

RESEARCH ARTICLE

Solophenyl Red 7BE Dye Biosorption Using Raw Sunflower Seed Hulls: Ionic Strength, Regeneration, and Real Textile Effluent Application

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ABSTRACT

Despite its widespread use in the textile industry, Solophenyl Red 7BE dye remains poorly studied. This study investigates the removal of the anionic dye, Solophenyl Red 7BE (Direct Red 227), from aqueous solutions and subsequently validates the process using a real textile effluent. The low-cost lignocellulosic agro-industrial by-product, raw sunflower seed hulls, was used as biosorbent. Batch experiments achieved over 97% removal at neutral pH (7–7.5), $24^{\circ}\text{C} \pm 3^{\circ}\text{C}$ temperature, 20 mg L^{-1} dye concentration, 2.5 g L^{-1} biosorbent, and 90 min contact time. The regeneration and reusability of the biosorbent were also evaluated using NaOH to assess its potential for repeated adsorption cycles. Results show that the removal is efficient until the third cycle. It is also found that NaOH is the suitable eluent for the desorption of anionic dyes. The adsorption process followed the pseudo-first-order kinetic model and was well described by the Temkin isotherm. Analysis techniques such as FTIR, XRD, XPS, BET, SEM-EDS, and thermogravimetric analysis (TGA) confirm the involvement of surface functional groups. Dye removal experiments from real textile effluent showed that adsorption increased from 17.6% to 84.6% after NaCl addition. This demonstrates that the controlled ionic strength is an effective operational strategy for enhancing the adsorption of Solophenyl Red 7BE under realistic wastewater conditions. These findings provide a practical basis for the sustainable treatment of saline textile wastewaters containing poorly investigated dyes and encourage further evaluation of low-cost biosorbents under realistic industrial conditions.

1 | Introduction

Environmental protection is an increasingly prominent issue in contemporary discussions and represents a key area of research for the scientific community. Water pollution remains a major concern worldwide, particularly due to the discharge of large quantities of colored wastewater from textile dyeing processes. This severely contaminates water resources (Bouhadja et al. 2021; Gupta et al. 2007; Majeed et al. 2026). In addition

to their intense coloration, textile effluents are characterized by complex chemical matrices. They often contain persistent organic compounds and high salt loads, which makes their treatment under real conditions particularly challenging.

Solophenyl Red 7BE (SR7BE) is the most used dye in the textile manufacture. It is a direct anionic double azo dye commonly employed for dyeing cellulose fibers. It may exhibit mutagenic and carcinogenic effects, posing serious risks to

Summary

- Solophenyl Red 7BE was efficiently removed by low-cost biosorption.
- High removal efficiency (> 97%) achieved under optimized conditions.
- Real textile effluent reduced adsorption without added electrolyte.
- Ionic strength markedly enhances dye removal in complex matrices
- Low-cost biosorption offers a sustainable wastewater treatment strategy.

human health and aquatic life (Collivignarelli et al. 2019). Its sulfonated structure confers high solubility and chemical stability, making it a highly recalcitrant compound difficult to remove from textile effluents. Despite its industrial relevance, the treatment of SR7BE has received limited specific attention compared with many other azo dyes, particularly regarding adsorption-based removal processes under realistic saline conditions.

Various treatment techniques have been investigated to remove such dyes, including photocatalysis (Cherif et al. 2022) and adsorption onto nanomaterials (Sanaullah et al. 2025) or biomaterials (Mehrez et al. 2024). Among these approaches, adsorption using biomaterials has attracted considerable attention due to its low cost, simplicity, and environmentally friendly nature (Grabi, Derridj, et al. 2021). Several studies have explored the use of agro-industrial waste as biosorbents for dye removal from textile effluents, including sugarcane bagasse (Tsai et al. 2001), orange peel (Li et al. 2007), cocoa pods (Bello and Ahmad 2011), banana peel (Mehrez et al. 2024), hydroxyapatite (Lemlikchi et al. 2012), date palm flower stalk (Arroussi and Laksaci 2024), and sunflower seed hulls (SSHs) (J  zwiak et al. 2020; Kocada  stan and Kocada  stan 2016; Osm   et al. 2007; Srisorrachatr 2011). These materials have demonstrated promising removal efficiency. In particular, SSHs are abundantly available worldwide, especially in Europe and Asia. Global sunflower seed production was estimated at 58 million tonnes in 2022, generating approximately 14 million cubic meters of sunflower hulls (Tsvetanova et al. 2025), making them an economically attractive resource for large-scale applications. Moreover, their valorization is feasible within existing sunflower oil production industries. The biosorbent dose is a critical parameter influencing process feasibility and scalability (Grabi, Lemlikchi, et al. 2021; Salom  n et al. 2020; dos Reis et al. 2020), and achieving high removal efficiency with minimal adsorbent dosage remains a key objective for cost-effective treatment systems (Yagub et al. 2014).

In facts, many studies shown that the presence of electrolytes (NaCl) can significantly influence adsorption processes by promoting or inhibiting removal efficiency of dyes (Aksu and Balibek 2010; Ansari et al. 2016; Luk       et al. 2012). As reported by Ansari et al. 2016, the ionic strength decreased the adsorption of methylene blue on a cauliflower leaf biomass. This has

been attributed to the screening of electrostatic interactions. However, the increase in the NaCl concentration reduced the biosorption of an anionic dye with a metal complex on the biomass of *Rhizopus arrhizus* (Aksu and Balibek 2010). On the other hand, the ionic force can also modify the electrical double layer and reduce electrostatic repulsions in systems involving species of the same charge, thus favoring adsorption (Luk       et al. 2012).

The aim of the present study is to evaluate the removal of SR7BE using raw SSHs as a low-cost and sustainable biosorbent, without chemical or thermal activation. To our knowledge, the use of SSHs for SR7BE adsorption has not been previously reported. In addition, the effect of the ionic strength on SR7BE adsorption by the SSHs has not been studied yet. This parameter is particularly relevant because textile dyeing effluents often contain high salt concentrations. Besides, most adsorption studies do not adequately reflect the complexity of real textile effluents since they are usually conducted under laboratory conditions using synthetic solutions. Adsorption studies using real textile effluents remain limited.

Experiments were conducted at room temperature ($24^{\circ}\text{C} \pm 3^{\circ}\text{C}$), while the effect of pH was specifically investigated over acidic to alkaline conditions. Particular emphasis was placed on assessing whether ionic strength can act as an operational lever to enhance adsorption efficiency under realistic conditions. The biomaterial was characterized using Fourier-transform infrared (FTIR), thermogravimetric analysis (TGA), X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS), BET, SEM-EDS, and ColorFlex analyses. The effects of adsorbent dosage, contact time, pH, and initial dye concentration were investigated. Kinetic and equilibrium modeling were performed to describe the adsorption behavior and evaluate the fitting of the experimental data. In addition, the regeneration and reusability of the biosorbent were investigated to assess its potential for repeated adsorption cycles and practical application. Finally, the applicability of the process was assessed using a real textile effluent containing SR7BE under selected experimental laboratory conditions.

2 | Materials and Methods

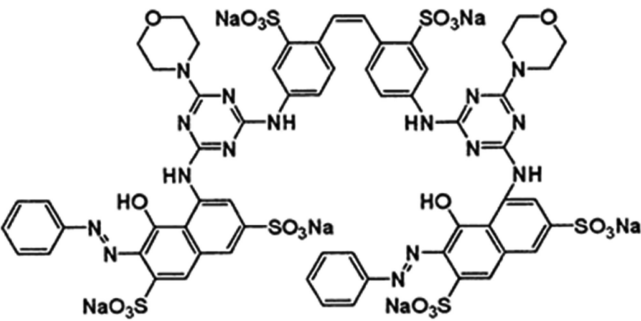
2.1 | Materials

The SSHs were purchased from a local supplier “shopi one” (Bir Mourad Rais, Algiers, Algeria).

The real textile effluent and SR7BE (Direct Red 227) dye were provided by a textile industry (TEXLAGE, Boufarik, Algeria). According to the product information supplied by the manufacturer, the dye was manufactured by BEZEMA AG (Kriessernstrasse 20, 9462 Montlingen, Switzerland). The structure and chemical characteristics of SR7BE are presented in Table 1.

1. Sodium chloride (NaCl, GPR Rectapur, purity > 98%), reference (27788.366), Batch (23E244115) was supplied by VWR Chemicals International (Leuven, Belgium) and was used as the supporting electrolyte throughout the adsorption experiments.

TABLE 1 | The structure of SR7BE dye and chemical characteristics.

Chemical name	Solophenyl Red 7BE
Scientific name	Direct Red 227
Class	Doubleazo
Structure	
Molecular formula	C ₆₀ H ₄₆ N ₁₆ Na ₆ O ₂₂ S ₆
Molecular weight (g·mol ⁻¹)	1673.43
Type of structure	Anionic
λ _{max} (nm)	542
Solubility	Soluble in water

- Hydrochloric acid (HCl, 0.1 mol L⁻¹, M = 36.46 g mol⁻¹) and sodium hydroxide (NaOH, 0.1 mol L⁻¹, M = 40.00 g mol⁻¹) were purchased from Panreac Química S.A. (Montcada i Reixac, Barcelona, Spain).
- Distilled water was produced in the Laboratory of Natural Molecules Chemistry, Gembloux Agro-Bio Tech, University of Liège (Gembloux, Belgium) and was used for the preparation of all aqueous solutions and adsorption experiments.

2.2 | Methods

Analytical grade NaCl and distilled water were used to prepare all solutions. A stock solution of SR7BE (1000 mg L⁻¹) was diluted with distilled water to obtain working solutions with different concentrations (10–400 mg L⁻¹). The biomaterial was prepared following the method of Grabi, Derridj, et al. (2021) with slight modifications. The SSHs were washed with distilled water, dried in a laboratory oven (Mettler GmbH + Co. KG, Schwabach, Germany) at 80°C, ground using an analytical mill (IKA A11 basic, IKA-Werke GmbH & Co. KG, Staufen, Germany), and sieved to obtain particles smaller than 0.5 mm using a laboratory sieve (VWR International, Leuven, Belgium). The material was then rewashed under agitation, dried at 60°C, reground, and sieved to 250 μm. The pH_{pzc} of the prepared SSHs powder was determined using the pH drift method (Altenor et al. 2009). Briefly, six 50 mL NaCl solutions (0.01 M) with initial pH values ranging from 3.16 to 9.82 were prepared by adjusting the pH with 1 N HCl or 1 N NaOH. After the desired initial pH (pH_i) had been reached, 0.05 g of SSH powder was added to each solution. The suspensions were stirred for 3 h at room temperature 22°C, after which the final pH (pH_f) was measured. The pH_{pzc} was determined from the intersection of the curve obtained by

plotting pH_f as a function of pH_i, corresponding to the point where pH_f = pH_i.

Characterization analyses were performed to evaluate changes in the material before and after adsorption. Functional groups were identified using a FTIR spectrometer (ALPHA-P, Bruker Optics GmbH, Ettlingen, Germany) operated in ATR mode over the spectral range 400–4000 cm⁻¹, with a resolution of 2 cm⁻¹ and 32× scans per spectrum. Thermal behavior was investigated using a simultaneous differential scanning calorimetry–thermogravimetric analyzer (SDT Q600, TA Instruments, New Castle, DE, United States). Measurements were performed under a nitrogen atmosphere (100 mL min⁻¹) from 22.5°C to 1291.8°C at a heating rate of 10°C min⁻¹, using approximately 7.5 mg of sample. Structural properties were examined by XRD using an Empyrean diffractometer (Malvern Panalytical B.V., Almelo, the Netherlands) equipped with a Cu Kα radiation source, operated at 45 kV and 40 mA, over a 2θ range of 5°–80° with a scanning step of 0.026°. Surface morphology and elemental composition were analyzed using a scanning electron microscope equipped with an energy-dispersive X-ray spectroscopy detector (SEM-EDS, Axia ChemiSEM, Thermo Fisher Scientific, Waltham, MA, United States). Prior to SEM observation, the samples were coated with a thin carbon layer using a Cressington 108 Carbon/A carbon coater (Cressington Scientific Instruments Ltd., Watford, Hertfordshire, United Kingdom) to improve surface conductivity. XPS analyses were performed using a JPS-9030 spectrometer (JEOL Ltd., Akishima, Tokyo, Japan) equipped with an Mg Kα X-ray source (1253.6 eV) operated at 120 W (12 kV, 10 mA) using an aperture diameter of 6 mm. The specific surface area was determined by nitrogen adsorption using an ASAP 2020 analyzer (Micromeritics Instrument Corporation, Norcross, GA, United States). Before analysis, samples were

automatically degassed at 80°C for 12 h. Nitrogen adsorption measurements were carried out at −195.8°C using approximately 0.2003 g of sample. UV-Visible spectra were recorded using an Ultrospec 7000 spectrophotometer (Biochrom Ltd., Cambridge (Waterbeach), United Kingdom). Absorbance was measured at the maximum absorption wavelength of SR7BE (542 nm) using optical glass cuvettes (320–2500 nm). Data acquisition and processing were performed using the instrument software. Colorimetric analysis was carried out using a ColorFlex EZ spectrophotometer (Hunter Associates Laboratory Inc., Reston, VA, United States) under illuminant D65 with a 10° observer angle, according to CIE and ISO standards. Measurements were conducted for samples loaded with 20 and 50 mg L^{−1} dye, and the CIE Lab color parameters were recorded, where L* represents lightness (0 = black and 100 = white), a* corresponds to the green (−) to red (+) chromatic axis, and b* to the blue (−) to yellow (+) chromatic axis.

2.3 | Batch Adsorption Experiments

Batch adsorption experiments were performed at 24°C ± 3°C in Erlenmeyer flasks containing 100 mL of SR7BE solutions. A schematic overview of the experimental design, including the biosorbent dosage, NaCl concentration, kinetic and equilibrium experiments, is presented in Figure 1. The pH was not adjusted and corresponded to the natural pH of the suspension (approximately 7–7.5). Erlenmeyer flasks contain 100 mL of SR7BE solutions. The agitation velocity was set to 250 rpm. All adsorption experiments were carried out in triplicate, and the results are reported as mean ± standard deviation. Control experiments

confirmed that NaCl alone did not induce dye removal, but enhanced adsorption performance. The biosorbent mass was tested between 0.0625 and 5 g per 100 mL of sample at 20 mg L^{−1} dye concentration and three NaCl levels (0, 0.01, and 0.1 M). The flasks were agitated for 4 h and then left to equilibrate for 24 h. Kinetic experiments were carried out with contact times from 5 to 240 min and 1440 min (24 h), using 0.25 g of SSHs powder per 100 mL of aqueous sample and 0.1 M NaCl in a 20 mg L^{−1} dye solution. In isotherm study, experiments were realized at different dye concentrations. To further investigate the contribution of electrostatic interactions to SR7BE adsorption, the effect of solution pH was evaluated in the absence and presence of NaCl. Batch experiments were conducted over a pH range of 2–9.5, using the same experimental conditions as those selected for the adsorption experiments (using 0.25 g of SSHs powder per 100 mL of aqueous, the mixtures were agitated for 90 min). The initial pH was adjusted to the desired value using 0.1 M HCl or 0.1 M NaOH. Experiments were performed both without NaCl (0.0 M) and in the presence of 0.1 M NaCl. Equilibrium adsorption isotherms were first established at 25°C using initial SR7BE concentrations of 10, 20, 30, 50, 100, 150, 200, 250, 300, 400 mg L^{−1} under the selected optimal conditions (0.25 g SSHs per 100 mL, 0.1 M NaCl, and neutral pH), were agitated for 4 h and subsequently allowed to equilibrate for 24 h. To investigate the temperature dependence of the adsorption equilibrium, the same series of initial SR7BE concentrations was subsequently investigated at 25°C, 50°C, 60°C, and 75°C under otherwise identical experimental conditions. For these temperature-dependent equilibrium experiments, the suspensions were agitated for 4 h and subsequently allowed to equilibrate for 24 h. Thus, a complete adsorption isotherm was obtained at each temperature.

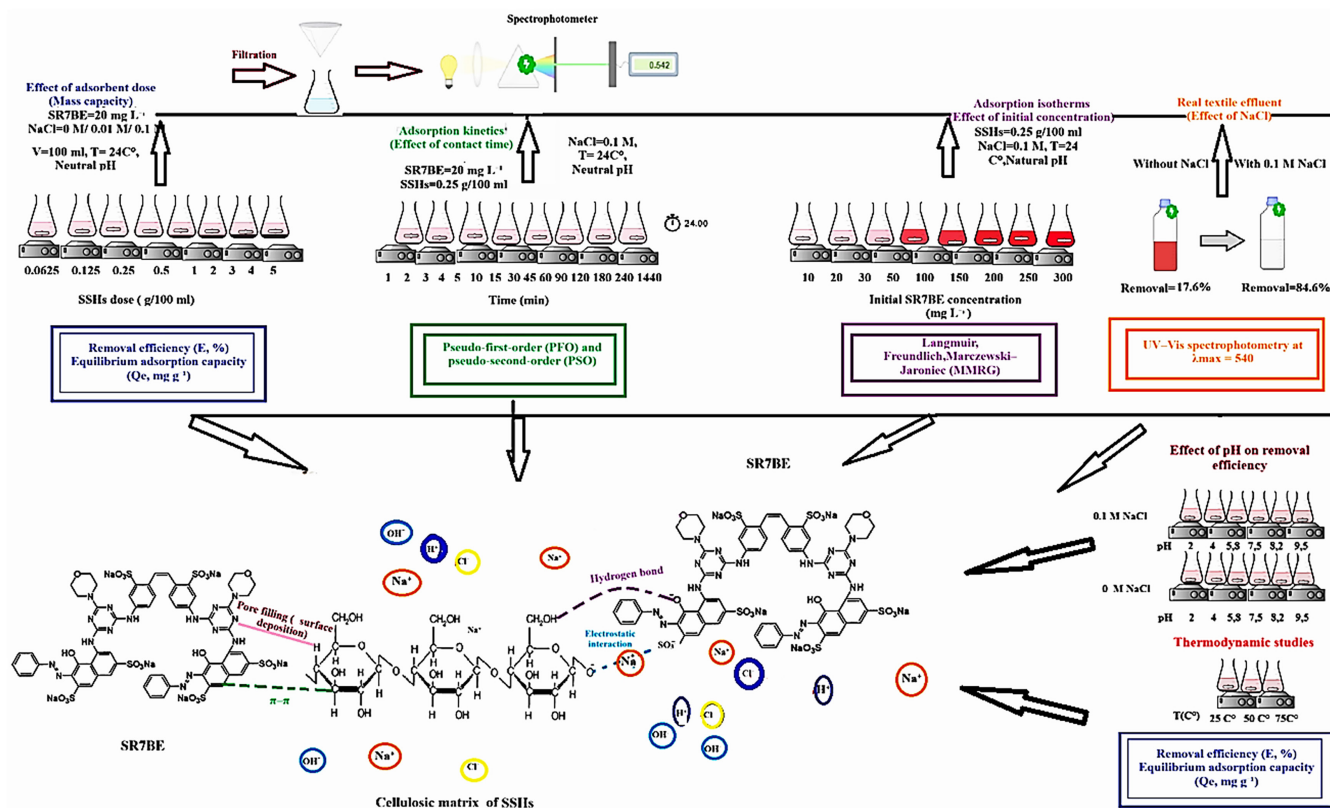


FIGURE 1 | Schematic representation of the adsorption experiments and the possible sorption mechanism of SR7BE onto sunflower seed hulls (SSHs).

After the contact period, all samples were filtered through MN 615 1/4 filter paper (125mm, MACHEREY-NAGEL GmbH & Co. KG, Düren, Germany) and analyzed using a UV-Vis spectrophotometer (Ultrospec 7000) at 542 nm. Residual dye concentrations were determined from a calibration curve established according to the Beer–Lambert Law. The equilibrium adsorption capacity (q_e) and the removal efficiency were calculated from the measured concentrations. Removal efficiency was determined from absorbance values at $\lambda_{\max}$, assuming a linear relationship between absorbance and concentration.

The applicability of the process was also evaluated using a real textile effluent. Prior to analysis, the effluent was pretreated by screening and sedimentation to remove suspended solids. Experiments were performed using the same experimental procedure as that applied to the synthetic dye solution (0.25 g/100 mL SSHs, 0 M NaCl and 0.1 M NaCl, 20 mg L⁻¹ of real textile effluent, neutral solution pH, 24°C ± 3°C and 24 h contact time). The maximum absorption wavelength was determined as 540 nm, and all measurements were conducted at this wavelength. Removal efficiency was estimated by comparing absorbance before and after treatment.

The removal efficiency (E , %) was calculated according to Equation (1) (Grabi, Lemlikchi, et al. 2021):

$$E (\%) = \frac{C_0 - C_e}{C_0} 100 \quad (1)$$

where C_0 and C_e (mg L⁻¹) are the initial and equilibrium dye concentrations, respectively.

The equilibrium adsorption capacity (Q_e , mg g⁻¹) was calculated using Equation (2):

$$Q_e = \frac{(C_0 - C_e) V}{m} \quad (2)$$

where V (L) is the solution volume and m (g) is the mass of biosorbent.

The adsorption capacity at time t (q_t mg g⁻¹) was determined using Equation (3):

$$q_t(t) = \frac{(C_0 - C_t(t)) V}{m} \quad (3)$$

where C_t (mg L⁻¹) is the dye concentration at time t and m is the mass of SSHs (0.25 g per 100 mL of aqueous sample in the actual case).

The thermodynamic parameters of SR7BE adsorption onto SSHs were determined following the approach proposed by Lima et al. (2019). In accordance with their recommendation, the equilibrium constant used in the Van't Hoff equation was obtained from the equilibrium constant of the selected adsorption isotherm model at each temperature rather than directly from the ratio q_e/C_e . The thermodynamic equilibrium constant, K_e° , was calculated as:

$$K_e^\circ = \frac{1000 K_{\text{model}} M C^\circ}{\gamma} \quad (4)$$

where K model is the equilibrium constant obtained from the selected isotherm model (L mg⁻¹), M is the molar mass of SR7BE (1673.43 g mol⁻¹), C° is the standard concentration (1 mol L⁻¹), and γ is the activity coefficient. Following Lima et al. (2019), γ was assumed to be unity under the dilute-solution approximation. The resulting K_e° is dimensionless and was subsequently used to calculate the standard Gibbs free energy change according to:

$$\Delta G^\circ = -RT \ln K_e^\circ \quad (5)$$

The standard enthalpy and entropy changes were determined from the Van't Hoff equation:

$$\ln K_e^\circ = -\frac{\Delta H^\circ}{R} \frac{1}{T} + \frac{\Delta S^\circ}{R} \quad (6)$$

where R is the universal gas constant (8.314 J mol⁻¹ K⁻¹) and T is the absolute temperature (K). A plot of $\ln K_e^\circ$ versus $1/T$ was used to determine ΔH° from the slope and ΔS° from the intercept.

2.4 | Kinetic and Equilibrium Modeling

Adsorption kinetics were evaluated using the pseudo-first-order (PFO), pseudo-second-order (PSO), Elovich, and Weber–Morris models, which are commonly used to describe adsorption rate behavior (Liu and Shen 2008; Musah et al. 2022; Weber and Morris 1963; Özmal and Tekke 2023).

Equilibrium data were fitted using the Langmuir, Freundlich, Multilayer Multisite Random Geometry (MMRG), Temkin, and Dubinin–Radushkevich (D–R) models (Langmuir 1916; Freundlich and Heller 1939; Bergaoui et al. 2018; Grabi, Lemlikchi, et al. 2021; Hayoun et al. 2020; Temkin and Pyzhev 1940; Dubinin and Radushkevich 1947). Statistical-physics-based approaches have also been applied to describe heterogeneous adsorption systems and molecular interactions (Dehbi et al. 2024).

Kinetic experiments were carried out using an initial SR7BE concentration of 20 mg L⁻¹, a biosorbent dose of 2.5 g L⁻¹ (0.25 g per 100 mL), and 0.1 M NaCl at the neutral solution pH and 24°C ± 3°C. Samples were collected after 1, 2, 3, 4, 5, 10, 15, 30, 45, 60, 90, 120, 180, 240, and 1440 min (24 h). The experimental adsorption capacity at time t (q_t) was calculated using Equation (3), and the adsorption kinetic data were fitted using the PFO, PSO kinetic models (Liu and Shen 2008) and Elovich, Weber–Morris models (Musah et al. 2022; Weber and Morris 1963; Özmal and Tekke 2023).

$$q_{PFO}(t) = Q_e (1 - e^{-k_1 t}) \quad (7)$$

$$q_{PSO}(t) = \frac{k_2 Q_e^2 t}{1 + k_2 Q_e t} \quad (8)$$

$$q_t = \frac{1}{\beta} \ln(1 + \alpha \beta t) \quad (9)$$

$$q_t = k_{id} t^{\frac{1}{2}} + C \quad (10)$$

where Q_e (mg/g) is the equilibrium adsorption capacity, k_1 (min^{-1}) is the PFO rate constant, and k_2 (g/mg·min) is the PSO rate constant. For the Elovich model, qt (mg g^{-1}) is the amount of adsorbate adsorbed at time t (min) is the contact time, α ($\text{mg g}^{-1} \text{min}^{-1}$) is the initial adsorption rate, and β (g mg^{-1}) is the Elovich constant related to the desorption process and surface heterogeneity. For the intraparticle diffusion model, qt (mg g^{-1}) represents the amount adsorbed at time t , k_{id} ($\text{mg g}^{-1} \text{min}^{-1/2}$) is the intraparticle diffusion rate constant, C (mg g^{-1}) is the intercept related to the thickness of the boundary layer, and t (min) is the contact time.

To evaluate the goodness-of-fit of the kinetic models, both the coefficient of determination (R^2) and the Akaike Information Criterion (AIC) were used. The AIC is a statistical criterion that compares model performance while accounting for the number of adjustable parameters; lower AIC values indicate a better balance between goodness-of-fit and model complexity.

Equilibrium experiments were performed using initial SR7BE concentrations ranging from 10 to 300 mg L^{-1} under the same experimental conditions (2.5 g L^{-1} SSHs, 0.1 M NaCl, neutral solution pH, $24^\circ\text{C} \pm 3^\circ\text{C}$ and 24 h contact time). The equilibrium adsorption capacity (Q_e) was calculated using Equation (2), and the equilibrium adsorption data were analyzed using the Langmuir (Langmuir 1916), Freundlich (Freundlich and Heller 1939), Marczewski–Jaroniec (MMRG) isotherm models (Bergaoui et al. 2018; Grabi, Derridj, et al. 2021; Hayoun et al. 2020), Temkin (Temkin and Pyzhev 1940), and Dubinin (Dubinin and Radushkevich 1947). The Langmuir model assumes monolayer adsorption on a homogeneous surface with a finite number of identical adsorption sites, whereas the Freundlich model describes adsorption on heterogeneous surfaces with non-uniform adsorption energies and possible multilayer adsorption. The MMRG model is a generalized adsorption isotherm that combines the characteristics of the Langmuir and Freundlich equations and is particularly suitable for describing adsorption on heterogeneous surfaces over a wide range of adsorption energies. The Temkin model considers adsorbate–adsorbent interactions and assumes that the heat of adsorption decreases linearly with increasing surface coverage, reflecting a gradual reduction in adsorption energy. The D–R model is commonly used to describe adsorption onto heterogeneous surfaces and to estimate the mean adsorption energy, which can provide an indication of the nature of the adsorption process. The corresponding equations are given below:

$$Q_e = \frac{Q_{\max} K_L C_e}{1 + K_L C_e} \quad (11)$$

$$R_L = \frac{1}{1 + K_L C_0} \quad (12)$$

$$Q_e = K_F C_e^{1/n} \quad (13)$$

$$Q_e = \frac{nNm}{\left[1 + w \frac{1-bC_e}{C_e} e^{k_B \frac{2a}{T} C_e} e^{-bC_e} e_{1-bC_e}\right]^n} \quad (14)$$

$$qe = B \ln(K_T C_e) \quad (15)$$

$$q_e = q_m \exp(-\beta \epsilon^2) \quad (16)$$

$$\epsilon = RT \ln \left(1 + \frac{1}{C_e}\right) \quad (17)$$

$$E = \frac{1}{\sqrt{2\beta}} \quad (18)$$

In the Langmuir equation (Equation 11), q_e (mg g^{-1}) is the equilibrium adsorption capacity, C_e (mg L^{-1}) is the equilibrium dye concentration, $q_{\max}$ (mg g^{-1}) is the maximum monolayer adsorption capacity, and K_L (L mg^{-1}) is the Langmuir affinity constant. The dimensionless separation factor R_L (Equation 12), was calculated to evaluate the favorability of the adsorption process, where C_0 (mg L^{-1}) is the initial dye concentration. In the Freundlich equation (Equation 13), K_F ($\text{mg g}^{-1} (\text{L mg}^{-1})^{1/n}$) represents the adsorption capacity, whereas n is the heterogeneity factor describing the adsorption intensity. The parameters of the MMRG model (Equation 14) (Nm , a , b , and w) characterize the adsorption capacity, surface heterogeneity, and adsorption energy distribution. For the Temkin model (Equation 15), q_e (mg g^{-1}) is the equilibrium adsorption capacity, C_e (mg L^{-1}) is the equilibrium dye concentration, K_T (L mg^{-1}) is the Temkin equilibrium binding constant, and B (J mol^{-1}) is related to the heat of adsorption. For the D–R model (Equation 16), q_e (mg g^{-1}) is the equilibrium adsorption capacity, q_m (mg g^{-1}) is the theoretical saturation capacity, β ($\text{mol}^2 \text{J}^{-2}$) is the D–R constant related to the adsorption energy, and ϵ (J mol^{-1}) is the Polanyi potential. In Equation (17), R ($8.314 \text{ J mol}^{-1} \text{K}^{-1}$) is the universal gas constant, T (K) is the absolute temperature, and C_e (mg L^{-1}) is the equilibrium dye concentration. The mean adsorption energy E (J mol^{-1}), calculated from Equation (18), is used to provide an indication of the nature of the adsorption process.

2.5 | Adsorbent Regeneration and Reuse Procedure

The regeneration and reusability of SSHs were evaluated over three successive adsorption–desorption cycles using the batch adsorption method. For the desorption step, distilled water, 0.1 mol L^{-1} HCl, and 0.1 mol L^{-1} NaOH were investigated as eluents. After each desorption step, the regenerated biosorbent was dried overnight and subsequently reused under the same experimental conditions for the following adsorption cycle using the best-performing eluent.

Adsorption experiments were performed using an initial SR7BE concentration of 20 mg L^{-1} and a biosorbent dosage of 0.25 g per 100 mL. NaCl (0.1 M) was used only during the adsorption step, while the solution pH was not adjusted and corresponded to the natural pH of the suspension. The adsorption–desorption experiments were conducted at $24^\circ\text{C} \pm 3^\circ\text{C}$, with a contact time of 90 min for each adsorption cycle.

The desorption efficiency, E (%) (des), was calculated according to Equation (19), following Allou et al. (2023) and Belounis et al. (2025):

$$E \% (\text{des}) = \frac{C_f}{C_i} \times 100 \quad (19)$$

where C_f is the concentration of SR7BE in the desorption solution after elution (mg L^{-1}) and C_i is the initial concentration of SR7BE adsorbed onto the biosorbent (mg L^{-1}).

The adsorption removal efficiency was calculated using Equation (1), as described above.

3 | Results and Discussion

3.1 | Physicochemical Study

Figure 2 shows the FTIR spectra of SR7BE dye and SSHs before and after adsorption at different dye concentrations. The SR7BE spectrum displays characteristic bands at 3388 cm^{-1} (O—H/N—H stretching), $2911\text{--}2846\text{ cm}^{-1}$ (aliphatic C—H stretching), and intense bands at 1615 , 1482 , and 1420 cm^{-1} , attributed to aromatic ring vibrations and azo (—N=N—) groups. The band at 1011 cm^{-1} corresponds to the asymmetric stretching vibration of sulfonate (S=O) groups, which are characteristic of sulfonated azo dyes (Sanaullah et al. 2025).

The spectrum of raw SSHs exhibits the characteristic features of lignocellulosic biomass, including a broad band at 3290 cm^{-1} assigned to O—H stretching of hydroxyl groups, a band at 2913 cm^{-1} corresponding to aliphatic C—H stretching, and absorption bands at 1720 and 1620 cm^{-1} , attributed to C=O stretching of carbonyl groups and aromatic C=C vibrations associated with lignin, respectively (Mousavi et al. 2022; Edokpayi et al. 2019).

After adsorption, a general decrease in the intensity of several absorption bands, particularly within the $1620\text{--}1011\text{ cm}^{-1}$ region, is observed, indicating the participation of hydroxyl, carbonyl, aromatic, and sulfonate groups in the adsorption process. These spectral changes suggest that SR7BE uptake is governed by the combined contribution of hydrogen bonding, electrostatic attraction, and $\pi\text{--}\pi$ interactions between the aromatic structure of the dye and the lignocellulosic matrix of the biosorbent. Similar FTIR modifications after dye adsorption onto lignocellulosic biosorbents have been reported by Mousavi et al. (2022)

and Tolkou et al. (2024), confirming the important role of surface functional groups in the adsorption mechanism.

Figure 3 presents the TGA and their derivatives (DTG) plotted for three samples: raw SSHs (Figure 3a), SSHs-SR7BE20 (Figure 3b), and SSHs-SR7BE100 (Figure 3c). All curves exhibit a typical three-stage degradation pattern of lignocellulosic materials (Tolkou et al. 2024; Gao et al. 2013; Liu et al. 2012; Roman et al. 2013; Bouhadjra et al. 2023; Liu et al. 2016). The first stage ($<200^\circ\text{C}$) corresponds to moisture loss (8%–10%). The main degradation ($200^\circ\text{C}\text{--}380^\circ\text{C}$) is attributed to hemicellulose and cellulose decomposition, with mass losses of 53%–58%. A slight increase in mass loss with dye loading is observed, suggesting dye incorporation into the biomass matrix. DTG peaks around $315^\circ\text{C}\text{--}351^\circ\text{C}$ confirm cellulose degradation. The third stage ($380^\circ\text{C}\text{--}650^\circ\text{C}$) corresponds to lignin decomposition, where higher mass loss is observed for dye-loaded samples. Overall, no new degradation peaks appear after adsorption, indicating that the thermal behavior of SSHs is preserved, which supports a mainly physical adsorption mechanism with possible weak interactions.

Figure 4 shows the XRD patterns of the biosorbent material before and after the adsorption at 300 mg/L of the SR7BE dye. The SSH powder exhibits three diffraction peaks at 2θ angles of 16.22° , 22.60° , and 34.94° . The highest diffraction peak ($\approx 22^\circ$) is attributed to the (002) plane of cellulose in our biosorbent. It is a characteristic peak of plant-based biomaterials (Edokpayi et al. 2019). Because of the non-crystalline nature of our dye, no new diffraction peak appears after adsorption. However, a clear decrease in the intensity of the diffraction peaks is observed following adsorption. According to Laggoun et al. (2025), this provides strong evidence of successful dye uptake demonstrating that the retention of SR7BE dye affects the microstructure of the biosorbent. These results show no new crystalline phases after adsorption, with only a decrease in peak intensity, indicating that SR7BE adsorption onto SSHs is mainly governed by physisorption.

SEM-EDS characterization (Figure 5) shows morphological and chemical changes in the SSHs powder surface before and after SR7BE adsorption. Before adsorption, the material exhibits agglomerated grains with a porous and irregular structure (Figure 5a), which may favor dye diffusion toward accessible surface and internal sites. After adsorption, the surface becomes smoother and more homogeneous (Figure 5c), suggesting pore filling and surface deposition of the dye. The EDS spectra before and after adsorption (Figure 5b,d, respectively) support these observations. The contents of C, N, and S increase after adsorption, indicating the incorporation of dye-related elements. The appearance of Cl and Na after adsorption is mainly associated with the NaCl electrolyte used during the adsorption experiments, while Na may also originate from the dye structure. In contrast, mineral elements such as Ca and Mg decrease or disappear, which may be related to surface masking by the adsorbed dye and/or possible redistribution of mineral species during adsorption.

Overall, these results show that SR7BE adsorption is accompanied by surface smoothing and chemical enrichment of the biosorbent surface, particularly through pore filling and dye

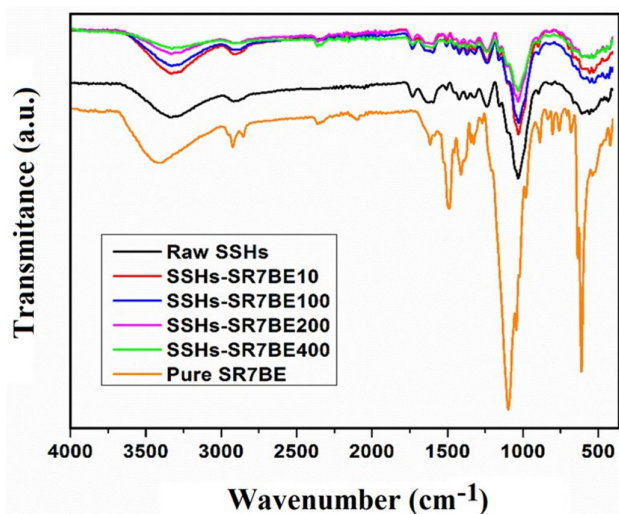


FIGURE 2 | FTIR spectra of pure SR7BE, SSHs before and after adsorption at 10, 100, 200, and 400 mg/L of SR7BE dye.

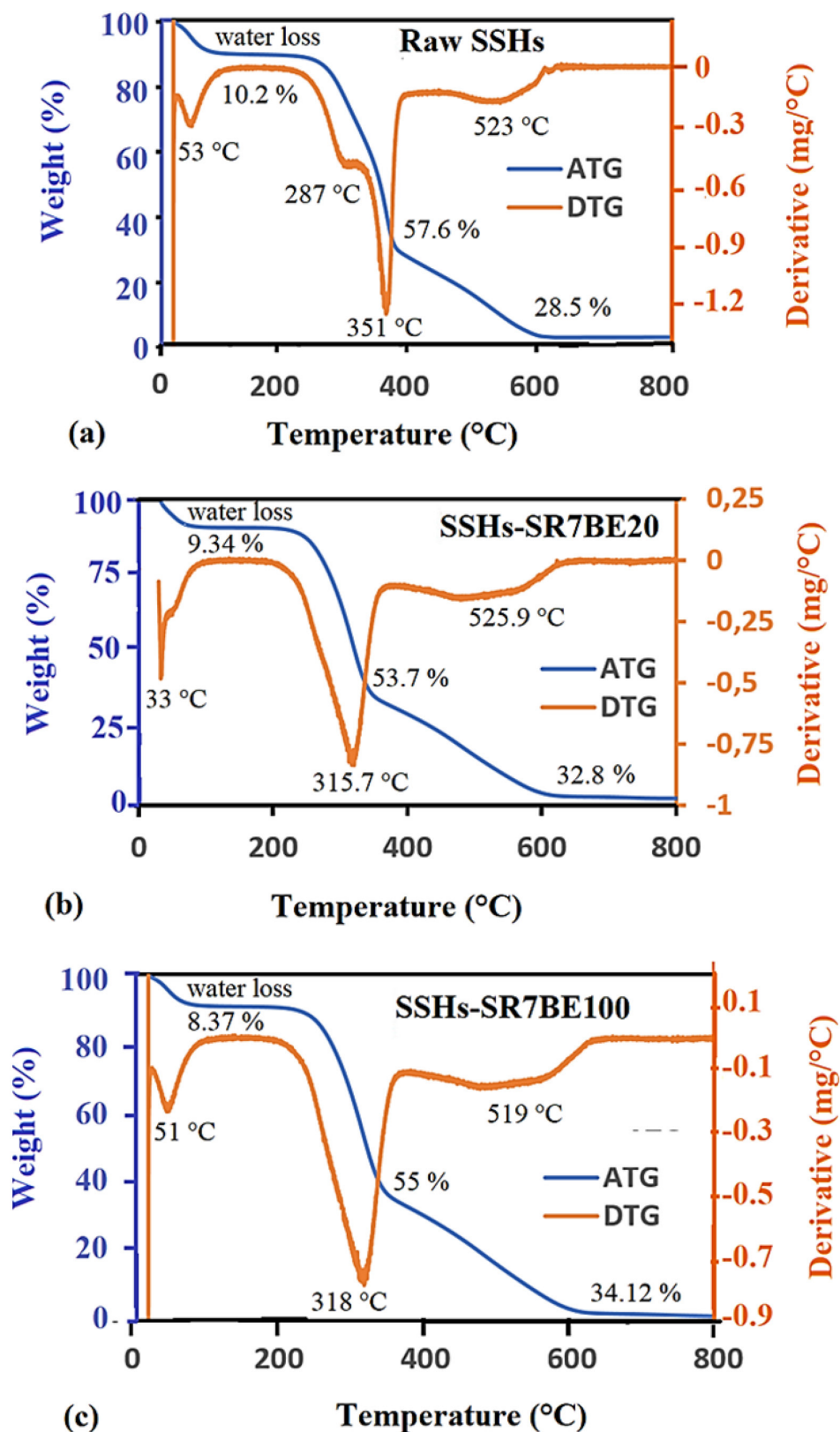


FIGURE 3 | ATG and DTG curves of: (a) Raw SSHs powder, (b) SSHs powder after adsorption at 20 mg/L, and (c) SSHs powder after adsorption at 100 mg/L ($V_i = 100 \text{ mL}$; $m = 2.5 \text{ g L}^{-1}$; pH 7.5; 25°C).

deposition. This observation is consistent with the findings of Grabi, Lemlikchi, et al. (2021), who reported surface deposition and pore filling associated with surface smoothing after dye adsorption.

XPS analysis provided further evidence of changes in the surface chemical composition of SSHs following SR7BE adsorption (Figure 6). Before adsorption, the main surface elements detected were C, O, N, and Si, in agreement with Jiang

et al. (2005), who characterized sunflower shells by XPS and identified C and O as the major surface elements, with N and Si as minor components. In the present study, additional contributions from Na, Mg, K, Ca, and S were detected, reflecting differences in the mineral composition of the SSHs and/or sample preparation conditions.

After adsorption, a pronounced increase in the relative surface N content was observed, from 5.56 to 13.95 at.%, while the S content increased from 1.70 to 2.04 at.%. The increase in nitrogen is consistent with the accumulation of SR7BE-derived species at the

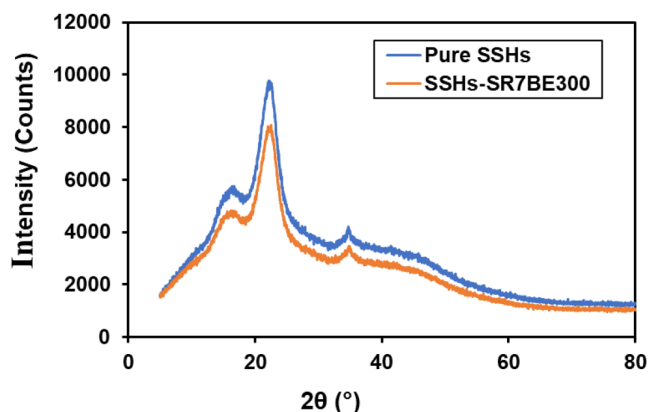


FIGURE 4 | XRD pattern before and after adsorption of SR7BE at 300 mg/L.

biosorbent surface, given the nitrogen-containing structure of the dye. Similarly, the increase in S is consistent with the presence of sulfonate ($-\text{SO}_3^-$) groups in SR7BE. These changes are consistent with the EDS results, which also showed increases in N (2.3 to 6.40 at.%) and S (0.20 to 0.43 at.%) after adsorption. The agreement between XPS and EDS therefore provides complementary evidence for the retention of SR7BE on the SSHs surface.

A decrease in the relative surface concentrations of Na, Mg, and Ca was observed after adsorption. In particular, Na decreased from 26.61 to 21.09 at.%, whereas EDS detected the appearance of Na after adsorption (1.40 at.%). This apparent difference is not necessarily contradictory, as EDS and XPS probe different sampling depths and have different analytical sensitivities. Moreover, XPS atomic percentages are normalized surface compositions; therefore, a decrease in the relative Na content does not necessarily imply an absolute loss of sodium. The decrease in the relative surface contribution of native mineral elements may also be associated with surface masking by the adsorbed dye and/or redistribution of inorganic species during the adsorption process.

Overall, the XPS results are consistent with the FTIR and SEM-EDS observations and provide complementary evidence of SR7BE retention at the SSHs surface. The increase in surface N and S contents is particularly consistent with the incorporation of dye-derived species, while the changes in the relative abundance of mineral elements indicate modification of the surface composition following adsorption. Together with the

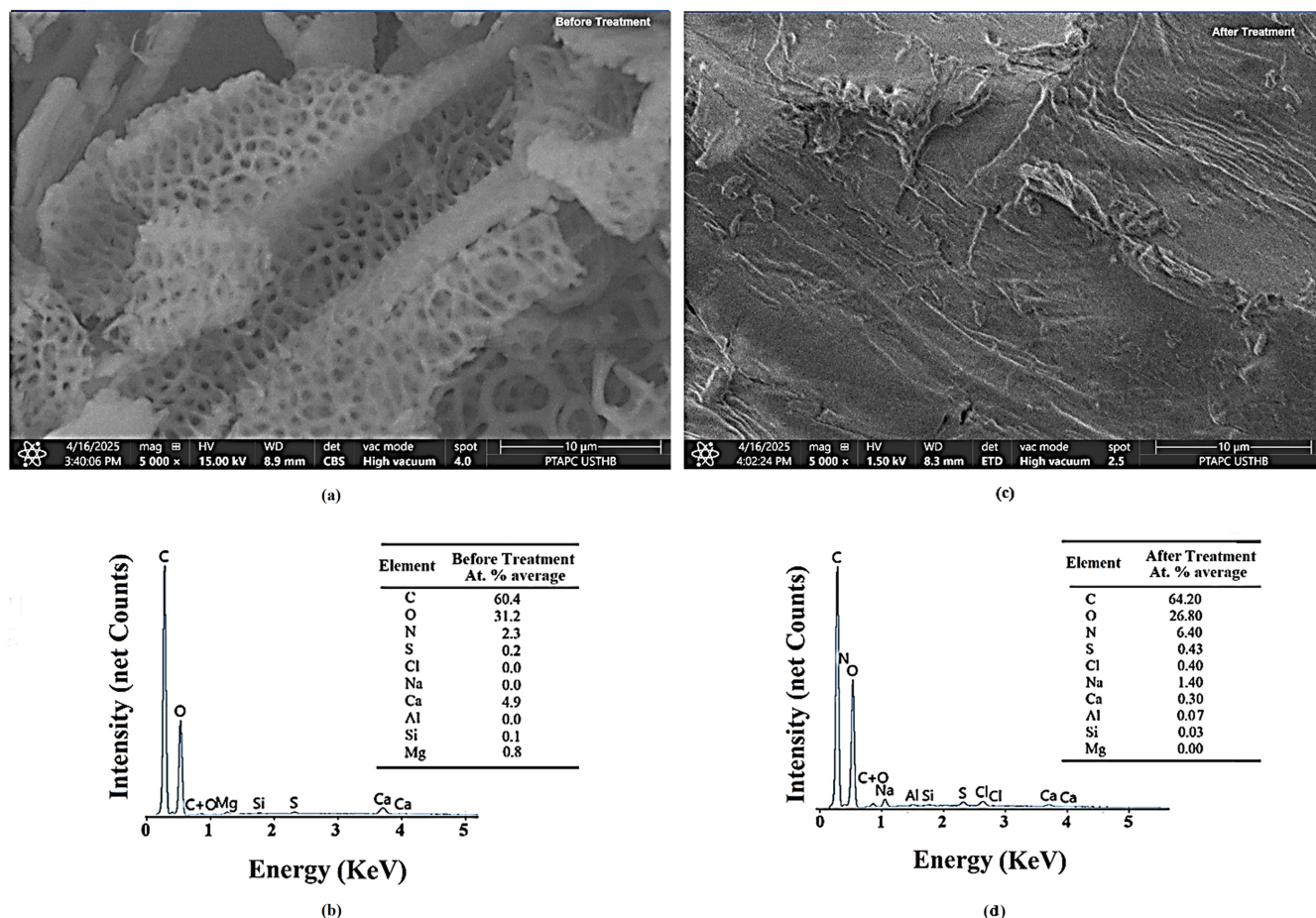


FIGURE 5 | SEM and EDS of SSHs before (a), (b) and after (c), (d) adsorption of SR7BE (400 mg/L).

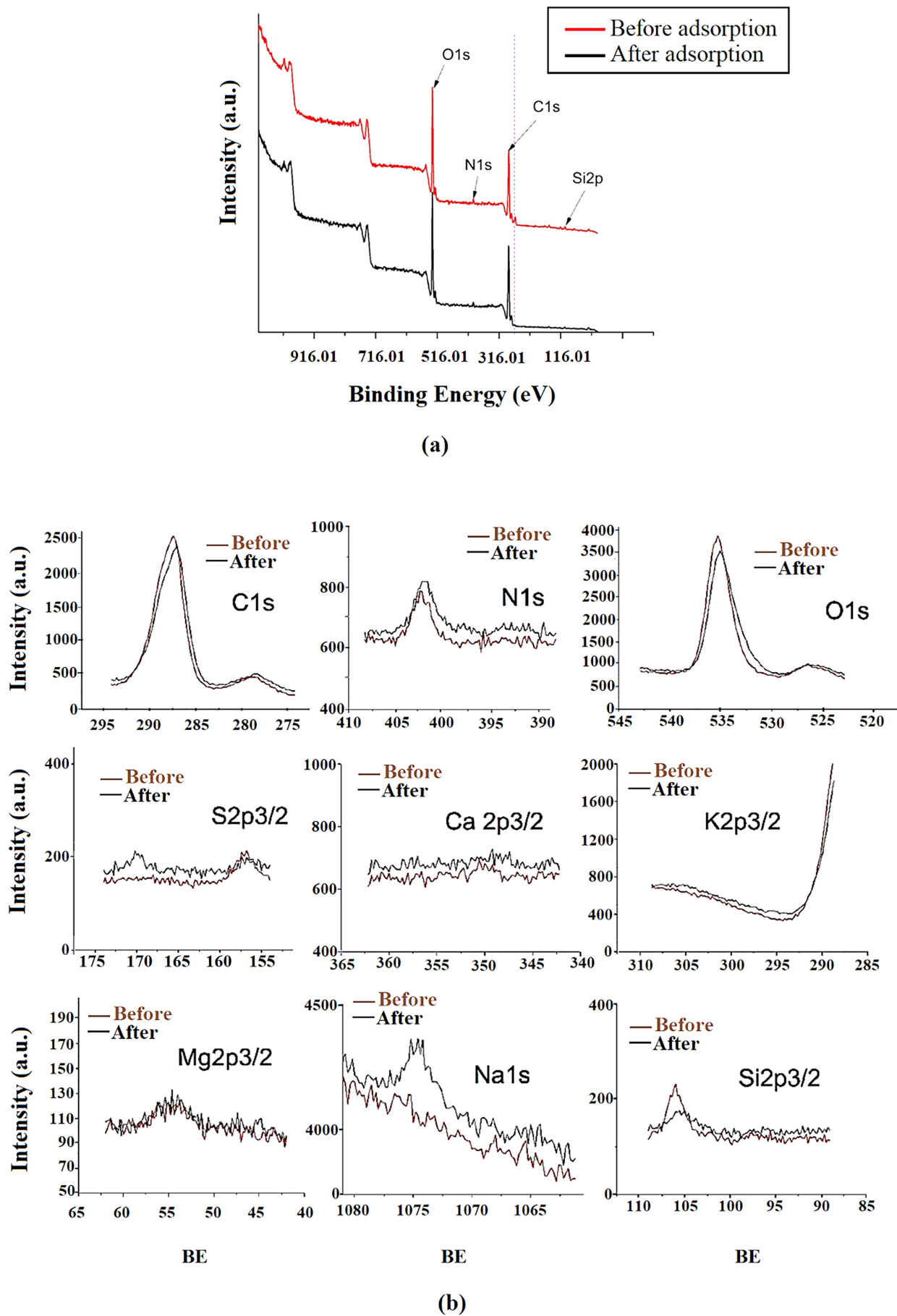


FIGURE 6 | Legend on next page.

FIGURE 6 | XPS characterization of SSHs before and after SR7BE adsorption (400 mg/L): (a) survey spectra and (b) high-resolution spectra of the main surface elements, highlighting the changes in N, S, Na, and Cl signals following adsorption.

XRD and TGA-DTG results, these findings support a multifactorial sorption process involving interactions with surface functional groups, retention on accessible surface regions, and ionic strength-dependent effects at the solid–liquid interface.

Nitrogen adsorption–desorption analysis was performed to determine the textural properties of the washed SSHs, including the specific surface area, pore volume, and average pore diameter. These parameters are important for evaluating the accessibility of adsorption sites and their potential influence on the adsorption performance of the biosorbent. The obtained textural characteristics are summarized in Table 2.

The washed SSHs exhibited a BET specific surface area of $3.40 \pm 0.08 \text{ m}^2 \text{ g}^{-1}$, a BJH adsorption pore volume of $0.00542 \text{ cm}^3 \text{ g}^{-1}$, and an average pore diameter of 9.56 nm, indicating a mesoporous structure according to the IUPAC classification.

The measured BET surface area is within the range reported for untreated sunflower-derived lignocellulosic biomasses. Ferreira et al. (2024) reported a BET surface area of $0.526 \text{ m}^2 \text{ g}^{-1}$ for raw sunflower seed husks, whereas Simón et al. (2022) summarized values of approximately $0.7 \text{ m}^2 \text{ g}^{-1}$ for untreated SSHs. Higher BET surface areas have been reported after physical or chemical modification, reaching $6.11 \text{ m}^2 \text{ g}^{-1}$ for raw shells and $16.6\text{--}27.1 \text{ m}^2 \text{ g}^{-1}$ after activation or chemical treatment (Hayoun et al. 2020; Moyo et al. 2013). These differences are mainly attributed to variations in biomass origin, particle size, washing and pretreatment procedures, drying and degassing conditions, as well as BET measurement protocols.

Although SSHs exhibit a relatively low specific surface area, they achieved excellent adsorption performance and a maximum adsorption capacity. This result suggests that SR7BE adsorption is governed not only by the available surface area but also by the accessibility of active sites and the physicochemical properties of the biosorbent. This interpretation is further supported by the complementary FTIR, XRD, SEM-EDS, TGA, and XPS analyses presented in the following sections, which collectively confirm the involvement of surface functional groups, preservation of the lignocellulosic structure, surface deposition of the dye, and the predominance of a physisorption mechanism.

The colorimetric parameters obtained from the ColorFlex analysis are presented in Table 3.

Color parameters (Table 3) show an increase in L^* , a^* , and b^* values with dye concentration, indicating progressive coloration of SSHs. The increase in a^* values confirms the predominance of the red color component associated with the adsorption of SR7BE onto the biosorbent. These results provide direct visual evidence of successful dye fixation and are in good agreement with the spectroscopic and structural characterization results. Importantly, this colorimetric analysis complements

TABLE 2 | Textural properties of raw washed sunflower seed hulls (SSHs) determined by N₂ adsorption–desorption analysis.

Parameter	Value
BET surface area ($\text{m}^2 \text{ g}^{-1}$)	3.396 ± 0.084
External surface area ($\text{m}^2 \text{ g}^{-1}$)	6.001
Total pore volume ($\text{cm}^3 \text{ g}^{-1}$)	0.00542
Average pore diameter (nm)	9.56

TABLE 3 | CIELAB color parameters (L^* , a^* , b^*) obtained from ColorFlex measurements (SSHs dose 2.5 g L^{-1} , 0.1 M NaCl, neutral pH, $t = 90 \text{ min}$, $24^\circ \text{C} \pm 3^\circ \text{C}$).

Concentration	L^*	a^*	b^*
20 mg/L	42.63	27.66	1.83
50 mg/L	48.08	33.03	2.91

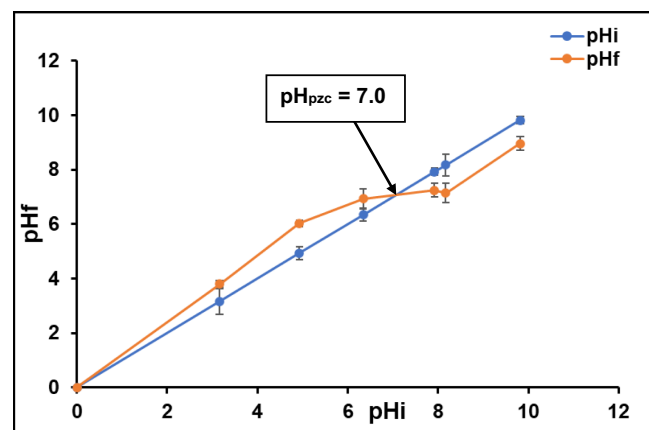


FIGURE 7 | Measurement of the pH at the point of zero charge (pH_{pzc}).

the spectroscopic and structural characterization techniques (FTIR, XRD, XPS, BET, and SEM-EDS), confirming the effective interaction between the dye molecules and the biosorbent. The ColorFlex results thus provide an additional validation of the adsorption process, particularly relevant for practical applications where visual decolorization is a key performance indicator.

3.2 | Adsorption Study

Figure 7 shows the variation of final pH as a function of initial pH for SSHs biosorbent. The material exhibits a pH_{pzc} close to 7. Below this value, the surface becomes positively charged, favoring the adsorption of anionic dyes such as SR7BE. This pH_{pzc} is consistent with the typical pH range of textile effluents (6–7),

indicating that the biosorbent can operate effectively under near-neutral conditions without pH adjustment.

Figure 8 shows the effect of biosorbent dosage under different salinity conditions. Removal efficiency increases with biosorbent dose due to the greater availability of active sites, before reaching a plateau corresponding to surface saturation. At 0 M NaCl, the maximum efficiency is $76.61\% \pm 3.04\%$ (3 g SSHs per 100 mL). At 0.01 M NaCl, a similar efficiency ($74.59\% \pm 4.28\%$) is achieved with only 1 g per 100 mL, while at 0.1 M NaCl, a maximum removal of $97.17\% \pm 0.81\%$ is obtained with 0.25 g per 100 mL, indicating a strong synergistic effect of ionic strength and adsorption capacity. The enhanced adsorption at higher NaCl concentration can be attributed mainly to electrostatic screening and compression of the electrical double layer, which may reduce the electrostatic repulsion between negatively charged surface sites of SSHs and anionic SR7BE species. In addition, Na^+ may act as a counter-ion and contribute to the local association of negatively charged species at the solid–liquid interface, thereby facilitating the approach and retention of SR7BE molecules. This possible counter-ion-mediated interaction can be schematically represented as $\text{SSHs}^- \leftarrow \text{Na}^+ \rightarrow ^-\text{O}_3\text{S}-\text{SR7BE}$ (Figure 1). However, this representation should be considered a plausible contribution to the adsorption mechanism rather than direct evidence of a specific Na^+ -mediated chemical bond.

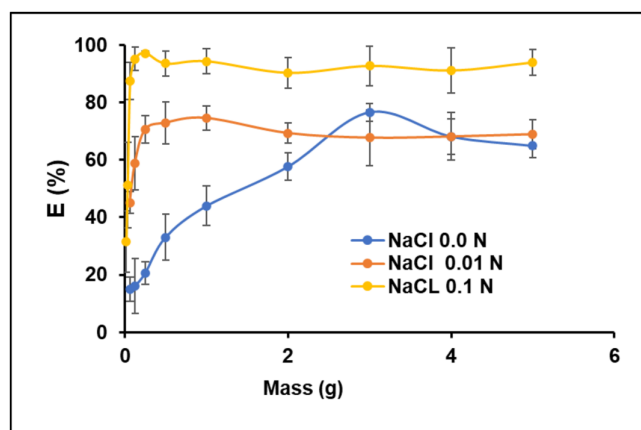


FIGURE 8 | Effect of SSHs mass on the removal of SR7BE efficiency at different electrolyte (NaCl) concentration ($C_i = 20 \text{ mg/L}$; $V = 100 \text{ mL}$, $\text{pH} = 7$; agitation: 250 rpm ; $t = 24 \text{ h}$; $T = 25^\circ\text{C}$).

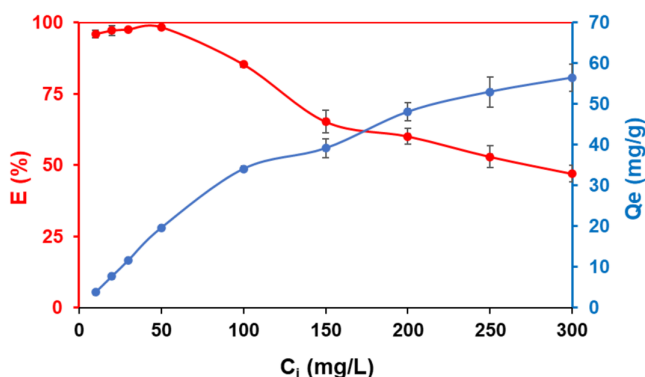


FIGURE 9 | Effect of the SR7BE initial concentration on the SSHs adsorption efficiency and capacity ($\text{pH} = 7$; $V = 100 \text{ mL}$; agitation: 250 rpm ; $t = 24 \text{ h}$; $m = 2.5 \text{ g/L}$; $T = 25^\circ\text{C}$; $\text{NaCl} = 0.1 \text{ M}$).

These observations are consistent with previous studies reporting enhanced adsorption of anionic pollutants onto lignocellulosic materials under increased ionic strength (Dhaouadi et al. 2020; Bentahar et al. 2024; Hu et al. 2013). Notably, the substantial removal observed even in the absence of NaCl (76.61%) indicates that electrostatic screening is not the sole mechanism governing SR7BE uptake. Non-electrostatic contributions, including interactions with surface functional groups, hydrogen bonding, and π - π interactions, may also contribute to dye retention. Thus, the strong increase in removal efficiency upon NaCl addition suggests that electrostatic effects are important, but are not the only interactions involved in SR7BE adsorption.

Figure 9 presents the effect of initial dye concentration (10 – 300 mg/L^{-1}) on removal efficiency and adsorption capacity.

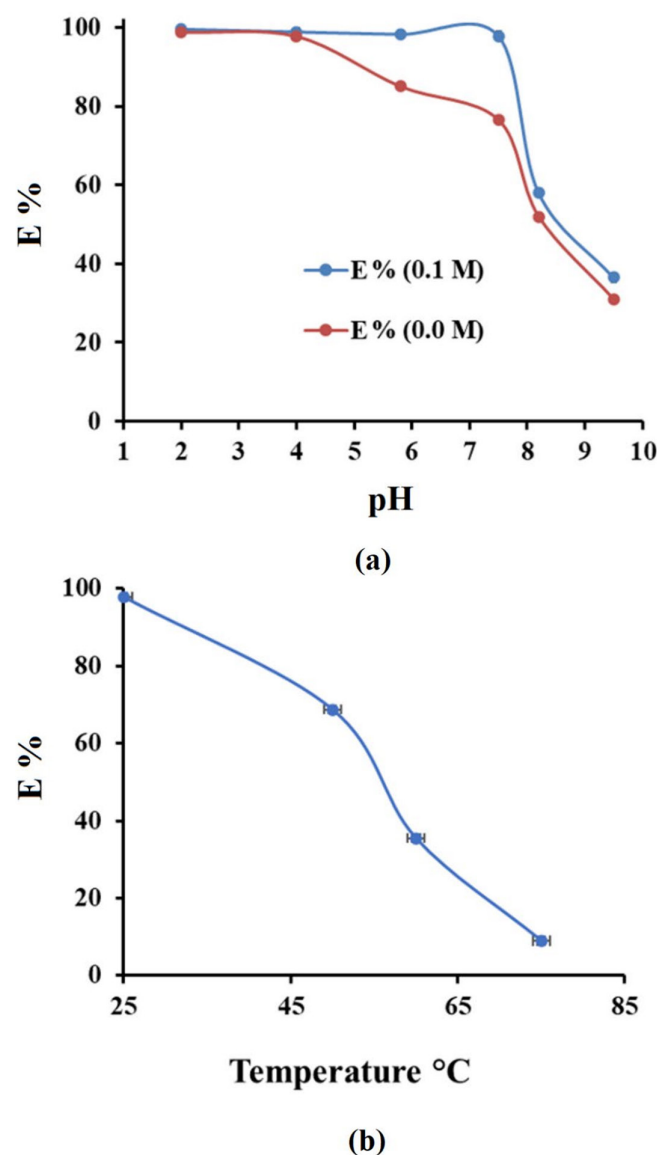


FIGURE 10 | (a) Effect of pH on the removal efficiency (%E) of Solophenyl Red 7BE by sunflower seed hull powder (SSHP) in the absence (0.0 M) and the presence (0.1 M) of NaCl; (b) effect of temperature ($T, ^\circ\text{C}$) on the removal efficiency (%E) of Solophenyl Red 7BE by sunflower seed hull powder (SSHP) in the presence of 0.1 M NaCl. ($\text{pH} = 7$; $V = 100 \text{ mL}$; agitation: 250 rpm ; $C_i = 20 \text{ mg/L}$; $m = 2.5 \text{ g/L}$; $T = 25^\circ\text{C}$; $\text{NaCl} = 0.1 \text{ M}$; $t = 90 \text{ min}$).

Under the selected experimental conditions (0.1 M NaCl, 0.25 g SSHs per 100 mL, neutral pH, 24 h contact time), maximum efficiency reaches $98.38\% \pm 0.43\%$ at low concentrations ($10\text{--}50\text{ mg L}^{-1}$), corresponding to a maximum adsorption capacity of $19.67 \pm 0.09\text{ mg g}^{-1}$. Beyond 50 mg L^{-1} , removal efficiency decreases due to progressive saturation of active sites on the biosorbent surface. This behavior indicates a capacity-limited adsorption process at higher dye loads and is consistent with previous reports on azo dye adsorption onto lignocellulosic materials (Grabi, Lemlikhi, et al. 2021).

Figure 10 shows the effects of pH and temperature on SR7BE removal by SSHP. Figure 10a illustrates the effect of pH on SR7BE removal in the absence and the presence of NaCl. High removal efficiencies ($98.8\% \pm 1\%$ – $99.5\% \pm 0.26\%$) were obtained under acidic conditions (pH 2–4), consistent with the pH_{pzc} of SSHs (≈ 7). Below the pH_{pzc}, the surface is expected to be positively charged, favoring electrostatic attraction with the anionic dye. This behavior is consistent with previous studies on the adsorption of anionic dyes onto agricultural biosorbents. Grabi, Derridj, et al. (2021) reported enhanced Cibacron Blue removal under acidic conditions and attributed this behavior to the positive surface charge of the biosorbent below its pH_{pzc}, which favors electrostatic attraction with the anionic dye.

Above pH 5.8, removal progressively decreased in the absence of NaCl, from $76.67\% \pm 0.9\%$ to $31.0\% \pm 2.8\%$ at pH 9.5, which can be attributed to the increasing negative surface charge and the resulting electrostatic repulsion with the anionic dye. In the presence of NaCl, removal remained higher up to pH 7 and then decreased, reaching $36.51\% \pm 0.15\%$ at pH 9.5, suggesting partial screening of electrostatic repulsion by the increased ionic strength.

Overall, the results confirm that electrostatic interactions strongly govern SR7BE adsorption, while the remaining uptake suggests additional non-electrostatic contributions.

Figure 10b shows the effect of temperature on SR7BE removal in the presence of 0.1 M NaCl. A clear decrease in removal efficiency was observed as the temperature increased. The removal efficiency decreased from 97.8% at 25°C to 68.8% at 50°C, 35.5% at 60°C, and only 9.0% at 75°C. This pronounced decrease indicates that increasing temperature is unfavorable for SR7BE adsorption onto SSHs, suggesting an exothermic adsorption process. The reduced removal at higher temperatures may be associated with a weakening of the interactions between SR7BE molecules and the active sites of SSHs, resulting in enhanced desorption of the adsorbed dye. Therefore, lower temperatures favor SR7BE uptake, with the highest removal efficiency obtained at 25°C.

Thermodynamic analysis was further performed using independent equilibrium data obtained at 25°C, 50°C, 60°C, and 75°C following the approach proposed by Lima et al. (2019). The dimensionless standard equilibrium constant (K°) was determined from the corresponding adsorption isotherms and used for the Van't Hoff analysis. The linear regression of $\ln K^\circ$ versus $1/T$ yielded an R^2 of 0.9172 (Figure S1). The calculated ΔH° and ΔS° values were $-73.59\text{ kJ mol}^{-1}$ and $-144.43\text{ J mol}^{-1}\text{ K}^{-1}$, respectively (Table 4). The negative ΔH°

TABLE 4 | Thermodynamic parameters for the adsorption of SR7BE onto sunflower seed hull powder.

Temperature	ΔG° (kJ mol ⁻¹)
25°C	-31.43
50°C	-25.15
60°C	-24.85
75°C	-24.80
ΔH° (kJ mol ⁻¹)	-73.59
ΔS° (J mol ⁻¹ K ⁻¹)	-144.43
Van't Hoff R^2	0.9172

confirms the exothermic nature of the adsorption process, consistent with the decrease in removal efficiency observed with increasing temperature. The negative ΔS° suggests a decrease in molecular freedom at the solid–solution interface during adsorption. The ΔG° values were negative at all investigated temperatures (-31.43 , -25.15 , -24.85 , and $-24.80\text{ kJ mol}^{-1}$ at 25°C, 50°C, 60°C, and 75°C, respectively), indicating that the adsorption remained thermodynamically favorable over the investigated temperature range. The progressive increase in ΔG° toward less negative values with increasing temperature further indicates that higher temperatures are unfavorable for SR7BE adsorption.

The detailed equilibrium constants and thermodynamic calculations are provided in Table S1, together with the Van't Hoff plot (Figure S1).

3.3 | Kinetic and Isothermal Study

Figure 11 shows the evolution of SR7BE removal efficiency and adsorption capacity (q_t) as a function of contact time. Experiments were conducted at 0.25 g biosorbent per 100 mL, 0.1 N NaCl, and 20 mg L^{-1} of SR7BE dye concentration. A rapid increase in SR7BE removal was observed during the first minutes of adsorption, with the removal efficiency increasing from 27.41% after 1 min to 84.67% after only 5 min, accompanied by an increase in adsorption capacity from 2.19 to 6.77 mg g^{-1} . Equilibrium was reached after approximately 90 min, corresponding to a maximum removal efficiency of $97.98\% \pm 0.40\%$ and an adsorption capacity of $7.83 \pm 0.03\text{ mg g}^{-1}$. This equilibrium time is substantially shorter than those reported for raw SSHs in previous studies, namely 210 min (Osma et al. 2007) and 150–240 min (Srisorrachatr 2011). After equilibrium, only slight fluctuations in both removal efficiency and adsorption capacity were observed up to 24 h. The complete experimental data are provided in Table S2.

These small variations may reflect the establishment of a dynamic adsorption–desorption equilibrium, slight desorption of weakly bound dye molecules, or other interfacial equilibrium phenomena occurring at the biosorbent surface. However, the overall adsorption performance remained essentially unchanged, indicating that the SSHs–SR7BE system was stable over the investigated contact time.

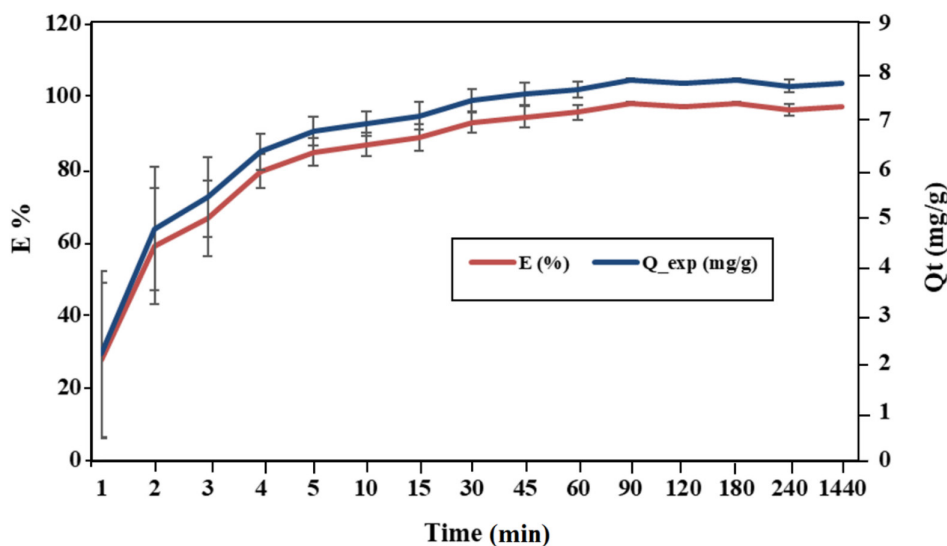


FIGURE 11 | Experimental adsorption kinetics aspect of biosorption dye SSHs-SR7BE (pH=7; V=100mL; agitation: 250rpm; Ci=20 mg/L; m=2.5 g/L; T=25°C; NaCl=0.1 M).

TABLE 5 | Models for the adsorption of ST7BE dye onto SSHs and adsorption performance.

Isotherm model	Parameter	Value	R ²	RMSE (mg/g)
Langmuir	Q_{max} (mg/g)	49.97	0.913	5.583
	K_L (L/mg)	0.33		
Freundlich	K_F (mg/g)	12.88	0.950	3.940
	$1/n_F$	0.30		
MMRG	N	0.60	0.947	4.410
	N_m (mg/g)	111.00		
	W	19.00		
	b (L/mg)	2.10×10^{-3}		
	a (J·L/mg)	4×10^{-24}		
Temkin	B (mg/g)	7.91	0.970	3.431
	K_T (L/mg)	5.97		
Dubinin–Radushkevich	q_m (mg/g)	47.17	0.884	6.706
	β (mol ² /J ²)	$2.68 \cdot 10^{-7}$		

Note: The bold values indicate the best results obtained under the corresponding experimental conditions.

The fast initial removal of SR7BE can be attributed to the high availability of accessible adsorption sites, whereas the subsequent slower stage may result from their progressive occupation.

To further characterize the adsorption kinetics of SR7BE, the experimental data were fitted to the PFO, PSO, Elovich, and Weber–Morris intraparticle diffusion models (Liu and Shen 2008; Musah et al. 2022; Weber and Morris 1963; Özmal and Tekke 2023). The PFO model assumes that adsorption depends on the difference between the equilibrium capacity and the amount adsorbed at time t , whereas the PSO model

describes adsorption according to a second-order rate equation. The Elovich model is commonly applied to heterogeneous adsorption systems, whereas the Weber–Morris model is used to assess the contribution of intraparticle diffusion to the overall adsorption process.

The kinetic results indicate that both the PFO and PSO models satisfactorily describe the adsorption of SR7BE onto SSHs. The PSO model showed a calculated equilibrium adsorption capacity ($Q_{e,cal} = 7.917 \text{ mg g}^{-1}$) close to the experimental value ($Q_{e,exp} = 7.83 \pm 0.03 \text{ mg g}^{-1}$). However, the PFO model provided better overall statistical indicators, with a higher R^2 (0.9656) and lower RMSE (0.284 mg g^{-1}), compared with the PSO model ($R^2 = 0.9421$; RMSE = 0.369 mg g^{-1}). The Elovich model showed a lower goodness of fit ($R^2 = 0.7138$; RMSE = 0.82 mg g^{-1}), while the Weber–Morris model provided the lowest R^2 (0.4476) and an RMSE of 1.138 mg g^{-1} . These results suggest that intraparticle diffusion alone does not adequately describe the overall adsorption kinetics. The complete kinetic parameters are provided in Table S3. Based on the statistical criteria, the PFO model provides the most appropriate empirical description of the adsorption kinetics under the investigated conditions. Nevertheless, kinetic modeling alone does not provide sufficient evidence to identify the adsorption mechanism or distinguish between physisorption and chemisorption (Brandani 2021). Mechanistic interpretation should therefore rely on complementary equilibrium modeling and physicochemical characterization (Ahmed et al. 2025).

The equilibrium adsorption data were analyzed using the Langmuir, Freundlich, MMRG, Temkin, and D–R isotherm models (Langmuir 1916; Freundlich and Heller 1939; Bergaoui et al. 2018; Grabi, Lemlikchi, et al. 2021; Hayoun et al. 2020; Temkin and Pyzhev 1940; Dubinin and Radushkevich 1947). The Langmuir model assumes monolayer adsorption onto a homogeneous surface, whereas the Freundlich model describes adsorption on heterogeneous surfaces with non-uniform adsorption energies and possible multilayer adsorption. The MMRG model considers heterogeneous surfaces with multiple adsorption sites and allows for multilayer adsorption. The Temkin

model considers a progressive decrease in adsorption energy with increasing surface coverage, while the D–R model describes adsorption on heterogeneous surfaces through an energy distribution approach.

The model-fitting results are summarized in Table 5. Among the tested models, the Temkin model provided the best overall statistical fit, with the highest R^2 (0.9700) and the lowest RMSE (3.431 mg g^{-1}). This result suggests that the adsorption energy progressively decreases as the surface coverage increases, which is consistent with interactions between adsorbed molecules and a heterogeneous distribution of adsorption energies.

The Freundlich model also provided a good fit, with $R^2=0.950$ and $\text{RMSE}=3.940 \text{ mg g}^{-1}$. Moreover, the value of $1/nF=0.2968$

(<1) indicates favorable adsorption and supports the heterogeneous nature of the SSHs surface. A similar application of Langmuir and Freundlich models to evaluate adsorption behavior was reported by Allwar et al. (2019). The MMRG model also showed a good fit ($R^2=0.947$; $\text{RMSE}=4.410 \text{ mg g}^{-1}$), further supporting the contribution of surface heterogeneity and concentration-dependent adsorption.

In comparison, the Langmuir model provided a lower fit ($R^2=0.913$; $\text{RMSE}=5.583 \text{ mg g}^{-1}$), indicating that a homogeneous monolayer model does not fully describe the experimental equilibrium data. The D–R model showed a lower statistical performance ($R^2=0.884$; $\text{RMSE}=6.706 \text{ mg g}^{-1}$), indicating that this model is less suitable for describing the experimental equilibrium data over the investigated concentration range.

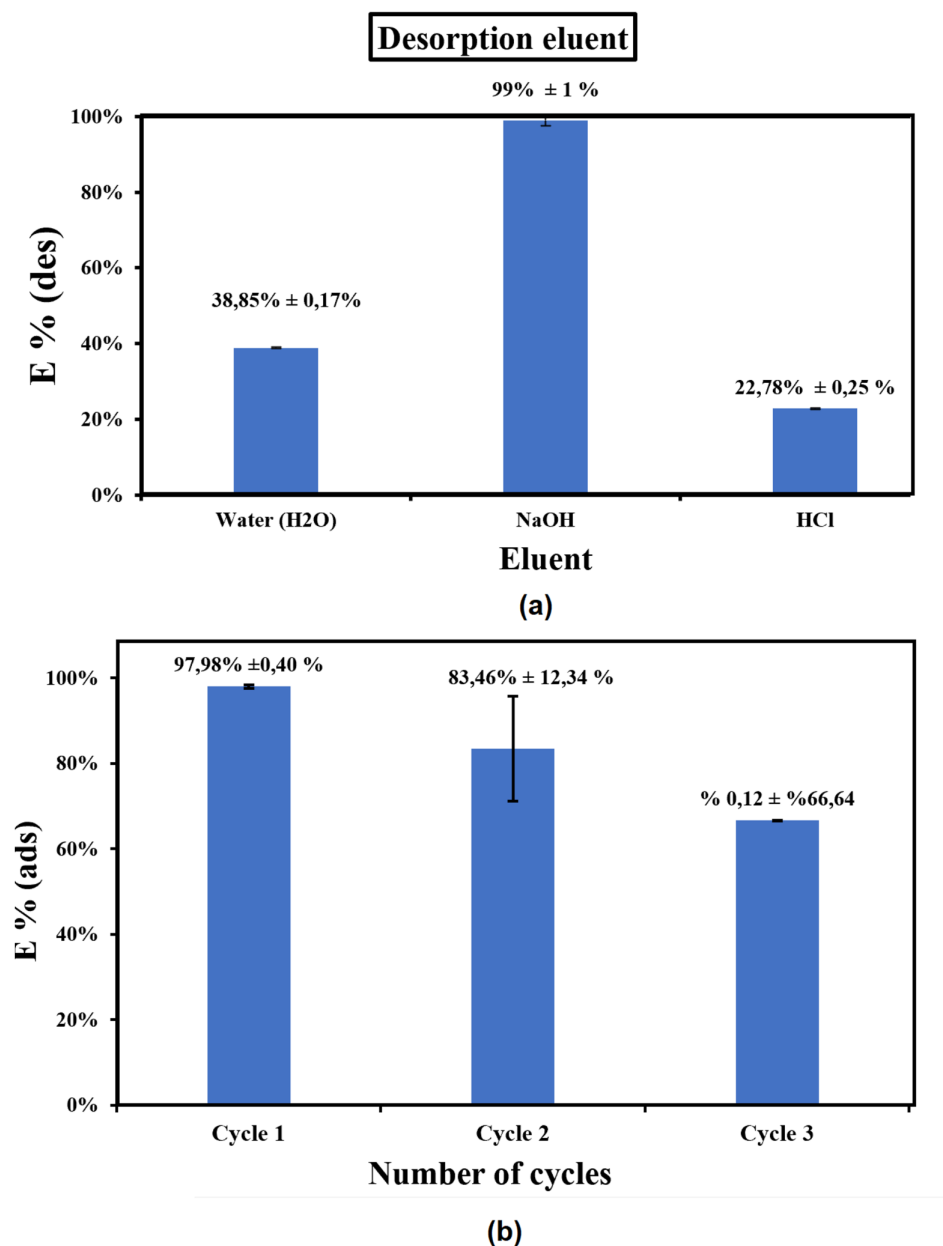


FIGURE 12 | (a) Effect of the eluents (water, NaOH, and HCl) on SR7BE desorption from SSHs; (b) reusability of SSHs over three consecutive adsorption–desorption cycles (initial SR7BE concentration: 20 mg L^{-1} ; biosorbent dosage: 0.25 g per 100 mL ; temperature: $25^\circ\text{C} \pm 2^\circ\text{C}$; contact time: 90 min).

Overall, the equilibrium analysis indicates that SR7BE adsorption onto SSHs is better described by heterogeneous surface interactions than by a simple homogeneous monolayer model. The superior performance of the Temkin model suggests a progressive decrease in adsorption energy with increasing surface coverage, whereas the Freundlich and MMRG models further support surface heterogeneity and possible multilayer adsorption. The separation factor (RL) values between 0 and 1 additionally confirm the favorable nature of the adsorption process.

These equilibrium models should not, however, be considered as definitive evidence of physisorption or chemisorption. When considered together with the complementary FTIR, XRD, XPS, and SEM-EDS analyses, the results suggest that several interactions may contribute to SR7BE retention, including electrostatic interactions, hydrogen bonding, and possible π - π interactions. These possible interactions are schematically represented in the proposed sorption mechanism (Figure 1).

3.4 | Regeneration and Reusability Performance of SSHs

Figure 12a shows the desorption efficiency of SR7BE using water, 0.1 M NaOH, and 0.1 M HCl. NaOH exhibited the highest desorption efficiency ($99\% \pm 1\%$), followed by water ($38.85\% \pm 0.17\%$), whereas HCl showed the lowest efficiency ($22.78\% \pm 0.25\%$). The different performances of the eluents indicate that regeneration efficiency depends on the chemical nature of the dye-adsorbent interactions and the chemical environment imposed by the eluent. The higher desorption obtained with NaOH may be associated with modification of the surface charge under alkaline conditions, which weakens the interactions between SR7BE and the SSHs surface. Similar eluent-dependent regeneration behavior has been reported for dye-adsorbent systems (Grabı, Derridj, et al. 2021; Belounis et al. 2025). Based on its highest desorption efficiency, NaOH was therefore selected for the reusability experiments.

Figure 12b shows the adsorption performance of SSHs over three successive adsorption-desorption cycles using NaOH for regeneration. The removal efficiency decreased from $97.98\% \pm 0.40\%$ in the first cycle to $83.46\% \pm 12.34\%$ in the second and $66.64\% \pm 0.12\%$ in the third cycle. This gradual decrease may be attributed to incomplete dye desorption and/or partial modification or blockage of accessible adsorption sites during successive regeneration steps. A decrease in adsorption performance over repeated cycles has also been reported for raw sunflower seed shell particles, which nevertheless retained their adsorption ability over several adsorption-desorption cycles (Mousavi et al. 2022).

3.5 | Application to Real Textile Effluent and UV-Vis Analysis

The applicability of the adsorption process was evaluated using a real textile effluent containing SR7BE under the corresponding laboratory conditions as for aqueous dye solution ($0.25\text{ g}/100\text{ mL}$ SSHs, 0 M NaCl and 0.1 M NaCl, 20 mg L^{-1} of real textile effluent, neutral solution pH, $24^\circ\text{C} \pm 3^\circ\text{C}$ and 24 h contact time). Removal efficiencies determined by UV-Vis spectrophotometry at $\lambda_{\text{max}} = 540\text{ nm}$.

The results show a strong limitation of the process in the absence of electrolyte, with a removal efficiency of 17.6% , highlighting the impact of matrix effects in real effluent conditions. In contrast, the addition of NaCl significantly enhanced performance, reaching 84.6% , corresponding to an increase of approximately 67% (Table S4).

UV-Vis spectra (Figure 13) confirm these observations. The untreated effluent shows a broad absorption band in the visible region, indicating the presence of colored organic compounds. After treatment, a marked decrease in absorbance intensity is observed, particularly in the presence of NaCl, confirming improved dye removal. This enhancement is attributed to electrical double layer compression, which reduces electrostatic repulsion between anionic dye molecules and the biosorbent surface, thereby facilitating adsorption. No significant shift in λ_{max} is observed after treatment, indicating that the process is governed by adsorption rather than chemical degradation, with dye molecules transferred from the liquid phase to the biosorbent surface without structural modification. Overall, these results demonstrate that matrix effects strongly limit biosorption efficiency under real conditions, but can be effectively mitigated by controlling ionic strength, thereby improving process applicability for textile wastewater treatment. To further confirm the absence of transformation products and strengthen mechanistic understanding, complementary techniques such as HPLC or LC-MS could be considered in future studies.

3.6 | Comparative Assessment With Previous Studies and Future Perspectives

To further assess the performance and practical relevance of the present approach, a comparison with previously reported studies using sunflower-based adsorbents was conducted. The main adsorption parameters, including pH, contact time, maximum

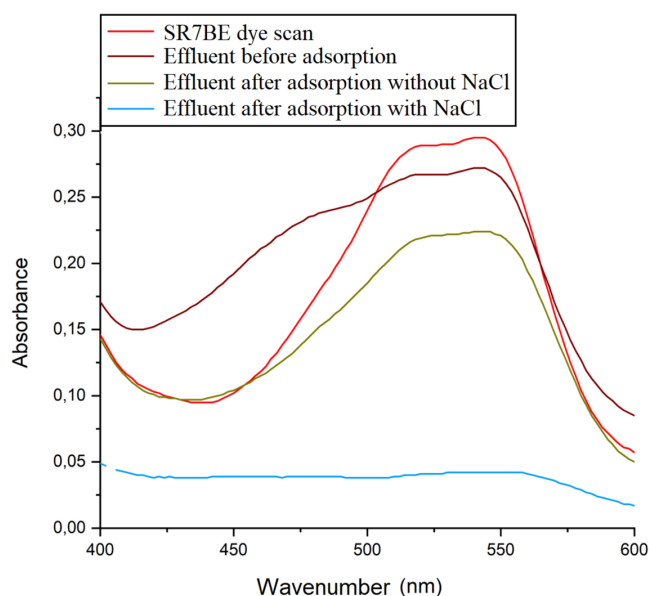


FIGURE 13 | Comparison of the UV-Visible spectra of the SR7BE dye, the real effluent before treatment, and after adsorption on SSHs with and without NaCl (pH = 7; V = 100 mL; agitation: 250 rpm; t = 24 h; m = 2.5 g/L; T = 25°C; NaCl = 0.1 M).

TABLE 6 | Comparison of dye adsorption performance of sunflower-based adsorbents reported in previous studies and in the present work.

Reference/Pollutant	Adsorbent/Process	pH	Contact time	Removal efficiency			Key outcome
				Q_{max} (mg g ⁻¹)	(E%)	Real effluent	
Jóźwiak et al. 2020—Reactive Black 5, Reactive Yellow 84, Acid Yellow 23, Acid Red 18	Aminated sunflower seed hulls	~3	90–240 min	51.02–63.27 44.78–42.19	80–90	No	Amination increased adsorption capacity by 15–24-fold, but required an additional chemical modification step.
Zhao et al. 2023—Acid Fuchsin (AF)	Sunflower straw activated carbon (SSAC4); H ₃ PO ₄ activation	–	200 min	2763.36	97.73	No	High adsorption capacity obtained after H ₃ PO ₄ activation; however, preparation requires chemical activation and therefore involves greater processing complexity than raw SSHs.
Kocadağistan and Kocadağistan 2016—Astrazon Red	H ₂ SO ₄ -treated sunflower seed shells	~7.4	120 min	284.45 mg/g	> 97	No	High removal after chemical treatment, but with additional chemical consumption and process complexity.
Srisorrachatr 2011—Methylene blue/Mexican red	NaOH/HCl-treated sunflower husk	2–10	350 min	67.114–42.017	95.87	No	Enhanced removal after chemical modification, requiring additional treatment steps.
Osma et al. 2007—Reactive Black 5	Raw sunflower hulls	2	210 min	Q _{e,exp} = 1.10	71–85	No	Good removal using untreated biomass with low process complexity.
This study—Solophenyl Red 7BE (Direct Red 227)	Raw sunflower seed hulls (SSHs); adsorption in the presence of NaCl electrolyte (0.1 M)	~7	90 min	49.97	97.98 ± 0.40	Yes; 84.6% with NaCl	High removal using an untreated, low-complexity biosorbent without chemical activation or thermal treatment; validated with real textile effluent.

adsorption capacity, removal efficiency, use of real effluent, and process complexity, are summarized in Table 6.

As shown in Table 6, previous studies have employed sunflower-derived adsorbents with different degrees of chemical or thermal modification. For example, H_2SO_4 treatment, HCl/NaOH modification, amination, and thermal activation have been used to enhance the adsorption performance of sunflower-based materials (J  zwiak et al. 2020; Kocada  stan and Kocada  stan 2016; Srisorrachatr 2011). Although such treatments can substantially improve adsorption performance, they require additional processing steps and may increase chemical consumption, energy demand, and overall process complexity. In contrast, Osma et al. (2007) demonstrated dye removal using raw sunflower hulls, although with a longer equilibrium time and lower removal efficiency than those obtained in the present study. Zhao et al. (2023) also reported a high adsorption capacity using H_3PO_4 -activated sunflower straw carbon, illustrating the substantial enhancement that can be achieved through chemical activation.

In the present study, untreated SSHs achieved a high SR7BE removal efficiency of $97.98\% \pm 0.40\%$ within 90 min, without chemical activation or thermal treatment. Moreover, the beneficial effect of NaCl was demonstrated as a simple operational strategy to enhance adsorption, without modifying the biosorbent itself. The process was further validated using a real textile effluent, achieving 84.6% dye removal under saline conditions. The regeneration experiments further demonstrated that the SSHs could be reused over three successive adsorption–desorption cycles, although a gradual decrease in adsorption performance was observed. The main advantages of the proposed approach are therefore its low processing complexity, the use of an untreated agricultural by-product, high removal efficiency, and applicability to real textile effluent. However, its main limitation is the relatively low adsorption capacity compared with some chemically or thermally modified sunflower-based adsorbents, as well as the decrease in removal efficiency observed in real effluent compared with synthetic solutions. Thus, the present approach offers a simpler and potentially more sustainable alternative for the treatment of a scarcely investigated anionic dye.

4 | Conclusion

This study demonstrated the potential of raw SSHs as a low-cost and sustainable biosorbent for the removal of the scarcely investigated azo dye SR7BE (Direct Red 227) from aqueous solutions and real textile effluent. Under the selected operating conditions (2.5 g L^{-1} SSHs, 20 mg L^{-1} SR7BE, 0.1 M NaCl , neutral pH, 90 min, and $24^\circ\text{C} \pm 3^\circ\text{C}$), a high removal efficiency of $97.98\% \pm 0.40\%$ and an adsorption capacity of $7.83 \pm 0.03\text{ mg g}^{-1}$ were achieved. The pH study showed higher SR7BE removal under acidic conditions, consistent with the positive surface charge of SSHs below their pH_{pzc} (≈ 7), whereas removal decreased under alkaline conditions. Kinetic modeling showed that the PFO model provided the best overall statistical fit, while equilibrium data were better described by the Temkin model, which is consistent with a progressive decrease in adsorption energy with increasing surface coverage, whereas the good fits obtained with the Freundlich and MMRG models further support surface heterogeneity and possible multilayer adsorption.

Complementary FTIR, XRD, XPS, SEM–EDS, BET, and thermogravimetric analyses provided complementary information on the physicochemical properties of SSHs and their changes following dye adsorption. The addition of NaCl markedly enhanced the adsorption performance, highlighting the crucial role of ionic strength in promoting dye–biosorbent interactions under realistic wastewater conditions. The successful application of the process to real textile effluent, together with the satisfactory regeneration performance of the biosorbent, demonstrates the practical feasibility of this simple and sustainable approach. Overall, raw SSHs constitute a promising, low-cost and environmentally friendly biosorbent for the treatment of textile wastewater containing scarcely investigated dyes and provide a solid basis for future scale-up and industrial applications. Future work should focus on improving the regeneration efficiency over successive cycles, evaluating the biosorbent with different real textile effluents and more complex wastewater matrices, and assessing the process at pilot scale to determine its long-term operational and economic feasibility. Further studies could also investigate the treatment of effluents containing mixtures of dyes and other coexisting pollutants to better assess the applicability of SSHs under realistic industrial conditions.

Author Contributions

Selma Si smail: conceptualization, formal analysis, visualization, writing – original draft, writing – review and editing, validation. **Wahiba Lemlikchi:** conceptualization, visualization, validation, methodology, supervision, resources. **Marie Laure Fauconnier:** conceptualization, visualization, methodology, supervision, validation, resources. **Safia Lemlikchi:** visualization, writing – review and editing, validation. **Messaoud Hachemi:** resources, validation, methodology, visualization. **Amina Belounis:** resources, visualization, validation. **Adhya-Eddine Hamitouche:** resources, validation, visualization.

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Artificial intelligence (AI) tools were used solely to improve the linguistic quality of the manuscript (grammar, spelling, and clarity of expression). AI was also used to generate two graphical elements in the graphical abstract (the NaCl bottle and the red-colored wastewater container). All scientific content, data analysis, interpretation, conclusions, and the overall design and assembly of the graphical abstract were performed by the authors.

Conflicts of Interest

The authors declare no conflicts of interest.

Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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Supporting Information

Additional supporting information can be found online in the Supporting Information section. **Table S1:** Standard thermodynamic parameters and dimensionless equilibrium constants for SR7BE adsorption onto raw sunflower seed hulls at different temperatures. **Table S2:** Kinetics of SR7BE dye removal by SSHs as a function of contact time. **Table S3:** Kinetic parameters for SR7BE adsorption onto SSHs obtained from non-linear model fitting. **Table S4:** Effect of ionic strength on dye removal efficiency in real textile effluent (UV-Vis analysis at $\lambda_{max} = 540\text{ nm}$). **Figure S1:** Van't Hoff regression plot of $\ln K^o$ versus $1/T$ for SR7BE adsorption onto sunflower seed hull powder.