ and a Langmuir constant K_L of 0.28 L.mg⁻¹. The Freundlich model also provided a high coefficient of determination R² = 0.97, while the adsorption intensity parameter 1/n = 0.42 indicated favorable adsorption under the experimental conditions. The Temkin model showed a slightly lower coefficient of determination R² = 0.97.

Overall, the Langmuir and Freundlich models described the experimental data similarly well and fitted slightly better than the Temkin model. However, the coefficients of determination alone are insufficient to establish which model provides the most appropriate representation of the adsorption equilibrium. Additional statistical error analysis would be useful to distinguish between the Langmuir and Freundlich models. (Hu Q. L., 2023).

Thermodynamic study

The effect of temperature on adsorption capacities was studied by performing a series of experiments between 283°K and 348°K. Thermodynamic parameters such as enthalpy (ΔH°), entropy (ΔS°) and free energy of Gibb's (ΔG°) were determined by Equation (13),(14) and (15).

Table 4 | Thermodynamic parameter for adsorption of Bezacryl Red 180 by sewage sludge-derived biochar

Parameters	Values			
T (K)	283	298	306	348
ΔH° (KJ.mol ⁻¹)	-20,68	0.153	0.146	0.142
ΔS° (kJ.K ⁻¹ .mol ⁻¹)	39.43			
ΔG° (kJ.mol ⁻¹)	-2,66	-31.84	-32,75	-34,40

The thermodynamic analysis indicates that the adsorption of BEZACRYL Red GRL 180 onto sewage-sludge-derived activated carbon is a spontaneous and exothermic process. The negative value of ΔH° (-20.68 kJ mol⁻¹) confirms the exothermic nature of the adsorption, while the positive ΔS° value (39.43 J K⁻¹ mol⁻¹) suggests an increase in the overall randomness of the system, which may be associated with the release and rearrangement of water molecules at the solid–liquid interface. Similar thermodynamic behavior, involving favorable Gibbs free-energy changes and entropy contributions during dye adsorption onto activated carbons derived from sewage sludge, has been reported in previous studies. The negative ΔG° values obtained over the investigated temperature range confirm the spontaneous nature of BEZACRYL Red adsorption. The moderate negative value of ΔH° also suggests that the process is associated with relatively moderate adsorbent–adsorbate interactions, although ΔH° alone cannot definitively distinguish physisorption from chemisorption. (Wen, 2019).

Conclusion

This study demonstrates the remarkable potential of sewage sludge as a low-cost and sustainable precursor for producing an efficient activated carbon for textile wastewater treatment. Under the optimized conditions, using 0.3 g of activated carbon to treat a 0.05 g L⁻¹ solution of the cationic dye BEZACRYL Red GR at pH 2, a removal efficiency exceeding 98% was achieved within only 30 min.

According to the thermodynamic results obtained, the adsorption process was spontaneous and exothermic, confirming the favorable nature of the interaction between the dye molecules and the activated carbon surface. Beyond its high adsorption efficiency, the main significance of this work lies in the valorization of sewage sludge into a functional carbonaceous material with environmental added value.

This waste-to-resource strategy provides a promising and sustainable approach for simultaneously addressing sewage-sludge management and textile dye pollution, highlighting the potential of the developed material for future wastewater treatment applications.

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