

Application of *Olea Europea L.* barks as eco-friendly adsorbent material: mechanism studies and efficiency evaluation

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ABSTRACT – REZUMAT

Application of *Olea Europea L.* barks as eco-friendly adsorbent material: mechanism studies and efficiency evaluation

The olive tree (*Olea europaea L.*) holds significant nutritional importance due to the therapeutic properties of virgin olive oil. Consequently, there has been a growing interest in exploring novel applications for the agricultural and industrial waste generated by the olive industry. This study investigates the performance of bark material derived from olive trees in removing dyes from textile wastewater. The study elucidates the influence of pH, ionic strength, adsorbent concentration, and chemical structure on the adsorption process. For direct dye removal, a basic pH value demonstrated superior depollution results. However, an acidic medium was necessary to achieve complete decolourisation of reactive dyes. Notably, the *Olea europaea L.* bark exhibited remarkable decolourisation and COD abatement capacities, surpassing 75% and 65%, respectively.

The experimental data were further validated by comparing them with classical calculations obtained using activated carbon. A relatively high level of agreement was observed, suggesting that bio-sorption using olive bark presents a promising alternative to the utilisation of expensive adsorbent materials.

Keywords: *Olea europaea L.*, olive bark, biomaterial, dye uptake, reactive dye, direct dye

Aplicarea scoarței de *Olea Europea L.* ca material adsorbant ecologic: studii privind mecanismul și evaluarea eficienței

Măslinul (*Olea europaea L.*) are o importanță nutrițională semnificativă datorită proprietăților terapeutice ale uleiului de măsline virgin. În consecință, a crescut interesul pentru explorarea de noi aplicații pentru deșeurile agricole și industriale generate de industria măslinelor. Acest studiu investighează performanța materialului din scoarță provenit de la măslini în eliminarea coloranților din apele uzate rezultate din industria textilă. Studiul elucidează influența pH-ului, a puterii ionice, a concentrației adsorbantului și a structurii chimice asupra procesului de adsorbție. Pentru îndepărtarea directă a coloranților, o valoare bazică a pH-ului a demonstrat rezultate superioare de depoluare. Cu toate acestea, a fost necesar un mediu acid pentru a obține decolorarea completă a coloranților reactivi. În mod remarcabil, scoarța de *Olea europaea L.* a prezentat capacități remarcabile de decolorare și reducere a COD, depășind 75% și, respectiv, 65%. Datele experimentale au fost validate în continuare prin compararea lor cu calculele clasice obținute folosind cărbune activ. S-a observat un nivel relativ ridicat de concordanță, ceea ce sugerează că biosorbția folosind scoarță de măsline reprezintă o alternativă promițătoare la utilizarea materialelor adsorbante costisitoare.

Cuvinte-cheie: *Olea europaea L.*, scoarță de măsline, biomaterial, absorbție de colorant, colorant reactiv, colorant direct

INTRODUCTION

The textile dyeing industry utilises substantial quantities of fresh water and generates substantial volumes of contaminated wastewater that contain residual colour, elevated salt concentrations, and high Biological Oxygen Demand (BOD) or Chemical Oxygen Demand (COD) values [1–6]. It is reported that there are over 100,000 commercially available dyes, with a production output exceeding 7×10⁵ metric tons annually. Consequently, 280,000 tons of textile dyes are discharged globally each year [7].

Although cotton is an environmentally friendly and sustainable textile, it contributes significantly to environmental emissions. Cotton factories generate substantial amounts of wastewater daily, which contains organic dyes such as Direct and Reactive dyes,

along with other contaminants exhibiting high levels of colour and total organic content [8].

Reactive dyes are typically azo-based chromophores combined with various reactive groups, including vinyl sulfone, chlorotriazine, trichloropyrimidine, and difluorochloropyrimidine [8, 9]. Direct dyes are predominantly azoic and also contain vinyl sulfone compounds. Direct and Reactive dyes are widely used in cotton dyeing factories due to their favourable characteristics, such as bright colours, simple application techniques with minimal energy consumption, and excellent fastness to washing and rubbing, particularly for reactive dyes [10–12]. However, these dyes pose significant environmental challenges. They are scarcely eliminated under standard aerobic conditions and can be transformed into carcinogenic

aromatic by-products. Furthermore, chemical coagulation is ineffective in removing reactive and direct dyes due to their high solubility in water [13–15]. Consequently, the rate of reactive dye discharge is low, and its hydrolysis cannot be avoided.

Consequently, highly coloured effluents that do not meet environmental standards and cannot be reused are discharged daily.

Adsorption has been extensively used in industrial processes for the separation and purification of coloured industrial effluents. A low number of applications has been noted for the case of textile effluents, where treatment is mostly based on coagulation and flocculation, followed by aerobic degradation techniques [16–20]. The implementation of biological treatments has inherent drawbacks of being technically unfeasible because of the non-biodegradability of such organic and recalcitrant compounds.

Adsorption is a more promising option for non-biodegradable organics. This technique requires less land area (half or a quarter of what is required in a biological system), gives lower sensitivity to diurnal variation, is not affected by toxic chemicals and ensures greater flexibility in the design and operation and superior removal of organic contaminants [21–24].

The activated carbon adsorbent is now considered to be the best prospect for eliminating residual colour. Despite its effectiveness and versatility, this adsorbent is prohibitively expensive and difficult to regenerate after use [25]. To be more attractive, there is a growing interest in using other low-cost adsorbents. Being an inexpensive and non-abundant adsorbent, some lower-cost bio-adsorption compounds have been proposed [9]. Natural compounds, including agricultural by-products such as nut shells, wood, bone, and peat, processed into activated carbons, have also been proposed [26–29]. For example, sawdust, which is an abundant and low-cost by-product, has been used as a precursor for the preparation of activated carbons and successfully explored as an adsorbent for the removal of dyes from wastewater [30]. Some other by-products, either synthetic but non-expensive, were also employed for the decolourisation of textile wastewater. The β -cyclodextrin grafted

nonwoven polypropylene (PP) was effective for the removal of cationic dyes [31]. N6-PPI nanofiber also showed its effectiveness and rapidity in removing anionic dyes from aqueous media [32, 33].

Olive (*Olea europaea* L.) has been cultivated in the Mediterranean region since ancient times. Olive trees are extremely long-lived (up to 1000 years). The consumption of Virgin Olive Oil is increasing worldwide due to its recognised nutritional benefits [34]. A young olive tree has smooth grey bark, but with age, the bark becomes gnarled and gradually disintegrates into rectangular pieces. It is believed that cuticular photosynthesis plays a vital role in recapturing respiratory CO_2 and, thus, contributes to internal CO_2 recycling in stems and overall annual carbon balance. Dry barks result from the photosynthesis of sun-exposed corticular [35]. The resulting biomaterial is considered waste and left without any valorisation. Therefore, such wastes are low-cost, available in abundance, and could be suitable for the decolourisation of cotton effluent thanks to their mainly cellulosic composition. So far, no studies have been reported on the mechanisms of adsorption.

To gain a comprehensive understanding of the potential applicability of olive tree bark, we have conducted a meticulous study on its absorbency capacity for the removal of direct and reactive dyes from synthetic dyeing wastewater. The effluent, which contains residual dyes and their associated auxiliaries, is substituted for actual dyeing wastewater from the textile industry to replicate comparable conditions. The impact of pH, chemical structure, and dye concentration was investigated to optimise dye removal, maximise adsorption capacity, and elucidate the underlying adsorption mechanism. Additionally, the adsorption process with activated carbon was examined for both tested dyes to provide comparative data.

EXPERIMENTAL

Chemicals

Commercial Reactive and Direct dyes were supplied from the Dyeing Factory and used directly for adsorption experiments without further purification. Chemical structures are presented in figure 1.

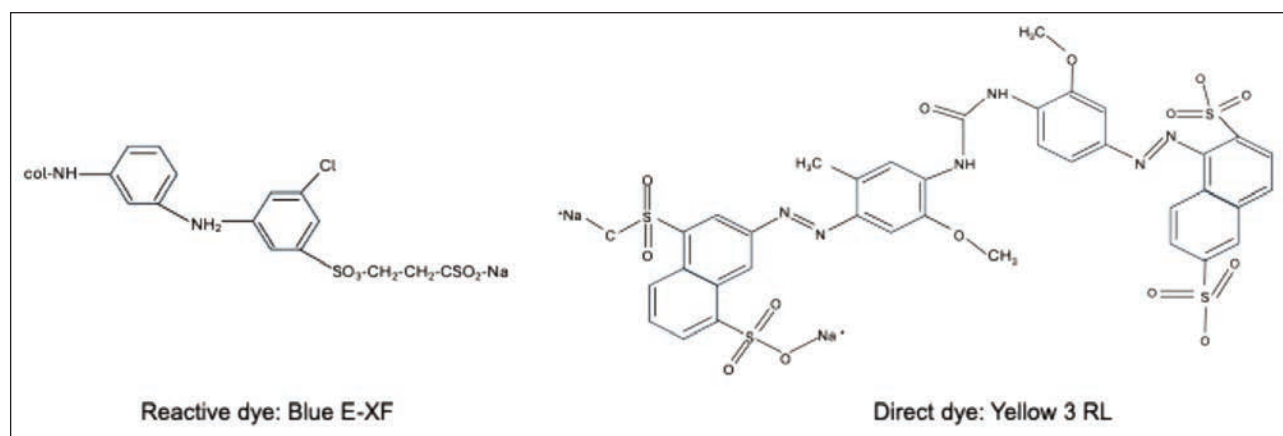


Fig. 1. Chemical structures of the tested dyes



Fig. 2. Adsorbent materials: *a* – activated carbon; *b* – olive barks slices; *c* – olive barks powder after preparation

Bioadsorbents preparation

Olive Barks were dried at 110°C for 12 hours, powdered and sieved to a particle size range of 40–60 meshes, or maintained as slices and then stored in sealed containers for experimentation. Activated carbon granulates were also used to make the comparison (figure 2).

Method

Batch absorption study

Adsorption experiments were determined by shaking a 500 ml vial with 1% (w/v) of adsorbent material collected from olive trees and previously treated with caustic soda (1M of NaOH for 2 hours). The dye concentration varied from 5 to 40 mg/l. Experiments were also conducted at different pH levels (from 2 to 10). The pH adjustments were done with 1.0 M HCl and 1.0 M NaOH.

After shaking at 90 rpm for 120 min, the samples were taken, and the solution was separated from the precipitate by centrifugation at a speed of 4000 rpm for 5 min. The remaining concentrations of dye were determined spectrophotometrically by monitoring the absorbance at $\lambda_{\max}$. The effects of the chemical structure of pollutants were also studied.

Evaluation of the adsorption efficiency

The surface images of the used adsorbents were captured by scanning electron microscopy (SEM). The SEM used was a Hitachi S-2360N.

The amount of dye adsorbed per unit of mass of adsorbent at equilibrium conditions Q_e (mg/g) was calculated as follows (equation 1):

$$Q_e = \frac{C_0 - C_t}{m} \times V \quad (1)$$

where C_0 (mg·l⁻¹) is the initial concentration of dye, C_t (mg·l⁻¹) is the residual dye concentration in solution at time t , V (l) is the volume of solution and m (g) is the mass of the adsorbent material.

The measurement of each sample was replicated twice and averaged. To calculate the dye adsorption efficiency by Activated carbon and Bark powder, the following equation 2 was used.

$$\text{Adsorption} = \frac{C_0 - C_t}{C_0} \times 100 \quad (2)$$

Evaluation of the depollution efficiency

pH measurements were performed with a pH meter (Model 2906, Jenway Ltd., UK). Chemical Oxygen Demand tests were performed by oxidation with dichromate according to Standard Methods of Examination of Water and Wastewater [36, 37].

Absorbance measurements were carried out with a UV-visible spectrophotometer (Shimadzu UV-256), recording the spectra over the 190–900 nm range. Direct and reactive dyes with different absorbance peaks were presented in the studied wastewater. The maximum absorbance of the real wastewater is nonstable. So, before each experiment, UV-Vis spectra of dye solutions were plotted to establish their maximum.

The disappearance of the absorbance peaks of the treated solutions was monitored, and the colour removal ratio was calculated as follows (equation 3):

$$\text{abs} (\%) = \frac{\text{abs}(\lambda_{\max})_{\text{ini}} - \text{abs}(\lambda_{\max})_t}{\text{abs}(\lambda_{\max})_{\text{ini}}} \times 100 \quad (3)$$

where $\text{abs}(\lambda_{\max})_{\text{ini}}$ is the average value of absorben-
cy at $\lambda_{\max}$ of the concerned wastewater, and $\text{abs}(\lambda_{\max})_t$ is the value obtained at time t .

Chemical Oxygen Demand tests were performed according to the Standard Methods of Examination of Water and Wastewater [38]. The percentage of dye mineralisation was evaluated from the measurement of COD removal using this formula (equation 4):

$$\text{abs} (\%) = \frac{\text{COD}_i - \text{COD}_t}{\text{COD}_i} \times 100 \quad (4)$$

where COD_i corresponds to the initial value and COD_t is the value obtained at time t .

RESULTS AND DISCUSSIONS

Physical character of adsorbents

The physical character of the tested adsorbents is detailed in table 1.

With a relatively similar physical property, the topography of each adsorbent was also investigated using a scanning electron microscope. Figure 3 shows the SEM images of microstructures of the *Olea europaea* L. Bark powder compared to the Activated carbon structure under different magnifications. Images show that the surface of activated carbon is fairly

Table 1

PHYSICAL PROPERTIES OF OLIVE BARKS AND ACTIVATED CARBON GRANULAR		
Properties	Olive barks	Activated carbon
Bulk density (kg/m ³)	1650	2000
BET surface Area (m ² /g)	479	698
Moisture (%)	8	4

smooth with few cracks and voids. The SEM of the Bark powder has an extensive external surface with quite irregular cavities and pores in terms of shape and length. The high porosity observed on the external surface of Bark powder can be attributed to the swelling step with caustic soda of this material, mainly cellulosic. High porosity was also noted for activated carbon.

This porosity results from the evaporation of the chemical reagent (H₃PO₄) during the preparation of the activated carbon [35]. The microstructure of each adsorbent has an important role in defining further the adsorption capacity.

Adsorption evolution experiments

For the adsorption study, two dyes (Direct and Reactive) and two adsorbent materials (Activated carbon and Olive tree bark) were considered. Initial experimental conditions were 120 min of absorbance duration, 10 mg/l of Dye, pH 6 and ambient temperature. At the selected conditions, the absorbance kinetics are described in figure 4.

In each studied case, the results demonstrated the feasibility of achieving a substantial colour removal value through the utilisation of Activated carbon or Olive bark. Notably, the evolution of absorbance curves revealed two distinct zones. The initial zone

exhibited a rapid decline in absorbance, attributed to the enhanced availability of vacuums and active sites on the adsorbent material.

Experimental results presented in figure 4 also showed that a good degree of colour removal might be reached using the bioadsorbent. High removal rates (over 70%) were achieved using Activated carbon for Direct and Reactive dyes. Using *Olea europaea* L. barks, decolourisation was similar for direct dyes, but lower performance was mentioned with the Reactive dye. Side experiments showed that this performance could be improved using massive dosages of adsorbent material.

Study of the experimental effect of pH, dye type and concentration

Effect of initial pH on dye adsorption

pH of the medium defines the surface charge of the adsorbent, which is related to the ionisation degree of the adsorbate. The relationship between the initial solution pH and dye removal is illustrated in figure 5. The experiments carried out at different pH values showed that there is a progressive increase in the capacity of removal of Direct dye over the entire pH range of 2 to 10. The interaction between dye and Olive bark powder is becoming stronger at higher pH values. Better results were reached with pH 8 and 10. However, at pH 10 and after 20 min of adsorption, the attached dye molecules showed a slight disturbance, which can be related to the swelling evolution of the introduced Olive barks in the basic medium. In fact, at the beginning of the process, the adsorbed dye molecules were attached to the surface by physical Van Der Waals bonds. By progressive swelling onto Olive tree barks, two simultaneous and antagonistic phenomena can take place:

- The absorbance of Direct dye is due to its affinity for cellulosic material.
- Physical repulsion between the nearest dye molecules and the surface of the cellulosic material due to its progressive swelling at basic pH.

After 30 minutes of the absorbency evolution, the adsorbed dye molecules formed stronger bonds. Indeed, the cellulosic adsorbent became deprotonated at this pH value. So, the electrostatic interaction between dye and adsorbent developed the main force controlling the absorbance. Figure 5 shows that the effect of pH on the amount of dye adsorbed was different for the Reactive dye. Better results were obtained in an acidic medium. When the solution pH was less than 6, the nitrogen groups, especially those involved in aromatic systems of the tested Reactive dye, became positively charged by protonation. This finding is in accordance with previous studies [39]. Subsequently, the attraction between reactive dyes (positively charged in an acidic medium) and Olive bark

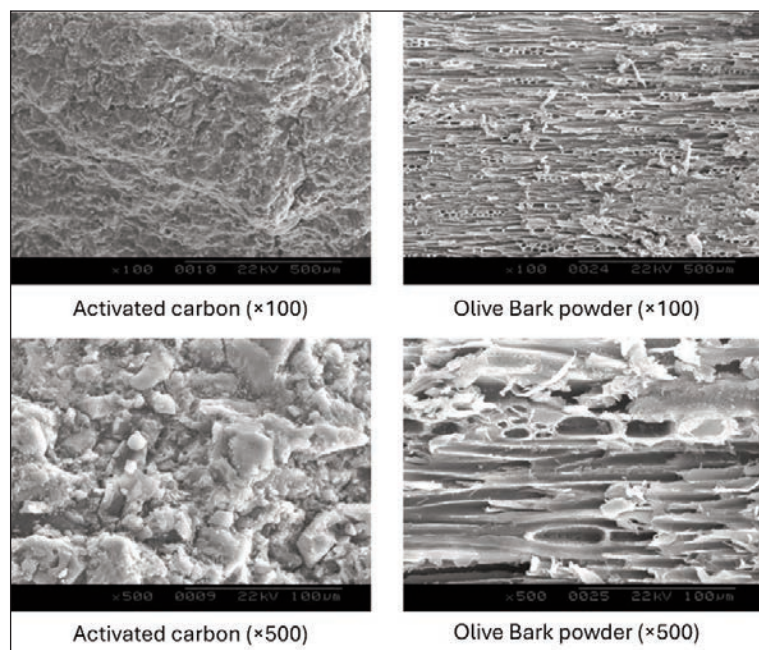


Fig. 3. SEM images of Activated carbon (at the left) and Olive Bark powder (at the right) under a magnification of $\times 100$ and $\times 500$

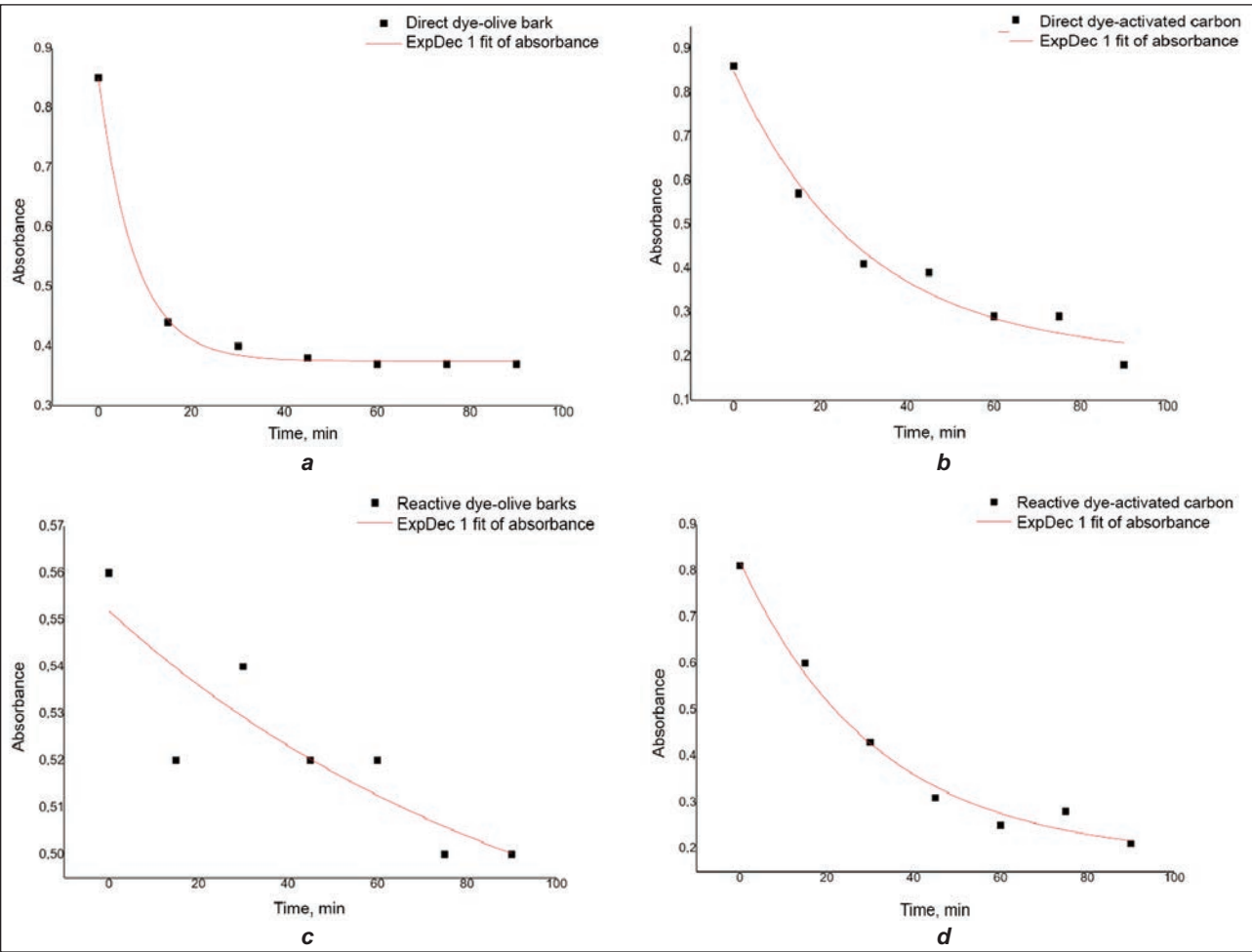


Fig. 4. Absorbance kinetics: *a* – Direct dye with Olive bark; *b* – Direct dye with Activated carbon; *c* – Reactive dye with Olive bark; *d* – Reactive dye with Activated carbon

increases, resulting in a slight increase in the amount of dye adsorbed.

The low dye removal at a highly basic solution was related to the strong repulsion interaction between the negatively charged adsorbent and the deprotonated reactive dye molecules. At the same time, hydroxide ion concentration increased with the incremental solution pH, and it could be adsorbed preferentially on the surface of the activated carbon.

In turn, variations in the pH value with Activated carbon did not evoke such a considerable increase in the adsorption affinity. Elsewhere, the adsorbent quantity was not explored to confirm its effect on the adsorption affinity.

Effect of dye concentration on the amount of dye adsorbed

Adsorption of dye on the adsorbent surface was determined for different concentrations of Direct and

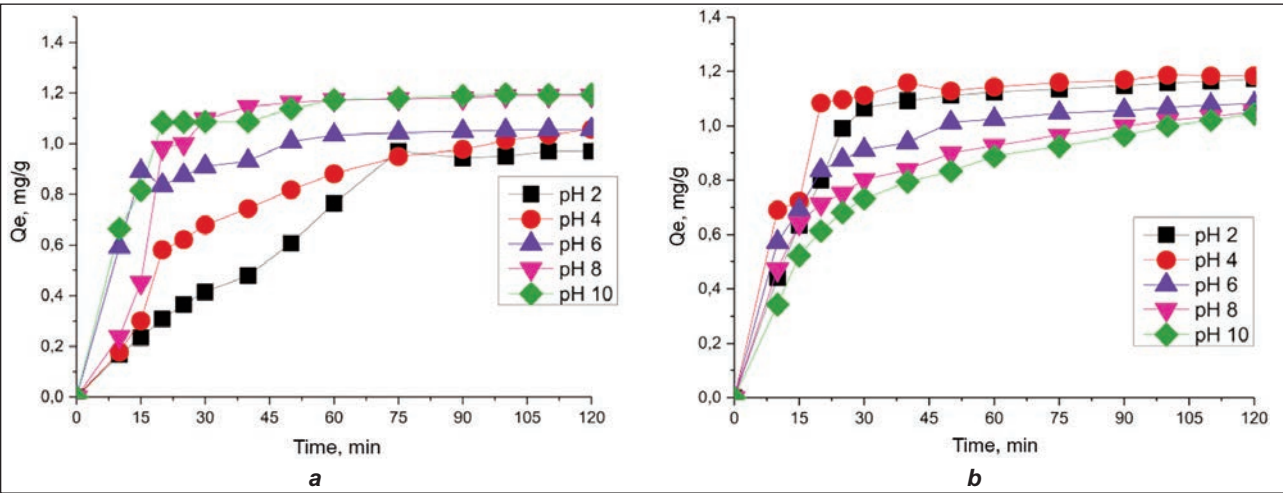


Fig. 5. Effect of pH on the dye uptake kinetics: *a* – Direct dye; *b* – Reactive dye

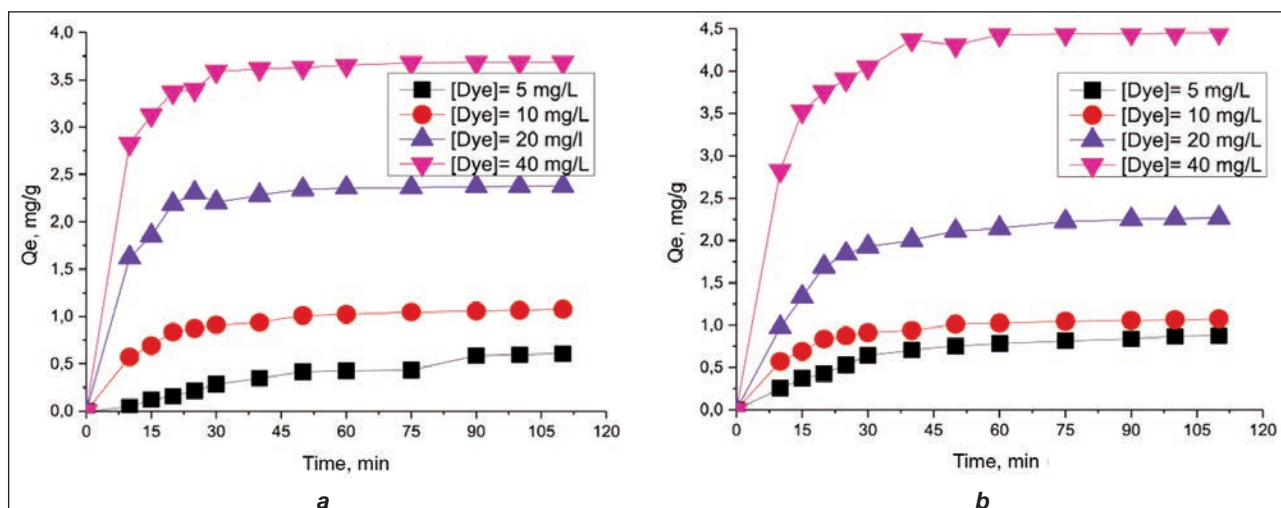


Fig. 6. Dye uptake kinetics as a function of dye concentration: a – Direct dye; b – Reactive dye

Reactive dyes while maintaining the same quantities of adsorbent. Results are presented in figure 6.

The effect of dye concentration was also investigated in the case of the Direct dye treated with Olive bark powder. Initially, during the adsorption process, the equilibrium value of the amount adsorbed was observed to increase with the increasing dye concentration. For dye concentrations exceeding 30 mg/l, the equilibrium value of the amount adsorbed reached its maximum within a few minutes. This phenomenon can be attributed to the rapid decrease in the availability of surface-active sites due to the increased dose and accumulation of the adsorbent. Consequently, the amount of the adsorbed dye exhibited exponential variation at these concentrations.

On the other hand, the contact time reached saturation within 60 minutes for all tested concentrations. Subsequently, the equilibrium value of the amount of adsorbed exhibited a smooth and continuous curve, indicating the formation of a monolayer coverage of the adsorbate on the outer surface of the adsorbent. This linearity at higher contact times confirmed the absence of desorption, which can also be attributed to the strong crosslinking between the dye and the adsorbents. Olive bark possesses a cellulosic composition. It is well-known that polysaccharides can establish physical and chemical interactions with a diverse range of molecules [40]. This property enhances the degree of crosslinking and effectively prevents desorption even during extended periods of adsorption.

The reactive dye concentration also exhibited a significant influence on its removal from the aqueous phase. This trend was attributed to the high driving force for mass transfer at a high initial dye concentration. Furthermore, for higher dye concentrations, a greater number of dye molecules surrounded the active sites of the adsorbent, leading to more efficient adsorption. Consequently, dye uptake increased with the increase in the initial dye concentration.

UV-Vis spectra changes

The spectrum of Reactive dye evolution in the visible region exhibits a main peak with a maximum at 610 nm. The decrease of the absorption peak at $\lambda_{\max}$ in figure 7 indicated a rapid adsorption of the dye in the first 15 min. The main absorbance peak disappeared within approximately 75 minutes. This result indicates that the colour removal by Olive bark powder is attributed to elimination without decomposition.

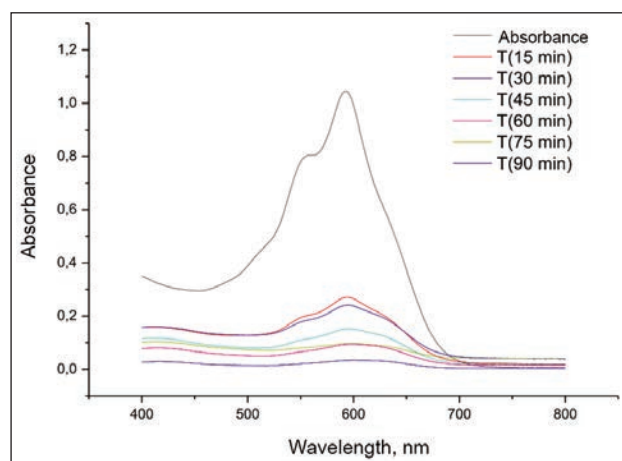


Fig. 7. UV-Vis evolution on Olive Bark support for Reactive dye

Adsorption experiments at optimal conditions showed that colour and COD removals of the direct dye were 87% and 70% respectively. As shown in table 2, Olive barks showed similar performance in terms of colour and COD removals, either for direct or reactive dye experiments. Absorbance measurements were also found to be in good accord with COD and colour results.

CONCLUSIONS

This research study investigated the potential of Olive Bark as an agro-waste and compared its performance with a commercial granular activated carbon (GAC) as a reference material in the adsorption

DEPOLLUTION RESULTS EVALUATION WITH BIO-ADSORBENT VERSUS CONVENTIONAL ADSORBENT MATERIAL				
Parameter	Direct Dye depollution results		Reactive Dye depollution results	
	Activated carbon	Olive Barks	Activated carbon	Olive Barks
Colour removal (%)	87	83	86	75
COD removal (%)	73	70	77	69

and depollution of wastewater contaminated with reactive and direct dyes. The findings demonstrated the feasibility of utilising Olive Bark powder and slides as effective adsorbents for Vinyl Sulfone reactive dyes and azoic direct dyes. The efficacy of the adsorption process was found to be influenced by the chemical structure of the adsorbate, the amount of adsorbent applied, and the pH value of the solution. The results indicated that Olive Bark powder exhibited a rapid, efficient, and economical adsorption process compared to conventional methods. For direct dyes, the adsorption treatment resulted in a faster and complete decolourisation of the contaminated wastewater. Colour and Chemical Oxygen Demand (COD) reductions were approximately 83% and 70%, respectively. Similarly, 87% and 73% colour and COD removals were achieved using granules of activated carbon.

The improvement in depollution parameters observed for the direct dye sample was permitted, considering its high affinity for the cellulosic composition. Furthermore, the selected direct dye exhibited enhanced reactivity due to its superior global electric charge.

Consequently, a comprehensive analysis of the adsorption kinetics is imperative and necessitates a substantial number of experiments under varying conditions. Additionally, the study of the isotherm and kinetics of the adsorption of both dyes onto the proposed adsorbent should be undertaken.

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