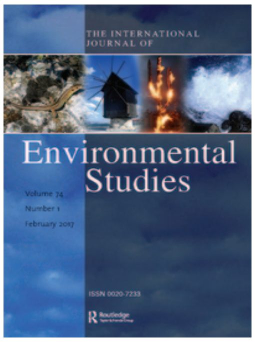

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An approach to textile dye removal using sawdust from *Aspidosperma polyneuron*

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ABSTRACT

The textile industry is responsible for discarding wastewater contaminated with dyes. The timber industry generates waste in the form of sawdust. The aim of the present study was to evaluate the adsorptive potential of sawdust obtained from the *Aspidosperma polyneuron* tree for the removal of the textile dye from wastewater. Sawdust was subjected to different pre-treatments (acid, alkaline and polyethyleneimine) in order to increase its adsorption capacity. Based on the results from the isotherms, treatment with polyethyleneimine (PEI) led to the greatest adsorption capacity and fits the Freundlich model, indicating cooperative adsorption. Other treatments with sawdust best fit the Langmuir model, but the untreated sawdust presented better results than the treated sawdust. These results were only surpassed by sawdust treated with PEI. *A. polyneuron* revealed good potential for use as an adsorbent to remove dyes, which is a novel result, since to date there is no study on its use as a sorbent material.

KEYWORDS

Adsorption; isotherm; polyethyleneimine; sawdust; wastewater

Introduction

Pollution of rivers is widespread because of illegal discharges from domestic sewage, industrial and biomedical waste, pesticides, etc. Freshwater supplies are becoming increasingly polluted throughout the world and thus unable to satisfy industrial demands [1]. Textile manufacturing is one of the most polluting industries, owing to the high volume and chemical composition of its effluents [2]. Dyes predominate in effluents from textile plants. More than 700,000 tons of dyes are produced annually [3]. During production and consumption, up to 10% of these dyes are discharged as wastewater, creating a variety of environmental problems [4]. The negative effects of dyes included impeding the penetration of sunlight, and affecting the solubility of gases; thus harming aquatic ecosystems. Moreover, dyes may be toxic and exert both mutagenic and carcinogenic effects on organisms [5]. Such effluents also persist longer because of their chemical composition containing large amounts of aromatic rings, amine linkages and sulfonic acid groups [6].

Adsorption is one of the most attractive methods for the removal of dyes from wastewater; there is no need for a large application area, treatment is rapid, and adsorption is cheap. The possibility of using existing waste products as adsorbent material is an additional

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advantage that reduces treatment costs [7]. A number of different products such as coal coke, bentonite, resin, cotton, sand, rice hulls, palm tree straw, steel slag, feathers, steel waste, tannery waste and the seed husks from *Araucaria* sp. [8] have been employed as adsorbent material.

The Brazilian timber industry generates 22.5 million tons of waste annually, which represents a serious economic and environmental problem [9]. Sawdust is also considered a waste presenting difficulties in proper disposal. Burning sawdust results in concentrated emissions, such as carbon dioxide [9]. In this way, the sawdust is often wasted in large quantities.

Aspidosperma polyneuron is a species of tree native to Brazil, popularly known as Pink Mahogany, and its biome of the Atlantic Forest origin. The *A. polyneuron* is very important for the Brazilian timber industry, since it is used in carpentry, civil engineering and building furniture. Accordingly, much sawdust from timber processing of *A. polyneuron* is generated during processing.

Therefore, the aim of the present study was to test sawdust from the timber tree *A. polyneuron* as a possible adsorbent material for the removal of a textile dye from an aqueous solution. To increase its adsorption capacity, the sawdust was submitted to both acid and alkaline treatments in addition to treatment with polyethyleneimine (PEI). The isotherm was studied using different mathematical models to understand the adsorption processes, and the effectiveness of the colour removal from solution was analysed by spectrophotometry.

Materials and Methods

Dye

The textile dye Direct Red 23 (DR 23), is a reactive azo dye with molecular mass of 813.72 g mol^{-1}, empirical formulation: $C_{35}H_{25}N_7Na_2O_{10}S_2$, and Colour Index: 29,160 (Figure 1), manufactured by Imperial Chemical Industries (ICI) was employed in the present investigation. The stock solution was prepared by diluting 500 mg of powdered dye in 500 mL of distilled water to a final concentration of 1 mg mL^{-1}.

As figure 1 shows, the DR 23 dye has a long chemical structure with several aromatic rings connected in their structure, and also has two binding groups azo –N=N–, typical of recalcitrant molecules, that are difficult to degrade. It may also be noted that the dye has two SO_3Na groups in its structure, confirming its solubility in water.

Source of adsorption material

Sawdust from the species *A. polyneuron* was donated by the company AM Resins & Varnish Ltd. (Brazil) and was sieved to achieve a standard particle size of 0.14–0.21 mm. The sawdust was washed with distilled water, and then placed in an oven at 100 °C for 24 h, to remove any impurity.

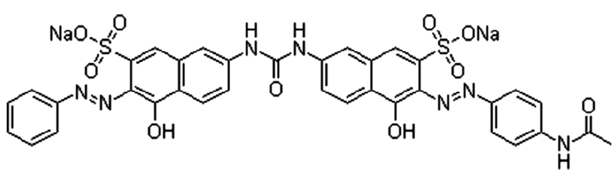

Figure 1. Chemical structure of DR 23 dye.

pH optimization

Many dyes undergo changes in their spectra and a slight shift in peak absorbance to shorter or longer wavelengths (hypsochromic and bathochromic shifts, respectively) as the pH of the environment changes [10]. To check the occurrence of these phenomena, different dye concentrations were tested by varying the pH from 2.50 to 6.50 and scans were performed in a UUV-vis spectrophotometer (Shimadzu 2401-PC) from 200 to 800 nm in quartz cuvettes with an optical path of 10 mm.

To determine the optimum pH for the adsorption of the dye using sawdust, tests were performed with different dry weights of sawdust from 2 to 10 mg in dye solutions at pH 2.50, 4.50 and 6.50 in 50-mL Erlenmeyer flasks containing 20 mL of dye at a concentration of 100 μg mL^{-1}. The dye remained in contact with the sawdust for 180 min at 30 °C. The sample was then centrifuged for 30 min at 5000 rpm to separate the adsorbate from the adsorbent and a reading was taken in the UV-vis spectrophotometer. Equation 1 was used to calculate the percentage of dye removed.

$$D(\%) = \frac{(Co - Ce) \cdot 100}{Co} \tag{1}$$

in which Co is the initial concentration of dye (μg mL^{-1}), Ce is the concentration of dye in equilibrium (μg mL^{-1}) and D(%) is the percentage of dye removed.

Chemical treatment

The sawdust was submitted to different chemical treatments to increase its adsorption capacity. For such, 1 g of dried sawdust was submitted to acid treatment with sulphuric acid (H_2SO_4), alkaline treatment with sodium hydroxide (NaOH) and a PEI treatment (Figure 2) with the aim of increasing the NH and NH_2 groups.

Thus, a 50 mL solution of each compound was prepared at a concentration of 2% (m v^{-1}). The sawdust remained in contact with these solutions for 24 h at 150 rpm and 20 °C. The sawdust was then washed with distilled water and placed in an oven at 65 °C for 24 h.

Adsorption tests using the sawdust submitted to different chemical treatments were performed with 20 mL of dye solution in 50-mL Erlenmeyer flasks with dye concentration of 100 μg mL^{-1}. The dry weight mass of sawdust modified used was 2–10 mg, and it was placed in contact with dye solution for 180 min at 30 °C. The pH that led to the best adsorption during initial test on pH optimization was used. To compare the results of the adsorption using the chemically modified sawdust, the research team used the same adsorption tests with sawdust without any chemical treatment (*in natura*). Equation 2 was employed to calculate the amount of adsorbed dye.

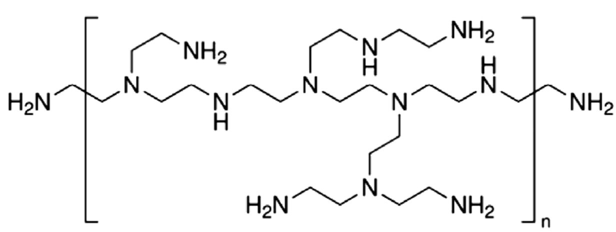

Figure 2. Chemical structure of PEI.

$$qe = \frac{V \cdot (Co - Ce)}{W} \quad (2)$$

in which qe is the equilibrium concentration of the adsorbate on the adsorbent ($\mu g\ mg^{-1}$), V is the volume of adsorbate (mL) and W is the mass of the adsorbent (mg).

All adsorption tests were made in triplicate. Graphs and chemical structures of this study were made with the Origin 6.0 and ACD/ChemSketch software programs.

Results and Discussion

Determination of optimum pH for adsorption

Spectrophotometry and linear regression coefficients demonstrated for each pH tested that the DR 23 dye was stable (no bathochromic or hypsochromic shift), at a maximum range at 507 nm. Table 1 shows the results obtained during the initial adsorption tests with variations in sawdust mass and pH.

Adsorption with the sawdust was most efficient at pH 2.50. Many binding sites are protonated because of an acidic adsorbent and are able to form more bonds with a dye [11]. This is demonstrated by the results obtained at pH 6.50, when adsorption occurred more inefficiently owing to the neutral solution. In solutions with pH close to neutral, there is less formation of hydrogen bridges or protonation binding sites, reducing interactions adsorbate/adsorbent, thereby reducing efficiency of adsorption [10]. Adsorption at pH 4.50 shows that the acid solution of dye makes for better adsorption when compared to the pH 6.50.

It was also possible to estimate the amount of sawdust necessary for the complete removal of dye for each treatment. At pH 2.50 and 4.50, 21 and 37 mg would be necessary, respectively, whereas 86 mg would be required at pH 6.50, which is more than twice the concentration of sawdust as that needed in acidic pH.

These results are very important because although there are many dye adsorption studies using sawdust [12–14], no work to date has used sawdust from *A. polyneuron* to remove dyes or any other contaminant. Even though a species of tree native to Brazil, it has not been studied by any other Brazilian researchers in respect to its adsorptive potential [14].

In the present investigation, rapid decanting of the sawdust was found; less than 10 min were needed for the entire mass of sawdust to be deposited on the bottom of the flask. Decantation is an important property in wastewater treatment [15].

Chemical treatment of sawdust

The Langmuir and Freundlich isotherms (mathematical models) were used to analyse the data obtained for absorption at pH 2.50 with the different sawdust treatments. An adsorption isotherm is important to describing how a solute interacts with an adsorbent [16].

Table 1. Adsorption results varying sawdust mass and pH of solution.

Sawdust (mg)	pH 2.50 (%)	pH 4.50 (%)	pH 6.50 (%)
2	9.12	8.36	0.00
4	26.81	14.60	0.00
6	30.55	17.76	2.08
8	40.48	23.99	3.59
10	46.94	30.50	10.25

The Langmuir isotherm [17], Equation 3, suggests that the adsorbate will bind to a specific site of the adsorbent and have a finite number of binding sites, forming a monolayer. The forces of interaction between the adsorbed molecules in the Langmuir model are negligible [18].

$$qe = \frac{qml \cdot Ce}{1 + l \cdot Ce} \tag{3}$$

in which l is the Langmuir constant related to affinity between the adsorbent and adsorbate (mL mg^{-1}) and qm is the maximum quantity of adsorbate adsorbed to the monolayer (μg mg^{-1}). The Langmuir mathematical model can be linearized (Equation 4). The slope of the (Ce/q) vs. Ce graph then allows the visualization of the balance parameters.

$$\frac{Ce}{qe} = \frac{1}{l \cdot qm} + \frac{1}{qm} \cdot Ce \tag{4}$$

The separation factor of the Langmuir constant, which is defined as Rl can then be calculated (Equation 5), thus enabling the determination of whether adsorption is favourable [19].

$$Rl = \frac{1}{1 + l \cdot Co} \tag{5}$$

The Rl value indicates whether the type of isotherm is irreversible ($Rl = 0$), favourable ($0 < Rl < 1$), linear ($Rl = 1$) or unfavourable ($Rl > 1$) [20].

The Freundlich model [21], Equation 6, was also employed. This model proposes that binding sites are occupied exponentially, forming multiple layers [22]. More energetic sites are occupied first, as the intensity of the interactions decreases with the progressive increase in the occupation of sorption sites [23].

$$qe = Kf \cdot Ce^{1/nf} \tag{6}$$

in which nf is the adsorption constant intensity and Kf is the Freundlich constant representing solid adsorption capacity (mL mg^{-1}).

The Freundlich mathematical model can also be linearized and rewritten (Equation 7). A lnqe vs. lnCe linear regression graph can then be created to determine the Kf and nf values.

$$\ln qe = \ln Kf + \frac{1}{nf} \cdot \ln Ce \tag{7}$$

The isotherm results (Table 2) indicate that adsorption was most efficient when the sawdust was treated with PEI, which best fits the Freundlich model (Figure 3), demonstrating the interaction of adsorbate with more than one active site (multiple layers). The chemical molecule of PEI offers many potential sites to bond with a dye, such as amine groups, thereby increasing the adsorption efficiency of adsorbents treated with this polymer [24].

The nf values greater than 1 for the PEI treatment also show the occurrence of lateral interactions with the dye molecules, demonstrating cooperative adsorption [25]. This also confirms the interaction between the dye and sawdust treated with PEI at heterogeneous sites. The data from the Langmuir isotherm for sawdust treated with PEI show that the highest value qm, confirming greater capacity to adsorb the dye. The values of Rl less than 1 indicate that the adsorption of the dye by sawdust treated with PEI is favourable.

Table 2. Results of isotherm study.

Different chemical treatments with sawdust	Langmuir isotherm				Freundlich isotherm		
	qm	*l*	*Rl*	R^2	*Kf*	*nf*	R^2
Sawdust *in natura*	5.560	0.051	0.163	0.86104	0.158	1.178	0.81092
Alkaline treatment	1.273	0.009	0.524	0.93787	0.002	0.598	0.87433
Acid treatment	1.552	0.025	0.285	0.94027	0.006	1.142	0.85591
Treatment with PEI	121.95	0.029	0.256	0.87936	0.083	2.217	0.89639

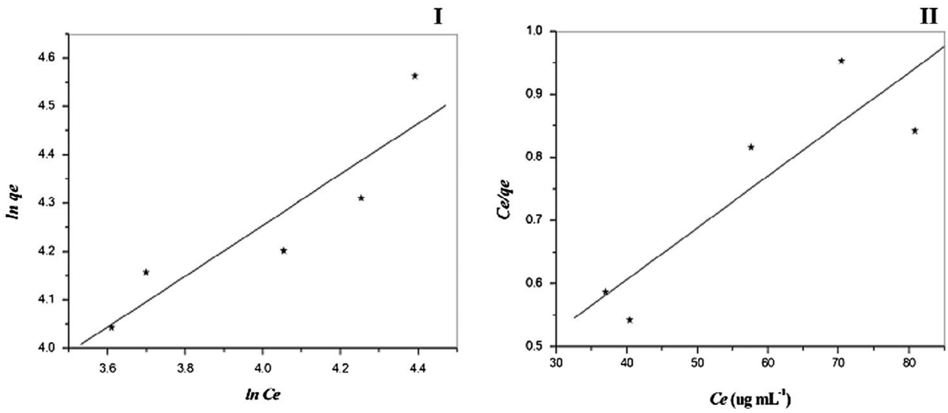

Figure 3. Adsorption isotherm models for sawdust treated with PEI: (I) Freundlich model and (II) Langmuir model.

Sawdust submitted to acid treatment (Figure 4) and sawdust *in natura* (without chemical treatment) (Figure 5) fit the Langmuir isotherm, indicating that only one adsorbent site interacted with the dye and formed monolayers. The *Rl* values for the two treatments also indicate favourable adsorption. But the *qm* values of sawdust *in natura* demonstrate greater adsorption capacity in comparison to the sawdust submitted to acid treatment. Although the results of these two treatments do not fit the Freundlich model, the *nf* values indicate lateral interactions with the dye, which likely occurred because of physical adsorption by van der Waals forces [26].

The alkaline treatment led to the lowest adsorption capacity, as demonstrated by the lowest *qm* value. Despite fitting the Langmuir model (Figure 6), the *Rl* value was the highest of the four treatments, indicating less favourable adsorption.

The *nf* less than 1 also indicates a lack of lateral interactions between the dye and adsorbent [27]. The acid and alkaline treatments likely affected the lignin and cellulose in the sawdust, modifying the original structure, with a decrease in binding sites and, hence, adsorption capacity [28]. Figure 7 shows the percentage of dye removed with each treatment.

The sawdust *in natura* presented a percentage of dye removal higher than chemical treatments which were acid and alkaline. Thus, untreated sawdust could be a good adsorbent material. This also highlights the use of sawdust from *A. polyneuron* treated with PEI, which can nearly triple the dye removal percentage. Chemical treatments in sawdust [12,29] or using the PEI [24] are very common, aiming to increase the adsorptive potential of the material, but these treatments end up increasing the cost of the material. Even so these

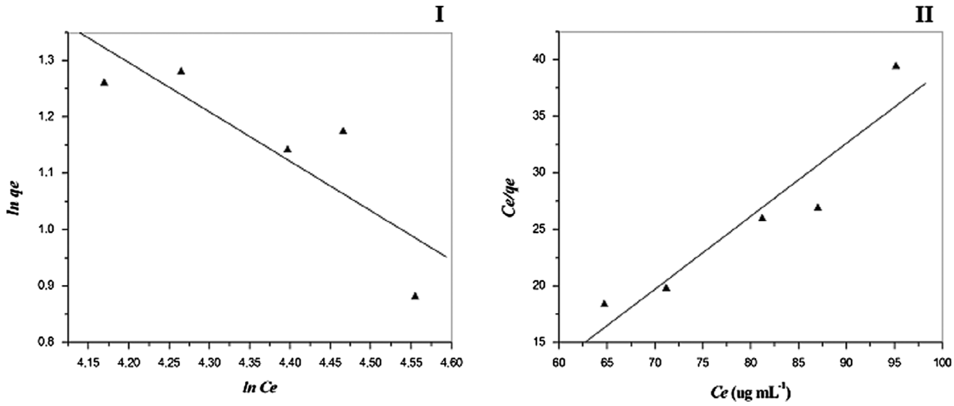

Figure 4. Adsorption isotherm models for sawdust submitted to acid treatment: (I) Freundlich model and (II) Langmuir model.

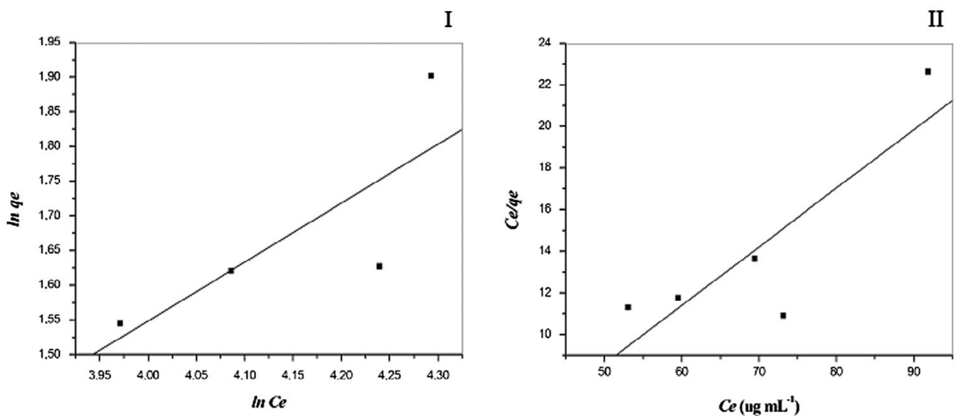

Figure 5. Adsorption isotherm models for sawdust *in natura*: (I) Freundlich model and (II) Langmuir model.

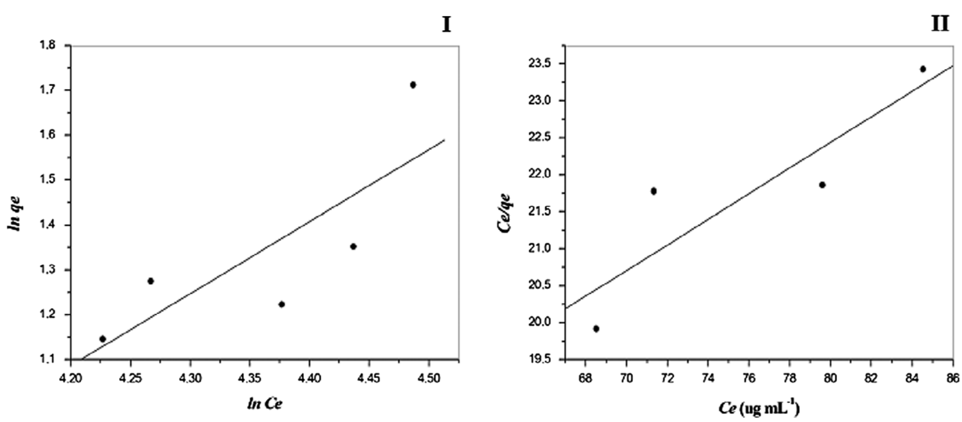

Figure 6. Adsorption isotherm models for sawdust submitted to alkaline treatment: (I) Freundlich model and (II) Langmuir model.

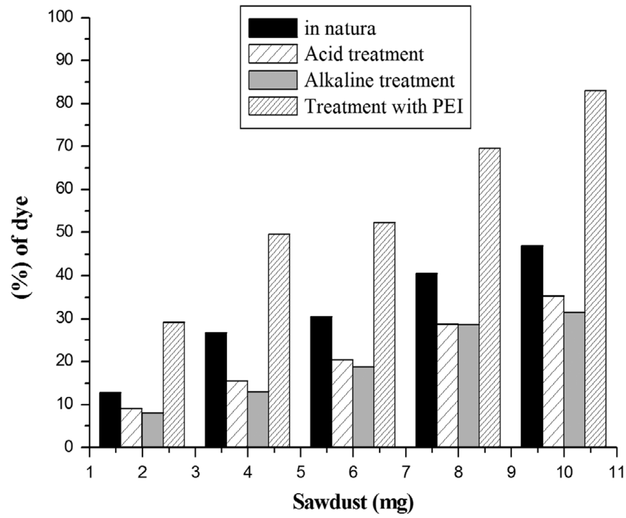

Figure 7. Percentage dye removal with different treatments.

results are important because until now no study has been done on the adsorptive capacity of *A. polyneuron* sawdust using any of these chemical treatments, which is a novel result.

The effectiveness of the colour removal from a solution can be evaluated spectrophotometrically using a standard defined in the literature [30]. The concentration of colour that is left by the dyes in the water is one of its main negative effects, not allowing the passage of sun light, impeding the activities of photosynthetic organisms from water, affecting the entire base of the food chain in this environment. Thus, it is possible to evaluate the degree of contamination through the absorbance of the dye solution, with absorbance of 0.060 considered tolerable [10]. Figure 8 shows absorbance of the permitted standard and that of the dye solution at a concentration of 100 μg mL^{-1} before and after adsorption to sawdust submitted to the different types of treatment tested in the present study.

Although no treatment achieved complete discoloration, treatment with PEI led to absorbance near the permitted standard (0.060). Since the use amount of sawdust treated with PEI was proportional to dye removal, it is possible to achieve even lower results than the permitted standard by increasing the mass of the adsorbent. Thus, 16 mg of sawdust treated with PEI can lead to the complete removal of the DR 23 dye. Sawdust *in natura* also exhibits a satisfactory result, since 21 mg leads to complete dye removal. One of the advantages of adsorption is the total removal of dye molecule, removing the coloration and toxicity of the solution [26].

For the acid and alkaline treatments, a total of 27 and 35 mg of dried sawdust would be required to remove all the dye from the solution. It is therefore preferable to employ sawdust *in natura*, which makes treatment of effluents polluted with dye much more economical. Indeed, the lower treatment cost is one of the advantages of using an industrial waste to remove a contaminant [12].

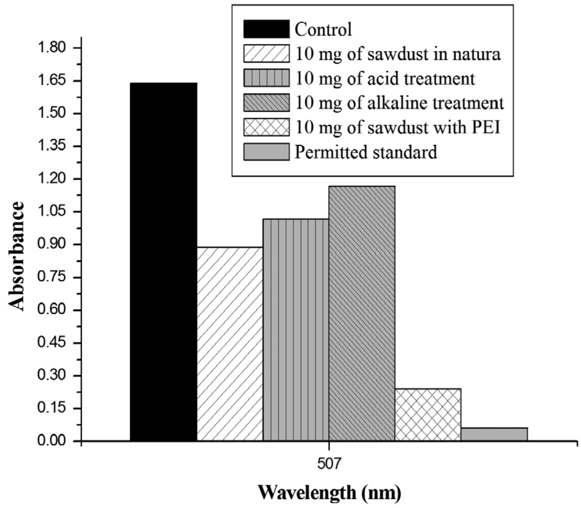

Figure 8. Graph of discoloration with different treatments in comparison to permitted standard.

Conclusions

The DR 23 textile dye was found to be stable, with no changes in its spectrum with the variation in the pH of the solution. Moreover, the dye demonstrated greater affinity to sawdust at pH 2.50. All the tested treatments with sawdust, except for the treatment with PEI, fit the Langmuir isotherm, indicating interaction with a specific site of the adsorbent to form monolayers. Treatment with PEI better fits the Freundlich isotherm, forming multiple layers, and indicating the occurrence of cooperative adsorption.

Sawdust treated with PEI also exhibits greater efficiency in adsorbing the dye in comparison to the other treatments tested. The comparison of the percentage of dye removed from the different treatments confirms the results of the isotherm studies, with the best results achieved using PEI to treat sawdust and with poorer results achieved using the acid and alkaline treatments in comparison to sawdust *in natura*. Although no treatment tested achieved the permitted colour standard of the solution, treatment with PEI and sawdust *in natura* could reach this standard with an increase in sawdust mass.

All studies in this paper were *in vitro*, and were not applied in the real world of industry. Industrial application is certainly desirable. The sawdust of *A. polyneuron* is a residue produced in large quantities in Brazil, and thus can be trialled at many sites as an adsorbent. Further, the sawdust treated with PEI can be applied to a decolouring treatment without causing toxic effects, since both materials can be removed before the effluent is disposed of in the environment, leaving no residue. It can therefore be said that sawdust from *A. polyneuron* is a good adsorbent material and its adsorption capacity increases when treated with PEI. This proves it to be a possible low-cost alternative for treating wastewater contaminated with textile dye on an industrial scale.

Disclosure statement

No potential conflict of interest was reported by the authors.

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