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ECO-FRIENDLY BIOSORBENT BASED ON LOCAL RAW MATERIAL: APPLICATION TO DYE REMOVAL

Purpose. A leading cause of water contamination is the widespread use of dyes, which are consumed in large amounts. The purpose of the present study was to investigate the potential of the Cactus paddle (CP) as an environmentally safe biosorbent for removing Basic Red 46 dye from aqueous media.

Methodology. Various analytical tools, comprising XRF, XRD, BET/BJH, FTIR, and pH_{PZC} methods, were used to examine the surface characteristics of the biosorbent. For assessing the ability of CP for BR46 adsorption, batch adsorption assays were performed.

Findings. An amount of 3.45 mg · g^{-1} of BR46 was adsorbed at pH 7, 500 rpm and 293 K within 180 minutes. The isothermal data was described by both the Freundlich and the Langmuir equations with $R^2 \geq 0.984$ and $\chi^2 \leq 0.01$. The kinetics was well described by a PSO model, suggesting that physico-chemical interactions govern the adsorption process.

Originality. For the first time, the effective use of a local raw material resource CP as a biosorbent for the removal of BR46 dye has been substantiated.

Practical value. As a result of the findings of this study, it seems possible that CP can be regarded as a sustainable resource, which would create a possibility for addressing environmental concerns in the future.

Keywords: *biosorbent Cactus paddle, adsorption, basic Red 46 dye*

Introduction. Water contamination is one of the most urgent problems facing the globe today. Dyes from the textile, printing and leather industries present one of the leading causes of water pollution. This negatively impacts both aquatic and terrestrial life. These substances can lead to various ailments and are extremely hazardous to the environment. They are also mutagenic and carcinogenic to living organisms. So, it is vital to lower the dye concentration before releasing it into water bodies.

The removal of dyes takes a lot of attention nowadays. For this purpose, an assortment of biological, chemical and physical techniques has been used. These techniques have benefits and drawbacks. The majority of them are costly and intricate; however, adsorption is the approach used the most frequently to purify water. Compared to other traditional procedures, this method operates effortlessly and is straightforward and efficient. Numerous adsorbents may be utilized to conduct dye adsorption from wastewater. Activated carbon, alumina, fly ash, silica gel, zeolites, and others are commercially available adsorbents. Most of them are non-renewable and expensive; their preparation is complicated and requires a lot of dangerous chemicals [1]. This is why the search for adsorbents with low investment costs and above all based on renewable and environmentally-friendly resources is in progress.

Biosorbents have emerged as a particular class of substances in adsorption processes due to their abundant availability in natural resources or waste and their commendable performance. Some naturally occurring substances have been examined as biosorbents for eliminating dyes within this context, and the data from these investigations showed promising results [2]. Prickly pear cacti are ubiquitously present in semi-arid regions across the globe and are characterized by their affordability and accessibility. Extensive research has been conducted in diverse industrial sectors, encompassing food and medicine. The dried biomass of the prickly pear cactus paddle has garnered interest as cost-effective and sustainable for eliminating heavy metals [3–5], particularly some anionic pigments [6, 7]. The available literature on the utilization of this substance for cationic dye removal is restricted.

Basic Red 46 (BR46) is a prevalent cationic dye found in the region's liquid discharges from the textile sector. It is especially noteworthy since Cactus paddle (CP) has never been previously applied as a biosorbent for removing this deleterious substance, thereby emphasizing the importance of this research. In this regard, this study determined how well local CP removed this dye from aqueous media. First, biomass was collected and pretreated to prepare a powder which was then characterized by XRF, XRD, BET/BJH, FTIR and pH_{PZC}. Subsequently, the tuning of the adsorption process parameters encompassing pH, CP mass, stirring speed, temperature, time and BR46 concentration was conducted by discussing the isotherm and kinetics of adsorption. Finally, an adsorption mechanism has been proposed. From this standpoint, this absorbent has the potential to serve as a feasible alternative to purifying water in rural areas and treatment facilities.

Experimental Part. ***BR46 solution.*** BR46 was purchased from a local textile factory and used without purification. A stock solution (1.0 g · L^{-1}) was prepared, diluted to the required concentrations, and adjusted with NaOH and HCl solutions (0.1 mol · L^{-1}).

Preparation and characterization of CP. Cactus paddles were harvested from the region of Souk El Teninein Bejaia (Algeria). Once freed of their thorns, the fresh cactus paddles were washed in tap water, slice into smaller pieces and subjected to drying in an oven at 80 °C for 24 hours. Right after the initial drying, the specimens underwent a subsequent washing utilizing hot distilled water, followed by a rinse with cold water

and dried at 80 °C for 24 hours. Afterwards, they were ground, sieved to <100 μm and stored in a hermetically sealed bottle.

CP's elemental composition was examined through a Rigaku ZSX Primus II X-Ray Fluorescence Spectrometer. XRD pattern was obtained from a Powder X-ray diffractometer (PANalytical Empyrean). N_2physisorption isotherm at 77 K was utilized to quantify textural features through a Micro Metrics ASAP surface area and pore size analyzer (BET/BJH). FTIR spectra were taken by applying a Nicolet IS5 Spectrophotometer. pH_{PZC} was determined according to a method already described in previous research [8].

Tests of adsorption. To investigate the effects of pH, CP mass, stirring rate, temperature, duration, and initial BR46 concentration, adsorption tests were carried out in batches. Furthermore, kinetics and isotherms were studied.

Dye concentration determined after centrifugation by SHIMADZU UV-1800 at 531 nm. The calculation of the adsorption uptake (q) and removal percentage (R) was carried out in the same detailed manner by a previous article [9]. The same thing for the study of the isotherm by the Langmuir, Freundlich and Temk in models, and the kinetic study by the pseudo-first order (PFO) and pseudo-second order (PSO) models. The adequacy of the models was evaluated through the drive coefficient values (R^2), Adj. R^2 and χ^2.

Results and Discussion. ***Characterization of CP.*** According to the literature, biomass comprises carbon, oxygen, nitrogen, and hydrogen. The chemical constitution of CP was predominantly composed of carbon and oxygen, with a carbon content of 21.87 % (Table 1).

As per the XRD measurements, there was no discernible crystal peak within 10–80°. The occurrence of cellulose was found to be responsible for the manifestation of two distinct broad peaks at 24.3 and 38.0°. The contents of lignin and hemicelluloses were determined through the utilization of heightened vibration and the concomitant overlap of additional peaks. The results suggest that CP, akin to other lingo cellulosic biomasses, exhibits an amorphous character. The amorphous structure of the material suggests that dye biosorption is more likely and that dye molecules could easily reach CP surfaces.

Table 1

The properties of CP

Parameter	Value
C (wt. %)	21.877
O (wt. %)	68.608
Others (wt. %)	9.515
BET surface area ($m^2 \cdot g^{-1}$)	4.8060
Total pore volume ($cm^3 \cdot g^{-1}$)	0.016564
Average pore diameter (nm)	19.1293
pH_{pzc}	6.72

Fig. 1 displays the N_2 adsorption-desorption isotherm of CP and the BJH pore size distribution. The isotherm may fall under category IV, which in IUPAC terminology refers to mesoporous material. The BET surface area and pore volume of CP are 4.806 $m^2 \cdot g^{-1}$ and 0.016 $cm^3 \cdot g^{-1}$, respectively, according to Table 1. The molecular dimensions of BR46 were also determined to be (1.55 × 0.59 × 0.49) nm [9], while CP had a size distribution that is panned from 1.91 to 220.58 nm with a mean pore size of 19.12 nm. This implies that the pores might be easily accessed by BR46 molecules.

The FTIR spectra of CP were made to identify the nature of the surface functional groups (Fig. 2).The FTIR spectrum of the CP prior to adsorption reveals the following primary peaks: the pre dominant peak observed at 3,445 cm^{-1} maybe the result of the overlap of the stretching vibrations of the OH and NH groups; and the C—H peaks exhibit an asymmetric stretching vibration, with respective wave numbers of 2,970 and 2,911 cm^{-1}.The peak witnessed at 1,627 cm^{-1} may be associated to C=O, C=C and C—O are linked to the peaks at 1,449 and 1,057 cm^{-1}, and CN stretching/NH bending modes maybe related to the peak at 1,318 cm^{-1} [10]. Following adsorption, nearly all peaks have changed locations, indicating that various functional groups on the CP surface may be interacting with dye molecules.

The point of zero charge is the pH at which there is no charge on the adsorbent surface (pH_{PZC}). CP's pH_{PZC} was found to be 6.72 (Table 1). This suggests that:

- when pH < pH_{PZC}, the functional groups of the CP surface are protonated and the surface charge then shifts to positivity;
- when pH exceeds pH_{PZC}, some of the functional groups on the adsorbent surface release H^+, and the surface acquires a negative charge.

Adsorption parameters.

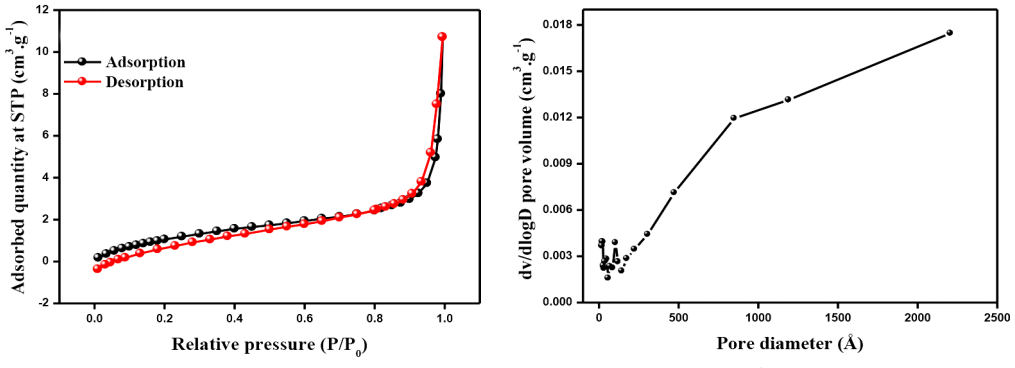
pH. The surface electrical charges of CP and the ionization of BR46 molecules are both influenced by the pH of the solution. The effect of pH (2–10) on BR46 adsorption and the pH–dependent distribution of BR46 species are shown in Fig. 3, *a*. Adsorption increased when the pH rose from 2 to 7 and then reduced.

1. The functional groups on the CP surface proton ate when pH < pH_{PZC} (6.72), obtaining a positive charge that prevents the BR46 cations from adhering through electrostatic repulsion.

2. As the pH increases, more OH^- ions are formed, encouraging the deprotonation of CP functional groups. This allows the surface to acquire negatively charged, electrostatically attract BR46 cations, and boost adsorption.

3. Higher pHs (pH > pKa) result in an abundance of OH- and neutralization of BR46, which prevents electrostatic interactions with the negatively charged surface of CP.

CP mass. Fig 3, *b* illustrates how CP mass affects the outcome. The percentage of sorption rose with an increase in CP



a *b*

Fig. 1. The N_2 sorption/desorption isotherm of CP (77 K) (a), and the pore size distribution (BJH) (b)

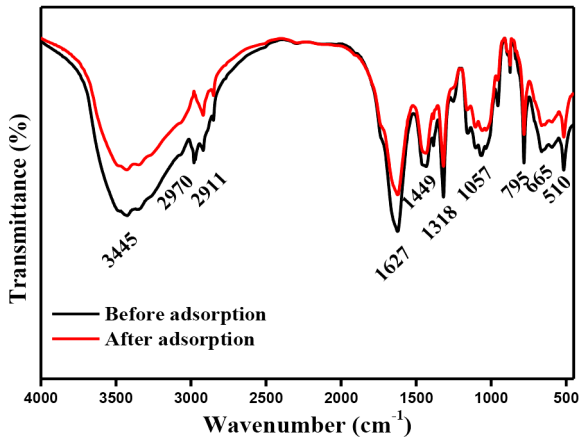


Fig. 2. Before and after adsorption FTIR spectra of CP

mass (0.1–6 g), according to experimental results. This was actually anticipated because increasing the biosorbent mass offers more sorption sites with a bigger surface area and a consistent dye concentration. A drop in the active surface area involved in the adsorption process can be employed to account for the loss in adsorption capacity with increasing biosorbent masses.

Agitation speed. Fig 3, *c* illustrates the impact of stirring speed (300–600 rpm) on BR46 uptake. By raising the stirring speed from 300 to 500 rpm, the adsorption capacity was marginally reduced. The CP particles are dispersed in the BR46 solution and their interactions are boosted when the stirring speed is increased. On the other hand, faster agitation speeds (>500 rpm) result in some biosorbent being rejected from the reactor's walls.

Temperature. Temperature: Different temperatures (25–50 °C) were considered to account for their impact on the ability of PC to adsorb BR46 (Fig. 3, *d*). Temperature-dependent

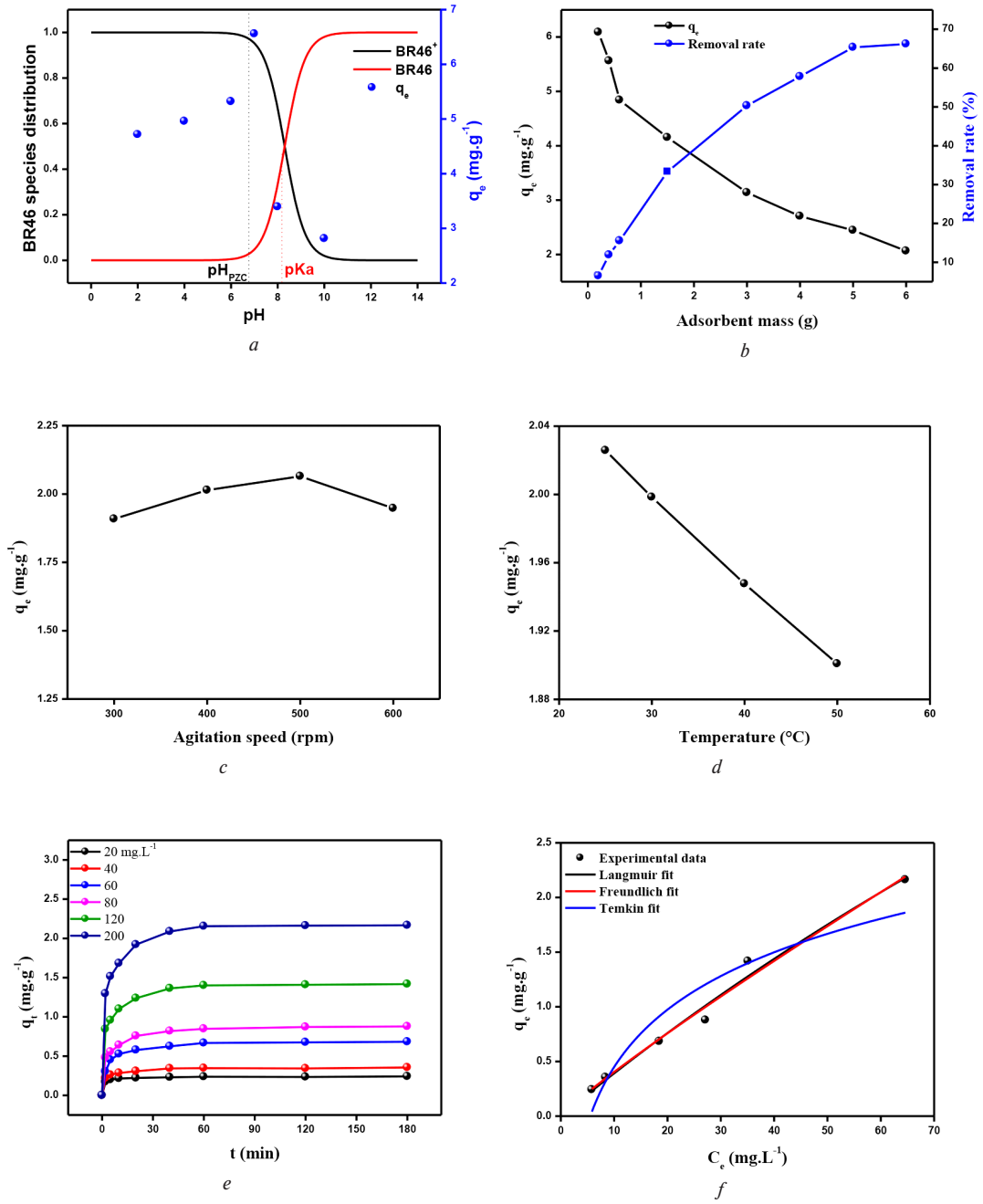


Fig. 3. The influence of: solution pH (a), adsorbent dose (b), agitation speed (c), temperature (d) and contact time and initial dye concentration (e); adsorption isotherm (f)

BR46 adsorption showed a modest reduction, indicating that the process works best at low temperatures and that adsorption can be exothermic. Additionally, the fact that adsorption capacity decreases with temperature may indicate the involvement of weak forces (van der Waals) in BR46 adsorption [11].

Time and BR46 concentration. The impact of time and BR46 concentration on uptake is depicted in Fig. 3, *e*. Adsorption started out quickly for the first 20 minutes before slowing down and eventually reaching equilibrium after 180 minutes. The decline in vacant sites over time may have caused the adsorption rate to decline.

According to the general law, the adsorption capacity rose as the concentration of BR46 did (20–200 mg · L^{-1}). The observed phenomenon could be elucidated by a concomitant augmenting in the driving force of the potential chemical gradient as a function of escalating initial dye concentrations.

Adsorption isotherms. Fig. 3, *f* displays the nonlinear fits of the Langmuir, Freundlich, and Temkin isothermal models to the empirical adsorption data. The isothermal profile might fall within Giles' classification of type L. This isotherm shows that the adsorbate molecules are horizontally adsorbed on the adsorbent surfaces and barely compete with the solvent molecules [12].

The empirical outcomes were accurately suited by both the Langmuir and Freundlich models. This is evident from the high and proximate values of R^2 and adj-R^2, coupled with the least values of χ^2.

According to the Langmuir model, the homogeneous distribution of sites on the surface leads to monolayer adsorption. No matter how many active sites are occupied, this surface has a fixed energy that does not change. Moreover, it is believed that all active sites are equivalent and have a finite capacity for adsorption. Nevertheless, molecules that have been adsorbed do not interact with one another. The Freundlich model characterizes heterogeneous surfaces that undergo multilayer adsorption processes with varying adsorption intensities. The degree of occupancy at the active locations is negatively correlated with the weakening of the affinity forces. Since there

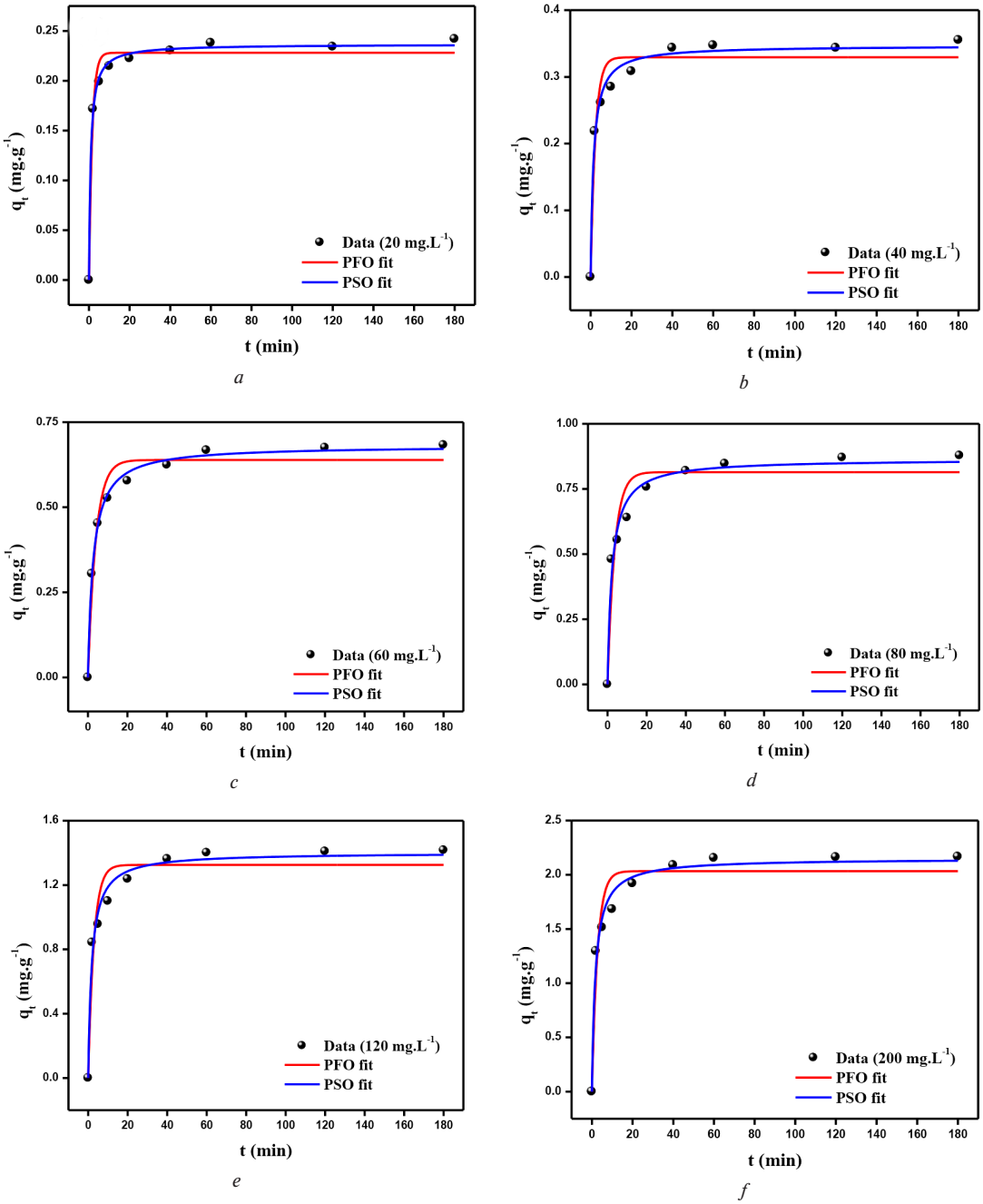


Fig. 4. Non-linear plots of the kinetic models

are only a few spots left, it is prioritized to occupy those with the strongest attractions. The good description of the experimental data by the two models (Langmuir and Freundlich) supposes that the adsorption process can be physico-chemical and contain several mechanisms.

There is some useful information provided by the Langmuir separation factor R_L which can be used to determine the feasibility of the adsorption process, which could be advantageous if $0 < R_L < 1$. The calculated R_L values ranged from 0.714 to 0.961, indicating favorable dye adsorption on CP.

Data obtained from Langmuir's model, such as monolayer adsorption capacity (q_m), are crucial. In one gram of adsorbent, this parameter indicates how much adsorbent is needed to occupy all the available active sites. Dye adsorption on CP had a q_m value of 13.45 mg · g^{-1}.

Adsorption kinetics. In Fig. 4 and Tables 2, 3, the nonlinear fits of the PFO and PSO models are displayed. The PSO model better matched the experimental data, as per the findings. Strong R^2 and Adj. R^2, and low χ^2 values, this PSO model appears to accurately capture the behavior of the adsorbate/adsorbent system across all concentrations investigated. Moreover, the q_e values derived from this model are nearer to the experimental ones.

The PSO model posits an interaction between the adsorbent and the adsorbate based on chemisorption assumptions. Moreover, the total number of binding sites is constrained at equilibrium by the amount of adsorbed adsorbate. Furthermore, k_2 values decreased from 5.110 to 0.269 g.mg^{-1} · min^{-1}, showing competition between dye molecules for adsorption sites.

Comparison of BR46 adsorption. CP has an average adsorption capacity of BR46 of 14.15 mg · g^{-1} compared to several adsorbents mentioned in the literature (Table 4). However, CP and other biosorbents like olive pomace are suitable for the adsorption of contaminants present in liquid media due to their eco-friendliness, cost-effectiveness, and lack of necessity for chemical or physical modification, in contrast to other adsorbents that may be relatively costly and necessitate intricate manufacturing procedures, substantial quantities of chemicals, and considerable energy inputs.

Table 2

Kinetic parameters for pseudo first-order model

Concentration (mg/L)	q_{eexp} (mg/g)	Pseudo first-order model				
		q_{ecal} (mg/g)	k_1 (min^{-1})	R^2	Adj. R^2	χ^2
20	0.242	0.228	0.634	0.979	0.976	1.394
40	0.355	0.329	0.436	0.949	0.942	7.299E–4
60	0.683	0.638	0.251	0.965	0.960	0.001
80	0.878	0.814	0.277	0.925	0.915	0.006
120	1.416	1.325	0.348	0.931	0.921	0.016
200	2.166	2.033	0.365	0.936	0.927	0.035

Table 3

Kinetic parameters for pseudo second-order model

Concentration (mg/L)	q_{ecal} (mg/g)	Pseudo second-order model			
		k_2 (g/mg · min)	R^2	Adj. R^2	χ^2
20	0.236	5.110	0.997	0.996	1.983E–5
40	0.346	2.022	0.988	0.986	1.691E–4
60	0.681	0.555	0.996	0.995	2.196E–4
80	0.864	0.509	0.979	0.976	0.001
120	1.401	0.395	0.981	0.797	0.004
200	2.149	0.269	0.984	0.982	0.008

Table 4

Comparison of BR46 dye adsorption

Adsorbent	Adsorption capacity(mg · g^{-1})	Reference
Biochar prepared from Chrysanthemum Morifolium Ramatstraw	53.19	[13]
Fe@graphite core-shell magnetic nanocomposite	46.7	[14]
Commercial activated carbon (Norit PK 1–3)	50.78	[15]
Olive pomace	14.15	[2]
Cactus paddle	13.45	This work

Probable mechanism. The adsorption mechanism is typically intricate and contingent upon multiple parameters. The findings of BET study showed that, notwithstanding the molecular size of BR46 (1.55 × 0.59 × 0.49 nm) is less than the typical pore size of CP (19.12 nm), BR46 molecules can enter mesopores of CP through the pore filling process. The findings derived from the isothermal and kinetic tests showed that both chemical and physical interactions control the process. According to studies on the impact of pH on BR46 adsorption that considered the pH_{PZC} of CP and the pKa of the BR46, electrostatic interactions were undoubtedly a component of the mechanism. An alternative possibility is that weak forces like van der Waals are involved in the adsorption, as evidenced by the observed reduction in the adsorption capacity of CPs with increasing temperature [11]. A variety of surface functional groups of CP were discovered using FTIR. The results following uptake supported the notion that, given the movement of their respective peaks, various groups can interact with BR46 molecules. Between the hydroxyl groups of CP (the H-acceptor) and the nitrogen atoms in BR46 molecules, the dipole-dipole hydrogen bond may occur. Meanwhile, the aromatic rings of BR46 and the hydroxyl groups of CP may connect by Yoshida hydrogen bonding. π–π interactions could take place among π aromatic rings donors of BR46 and π acceptors of CP. n–π interactions among the oxygen groups of CP (electron donors) and the aromatic rings of BR46 (electron acceptors) may also occur.

Conclusion. In this study, the effectiveness of using locally accessible CP as a biosorbent to remove the dye BR46 was examined. XRF, XRD, BET/BJH, FTIR, and pH_{pzc} were used to describe the physicochemical characteristics of CP. The findings demonstrate that its surface is amorphous, abundant in functional groups, and provides a conducive environment for the uptake of dye molecules with amedian pore size of 19.12 nm. An adsorption capacity of 13.45 mg · g^{-1} was obtained under a batch system, while the operating parameters comprising contact time, solution pH, CP mass, agitation speed, temperature, and beginning BR46 concentration, were examined. Freundlich and Langmuir's models characterized the isothermal data, whereas the PSO model fits the kinetics well, indicating that physicochemical interactions control adsorption. The findings indicate that using CP as a feasible and environmentally sustainable material for remediating textile effluent represents a propitious avenue for further investigation.

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Declaration of interest statement. *There is no conflict interest that could have appeared to influence the work reported in this paper.*

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Екологічно безпечний біосорбент на основі місцевої сировини: застосування для видалення барвників

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Мета. Основною причиною забруднення води є широке використання барвників, що споживаються у великій кількості. Метою даного дослідження було дослідити можливість використання кактусів, род Cactus paddle (CP), як екологічно безпечного біосорбенту для видалення барвника типу Basic Red 46 із водних середовищ.

Методика. Для дослідження поверхневих характеристик біосорбенту використовували різні аналітичні інструменти, включаючи рентгено-флуоресцентний аналіз (XRF), рентгеноструктурний аналіз (XRD), метод вимірювання площі поверхні БЕТ/метод Баррета-Джойнера-Галенда (BET/BJH), інфрачервоний аналізатор Фур'є (FTIR) і потенціал нульового заряду (pH_{PZC}). Для оцінки здатності CP до адсорбції барвника BR46 проводили аналіз періодичної адсорбції.

Результати. 13,45 мг · $г^{-1}$ барвника BR46 був адсорбований за рівнем pH 7,500 об/хв. і 293 K протягом 180 хвилин. Ізотермічні дані описувалися за допомогою як рівнянь Фрейндліха, так і рівнянь Ленгмюра з $R^2 \geq 0{,}984$ та $\chi 2 \leq 0{,}01$. Кінетика була добре описана моделлю PSO (фосфіну янтарного олігомера), припускаючи, що процесом адсорбції керують фізико-хімічні взаємодії.

Наукова новизна. Уперше обґрунтоване ефективне застосування місцевого сировинного ресурсу (Cactus paddle) як біосорбенту для видалення барвника BR46.

Практична значимість. За результатами підсумків цього дослідження здається можливим, що CP можна розглядати як стійкий ресурс, який створить можливість для вирішення проблем екології у майбутньому.

Ключові слова: *біосорбент Cactus paddle, адсорбція, основний барвник Red 46*

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