



Chitosan and polycaprolactone electrospun nanofibrous composites for sustainable decolorization of textile wastewaters

Óscar Martínez-Rico^a, Rui P.C.L. Sousa^{b,*}, Joana C. Araújo^b, Sofia M. Costa^b,
Begoña González^a, Diana P. Ferreira^{b,*}

^a Department of Chemical Engineering, Universidade de Vigo, Vigo, 36310, Spain

^b Centre for Textile Science and Technology (2C2T), University of Minho, Guimarães, 4800-058, Portugal

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ABSTRACT

A new approach for textile wastewater remediation via adsorption with electrospun nanofibers is presented in this work. Chitosan (Ch) and poly-ε-caprolactone (PCL) were electrospun to obtain a nanocomposite, and different deposition methods of the polymers were tested in order to optimize their mechanical and adsorptive properties. A layered structure showed higher capacities for dye adsorption compared to the monolayer structures, achieving full decolorizations with efficiencies over 98%, for a wide range of concentrations and temperatures. The developed fibrous structures were characterized via ATR-FTIR, SEM, TGA and measurements of the wettability, porosity and mechanical performance. Experimental data best fit to the pseudo-second order kinetic model and the Langmuir isotherm, with the maximum adsorption capacities found to be 69.5 mg/g at 25 °C. Desorption was possible using alkali media, recovering up to 25% of the dye. Sorbents could be used for at least 3 cycles without loss on the removal efficiency, which shows the potential of these new nanocomposites as reusable, versatile and stable eco-friendly adsorbents.

1. Introduction

The growing industrialization of the world is exacerbating water scarcity due to high industrial demand. Furthermore, the inability to effectively eliminate contaminants, coupled with irregular remediation efficiencies, deteriorates water quality, which in turn has severe consequences in aquatic and human life. One of the most important challenges mankind must face is the access to clean water, hindered by the difficulty in the removal of pollutants like phenolic compounds, dyes, heavy metals or pesticides derived from industrial activity (Saravanan et al., 2021; Rodríguez-Narvaez et al., 2017). In particular, the textile industry is one of the most important contributors to worldwide water pollution, as an estimate of 20% of the global water pollution is caused by dyeing and finishing textile products (European Environment Agency, 2019). During the dyeing processes, up to half of the colorant content can end up in the wastewaters (Forgacs et al., 2004; Przystas et al., 2012; Martínez-Rico et al., 2025), posing environmental problems as some dyes are highly toxic and affect the transparency of water bodies at even very low concentrations, which for some dyes may be lower than 1 mg/L (Wijetunga et al., 2010).

Dyes can be classified according to their molecular structures, where the azo type accounts for around 80% of the total amount used in the sector, although other ones like anthraquinones or triarylmethanes are still relevant (Kishor et al., 2021). Research for dye removal from wastewaters is vast, and it includes an extensive array of techniques ranging from conventional methods—such as adsorption, filtration, coagulation, liquid-liquid extraction or precipitation—to more intricate ones like photooxidation or biological treatments (Arslan et al., 2016; Aramesh et al., 2021; Villar et al., 2022; Asla et al., 2023; Martínez-Rico et al., 2024). Chemical approaches like ozonation or oxidation are rather specific but generally end up rendering byproducts like sludges. Physicochemical ones have high removal efficiencies but tend to have a high cost due to the complexity of the materials employed.

Biological treatments are not adequate for most dyes and require elaborate enzymatic productions (Arslan et al., 2016). Adsorption stands out from all these processes due to its efficient ability to remove waste compounds in effluents (Yagub et al., 2014). This extraction is due to the adhesion of these compounds to the solid surface of the adsorbent, which takes place due to chemical, van der Waals or electrostatic interactions. The main advantage is its simple operation mode for a wide

* Corresponding authors.

E-mail addresses: rui.sousa@2c2t.uminho.pt (R.P.C.L. Sousa), diana.ferreira@det.uminho.pt (D.P. Ferreira).

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range of pollutants. This is the reason why in the textile industry it is usual to employ, for instance, commercial activated carbon as an adsorbent. Nevertheless, there is intensive research to develop new adsorbents that are environmentally friendly while showing economic advantages. For example, agricultural/industrial byproducts or biomass substrates are remarkable starting materials for this purpose (Bello et al., 2013; Rehman et al., 2012; Demirbas, 2009).

Recently, nanocomposite materials have received a lot of attention due to their broad range of applications in water purification (Li et al., 2021). One of the most relevant techniques for their synthesis is electrospinning, the only method that allows to obtain fibers in the micro to nano size range in a continuous manner (Greiner and Wendorff, 2007). Plus, it is adequate for many materials, cost-effective and easily accessible given that the experimental setup does not present a high complexity (Patel et al., 2020). Electrospun mats feature attractive mechanical properties, high surface area, a low-weight porous structure, and significant porosity. To customize nanofiber mats for specific applications (Nasreen et al., 2019; Rocha et al., 2024), functional additives such as surface coatings or active species can be incorporated into the electrospinning solution. These modifications enhance the utility of electrospun nanofibers, making them particularly advantageous for applications like water purification by adsorption (S. et al., 2021; Zia et al., 2020).

Chitosan (Ch) has been studied broadly lately due to its green features and high adsorption abilities (Martínez-Rico et al., 2024; Martínez-Rico et al., 2023; Blanco et al., 2023). It is obtained by deacetylation of chitin, the second most abundant polymer in nature. Due to the high number of amine and hydroxyl groups in its molecular structure, this abundant biopolymer has many available adsorption sites. It also has a highly tunable structure which allows for modifications to improve its properties, so the literature that describes combinations of chitosan to produce hybrid materials for adsorption is extensive (qi Liu et al., 2022; Saeed et al., 2022). Nevertheless, the fabrication of pure chitosan nanofibers presents difficulties due to its chain stiffness and its polycationic nature that gives rise to strong hydrogen bond interactions. These hydrogen bonds prevent the free movement of the polymeric chain segments that are exposed to the electric field, causing the jet to break up during the formation. Also, repulsions among the ionic groups have a negative influence in the formation of chain entanglements, which are necessary to allow stable fiber formation (Pakravan et al., 2011; Hasanpour Ardekani-Zadeh and Hosseini, 2019). That is why additives that favor the spinnability of chitosan are usually employed, such as polyethylene oxide (PEO) or polyvinyl acetate (PVA) (Shalumon et al., 2010). PEO is usually chosen as a co-spinning agent for chitosan given that it is non-toxic, biocompatible and biodegradable (Amiri et al., 2020). Additionally, cellulose nanocrystals (CNC) are known to stabilize the morphology of nanofibers, decreasing their diameters. They provide additional strength and a large surface area, while also being biodegradable (Ridolfi et al., 2017). This polymer blending yields new materials whose physicochemical properties can be tailored depending on the purpose. Poly-ε-caprolactone (PCL) is another attractive choice for use in nanofiber structures due to its biodegradability, biocompatibility, good mechanical properties, and non-toxicity (Steyaert et al., 2012). It has been widely used in biomedicine because it can be biodegraded by living organisms like bacteria and fungi, with faster degradation rates when adding hydrophilic copolymers like PEO. In addition, it is relatively easy to electrospin (Cipitria et al., 2011), and PCL blends show mechanical upgrades (e.g. higher elastic modulus) with additions of <10 wt% PCL (Guarino et al., 2017). Recent studies have integrated functional nanostructures into electrospun chitosan blends such as Ch/PCL or Ch/PVA matrices for stimuli-responsive therapeutic delivery (Guesmi et al., 2026; Ben Hamadi et al., 2025). However, unlike these single-step blend configurations that tend to bury functional groups within the bulk matrix, this work employs an asymmetric layered architecture to increase surface accessibility of the chitosan active sites while

preserving mechanical integrity.

In this work, a solution of Ch mixed with PEO and CNC was electrospun with PCL to form a nanocomposite that shows efficient adsorption towards dyes, in particular Remazol Brilliant Blue R, which was chosen because it is a reactive dye very commonly used in the industry (Sathishkumar et al., 2012) and it represents an important class of toxic organic pollutants (Mechichi et al., 2006). Despite the high potential of these individual components, the challenge remains in synthesizing a material that maintains high adsorptive capacity (provided by Ch) without sacrificing mechanical durability in aqueous environments (provided by PCL). This study addresses this gap by proposing a novel nanocomposite that takes advantage of the synergistic effects of chitosan and a PCL reinforcing scaffold. While combining PCL and chitosan has been explored for biomedical applications, their use as adsorbents usually results in reduced functional group availability or requires chemical crosslinkers that block active sites. The novelty of this work lies in the treatment of the fragility of the chitosan nanofibers via a non-interfering PCL backing scaffold, delivering a green composite capable of high-affinity dye removal. First, the parameters that affect the production of the composite were optimized: the ratio of Ch/PEO/CNC and PCL to be electrospun and the way they are deposited (intertwined or a sandwich-like structure). Also, the equipment conditions to obtain a stable jet and the production of defect-free nanofibers were assessed. After obtaining stable materials, they were physically characterized with Scanning Electron Microscopy (SEM), Attenuated Total Reflectance-Fourier Transform Infrared Spectroscopy (ATR-FTIR), Thermogravimetric Analysis (TGA), Water Contact Angle (WCA) analysis, Brunauer-Emmett-Teller (BET) surface area and Barrett-Joyner-Halenda (BJH) pore size and volume analysis so as to understand the composition of the materials obtained. Moreover, adsorption efficiencies were assessed by optimizing the effects of contact time, adsorbent dose, initial dye concentration and kinetics and isotherms. Overall, this research presents and characterizes a new combination of Ch/PEO/CNC and PCL with great potential for textile wastewater remediation.

2. Experimental section

2.1. Materials and reagents

The reagents used in this article can be found in Table 1 along with the supplier, purity, CAS number and molecular weight (when corresponding). Milli-Q water was employed to prepare the aqueous solutions.

2.2. Preparation of Ch/PEO/CNC and PCL solutions

A Ch/PEO solution with a 1:1 wt ratio of both polymers was prepared in a 50% (v/v) acetic acid aqueous solution until reaching a 2% total polymer concentration, according to the study by Ribeiro et al. (Ribeiro et al., 2021). After stirring this mixture for 2 h, CNC was added slowly to avoid agglomeration until having a 1% CNC (w/v) concentration, and then it was left overnight under vigorous stirring. Similarly, the PCL solution was prepared following other studies (Francavilla et al., 2021): PCL pellets were dissolved in chloroform (CHF) under stirring and heating at 35 °C for at least 3 h until a homogeneous solution was obtained. Then, *N,N*-dimethylformamide (DMF) was incorporated, giving rise to a 15% (w/v) PCL solution in CHF/DMF (4:1).

2.3. Electrospinning process

Solutions were electrospun in an electrospinning NF-103 equipment from MECC. The optimal parameters employed for the process for each solution are described in Table 2. Temperature and ambient humidity have a strong influence in the nanofiber formation (De Vrieze et al., 2009), so they were closely monitored so as to ensure that humidity

Table 1

List of the chemicals employed in this work with their supplier, purity and CAS number as stated by the supplier.

Chemical name	Molecular weight (kDa)	Supplier	Purity	CAS number
Chitosan	310–375	Sigma Aldrich	≥ 99.9%	9012–76–4
Polyethylene oxide	900	Sigma Aldrich	≥ 99.9%	25322–68–3
Cellulose nanocrystals	-	Celluforce	≥ 99.9%	-
Poly-ε-caprolactone	80	Sigma Aldrich	≥ 99%	24980–41–4
Acetic acid	-	ThermoFischer Scientific	≥ 99.7%	64–19–17
Chloroform	-	VWR Chemicals	≥ 99%	67–66–3
N,N-Dimethylformamide	-	ThermoFischer Scientific	≥ 99.8%	68–12–2
Remazol Brilliant Blue R	-	Dystar	≥ 99%	2580–78–1

Table 2

Electrospinning conditions employed for the fabrication of Ch/PEO/CNC and PCL composites.

Parameters	Optimal value	
	Ch/PEO/CNC	PCL
Voltage	29 kV	25 kV
Flow rate	4 mL/h	1 mL/h
Tip-collector distance	170 mm	260 mm
Needle diameter	0.41 mm	
Cleaning frequency	90 s	
Head transverse speed	50 mm/s	
Head transverse distance	150 mm	
Type of collector	Static	

was below 60% at all times and temperature was kept between 20–25 °C.

Several deposition methods of these two solutions were assessed in an attempt to maximize adsorption efficiencies while maintaining good mechanical properties, following similar works (Li et al., 2016). The subsequent mats were successfully produced:

- Chitosan mat. Obtained by electrospinning of a Ch/PEO/CNC solution for 6 h using conditions from Table 2.
- PCL mat. Fabricated by electrospinning of a PCL solution for 6 h under Table 2 conditions.
- Multilayered mat. PCL and Ch were combined in a layered structure (PCL/Ch/PCL/Ch/PCL) one on top of the other, each deposition lasting 1.5 h.
- Layered mat. Similar sandwich-like arrangement: Ch/PCL/Ch, done for 2.5 h for each layer.
- Simultaneous mat. Both solutions were electrospun simultaneously for 6 h, each with its optimal parameters from Table 2, except for the tip-collector distance and the voltage that were kept at 170 mm and 29 kV, respectively.

2.4. Nanofibers characterization

2.4.1. Attenuated total reflectance-Fourier transform infrared spectroscopy (ATR-FTIR)

Attenuated Total Reflectance-Fourier Transform Infrared Spectroscopy (ATR-FTIR) was carried out in several spots along the surface with an IRAffinity-1S SHIMADZU equipment. Spectra were collected using diamond ATR crystal cell by accumulation of 45 scans with a resolution of 8 cm⁻¹, in the 400–4000 cm⁻¹ wavenumber range.

2.4.2. Scanning electron microscopy (SEM)

The diameter and morphology of the nanofibers were assessed by SEM, using a FlexSEM 1000 II (HITACHI). After removing the composites from the aluminum collector, the samples were coated with a very thin layer of gold (Au) with a Quorum MiniQS sputter coater. An acceleration voltage of 5 kV was used to obtain the secondary electron pictures, which were then used to determine the diameters of the nanofibers with Image J software by measuring the diameters at 100

different locations.

2.4.3. Thermogravimetric analysis (TGA)

Thermal analysis was carried out on a STA 700 SCANSCI device. An atmosphere of N₂ was used in the 30–600 °C range with a constant heating rate of 10 °C/min.

2.4.4. Water contact angle (WCA) analysis

The water contact angle was measured in ten different spots of every sample using a Contact Angle System OCA 15 (dataphysics) attached to a high-resolution camera. Drops of 5 µL of distilled water were deposited on the surface of the samples and the contact angles were measured through the drop shape analysis.

2.4.5. Brunauer-Emmett-Teller (BET) and Barrett-Joyner-Halenda (BJH) analysis

The ASAP2020 system (Micromeritics) was used for the analysis of the BET surface area and the BJH pore volume. Samples were degassed for 3 h at 25 °C, and the isotherms were acquired using nitrogen gas immersed in liquid nitrogen.

2.4.6. Mechanical tests

Tensile tests were performed to assess the mechanical properties of the different composites. The experiments were carried out using a Hounsfield Tinius Olsen H100KS testing machine fitted with a 250 N load cell. Samples measuring 90 × 30 mm were evaluated at a strain rate of 30 mm/min in accordance with ASTM D5035 standards. Each test was conducted in triplicate to ensure consistency. Key mechanical properties such as tensile strength, yield point, strain at rupture, and Young's modulus were determined from the stress-strain curves. The tensile strength represents the highest stress recorded, while strain at rupture break was obtained from the total elongation at the point of rupture. The yield point was calculated as the end of the elastic deformation range and the Young's modulus was the slope of the curve in this elastic range. The stress-strain curves were generated by measuring the applied force and the corresponding filament elongation up to failure. The calculations for stress (σ), strain (ε) and Young's modulus (E) followed Eqs. (1)–3.

$$\sigma = \frac{F}{A} \quad (1)$$

$$\varepsilon = \frac{\Delta L}{L_0} \quad (2)$$

$$E = \frac{\Delta \sigma}{\Delta \varepsilon} \quad (3)$$

Where F represents the applied force (N), A is the initial cross-sectional area of the sample (mm²), ΔL is the change in length (mm) and L₀ is the initial sample length (mm). Δσ and Δε correspond to the variations in stress and strain within the elastic deformation range.

2.5. Adsorption experiments

Dye solutions were prepared by direct weighing with concentrations from 50 to 200 mg/L and used in batch adsorption experiments with varying conditions: initial dye concentration (50–200 mg/L), adsorbent dosage (0.5 to 20 mg), shaking time (15 min to 24 h) and temperature (25 to 45 °C). The volume of dye solution for each assay was kept constant at 3 mL. Agitation was set to 350 rpm in a Ohaus orbital shaker model ISHD16HDG. Dye concentration in the aqueous phases was analyzed by spectrophotometry using a JASCO V-750 with an uncertainty of ± 0.002 abs from 0 to 0.5 abs and ± 0.003 abs from 0.5 to 1 abs, respectively. Before the analysis, a calibration curve of the dye at its maximum absorbance wavelength (603 nm) was obtained. The dye removal percentage was calculated as determined in Eq. (4), and the adsorption capacity (q_t) at time t as in Eq. (5).

$$\% \text{ Dye removal} = \frac{C_0 - C_t}{C_0} \times 100 \quad (4)$$

$$q_t = \frac{V(C_0 - C_t)}{m} \quad (5)$$

Where C_0 and C_t are the dye concentrations at the beginning and at time t , V is the volume of solution and m is the mass of adsorbent in grams. At least two replicates of these assays were performed until a variation coefficient below 10% was attained.

2.5.1. Adsorption kinetics and isotherms

Diverse adsorption kinetic theories were tried in order to gain insight about the mechanism of the adsorption process. Namely: pseudo-first (equation S1), second-order (equation S2), Elovich (equation S3) and intraparticle diffusion (equation S4) models (Wu et al., 2009; Weber and Morris, 1963). Moreover, a similar approach was followed with the isotherms, as several models were tested: Langmuir (equation S5), Freundlich (equation S6), Temkin (equation S7), Elovich (equation S8) and Dubinin-Radushkevich (equation S9) (Langmuir, 1918; Freundlich, 1906; Temkin and Pyzhev, 1939; Dubinin and Radushkevich, 1947; Elovich and Larinov, 1962; Ayawei et al., 2017).

2.5.2. Adsorbent reusability and desorption

The desorption abilities of the adsorbents were assessed with 3 mL of several solutions: NaOH (0.1 and 1 M), H₂SO₄ 0.1 M and NH₄OH 0.1 M. Furthermore, the reusability was also tested by putting them again in contact with 3 mL of the 100 mg/L dye solution for 6 h at 25 °C.

3. Results and discussion

3.1. Adsorbent characterization

3.1.1. Attenuated total reflectance-fourier transform infrared spectroscopy (ATR-FTIR)

The combinations of the two polymers were produced as described in Section 2.3, and their FTIR spectra were acquired to obtain information about their composition. First, Figure S2 shows the spectrum of the chitosan mat (Ch/PEO/CNC), which contains the expected signals for Ch and PEO: a broad band in the 3000–3600 cm⁻¹ region due to the stretching vibration of N–H and –OH groups (and also their interaction with the carbonyl groups) and a peak at 2879 cm⁻¹, due to the C–H stretching of Ch and PEO (Kowalonek, 2016). Moreover, the three peaks at 1101, 1058 and 1031 correspond to C–O and C–C, C–O–C, C–O and C–C stretchings from nanocrystalline cellulose (Mohamed et al., 2015). The analysis was carried out on both sides of the composite and in several different spots to ensure homogeneity. Therefore, it can be confirmed that the three polymers from the initial solution are still present in the nanofibers. Furthermore, the spectrum of the PCL mat (Figure S3) presents the characteristic peaks of this polymer (Phillipson et al., 2014): a doublet at 2943 and 2866 cm⁻¹ due to the stretching of

C–H bonds, a sharp singlet at 1720 cm⁻¹ from the carbonyl group, another singlet at 1161 cm⁻¹ caused by the CH₂ deformation, two bands at 1240 and 1294 that can be assigned to the stretching of the ester group, and a peak at 960 cm⁻¹ from the bending of the ester moiety. This corroborates that the electrospinning of both polymers produces nanofibers without alterations in their integrity.

Next, the different combinations of Ch+PCL were evaluated. The multilayered mat was fabricated with the 5-layered sandwich structure described in the previous section, and rendered the ATR-FTIR spectrum in Figure S4. It can clearly be seen that it resembles the pure PCL spectrum with no traces of Ch (doublet at 2943 and 2866 cm⁻¹, sharp peak at 1722 cm⁻¹ and absence of bands in the 3000–3600 cm⁻¹ region). This points to the fact that PCL is the main component in the outer part of the material and chitosan remains in the inner part, which, as discussed later, translates to a lower decolorization efficiency. The polymer combination was optimized with a modification on the deposition mode: three layers were electrospun now (Ch/PCL/Ch). The spectra obtained were different depending on the side: on one side (Figure S5a) the bands match the spectrum of pure chitosan (Figure S2), and the other side is analogous to that of pure PCL (Figure S3). This allows to conclude that one of the Ch layers is lost when recovering the composite from the aluminum collector, which was within the expected outcomes due to its structural fragility (Xu et al., 2024). The final deposition manner was the simultaneous mat, where both syringes were used simultaneously. Its ATR-FTIR spectrum (Figure S6) coincided with the pure PCL one, in which, similarly to the multilayered one, the chitosan fibers are relegated to the core of the composite, which should lower its dye removal abilities.

3.1.2. Scanning electron microscopy (SEM)

The surface morphology of the nanocomposites was analyzed via SEM, with the images shown in Fig. 1 and the histograms with the distributions of fiber diameters in Fig. 2. First, the chitosan mat, composed of Ch/PEO/CNC showed the formation of thin nanofibers that deposited random and homogeneously with an average diameter of 156 nm, with almost no bead formation, as seen in Fig. 1a and b. The measured average diameter of these fibers falls within the expected nanoscale range, ensuring a high surface area-to-volume ratio beneficial for applications that rely on efficient surface interactions. In the layered mat (Fig. 1c and d), made up of Ch on one side and PCL on the other, the average diameter decreased to 124 nm and the fiber entanglement increased significantly as it can be appreciated in the pictures. This points to the fact that PCL acts as mechanical support of the Ch moiety, which should yield an improvement in the adsorption abilities, as it will be discussed later. Similar reductions in the diameter have been reported by others: for example, Ji et al. (Ji et al., 2014) added chitin nanofibrils to a PCL mat and reasoned this decrease with the additional electrostatic charge density of the electrospinning solutions, which causes more extensive filament stretching.

The other type of combination was the one obtained by simultaneous electrospinning of both polymers, whose SEM images (Fig. 1e and f) were significantly different: as expected, both fibers are spun together, which causes a wider diameter distribution (Fig. 2c) with a much higher mean fiber diameter (1.29 μm). Two types of fibers can be observed: the Ch (hundreds of nm) and the PCL ones (much thicker, in the μm range), whose dimensions match the ones reported by other works (Ji et al., 2014).

Furthermore, to confirm the layered structure, which in principle should be the most effective for adsorption due to its lower diameters, sectional images were taken and are presented in Fig. 3. It can clearly be seen that two different layers have been formed: the one on top on these images corresponds to PCL, which has larger diameters, and the one below that is made up of chitosan, with its thinner diameters (that is why the fibers are only perceptible with higher magnifications as in Fig. 1b).

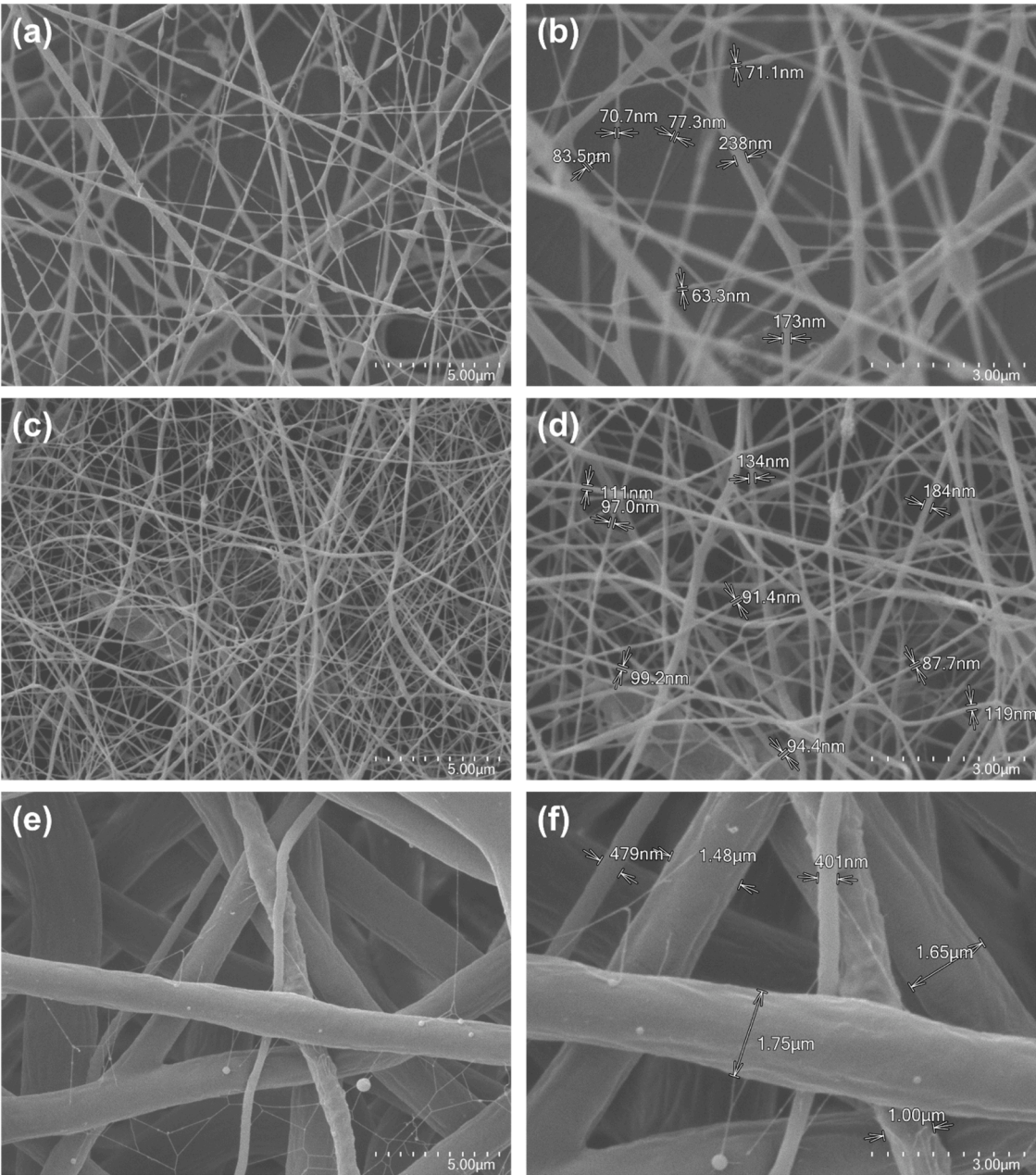


Fig. 1. SEM images of the developed electrospun nanocomposites: chitosan mat (a) and (b), layered mat (c) and (d), and simultaneous mat (e) and (f). For each material, images were acquired at two different magnifications: micrographs (a), (c), and (e) were obtained at 17,000 × magnification (scale bar: 5 μm), while micrographs (b), (d), and (f) were acquired at 35,000 × magnification (scale bar: 3 μm).

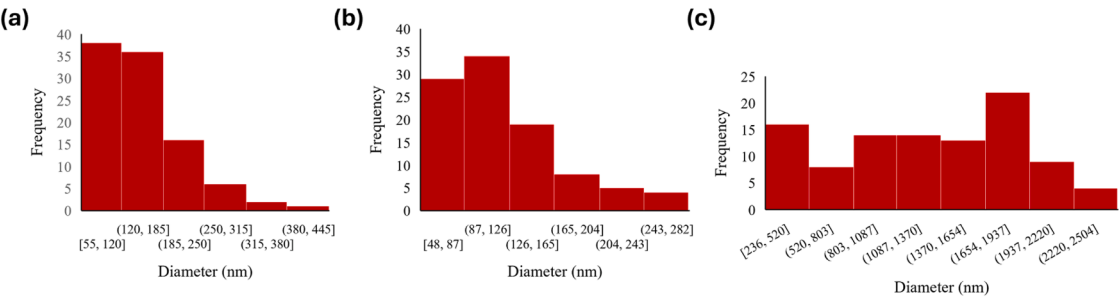


Fig. 2. Fiber diameter histograms of the chitosan mat, (a) the layered mat, (b) and the simultaneous mat (c).

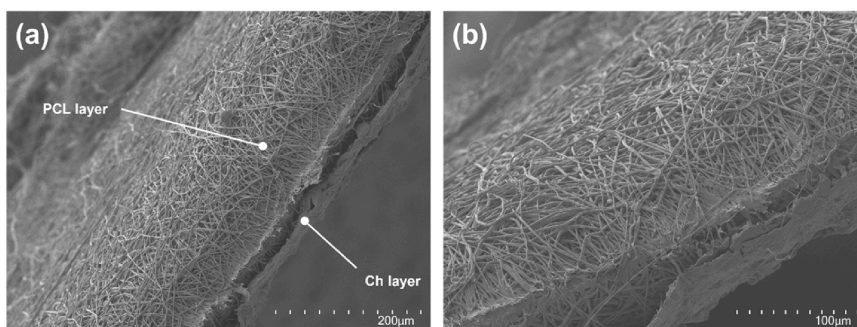


Fig. 3. Transversal SEM image of the layered mat, showing the PCL layer on top and the chitosan layer below, with: a) 600x and b) 1000x magnifications.

3.1.3. Thermal analysis

The thermal decomposition behavior of the nanofibers with the different deposition modes was studied and is depicted in Fig. 4. The Ch/PEO/CNC mat showed the expected mass losses for these compounds (Neto et al., 2005): the first weight loss starts at 61 °C and is due to water evaporation, which is followed by a second stage between 275 and 359 °C due to the thermal and oxidative decomposition of Ch and CNC. Finally, the maximum degradation peak at 415 °C is due to the degradation of PEO. Both mats that combine Ch and PCL showed a single abrupt decrease at 413 and 415 °C, which correlates well with the PCL degradation, which showed one main degradation peak at 420 °C (Persenaire et al., 2001). Moreover, layered and simultaneous depositions showed similar trends, albeit in the layered one the degradation process associated with the decomposition of Ch and CNC (275–359 °C) can be appreciated due to the higher chitosan content compared to the simultaneous, in which it was not visible. No other significant mass losses can be appreciated, which can be explained with three factors: the added stability provided by PCL, the fact that the chitosan content is much lower in comparison and the peak overlaps in this region.

3.1.4. Water contact angle

The behavior of these nanocomposites against water has special relevance given that their main application in this work is dye removal from aqueous matrices. For that, the surface hydrophilic character of the different composites was assessed in Table 3. As expected, the chitosan nanofibers showed surface hydrophilicity with an angle below 90°, and the pure PCL mat was strongly hydrophobic (Ribeiro et al., 2021; Ji et al., 2014). The layered configuration suggested by the ATR-FTIR and SEM analyses should cause different wettabilities on each side. Indeed, the chitosan side had an angle of 69.3° (hydrophilic) and the PCL one showed a contact of 122.1° (hydrophobic). Generally, the contact angle depends both on the surface morphology and the material composition, so the contact angle of the simultaneous composite -a homogeneous mix of both polymers- renders an angle of 95.7°. This puts it between the Ch and the PCL values, which is consistent to the

Table 3

Angles of contact of the different composites with water. Layered mat shows a value for each of its sides.

	Chitosan mat	PCL mat	Layered mat	Simultaneous mat
Angle	69.2° ± 1.5	145.1° ± 3.1	Upper side: 69.3° ± 1.6 Lower side: 122.1° ± 2.5	95.7° ± 1.8

composition suggested by the SEM images: an intertwined mat of Ch and PCL fibers. When it comes to the adsorption, these differences in the surface wettability are expected to influence dye uptake, given that more hydrophilic surfaces facilitate water spreading and thus closer interaction among aqueous dye molecules and the functional groups on the surface, which enhances adsorption through electrostatic interactions and hydrogen bonding. In contrast, more hydrophobic surfaces have lower affinity for water, which can limit dye accessibility to the adsorption sites. These trends are consistent with reports that show that increasing surface hydrophilicity favors the adsorption of polar and ionic dyes by improving interfacial wetting, as reflected by lower contact angle values (Law, 2015; Arévalo-Fester and Briceño, 2023).

3.1.5. BET and BJH analysis

BET and BJH analysis was conducted to determine the surface area and pore volume of the adsorbents, and the results are shown in Table 4. The porosity of the pure nanofibers (chitosan and PCL) compares well to other similar electrospun nanocomposites, which have surface areas in the range of 10 m²/g (Zhang et al., 2006). Combining both polymers causes a reduction both in the surface area and the micropore volume, but this decrease is much less acute for the layered mat, which suggests superior adsorption abilities. The seemingly low specific surface areas do not totally represent the functional performance of these electrospun mats, as the adsorption capacity is primarily driven by the macroporous nature of the fibers rather than microporous density. While BET measurements are quite sensitive to micro and mesopores, they do not describe as accurately the contribution of macropores, which act as large surface openings that facilitate the diffusion of the dyes into the fiber structure (Chen et al., 2023). Consequently, the high adsorption capacity is a result of the availability of sites on the surface rather than an

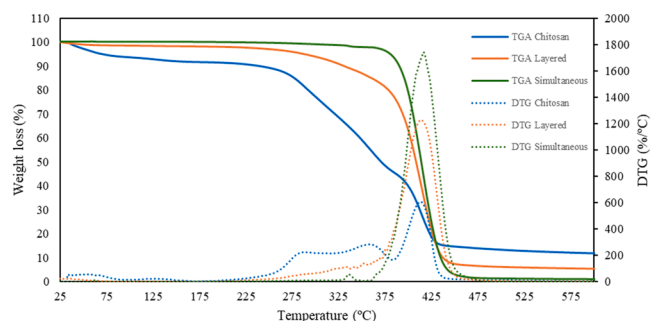


Fig. 4. TGA and DTG curves of the chitosan mat, the layered mat, and the simultaneous mat.

Table 4

Brunauer-Emmet-Teller (BET) and Barrett-Joyner-Halenda (BJH) analysis of the surface area and porosity of the five adsorbents.

	Micropore volume (cm ³ /g)	Micropore surface area (m ² /g)
PCL	6.893·10 ³	16.8457
Chitosan	6.221·10 ³	14.8077
Multilayered	0.664·10 ³	0.9977
Layered	4.139·10 ³	8.9189
Simultaneous	0.393·10 ³	0.8519

effect of having a highly textured surface. The combination of easy macropore access and dense surface functionalization (as is the case of chitosan) allows these materials to have strong adsorption rates even if the surface areas are not as pronounced (Scaffaro et al., 2024).

3.1.6. Mechanical tests

The mechanical behavior of the membranes was assessed through tensile tests, considering tensile strength, yield point, strain at rupture, and Young's modulus. The obtained results are summarized in Table 5.

The PCL membrane displayed a very ductile behavior, characterized by an elastic phase and a plastic phase where stress continued to increase, albeit at a lower rate (*vide* Figure S8). The tensile strength (3.49 MPa) was significantly higher than the yield point stress (1.56 MPa), indicating that after yielding, the material could sustain further loading before rupture, due to molecular orientation within the amorphous regions and the gradual stretching and rotation of crystalline lamellae. This behavior is typical of semicrystalline polymers like PCL, where the amorphous phase governs the large deformability, while the crystalline domains act as physical crosslinks that progressively align and reorganize under tensile loading (Baker et al., 2016; Alharbi and Guthold, 2024). Additionally, the high strain at rupture (373.2%) demonstrates its excellent stretchability and toughness, reflecting the dominance of chain mobility and alignment processes during deformation, as reported in the literature (Arrieta et al., 2020). The low Young's modulus (10.31 MPa) confirms its highly deformable nature. The uniformity of the PCL mat is confirmed by the low standard deviations in the mechanical properties of PCL and the consistent results across replicates.

The chitosan membrane exhibited a predominantly brittle behavior, characterized by a single elastic phase followed by immediate rupture, as seen on Figure S9. The tensile strength (2.35 MPa) coincided with the yield point, as no significant plastic deformation was observed. Once the material reached its maximum stress, failure occurred abruptly, with a strain at rupture of only 2.43%, confirming its limited elongation capacity. This membrane showed the highest Young's modulus (156.22 MPa), indicating a stiff structure with low flexibility. Similar behavior has been reported in electrospun chitosan nanofiber mats, which exhibited tensile strengths around 2.41 MPa and elongations at break of approximately 14.1% (Gu et al., 2013). In this case, the brittle nature of the mat is further enhanced by the presence of CNC, which is known for increasing material stiffness (higher Young's modulus) and reducing ductility, leading to more brittle behavior (Rocha et al., 2025).

Regarding the PCL/Ch nanocomposites, the simultaneous deposition membrane exhibited a mechanical profile similar to PCL, yet with notable differences due to the presence of chitosan (Figure S10). The Young's modulus (67.64 MPa) was significantly higher than that of pure PCL, meaning the membrane was stiffer and less prone to deformation. The yield point (2.32 MPa) was reached earlier than in PCL, suggesting that the presence of chitosan increased the structural rigidity, limiting the initial plastic deformation. Although a plastic region was still present, the increase in stress after yielding was much less pronounced than in PCL, indicating a restricted capacity for further deformation. Furthermore, the strain at rupture (34.63%) was significantly lower, aligning with expectations since chitosan is less ductile. The combination of both materials in a simultaneous deposition process explains the PCL-like mechanical profile, but with reduced elongation and increased stiffness. On the other hand, the layered membrane presented a distinct

mechanical behavior, combining characteristics of both chitosan and PCL membranes (Figure S11). The elastic phase was much shorter, resembling the chitosan membrane, with the yield point (2.62 MPa) being reached quickly (2.88%). Unlike the simultaneous deposition membrane, micro-fractures were observed immediately after yielding, likely due to the rupture of the chitosan layers within the structure. This suggests that, since the layers are stacked but not nano-scaled interwoven, the chitosan layers fail first, while the PCL component continues to sustain stress. After the initial micro-ruptures, the membrane entered a plastic phase more similar to PCL, where it maintained its integrity under further deformation. The strain at rupture (47.57%) was higher than in the simultaneous deposition membrane but still significantly lower than in pure PCL, reinforcing the idea that layer separation contributes to mechanical instability at higher strains.

Overall, both the simultaneous and layered mats increased the mechanical properties of chitosan membrane, with higher values for tensile strength and especially for strain at rupture. Although the simultaneous deposition membrane retained a more PCL-like profile and did not exhibit phase-specific failure, the layered membrane showed higher yield point stress and strain at rupture values. These findings suggest that both membranes can show the mechanical resistance for adsorption and filtration applications.

3.2. Preliminary screening and selection of the best adsorbent

The different nanocomposites were tested for adsorption of the dye Remazol Brilliant Blue R with an initial concentration of 100 mg/L and an adsorbent mass/solution volume of 4 g/L, a common starting value in the literature (Soon et al., 2019). Fig. 5 shows the dye removal efficiencies as well as the adsorption capacities (defined in Eqs. (4) and 5) for all sorbents except the pure PCL one, which showed a negligible removal.

In terms of removal percentages, the chitosan and the layered composites showed a much higher performance compared to the others, as they both achieve decolorizations above 95%, certifying that chitosan is the agent responsible for the dye elimination. The process occurs swiftly in the first 30 min of the experiment and then quickly reaches saturation. In contrast, the multilayered and the simultaneous mats, that have a higher PCL content, reach their plateau later but with remarkably lower efficiencies (35 and 20%, respectively). As far as the adsorption capacities are concerned, once again chitosan shows superior values around 80 mg/g, followed by the multilayered and layered ones with q_m values around 25 mg/g and by the simultaneous composite that was stagnant at 5 mg/g. All in all, these studies confirm that chitosan content is crucial for adsorption: the higher the chitosan content, the stronger the adsorption ability. PCL barely plays any role in it, given that its main role is providing mechanical strength as discussed in Section 3.1.6. It should also be mentioned that the samples of the chitosan mat employed for these tests suffered significant degradation due to the agitation. That is why the layered composite was chosen as the optimal adsorbent given that, despite its lower q_m , it still ensured full decolorization while being resistant to the agitation. Hence, it was selected for further study of the adsorption equilibria and kinetics.

Table 5

Mechanical analysis (tensile strength, yield point, strain at rupture, and Young's modulus) of chitosan, PCL, Layered and Simultaneous nanocomposites.

	Tensile Strength (MPa)	Yield Point Stress (MPa)	Yield Point Strain (%)	Strain at Rupture (%)	Young's Modulus (MPa)
PCL	3.49 ± 0.01	1.56 ± 0.04	22.20 ± 0.85	373.20 ± 10.18	10.30 ± 0.37
Chitosan	2.35 ± 0.58	2.35 ± 0.58	2.38 ± 0.35	2.43 ± 0.44	156.22 ± 44.82
Layered	2.95 ± 0.64	2.61 ± 0.98	2.89 ± 0.33	47.57 ± 15.16	133.76 ± 63.46
Simultaneous	3.37 ± 1.40	2.32 ± 0.99	5.01 ± 1.10	34.63 ± 6.90	67.64 ± 39.90

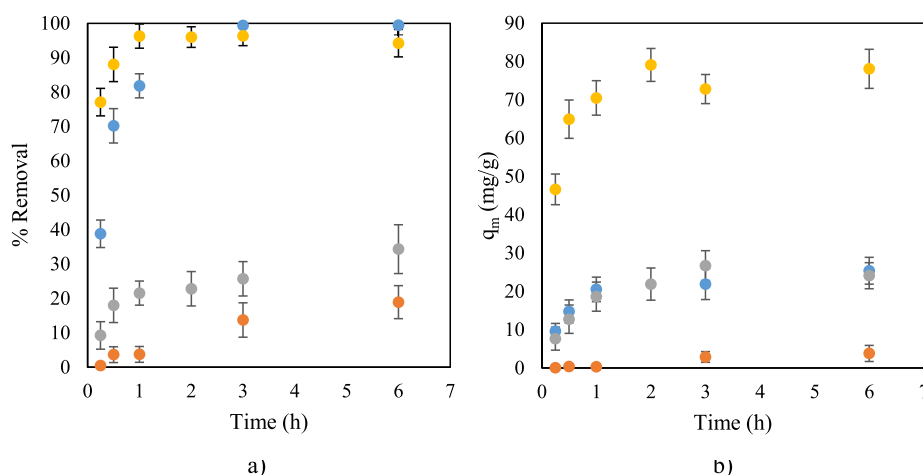


Fig. 5. Dye removal efficiencies (a) and adsorption capacities (b) at 25 °C, with a 100 mg/L dye concentration, a 4 g/L ratio of adsorbent mass/solution volume for the chitosan (●), the multilayered (●), the layered (●) and the simultaneous (●) mats.

3.3. Factors affecting the adsorption process

3.3.1. Adsorbent dosage

Minimizing the consumption of adsorbent is crucial for many industrial processes, so the dosage is frequently optimized in order to obtain a good compromise between total removal and material cost. For this, different amounts of nanocomposite were tested to adsorb the dye, as depicted in Fig. 6. Increasing the dose yields higher removal rates, while the adsorption capacity per weight of adsorbent decreases, as expected with the increased availability of adsorption sites provided by a higher dose.

The most efficient dosage for the experiments was chosen to be 10 mg of nanocomposite per 3 mL of dye solution, given that the removal at equilibrium is over 95%, and a higher mass does not cause a significant increase in the percentage. At the same time, the adsorption capacity is maximized, because at a higher dose there is a screening effect of adsorbent sites which may lower their availability to interact with the dye molecules in solution. This competition of the dye molecules for a limited number of available binding sites, along with an increased diffusion path length, provokes a suboptimal adsorption rate (Mohamed et al., 2022).

3.3.2. Dye concentration and contact time

Fig. 7 represents the removal of different concentrations of Remazol Brilliant Blue R at different times. During the first instances of the process, adsorption was fast for all cases, albeit slightly slower for the higher concentrations. Full decolorization was achieved (> 96%) for all

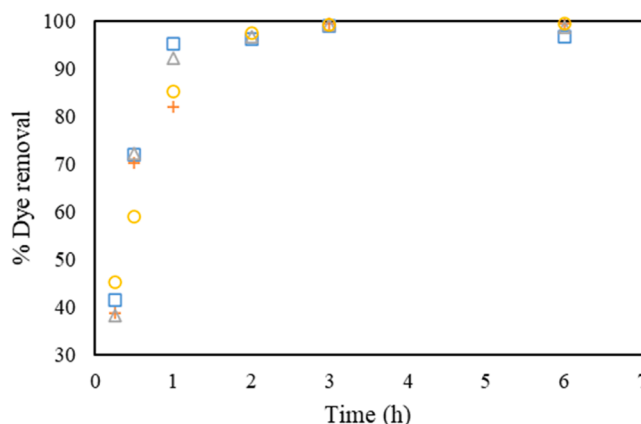


Fig. 7. Percentages of dye removal using 10 mg of the layered mat at 25 °C for different times and dye concentrations: (□) 50 mg/L, (+) 100 mg/L, (△) 150 mg/L and (°) 200 mg/L.

concentrations at times as short as 2 h, although experiments were prolonged to 6 h to ensure equilibrium was reached. Dye concentrations above 200 mg/L were not tested, as legislation requires textile wastewaters not to have values above this concentration range (Ananthashankar, 2013). The swift elimination observed in the initial steps of the process is explained with the abundance of active adsorption sites on the surface of the adsorbent and the strong concentration gradient that favors mass transfer between the solution and the adsorbent (Sadiq et al., 2021). The plateau is reached after the first hour of contact, when extraction significantly slows down as these two factors have decreased their magnitude. The adsorption capacities achieved were 12.2, 25.4, 43.3 and 56.2 mg/g for dye concentrations of 50, 100, 150 and 200 mg/L, respectively.

3.3.3. Temperature effect

The influence of temperature in the adsorption was tested by repeating the process under optimal conditions but modifying temperatures: in particular at 25, 35 and 45 °C. Adsorption efficiencies remain steady, as eliminations still hover around 96–98% in all cases for concentrations between 50 and 200 mg/L. The modifications of the conditions caused by temperature like the increase in diffusion rate seem to have little influence in the global percentage of removal, as the affinity of the nanofibers to capture the dye seems high at all temperatures tested.

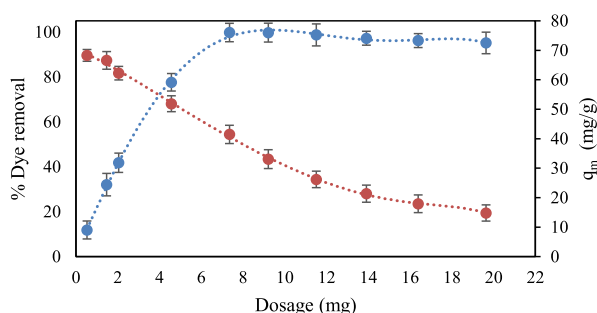


Fig. 6. Percentage of dye removal (●) and adsorption capacities (q_m , ●) with a varying adsorbent dose for 6 h of contact time at 25 °C and 100 mg/L dye concentration.

3.3.4. Adsorption kinetic models

Understanding and modeling the adsorption process is crucial to knowing its mechanism and the potential controlling steps. There are many theories that provide a theoretical foundation to how these processes may occur, so the ones mentioned in Section 2.5.1 were tested and fit to the obtained data.

As it can clearly be seen in Table 6, the pseudo-second order kinetic model provides the best correlations, given that the R^2 values are over 0.99 for all concentrations, significantly higher than the other ones (for comparison, results of the intraparticle diffusion model are provided in Figure S1). Furthermore, the experimental and the calculated q_e parameters agree solidly, as the overestimation by the model is not above 10%, and, as reported in the literature (Soliman et al., 2021), there is an inversely proportional relation between the pseudo-second-order rate constant (k_2) and the concentration of dye. According to this model, the rate of adsorption onto the fibers depends on the number of available sites and the concentration of adsorbate in the solution, i.e. surface adsorption is the dominating factor. The rate-determining step is governed by chemical adsorption, which depends on the electrostatic forces established by electron exchanges (Patinõ-Ruiz et al., 2020). The other models did not produce strong correlations for more than a single concentration value, so their contribution to rationalizing the process is not as relevant.

3.4. Adsorption isotherms

Three different temperatures were considered for obtaining the isotherms of adsorption: 25, 35 and 45 °C. Then, data was fit to the models listed in Section 2.5.1, and the results can be observed in Table 7. The best correlations were obtained with the Langmuir model, significantly better than with the other models, which were not taken into account. It still presented the strongest match at higher temperatures, but the R^2 coefficient dropped notably. This suggests that the adsorption process occurs by the formation of a monolayer of dye molecules that are uniformly distributed on the surface of the adsorbent with no interactions among them (Langmuir, 1918). At equilibrium, the saturation of the monolayer is complete. The separation factor constant (R_L) can be used to assess the favorability of the adsorption. If it is between 0 and 1, the process is very favorable, which was the case for the three temperatures considered.

Finally, thermodynamic parameters (Gibbs free energy, enthalpy and entropy changes) were calculated and are shown in Table S1. The negative values obtained for ΔG across all tested temperatures confirm the spontaneous and thermodynamically favorable nature of the dye

Table 6

Obtained parameters of the kinetic models for the dye adsorption on the layered mat.

	Dye concentration (mg/L)			
	50	100	150	200
q_e exp (mg/g)	12.2	25.4	43.3	56.2
Pseudo-first order kinetic model				
k_1 (min ⁻¹)	0.1037	0.4262	0.2386	0.0083
q_e (mg/g)	3.91	12.29	7.12	21.13
R^2	0.0798	0.9168	0.0985	0.6107
Pseudo-second order kinetic model				
k_2 (mg/(g·min))	0.6998	0.0964	0.0856	0.0489
q_e (mg/g)	12.53	27.32	46.95	59.88
R^2	0.9935	0.9983	0.9901	0.9933
Elovich order kinetic model				
β (g/mg)	3.865	5.015	11.668	0.094
α (mg/(g·min))	$5.49 \cdot 10^{-6}$	$4.48 \cdot 10^{-9}$	$3.96 \cdot 10^{-16}$	568.84
R^2	0.4434	0.9232	0.4761	0.8389
Intraparticle diffusion kinetic model				
k_3 (mg/(g·min ^{1/2}))	1.0277	6.9828	6.7718	15.893
I	7.8906	9.5037	27.113	24.035
R^2	0.4101	0.8121	0.2661	0.716

Table 7

Isotherm parameters for the dye adsorption on the layered mat.

Isotherm	Parameters	25 °C	35 °C	45 °C
Langmuir	k_L	0.2593	0.4295	0.3034
	q_m	69.4636	58.1284	108.9212
	R_L	0.0367	0.0225	0.0315
	R^2	0.9917	0.9812	0.7399
Freundlich	n	12.9120	12.9145	7.2983
	k_F	45.7426	48.4286	59.7392
	R^2	0.8903	0.4350	0.5928
Temkin	B (J/mol)	4.0798	4.2584	10.5838
	k_T (L/mol)	290.0689	433.5895	1.8514
	R^2	0.8489	0.3464	0.3165
Elovich	k_e	5.2751	15.3119	49.2956
	q_m (mg/g)	37187.7373	20.0954	0.9220
	R^2	0.8272	0.2599	0.1822
Dubinin-Radushkevich	β (kJ/mol)	$9.3116 \cdot 10^{-5}$	$1.661 \cdot 10^{-4}$	$1.570 \cdot 10^{-4}$
	E (kJ/mol)	73.2778	54.8697	56.4390
	q_m (mg/g)	62.2480	68.4891	101.0386
	R^2	0.7495	0.6318	0.3704

adsorption process. Furthermore, the positive enthalpy (ΔH) and entropy (ΔS) changes confirm an endothermic process with increased randomness, which supports the conclusions reached from the kinetic and isotherm studies.

3.5. Reusability and desorption

The adsorption of Remazol Brilliant Blue R onto the layered Ch/PCL electrospun mats operates through a multi-stage mechanism involving external film diffusion, microporous intraparticle mass transfer, and multi-site chemical interactions, consistent with multi-mechanism pathways reported for related functionalized electrospun Ch/PCL nanocomposites (Reghioua et al., 2021). First, anionic dye molecules diffuse rapidly from the bulk solution across the boundary layer to the nanofiber surface. Once at the active interface, the adsorption is primarily driven by strong electrostatic attraction between the negatively charged sulfonate groups of the dye ($R-SO_3^-$) and the protonated amines ($-NH_3^+$) and hydroxyls ($-OH_2^+$) of the accessible chitosan layer (Alghamdi et al., 2026). Then, secondary interactions stabilize the adsorbate complex. Namely, intermolecular hydrogen bonding established between the acidic protons of chitosan and the electronegative atoms on the anthraquinone backbone, and π - π and π - π interactions occurring between the electron-donating domains of the polymer and the electron-withdrawing aromatic rings of the dye. A graphical representation can be seen in Figure S12. This chemisorption-dominated, multi-stage mechanism is well supported by the high fits with the pseudo-second-order kinetic model and the Langmuir isotherm.

As expected from the high dye-chitosan compatibility, the reverse process was not as favorable. Alkaline solvents have been reported to desorb anionic dyes (Hamd et al., 2022; Martínez-Rico et al., 2024), given that the OH^- anions can compete with the adsorbate and replace it by overcoming the electrostatic interactions between the sulfonate groups on the dye molecules ($R-SO_3^-$) and the protonated $-NH_3^+$ and $-OH_2^+$ from chitosan (Wu et al., 2019), which has been reported to be the main interaction driving separations such as those described in this work (Martínez-Rico et al., 2024; Règo et al., 2013). These positively charged groups are deprotonated by hydroxyls so that the electrostatic interaction between chitosan and the dye weakens and the adsorbed dye leaves the adsorption site (Chiou and Li, 2003). Therefore, NaOH with different concentrations (0.1 and 1 M) was tested, as well as NH_4OH 0.1 M and H_2SO_4 0.1 M. Sulfuric acid produced a negligible desorption ($< 2\%$), while the basic solutions presented slightly better results: NaOH 0.1 M allowed to recover around 8% of the dye and NH_4OH a 13%. The best desorption was achieved with NaOH 1 M, which was able to transfer back to solution a 25% of the dye content. These findings can be explained by the fact that hydroxyl ions and the anionic dye molecules

compete among them (Gu et al., 2013), causing a partial desorption at higher OH⁻ concentrations, albeit not complete due to the significantly stronger dye-chitosan interaction.

Moreover, the results of the reuse of the adsorbents are shown in Fig. 8. It can be seen that the adsorption capacities and the removal percentages are maintained up to a fourth cycle, when a slight decrease to 85% is observed, leaving a slight hue of color in the aqueous phase.

Beyond retaining functional dye uptake, the structural and chemical robustness of the adsorbent after desorption and reuse cycles is critical for practical water remediation. After desorption in alkaline medium and subsequent washing, the layered Ch/PCL mat preserved its macroscopic integrity without experiencing degradation. This structural endurance is primary enabled by the stable, insoluble PCL supporting layer, which prevents the degradation or swelling-induced dissolution commonly reported for pure chitosan matrices. A similar behavior was reported by Guesmi et al. for an electrospun Ch/PCL nanofibrous membrane (Ce-MOF Ch/PCL/HA) where the polymeric matrix retained its structural morphology and chemical integrity after NaOH treatment (Guesmi et al., 2026). These observations confirm that the combination of chitosan with PCL provides sufficient chemical and mechanical stability.

3.6. Comparison with other adsorbents

As summarized in Table 8, electrospun nanofibrous adsorbents reported in the literature typically exhibit adsorption capacities below 70 mg/g when used against textile dyes. This behaviour is consistent across a wide range of polymeric systems, including polyacrylonitrile nanofiber mats, biodegradable polyhydroxybutyrate-based composites, silk nanofibers, and functionalized cellulose acetate membranes. In these materials, adsorption is primarily governed by surface chemistry rather than by exceptionally high specific surface area, with electrostatic or hydrogen bonding interactions playing key roles in dye uptake and their fibrous morphology ensuring good accessibility of functional groups and efficient mass transfer. Therefore, the adsorption performances observed in this study are in good agreement—often with slightly better values—with those reported for comparable electrospun nanofibrous materials, confirming that adsorption capacities hover around these ranges for this sort of dye adsorbents.

Beyond dye uptake performance, the economic feasibility of the layered Ch/PCL composite represents a key parameter for practical textile wastewater remediation. The cost-effectiveness of this system is based on three main factors. First, the low precursor cost, given that chitosan and cellulose nanocrystals are sourced from abundant industrial byproducts (crustacean waste and biomass), reducing reliance on expensive synthetic precursors. Then, savings on the recovery, as this nanofibrous mat can be easily recovered from solution with zero sludge production, as opposed to activated carbon or colloidal nanoparticles which require energy-intensive secondary treatments. Finally, its reusability, because the >98% removal capacity across multiple cycles and the possibility for dye recovery using low-cost NaOH solutions amortizes the initial manufacturing footprint, markedly reducing the adsorbent cost per volume of treated effluent.

4. Conclusions

This research highlights the development of a nanocomposite comprising environmentally-friendly chitosan and PCL, which exhibited superior adsorption properties compared to the initial materials. These adsorbents were easily retrievable and reusable, with the added advantage of enabling dye recovery through desorption. The inclusion of a PCL layer enhanced the structural stability of the material, resulting in highly effective removal of Remazol Brilliant Blue R from aqueous solutions. Even at concentrations reaching 200 mg/L, the elimination efficiency remained above 98%, leading to complete decolorization. The adsorption behavior aligned with the pseudo-second-order kinetic

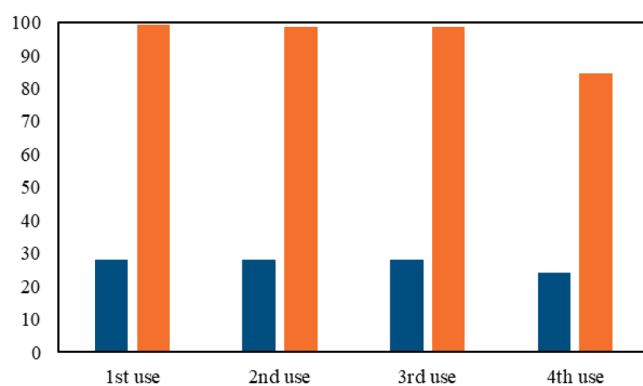


Fig. 8. Percentages of decolorization (●) and adsorption capacities (●) of the layered mat for 6 h at 25 °C against a dye concentration of 100 mg/L for each removal cycle.

Table 8

Comparison of the dye removal efficiencies with other similar materials.

Electrospun adsorbent	Dye	q _m (mg/g)	% Removal	Ref.
Cellulose acetate coated with polydopamine	Methylene Blue	69.89	90.77	(Chen et al., 2020)
Cellulose acetate coated with polydopamine	Congo Red	67.31	95.03	(Chen et al., 2020)
Cellulose/Chitosan hydrogel	Congo Red	40	89.6	(Li et al., 2016)
Silk nanofibers	Reactive Black 5	62.2	98	(Ghaffar et al., 2023)
Polyacrylonitrile	Congo Red	50.8	-	(Jadoo and Naser, 2019)
Poly-β-hydroxybutyrate with montmorillonite	Congo Red	52.7	-	(Penchev et al., 2023)
Polyvinylformate with montmorillonite	Congo Red	69.1	-	(Penchev et al., 2023)

model and the Langmuir isotherm, indicating monolayer adsorption. The adsorbents retained their performance over at least 3 reuse cycles. This process operated efficiently under mild conditions, with maximum efficiency achieved at room temperature. Furthermore, the approach can be extended to other anionic dyes, including azo dyes, offering a promising pathway for sustainable wastewater treatment that aligns with the principles of a circular economy in the textile industry.

CRediT authorship contribution statement

Óscar Martínez-Rico: Writing – original draft, Investigation. **Rui P. C.L. Sousa:** Writing – review & editing, Writing – original draft, Supervision, Investigation, Formal analysis, Conceptualization. **Joana C. Araújo:** Writing – review & editing, Investigation, Formal analysis. **Sofia M. Costa:** Writing – review & editing, Supervision, Formal analysis. **Begoña González:** Writing – review & editing, Supervision, Project administration, Formal analysis. **Diana P. Ferreira:** Writing – review & editing, Supervision, Project administration, Funding acquisition, Formal analysis, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at [doi:10.1016/j.hazadv.2026.101644](https://doi.org/10.1016/j.hazadv.2026.101644).

Data availability

No data was used for the research described in the article.

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