Ability to Remove Azo Dye from Textile Dyeing Wastewaters of Carbonaceous Materials Produced from Bamboo Leaves

Thi Thu Huong Tran, Ngoc Toan Vu, Thanh Nga Pham, and Xuan Tong Nguyen

Abstract Textile dyeing wastewaters is one of the most difficult to handle. The aim of this paper to investigate the removal capacity of two azo dyes, including Brilliant green and Alizarin red S from the aqueous solution by three types of activated carbon prepared from bamboo leaves AC30 (650 °C/30 min), AC45 (650 °C/45 min) and AC60 (650 °C/60 min). All three samples displayed the functional group represented by the azo dye removal capacity such as C=C; C–O–C, O–H and showed high C content (over 72%). With the highest BET surface area up to 108.9202 m^2/g, the AC60 material sample recorded the maximum efficiency of 100% at the reaction time of 30 min with the volume ratio of azo dye/distilled water of 2/18 (mL), pH 9, the absorbent amount of 0.5 and 2 g for Brilliant green and Alizarin red S, respectively. This study is an overview of the azo dye removal capacity in initial textile dyeing wastewater sample and compares the quality of AC60 material sample to four commercial activated carbon samples, including AC-R (Russian activated carbon), AC-C (Chinese activated carbon), AC-F (French activated carbon) and AC-Tra Bac (Vietnamese activated carbon from coconut shell). The analysis results also showed that the azo dye removal efficiency of the AC60 material synthesized from bamboo leaves is higher than some previously prepared material (AC-C material reached only 90.5 and 82.55% for Brilliant green and Alizarin red S, respectively) and achieved the maximum adsorption efficiency 100% after 30 min reaction time. These findings indicated that the removal efficiency for azo dye depends on experimental conditions

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S. S. Muthu and A. Khadir (eds.), *Novel Materials for Dye-containing Wastewater Treatment*, Sustainable Textiles: Production, Processing, Manufacturing & Chemistry,
https://doi.org/10.1007/978-981-16-2892-4_8

such as reaction time, pyrolysis temperature, pH… and the source of the raw materials also has a great influence on the carbonaceous material structure, determining the treatment efficiency as well as the removal capacity for pollutants.

Graphic Abstract

Keywords Activated carbon · Bamboo leaves · Azo dyes · Adsorption efficiency · Adsorption capacity · Brilliant green · Alizarin red S · Textile wastewater · Carbonaceous material · Characteristic material

1 Introduction

Activated carbon (AC) is well known as the most commonly used adsorbent in water and wastewater treatment. ACs characterized by their extended specific surface reactivity, good internal porous structure and high chemical, mechanical and thermal stability [15] which be used in a variety of applications such as removal of organic pollutants, heavy metals, medical application, catalysis, electrode materials in electrochemical devices [5]. In recent years, the adsorption techniques of ACs have been widely applied in water treatment due to fast adsorption kinetics, simplicity of design, initial cost, high removal efficiency, and insensitivity to toxic substances. However, the high cost of ACs makes it increasingly necessary to produce and regenerate [7, 39]. The carbonaceous material has a wide range of sources, including industrial waste [13], waste of biodiesel industry [36], agricultural by-products (pineapple waste [32]; soybeans, corn stalks, corn stalks [29], corncob [49]), livestock manure (poultry manure, poultry manure, poultry manure [29, 54]), vermicompost [51] sludge. The carbonaceous material itself has the advantages of large pores, high surface area, and variety surface functional groups. The variety surface functional groups with

high porosity are considerably crucial in the adsorption process of dye, especially azo dyes. Azo dyes are widely used in various industries such as textiles, leather, plastics, and paper and manufacturing of inks, which account for more than 60% of total dyes.

Azo dyes characterized with the presence of one or more azo functional groups (R_1–N=N–R_2), where R_1 and R_2 are aromatic groups, can be substituted by some combinations of functional groups such as amino (–NH_2), chlorine (–Cl), hydroxyl (–OH), methyl (–CH_3), nitro (–NO_2), sulphonic acid and sodium salts (–SO_3Na) [8]. However, azo dyes as wastewater pollutants represent a big concern for the environment, and currently the most attractive issue is finding the methods to remove these substances from water. Most dyes are toxic, carcinogenic or mutagenic [20] and can pose a hazard to health, like causing chromosomal damage [2]; affecting the eyes and skin, damage to internal organs such as liver and kidney [3]; disrupting the photosynthesis process in water bodies [38]. Consequently, it is very essential to treat the textile wastewater or find the new material to absorb or support the azo dye removal from wastewater. Several techniques were investigated to remove the azo dye species from the wastewater [45], including chemical, physical and biological treatments or a combination of these processes such as: adsorption [10, 26], hydrogen peroxide [25], biological treatment [24], membrane filtration [28]. Among the physical processes, adsorption is considered an effective separation technique in terms of low cost, flexibility, simplicity of design, ease of operation, and sensitivity to toxic substances [1, 20].

Bamboo is a large, woody-grass member of the family of Bambusoideae encompassing about 1250 species within 75 genera worldwide [16]. The raw materials used in the paper industries, cottage industries, domestic commodities, board and charcoal are gradually being replaced by bamboo material [30]. Vietnam is the fourth country in the world in terms of area planted to bamboo and production of bamboo, primarily grown in Lam Dong, Da Lat. Vietnamese people use bamboo in many ways, such as sharpened bamboo trees to make weapons in the wars, and now, bamboo trees are used as materials to build houses, make furniture, and other useful everyday items baskets, chopsticks, fans.

Bamboo is considered one of the ideal materials for the twenty-first century because it is environmentally friendly. Nowadays, there has been increasing interest in the research of the production of low-cost activated carbon from bamboo. Many studies have been conducted to evaluate the ability of using bamboo in the synthesis of carbonaceous materials for the removal of dyes [14, 52]. In this study, the carbonaceous materials were synthesized from bamboo leaves at 650 °C with three ranges of activation time 30, 45, and 60 min. The adsorption capacity of the activated carbon material in current study was evaluated through the use of two industrial dyes (namely, Brilliant green and Alizarin red S). The characteristics and structure of two selected commercial azo dyes are shown in Table 1 and Fig. 1 below.

Various researches were carried out to remove these dyes from wastewater by the carbonaceous material synthesized from other residues or agriculture waste, such as: studies for the treatment of Naphthol B. green by the red mud [25], removal of Alizarin red S reviewed by the activated carbon/γ-Fe_2O_3 nano-composite [9, 23],

Table 1 Characteristics of two azo dyes used in the current study

Dye	Brilliant green	Alizarin red S
Molecular formulae	$C_{27}H_{33}N_2HO_4S$	$C_{14}H_7NaO_7S$
IUPAC name	4-{[4 (diethylamino)phenyl](phenyl)methylidene}-N,N-diethylcyclohexa-2,5-dien-1-iminium	1,2-dihydroxy-9,10-anthraquinonesulfonic acid sodium salt
Characteristic	It is a cationic dye. It causes an allergic reaction on the human body, eye irritation and even leads to blindness, directly affects in the liver and kidney failure, cancer-causing agent, and even mutations in human beings [27, 34]	It is a water-soluble, anionic anthraquinone dye and usually used as a staining agent in textile industries. This dye is also used to stain biological specimens such as mineralized bones invertebrate groups and tiny invertebrate embryos [42]

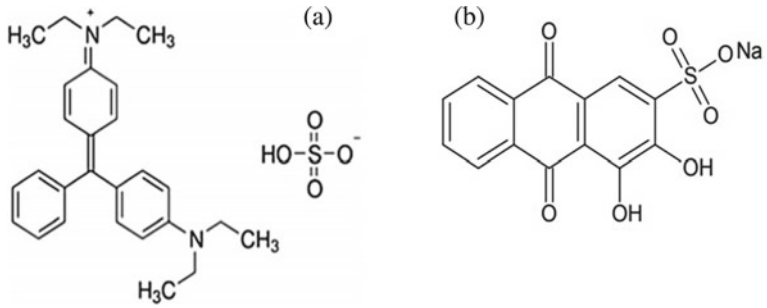

Fig. 1 Structure of two selected commercial azo dyes: **a** Brilliant green [27] and **b** Alizarin red S [42]

removal of Chrysoidine Y by the activated Sawdust [6, 35]. However, not much have been published on using ACs material obtained from bamboo leaves to adsorb these above dyes from industrial effluents. Therefore, this study was conducted to evaluate the ability to remove two azo dyes from the aqueous solution, including Brilliant green and Alirazin red S by carbonaceous materials produced from bamboo leaves. The study was designed to (1) assess the azo dye removal efficiency (according to the amount of the adsorbent material and the reaction time) of three activated carbon material and (2) examine the actual wastewater sample and (3) assess the quality of the synthesized material by comparing the adsorption efficiency to some commercial carbonaceous material.

2 Materials and Methods

2.1 Preparation of Activated Carbon from Bamboo Leaves

Fresh bamboo leaves as raw materials were collected at Lang area, Thach That District, Hanoi city, Vietnam and moved to laboratory, clean washed and naturally dried at room temperature for 24 h. Bamboo leaves were then chopped and soaked with 5% H_3PO_4 solution for 1–2 h, took out to drain (the aim of this technical step to clean impurities on bamboo leaves and prevented the natural burning under heating effect). Transfer the bamboo leaves to the tube containing samples of the oven and carbonized them at a temperature of 225–230 °C/4 h. Afterwards, down the temperature of the oven to 150 °C and took the carbonized bamboo leaves out of the oven. Then, put these bamboo leaves into distilled water to reduce heating, soon after took out the basket to drain naturally. After draining, put carbonized bamboo leaves into each inox tray in the drying oven, dried at 60 °C for 12 h. The final, transfer the carbonized bamboo leaves to each ceramic bowl to activate in the UAF furnace at temperature 650 °C for 30, 45, and 60 min, respectively. The washed material samples were dried at 105 °C for 12 h, finely ground and stored in ceramic boxes.

The yield of activated carbon in the current study calculated to be 38.71 [19]. The three material samples will be encoded as AC30, AC45, AC60 and used for further experiments. All the chemicals in this study supplied by Merck.

2.2 Identification of Material Characteristics

The activated carbon material also determined for its surface morphological characteristics and the chemical composition using a JSM 6380 Scanning Electron Microscope (SEM) and Energy-dispersive X-ray spectroscopy (EDX). The functional groups on the surface of each material sample were identified using the Fourier Transform Infrared spectroscopy (FTIR) TENSOR II instrument. The BET- and t-plot method was used to determine the total surface area, average pore radius and micropore volume of samples by Micromeritics Tristar 3000 instrument. Besides, the particle size is also determined to have an overview of the structure of the three activated carbon materials. All samples in this study were measured at the Institute of Tropical Technology, Vietnam Academy of Science and Technology and Hanoi National University of Education.

2.3 Experimental Setup

2.3.1 Preparation of Azo Dyes (Brilliant Green and Alizarine Red S)

The two azo dyes used in current study (Brilliant green and Alizarin red S) were supplied by Sigma–Aldrich. Their chemical structure is shown in Fig. 1. Weigh 0.01 g each compound Brilliant green and Alizarin red S separately, dissolve in 100 mL of distilled water, shake well and store in a dark bottle as a stock solution.

2.3.2 Experimental Steps

Our steps proceed very much in the same way follow what is indicated in Tran et al. [48]. Three activated carbon materials were evaluated for the adsorption capacity in batches in 100 mL flasks. At first, carried out experiments to select the optimization volume of azo dye in solution. 0.5 g activated carbon added into 20 mL solution containing azo dye (Brilliant green and Alizarin red S) and distilled water according to the ratio of $V_{\text{azo dye solution}}$: $V_{\text{distilled water}} = 2{:}18; 3{:}17; 4{:}16; 5{:}15$ and no pH adjustment. Then, shake the samples well by a horizontal shaker with speed of 150 r/m for 30, 60, 90, 120 min and measure the pH of the newly mixed samples. The filtered samples have analyzed the concentration of azo dye remaining by HPLC instrument at 360 nm (Brilliant green) and 260 nm (Alizarin red S) in Institute of Chemistry - Material, Institute of Military Science and Technology, Hanoi, Vietnam. The ratio

of $V_{azo\ dye\ solution}$: $V_{distilled\ water}$ with the highest adsorption efficiency was selected to evaluate for further experiments.

Subsequently, 0.25; 0.5; 1; 2 g of three material samples added into every flask contains 20 mL solution with selected optimization ratio and continue to carry out experiments at 30, 60, 90, 120 min. Adsorption capacity q_e (mg/g) and adsorption efficiency H (%) at equilibrium time are calculated according to the formula of Tran et al. [48] as follows:

$$q_e = (C_o - C_e) \cdot V/m\ (mg/g)$$

$$H(\%) = (C_o - C_e)/C_o \cdot 100\ (\%)$$

where q_e is the adsorption capacity at equilibrium (mg/g), C_o: initial concentration (mg/L), C_e: concentration at equilibrium (mg/L), V: volume of solution (L), m: mass of absorbent material (g).

In this study, V (the adsorption volume, 20 mL), C_o (the concentration of the initial adsorbent solution, mg/L), and m (the amount of the adsorbent, 0.25; 0.5; 1 and 2 g) were fixed. The ratio volume of azo solution/distilled water and adsorption time will be changed various from 2:18; 3:17; 4:16 và 5:15 mL and t = 30; 60; 90; 120 min, respectively.

The experimental procedure is summarized in Fig. 2 below. The newly synthesized activated carbon with highest adsorption capacity will be used to compare the quality with some available commercial products (from Russia, China, France and the Tra Bac of Vietnam). A textile wastewater sample was collected at the local textile factory in Long Bien, Hanoi and transfer to the laboratory. Analyze the initial

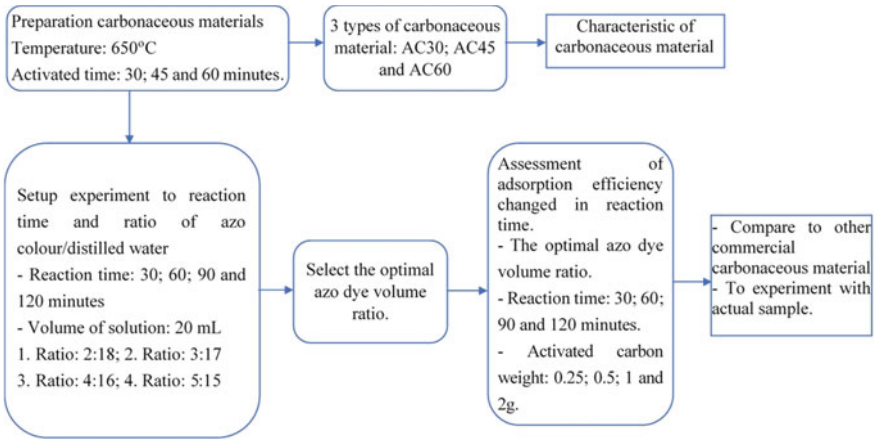

Fig. 2 Setup the experimental conditions

pollution concentration and test with the optimization conditions. Evaluate the azo dye adsorption capacity of the material.

2.4 Statistical Analysis

Statistical significance was analyzed through the use of Origin 2019 and GraphPad software.

3 Results and Discussion

3.1 Characteristics of Activated Carbon Material

In this study, the material characteristics of the three activated carbon samples is shown in Table 2 and Figs. 3, 4, 5. The C content of all material samples is relatively high, in which the AC60 sample has the highest C surface content of 80.18%,

Table 2 The physical and chemical properties of three activated carbon material

Code sample	Activated temperature and time (°C)	Size of particles (μm)	BET surface area (m^2/g)	Surface elements			
				C (%)	O (%)	Si (%)	P (%)
AC30	650 °C/30 min	17.4889	25.2337	72.45	23.91	3.42	0.22
AC45	650 °C/45 min	23.3497	44.6221	74.20	22.52	3.09	0.19
AC60	650 °C/60 min	18.7503	108.9202	80.18	17.82	1.04	0.96

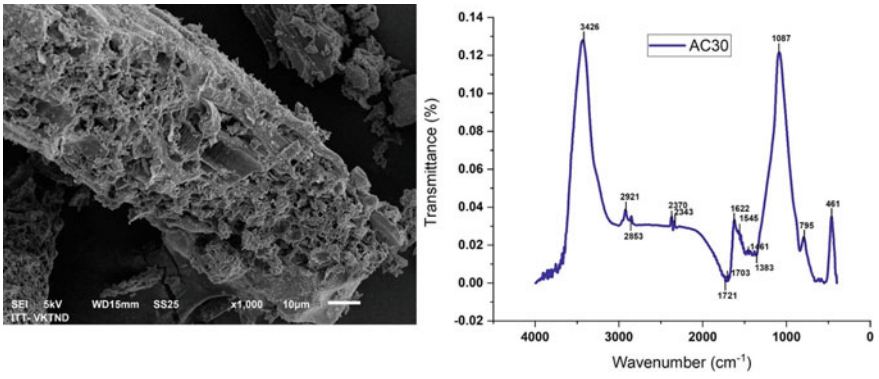

Fig. 3 SEM images and FTIR spectra of AC30 material activated from bamboo leaves at 650 °C/30 min

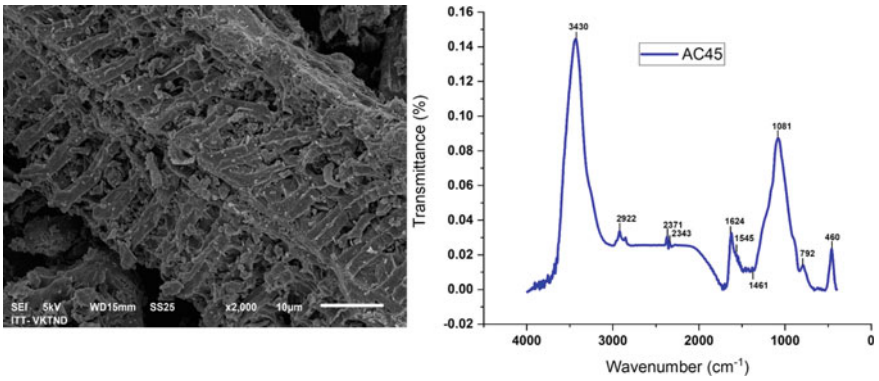

Fig. 4 SEM images and FTIR spectra of AC45 material activated from bamboo leaves at 650 °C/45 min

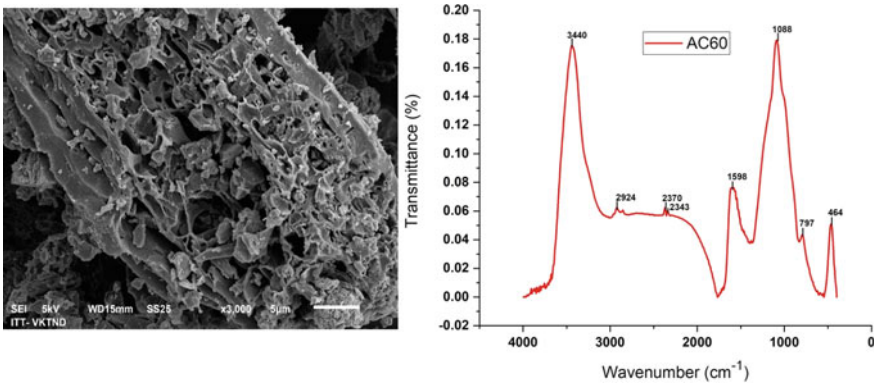

Fig. 5 SEM images and FTIR spectra of AC60 material activated from bamboo leaves at 650 °C/60 min

followed by the AC45 and AC30 samples with 74.2 and 72.45% C, respectively. The C content plays an important role in creating small porous with large volume to increase the adsorption surface area for activated carbon material [41]. The elemental percentage composition in Table 2 also showed that three activated carbon material AC30, AC45, AC60 contained other elements such as: O were found to be with the lowest percentage from 17.82% (AC60 sample) to the highest of 23.91% (AC30 sample); element Si and P have lower percentages from 1.04 to 3.42% (Si) and 0.19 to 0.22% (P) respectively. All three samples have a relatively high BET surface area, special the AC60 sample has a BET value of up to 108.9202 m^2/g. With the average particle size ranging from 17.4889 μm (AC30) to 23.3497 μm (AC45) showed that the material is smooth particles and quite similar together, the particle size of the AC60 sample is more uniform [11]. The small particle size combined with the large

specific surface area will affect the adsorption and removal of the material's pollutants [19].

The surface morphology of three activated carbon was studied by SEM technique, and the results showed that the surface structure of the material samples somewhat different and parallels with BET measurement results. It can be seen that the material structure in three samples is heterogeneous porous. However, the AC60 sample was observed to be most homogeneous, and it had a larger porous structure, more porous and contained the most significant surface area among the three samples. There was a significant positive between the current study and previous results [12, 47, 49]. It can be clearly observed the microporous with a larger pore diameter will lead to reduction in surface area and an increase in porous structure [49], improve adsorption capacity for azo dye. Compared to the raw materials, the activated carbon material has a more strongly adsorption capacity through a rough surface with tiny channels and porous structure [12, 29]. Similarly, the FTIR measurement results in Figs. 3, 4 and 5 indicated that the activation time and temperature influenced the material characteristics and the formation of functional groups on the material surface [37, 40]. All three samples have adsorption spectrum from 400 to 4000 cm^{-1} with characteristic bonds such as C=O, O–H, C–C, C=C… Apart from some functional groups, characteristic bonds lost or newly formed depending on activation time from 30, 45 or 60 min, the intensity of the characteristic peaks of samples also changed. The FTIR spectrum obtained for all three samples displayed the following bonds such as O–H stretch groups (peak at 3440–3426 cm^{-1}) and C–N amine (peak at 1088–1881 cm^{-1}). The absorption peaks at 2924–2853 cm^{-1} and 1680–1620 cm^{-1} are attributed to the C–H stretch and C=C groups, respectively. The FTIR results also show peaks varied from 797 to 792 cm^{-1} for aromatics out of plane bend C–H. The C–halogen (Br/I) bonds appeared with a peak at 464–460 cm^{-1} or N=O bonds representing spectral range from 1550 to 1300 cm^{-1}. The C–O–C group was also presented with a peak at 1120 to 850 cm^{-1}. However, the C=O bond as ketone group (peak at 1725–1705 cm^{-1}) was only observed in the AC30 sample. The AC30 sample had lower purity than the two remain samples and containing many functional groups on the material surface, leading to dispersed and reduced treatment efficiency. Moreover, according to Pavia et al. [40] FTIR spectrum of three activated carbon samples in this study is different from many other samples because of containing bonds around 2350 cm^{-1} representing the CO_2 group. Our results corroborate with published studies [11, 41]. According to Chan et al. [11], the C=C, O–H, C–O–C groups play an important role in characteristic dye adsorption were represented for peaks at 1650, 3450, 1120 cm^{-1}, respectively. Furthermore, the study of Pongener et al. [41] also showed that the surface structure and pore size of the material are highly dependent on additional precursors during activation processing, in which the presence of H^+ ions will increase particle distribution, leading to an increase in porous structure and the surface area of the material.

3.2 Removal Efficiency of Azo Dye by Three Activated Carbon Materials

3.2.1 Brilliant Green

Effect of Initial Azo Dye Volume and Reaction Time on Adsorption Capacity

To study the effect of initial azo dye volume and reaction time on adsorption capacity, 0.01 g Brilliant green dye was diluted in 100 mL with distilled water and analyzed the initial concentration by HPLC instrument at wavelength 360 nm. The analyzed result recorded the actual concentrations of Brilliant green in the stock solution was 85.97 mg/L. The amount of Brilliant green dye adsorbed at the equilibrium by the three materials AC30, AC45 and AC60 is shown in Table 3. All samples recorded pH value changed from 9 to 9.4. When the dye volume added into the samples changed from 2, 3, 4 to 5 mL, and the reaction time increased from 30 to 120 min, the azo dye removal efficiency also increased accordingly. At the reaction time of 30 min, the adsorption capacity (q_e) of the AC30 sample was the lowest with 95.40 mg/g and the highest value was observed for the AC60 sample with 293.71 mg/g when added 2 and 5 mL Brilliant green, respectively. The adsorption capacity of Brilliant green azo dye reached a maximum value of 343.88 mg/g for the AC60 sample at reaction time 120 m with every volume ratio (2, 3, 4 and 5 mL). Similarly, the AC45 sample also recorded the maximum adsorption capacity (343.88 mg/L) when adding 2 and 3 mL Brilliant green at the reaction time 120 min, but when the volume increases to 4 and 5 mL, the efficiency decreased to 327.8 and 320.92 mg/g, respectively. With different reaction times (from 60 to 90 min), the complete removal capacity of azo

Table 3 The adsorption capacity q_e (mg/g) of Brilliant green dye by 3 activated carbon materials

Type of materials	$V_{azo\ dye}$ (mL)	$m_{activated\ carbon}$ (g)	q_e (mg/g)			
			Reaction time (minute)			
			30 min	60 min	90 min	120 min
AC30	2	0.5	237.95	265.28	297.00	343.88
AC45	2	0.5	255.57	299.86	333.93	343.88
AC60	2	0.5	293.71	337.37	343.88	343.88
AC30	3	0.5	186.11	201.89	257.95	326.27
AC45	3	0.5	201.92	286.47	320.92	343.88
AC60	3	0.5	285.96	328.04	343.88	343.88
AC30	4	0.5	166.40	197.95	237.96	309.43
AC45	4	0.5	186.11	265.00	299.85	327.80
AC60	4	0.5	265.00	324.74	343.88	343.88
AC30	5	0.5	95.40	124.00	151.16	297.78
AC45	5	0.5	146.67	229.78	265.00	320.92
AC60	5	0.5	257.62	297.43	320.91	337.37

dye was found for the AC60 sample with 2 mL Brilliant green adding and reaction time 60 min. Unlike the results for the AC60 sample, the two samples were AC30 và AC45 had reported the lowest adsorption capacity of 124 and 333.93 mg/g when added 5 mL (AC30 sample, reaction time 60 min) and 2 mL azo dye (AC45 sample, reaction time 90 min), respectively. It can be clearly seen that the volume ratio of Brilliant green: distilled water (mL) of 2:18 recorded the maximum removal capacity because the adsorption capacity has obtained 237.95 mg/g after only 30 min reaction time and reached the maximum capacity of 343.88 mg/g at all remaining reaction times (60, 90 and 120 min).

Effect of Adsorbent Material Mass and Reaction Time on Adsorption Capacity

Based on the result obtained in Sect. 3.2.1, the volume ratio of brilliant green: distilled water (mL) of 2:18 selected for further experiments. Figure 6 showed that when increasing the amount of the adsorbent from 0.25; 0.5; 1 to 2 g, the adsorption efficiency (H%) for azo dye increased as well. Only the adsorption efficiency obtained from the AC60 sample after 30 min reached 97.4% (with 0.25 g adsorbent material) and up to 100% as soon as this amount was increased to 0.5 g, while the remaining

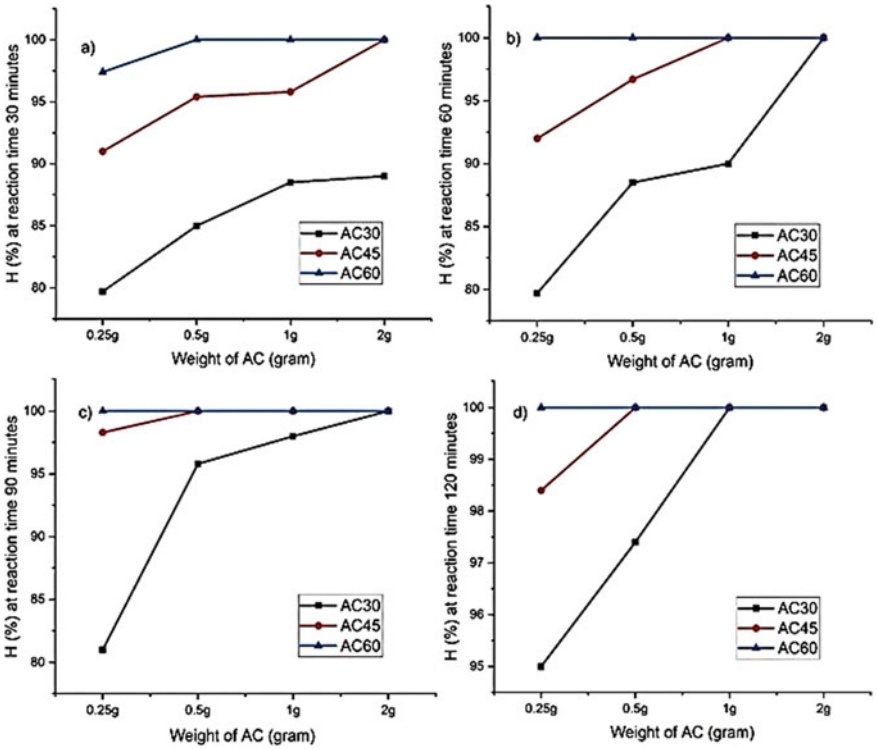

Fig. 6 The adsorption efficiency (H%) of Brilliant green dye from aqueous solution when adsorbed with 0.25, 0.5, 1 and 2 g activated carbon material

two samples AC30 and AC45 removed only 79.7 and 100% Brilliant green when added 0.25 and 2 g respectively, and even the AC30 sample achieved only 89% with the amount of adsorbent material 2 g. When the reaction time varied from 60 to 120 min and with just 0.25 g of adsorption material, the AC60 is the only sample that recorded the maximum adsorption efficiency of 100%, and the two remaining samples obtained the values of 79.7; 81; 95% (AC30 sample) and 94; 98.3; 98.4% (AC45 sample) with the reaction time 60, 90 and 120, respectively. Unlike the results for 0.25 g absorbent material, the adsorption efficiency with 0.5, 1 and 2 g for three activated carbon samples is very different together. The azo dye removal efficiency of the AC45 sample was recorded value up to 100% just as 0.5 g at reaction time 60 min, whereas the AC30 sample reached only the value of 100% with 2 g at 60 min. With the reaction time of 120 min and the amount of absorbent material 2 g, all three materials observed to able to remove 100% of Brilliant green azo dye from water. Of these, the AC60 is the only sample that obtained the completely removal efficiency of 100% with 0.5 g absorbent material at the reaction time of 30 min.

3.2.2 Alizarin Red S

Effect of Initial Azo Dye Volume and Reaction Time on Adsorption Capacity

Similarly, the initial concentration of Alizarin Red S solution analyzed by HPLC at wavelength 260 nm was 98.6 mg/L. The pH value in every sample measured around 8.9 to 9.2. The Alizarin Red S azo dye removal capacity at different time periods was lower than Brilliant green although the maximum adsorption capacity value of the material is higher. The results in Table 4 showed that when the 2 mL of Alizarin Red S azo dye was added, the removal capacity of all three materials recorded the rather high value and reached the maximum adsorption capacity of 394.4 mg/g at 60, 90 and 120 min by AC360, AC45 and AC30 samples, respectively. However, when the azo dye volume was altered from 3, 4 to 5 mL, the removal capacity of all samples was significantly reduced. As seen for the AC60 sample, the removal capacity was the highest for 5 mL of 118.32 mg/g and the lowest for 3 mL of 78.88 mg/g, respectively; with 4 mL Alizarin Red S, the adsorption capacity was only reached 177.48 mg/g (at 30 min) to 216.92 mg/g (at 120 min). The remaining samples showed the removal capacity for Alizarin Red S really reduced. As with 30 min reaction time and the volume of Alizarin Red S ranged from 3, 4 to 5 mL, the AC30 and AC45 samples only reached 134.10; 70.99; 7.89 mg/g and 248.47; 118.32; 27.61 mg/g, respectively. When the reaction time increased to 120 min, the adsorption capacity increased as well but only reached 39.44 to 256.36 mg/g (AC30 sample) and 78.88 to 343.13 mg/g (AC45 sample), respectively. It is also clear that the azo dye volume needed to reach the highest adsorption capacity value was 2 mL Alizarin Red S. This volume ratio would be selected for further experiments.

Table 4 The adsorption capacity q_e (mg/g) of Alizarin red S dye by activated carbon material

Type of materials	$V_{azo\ dye}$ (mL)	$m_{activated\ carbon}$ (g)	q_e (mg/g)			
			Reaction time (minute)			
			30 min	60 min	90 min	120 min
AC30	2	0.5	262.96	302.80	367.28	394.40
AC45	2	0.5	311.00	377.40	394.40	394.40
AC60	2	0.5	389.55	394.40	394.40	394.40
AC30	3	0.5	134.10	181.42	248.47	256.36
AC45	3	0.5	248.47	339.18	343.13	343.13
AC60	3	0.5	291.86	339.18	351.02	354.96
AC30	4	0.5	70.99	78.88	106.49	118.32
AC45	4	0.5	118.32	138.04	145.93	157.76
AC60	4	0.5	177.48	185.37	197.20	216.92
AC30	5	0.5	7.89	19.72	39.44	39.44
AC45	5	0.5	27.61	39.44	39.44	78.88
AC60	5	0.5	78.88	78.88	98.60	118.32

Effect of Adsorbent Material Mass and Reaction Time on Adsorption Capacity

As with Brilliant green azo dye, the adsorption efficiency for Alizarin Red S increases when increasing the reaction time and adsorbent material. The result of the experiments in Fig. 7 showed that the effect of adsorbent material mass and reaction time on adsorption capacity was lower than Brilliant green azo dye. With the volume ratio of Alizarin Red S: distilled water is 2:18 and the amount of the activated carbon material varied from 0.25 to 2 g, the adsorption efficiency of the material samples increases accordingly. When 0.25 g adsorption material was added, the AC30 sample recorded the lowest efficiency of 2%, and the highest was the AC60 with 30% at reaction time of 30 and 120 min, respectively. At a reaction time 30 min and the amount of adsorbent material increases from 0.5 to 1 g, the AC60 sample recorded the highest azo dye removal efficiency 74% (with 1 g adsorbent material) and the lowest value of 45% (with 0.5 g adsorbent material), the remaining two samples have the lowest removal capacity of 18% (with 0.5 g AC30 material sample) and the highest is 63% (with 1 g AC45 material sample). Between 60 and 120 m, the removal rate increases steadily but quite slowly. The maximum efficiency reached to 30, 45, 55% and 65, 87, 90%, respectively when added 0.5 and 1 g adsorbent material AC30, AC45, AC60 at reaction time 120 min. However, when the adsorbent material increased up to 2 g, the adsorption efficiency of all three samples was achieved to the maximum value, completely removing 100% of azo dye at all four reaction times (from 30 to 120 min) except for the AC30 sample with only 98% at 30 min.

In this way, it can be seen that the removal efficiency for Brilliant green azo dye better than Alizarin red S. The results also showed that the optimal experimental condition for the azo dye volume ratio/distilled water of 2/18 (mL). The adsorption

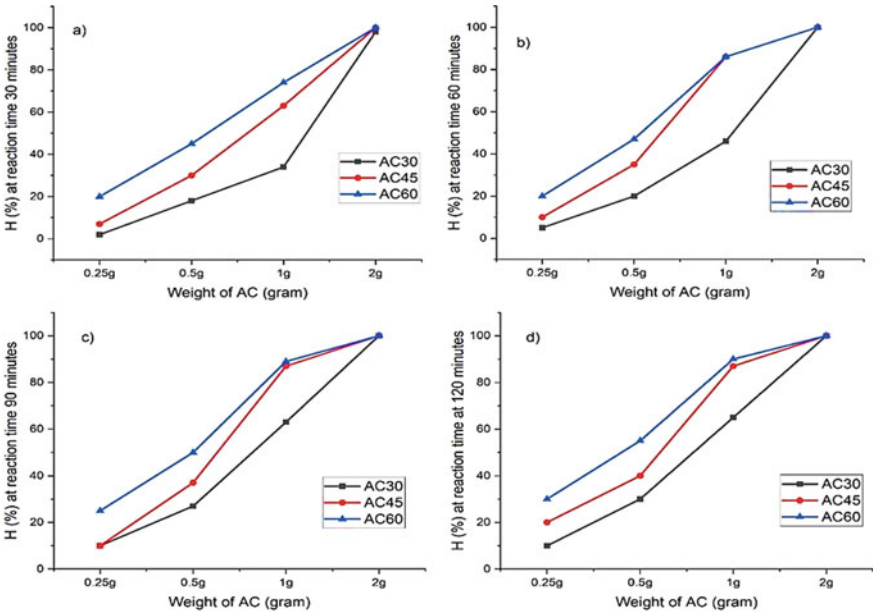

Fig. 7 The adsorption efficiency (H%) of Alizarin Red S dye from aqueous solution when adsorbed with 0.25, 0.5, 1 and 2 g activated carbon material

efficiency increases linearly with the reaction time and the weight of the adsorbent material added. However, only the AC60 sample achieved the maximum efficiency of 100% with both two azo dyes at reaction time of 30 min when the 0.5 and 2 g absorbent material for Brilliant green and Alizarin red S were added to the solution, respectively. This experimental result matches with the material structure characteristic of the AC60 sample. Further tests carried out with the optimization experimental conditions confirmed with our initial findings.

Azo dye is a group of dyes represented by a functional group (–N=N–) and the removal of this group from textile dyeing wastewater is a very difficult task. Our results have a number of similarities with some previously published findings [11, 19, 22]. Kaya and Uzun [22] used the carbonaceous material consist of pine cone, walnut shell, and hazelnut shell that were synthesized at different temperature range from 400 to 700 °C for 60 min to remove Alizarin yellow GG azo dye from the aqueous solution. When 20 ppm Alizarin red S and 8 g/L adsorbent material added to the solution at pH 3, the activated carbon sample from the walnut shells with the largest material surface area of 259.74 m^2/g had recorded the maximum adsorption efficiency of 82%. Similarly, Chan et al. [11] indicated that the dyes with a smaller molecular size would more easily adsorb onto the carbonaceous material and the dye removal efficiency depends on the appearance of the adsorbent functional groups on the material surface. In his investigation into the removal percentage of two azo dyes, Acid Yellow 117 (AY117) and Acid Blue25 (AB25), Chan et al. (2006) shows that the AB25 dye

with smaller molecular size readily adsorbed onto the carbonaceous material than AY117 dye with larger size showed minimal adsorption. The results of material characterization also showed that the FTIR spectrum of activated carbon samples appeared dye adsorption functional groups such as C=C, O–H, C–O–C represented for peaks at 1650, 3450, 1120 cm^{-1}, respectively. The surface structure and pore size of the material play an important role in the adsorption and removal of the dye. Hameed and El-Khaiary [19] demonstrated that activated carbon samples have a high BET specific surface area, large pore size, the adsorption capacity with pollutants also increases accordingly. In his analysis, Hameed and El-Khaiary [19] point out the adsorption speed of malachite green (MG) is controlled by the pore structure of the material and the maximum removal efficiency reaches 263.58 mg/g with 300 mg/L of the absorbent material (synthesized from bamboo leaves at 850 °C/2 h) at reaction time 230 min.

For batch adsorption, the reaction time and the concentration of the initial adsorbent are important factors in determining the adsorption capacity value (q_e). When the adsorption time increases, the adsorption efficiency also increases accordingly [27, 43]. These fit well with the statement of Saeed et al. [27], Laskar and Kumar [43] and also confirm our findings. When the amount of the initial adsorption time increases from 0.25 to 2 g, the AC30 sample also recorded maximum efficiency, completely removing 100% azo dye after only 30 min of reaction time (BG dye) and 60 min (AR dye), the AC60 and AC45 samples also reached this value with 0.25 and 2 g, respectively, after only 30 min of reaction time. As mentioned by Laskar and Kumar [27], the removal efficiency of Brilliant green dye by the activated carbon material synthesized from Bambusa Tulda bamboo up to 98% only with 10 g/L of the absorbent material at the reaction time of 60 min. The Brilliant green removal percentage has been reduced to 83.74% when the amount of adsorbent material is reduced to 50 mg/L accordingly. Furthermore, the different activated carbon material types exhibit different adsorption efficiency. Laskar and Kumar [27] also demonstrated that the Brilliant green azo dye removal rate reduced from 136 mg/L (absorbed with the material modified with sodium carbonate) to 72 mg/L (the material modified with hydrochloric acid). Our findings appear to be well supported by Saeed et al. [43]. He underlines that the sorption equilibrium of crystal violet dye (CV) by the activated carbon from grapefruit peel reached rapidly to 96% after 60 min. The maximum adsorption capacity increases gradually from 60.42 to 254.16 mg/g as the adsorbent material content increases from 25 mg/L to 1 g/L, and the amount of CV added to the solution increases correspondingly from 10 to 600 mg/L.

As with changes in the reaction time, the solid/solution ratio also is an important factor in calculating the capacity of adsorption of azo dye in a batch treatment system. This confirms previous findings in the literature of Ahmad et al. [3], Al-Da'amy and Al-Shemary [4], Xu et al. [50]. According to Xu et al. [50], the methyl violet (MV) adsorption of carbonaceous materials synthesized from 156 g rice husks achieved the highest efficiency with the ratio of MV/distilled water of 1/18.2 (mmol/L). Xu et al. [50] demonstrated that the adsorption of methyl violet by the carbonaceous material is related to electrostatic attraction, surface precipitation, the specific interactions between dyes and hydroxyl, and carboxylate groups on the surface material.

Ahmad et al. [3] also showed that methylene blue (MB) was completely removed by the carbonaceous material prepared from rice husk, cow dung and sludge. When the 0.5, 1.0, 1.5, 3.0, and 6.0 g of the absorbent material added to 100 mL solution containing 100 mg/L of MB, the maximum efficiency achieved with three materials: rice husk, cow dung and sludge of 97.0–99.0, 71.0–99.0 and 73.0–98.9%, respectively. Similarly, the removal capacity of Alizarin yellow R and Alizarin red S by the absorbent material from snail shells recorded the maximum value after 30 min with the amount of initial absorbent material of 0.01 g and the initial dye concentration of 30 mg/L [4].

The azo dye removal efficiency of the carbonaceous materials also depends on the adsorption mechanism. Mukarram et al. 2020 reported on adsorption for methyl orange (MO) and Eriochrome Black-T (EBT)—and cationic dyes—methylene blue (MB) and crystal violet (CV) and concluded that the maximum adsorption capacity reached up to 206.61, 309.59, 163.132 and 934.57 mg/g when supplemented with 200 mg/L MB, EBT, MO, and CV, respectively. According to Mukaram et al. (2020), the adsorption mechanism is mainly based on the Redlich-Peterson isotherm model ($R^2 > 0.95$). However, Saniyaa et al. [44] used the isothermal model Langmuir and Freundlich to demonstrate the optimal experimental parameters to remove crystal violet (CV) dye from the textile dyeing wastewater. Saniyaa et al. [44] showed that the maximum removal efficiency of 70% had obtained after reaction time of 60 min with 100 mg/L of the carbonaceous material synthesized from curry bark and the initial dye concentration of 50 mg/L. He comes to reach the conclusion that the Langmuir isothermal model is most suitable in his work. Three kinetic models,namely, pseudo-first-order, pseudo-second-order, and intra-particle diffusion were used to analyze the adsorption mechanism. The kinetic results showed that the pseudo-second-order equation was the best model. The isotherm analysis indicated that the equilibrium data were well fitted to the Langmuir isotherm model, showing a monolayer adsorption manner of the dyes onto a homogeneous surface of the modified nanoparticles. According to the experimental results, about 97.8% of alizarin yellow and 78.7% of alizarin red were removed from aqueous solutions under optimal conditions [18].

3.3 Effect to Actual Textile Dyeing Wastewater Sample and Compare the Material Quality with Some Commercial Activated Carbon Samples

3.3.1 Effect on Initial Textile Dyeing Wastewater Sample

To evaluate the application capacity of the activated carbon sample, an initial wastewater sample (NT0) was collected from a factory of jeans in Long Bien district, Hanoi city, Vietnam and moved to the laboratory. Initial wastewater samples were measured for pH and analyzed the concentration of azo dye in the solution. The experimental

Table 5 The analysis results with initial wastewater samples at different time periods

No	Items	NT0	NT30	NT60	NT90	NT120
1	pH	8.4	9.2	9.0	9.0	9.0
2	BG	66.29 mg/L	0	0	0	0
3	AR	0 mg/L	0	0	0	0

optimization conditions selected in Sect. 3.2 will be applied with the initial wastewater samples at different time range from 30 min (NT30), 60 min (NT60), 90 min (NT90) to 120 min (NT120) and 0.5 g the absorbent material AC60. The analysis results are shown in Table 5 and Fig. 8.

The analysis result showed that the Brilliant green dye was completely removed from the solution after only 30 min of reaction time, HPLC spectrum did not record the appearance of a peak at the retention time of 16.232 min for samples absorbed at 60, 90, and 120 min, respectively. As reported above, this is a factory of jeans so the analysis result was no peak of AR dye. Both samples before and after absorbed by AC60 material did not record a peak at the retention time of 3.364 min.

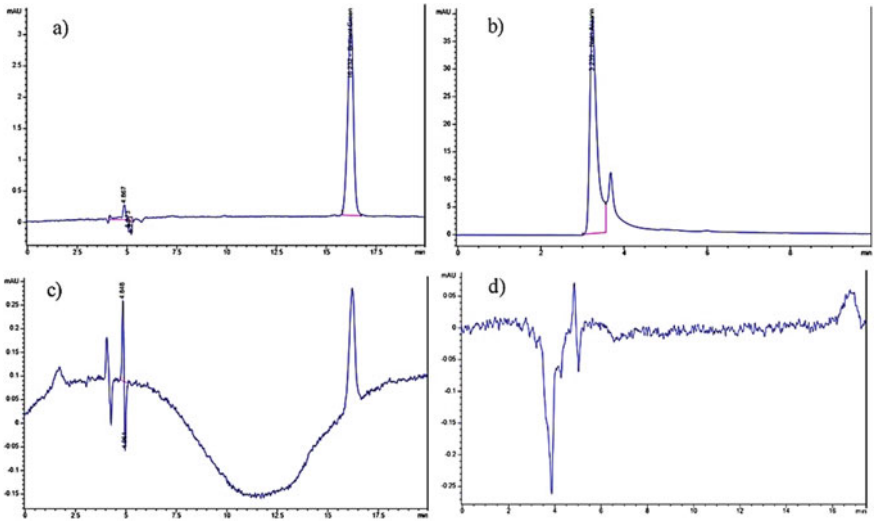

Fig. 8 HPLC (High Performance Liquid Chromatography) spectrum of standard and initial sample: **a** standard spectrum with Brilliant green dye; **b** standard spectrum with Alizarin red S dye; **c** spectrum of wastewater sample before adsorption to AC60 material and **d** spectrum of wastewater sample after adsorption with 0.5 g of AC60 material at 30 min

3.3.2 Compare the Material Quality with Some Commercial Activated Carbon Samples

To evaluate the quality of material in the current study, the AC60 sample with the best treatment efficiency would be compared with four other commercial material types available in the Vietnam market consists of AC-R (Russian activated carbon), AC-C (Chinese activated carbon), AC-F (French activated carbon) and AC-Tra Bac (Vietnamese activated carbon from coconut shell). The adsorption efficiency of four materials for Brilliant green and Alizarin red was performed according to the experimental optimization condition in Sect. 3.2. The results are shown in Table 6 and Fig. 9 as follows:

From the comparison results, it can be seen that the adsorption efficiency for BG and AR dyes by four commercial activated carbon materials share a number of similarities with our findings with AC60 material in the same optimization experimental conditions. The analysis results also showed that the azo dye removal efficiency of the AC60 material synthesized from bamboo leaves is higher than some

Table 6 The adsorption capacity (q_e) for BG and AR by four commercial activated carbon material compared to AC60 material

No	Type of materials	$V_{azo\ dye}$ (mL)	$m_{activated\ carbon}$ (g)	pH	q_e (mg/g)	
					Brilliant green (at 30 min)	Alizarin red S (at 60 min)
1	AC-C	2	0.5	4	311.20	325.60
2	AC-R	2	0.5	4	343.88	394.40
3	AC-F	2	0.5	3	343.88	394.40
4	AC-Tra Bac	2	0.5	5	343.88	394.40
5	AC60	2	0.5	9.1	343.88	394.40

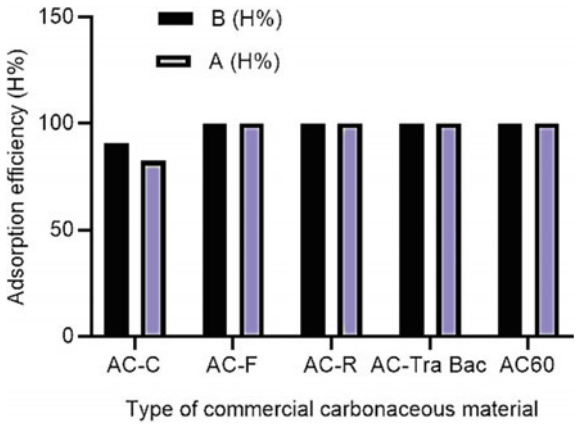

Fig. 9 The adsorption efficiency of four commercial activated carbon materials compared to AC60 material

previously prepared material (AC-C material reached only 90.5 and 82.55% for Brilliant green and Alizarin red S, respectively). As anticipated, our experiments prove that the removal efficiency for azo dye depends on experimental conditions such as reaction time, pyrolysis temperature, pH … and the activated carbon material quality. According to Mahdi et al. [33], the pyrolysis time will determine the material recovery productivity, affect the structure of cellulose and hemicellulose and release of organic molecules. The formation of functional groups (C=O, C–O–C and C–O) will determine the azo dye removal efficiency and therefore improve the material's surface characteristics (surface area and pore volume). The removal efficiency of pollutants depends on the functional groups on the carbonaceous material surface such as hydroxyl, carboxyl and –OH, C–H, C=O… These functional groups will have a strong influence on ion adsorption capacity [21].

The material synthesis environment and the experimental conditions were greatly affected the maximum adsorption capacity [31]. The pH value in the experimental medium is in contradiction with previous results reported in the documents about its effect on pollutant removal efficiency. Gautam et al. [17] suggested that the acidic environment enhances sorption processing. The maximum adsorption capacity of material from different waste plant biomass (consists of orange peel, flower waste, and environmentally harmful Alligator weed) recorded at 50 °C were 42.58 and 68.78 mg/g for Alizarin red S and Tartrazine at pH 4.0 and 2.0, respectively. Our findings do not support the data provided by Gautam et al. [17] in this area. In fact, in contrast with what was previously data, we found that the pH value in the current study change around pH 9 and our findings appear to be well supported by Kyi's report [26]. Kyi et al. [26] carried out the sorption experiment to remove crystal violet from wastewater with adsorbent material from palm kernel shell. The results showed that the removal percentage and adsorption capacity of the material increases linearly with the pH value in the solution. These differences can be explained partly by the negative charges on the surface of carbonaceous materials at high pH enhance electrostatic attraction between azo dye molecules and carbonaceous material.

According to Sumalinog et al. [46], the removal efficiency of acetaminophen (APAP) and methylene blue (MB) dyes from aqueous solution were affected by changes in pH value. The removal efficiency of MB dye (99.9%) recorded the higher value than APAP dye (only reached 63.7%). While the adsorption capacity of APAP was strongly reduced by pH change, MB adsorption found to be unaffected when pH increases from 2 to 12. Chahinez et al. [10] argued that the concentration of crystal violet dye (CV) adsorbed into the carbonaceous material derived from date palm petioles increased by 46% (from 18.8 to 27.4 mg/g, an average of 24.36 mg/g) when the solution pH increased from 2.0 to 12. Zazycki et al. [53] showed that the treatment capacity of methyl violet dye (MV) is higher in alkaline medium. Surprisingly, for maximum adsorption capacity of the carbonaceous material from chitin was found up to 1120.8 mg/g with pH changed from 6.8 to 9. Consequently, it can be seen that the characteristics and source of the raw materials have a significant influence on the carbonaceous material structure, determining the treatment efficiency as well as the removal capacity for pollutants. Our study provides additional further evidence for insight into the material synthesized conditions such as reaction time, pyrolysis

temperature as well as the volume ratio of dye/water added to the solution for azo dye removal in textile wastewater.

4 Conclusion

This study investigated the removal capacity of two azo dyes, including Brilliant green and Alizarin red S, by three types of activated carbon prepared from bamboo leaves, AC30 (650 °C/30 min); AC45 (650 °C/45 min) and AC60 (650 °C/60 min). The obtained materials have relatively high C content (AC30 is 72.45%; AC45 is 74.30% and AC60 is 80.18%) and contain the characteristic bonds represented by the azo dye removal capacity C=C; C–O–C, –OH. The results also showed that the AC60 material sample has the best adsorption efficiency reached 100% at the reaction time of 30 min with the volume ratio of azo dye/distilled water of 2/18 (mL), pH 9, the absorbent amount of 0.5 and 2 g for Brilliant green and Alizarin red S, respectively.

The analysis results of initial textile dyeing wastewater sample with four commercial activated carbon samples showed that the material source and the experimental conditions in the current study are suitable for the azo dye removal application from industrial textile dyeing wastewaters. Despite this, we believe our work could be a starting point for further studies on the adsorption mechanism, the wastewater properties, desorption/adsorption… need to be thoroughly conducted before application in factories or actual conditions.

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