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Adsorption of Remazol Blue and Indigosol Yellow Mixed Dyes Using Bidara Arab Leaves *(Ziziphus spina-christi)*

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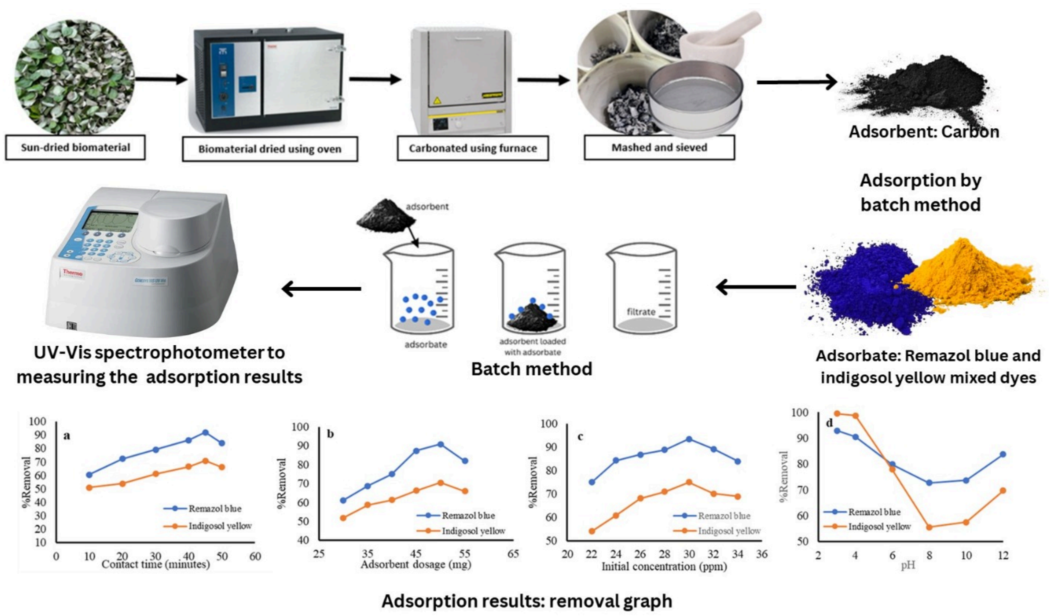
GRAPHICAL ABSTRACT



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ABSTRACT

In this study, the adsorption of Remazol Blue and Indigosol Yellow using Bidara Arab leaves *(Ziziphus spina-christi)* as adsorbents was investigated through batch adsorption. The experimental parameters included contact time, adsorbent mass, initial concentrations, and pH. UV-Vis spectrophotometer was employed to measure the adsorption results at the maximum wavelength of each dye. The research was conducted in triplicate, and a t-test, "Paired Two Sample for Means", with a standard deviation <2% was employed for statistical analysis. Optimal adsorption conditions were achieved at a contact time of 45 minutes, an adsorbent mass of 45 mg, an initial concentration of 30 ppm, and a pH of 3. The results indicated that Bidara Arab leaves effectively reduced the concentration of the mixed dyes with %decoloration up to 99,79% on Remazol Blue and 99,50% on Indigosol Yellow.

1. INTRODUCTIONN

Formerly known as one of the largest prostitution sites in Southeast Asia, Dolly localization in Putat Jaya, Sawahan, Surabaya, has transformed into a tourism village. The establishment of Dolly Tourism Village aimed to change its previous image. Among the home industries in this village is the Putat Jaya Batik House, which produces a variety of batik patterns and colors using synthetic dyes, including Remazol, Indigosol, and Naphthol. However, the textile industry, including batik production, is a significant contributor to dye waste generation [1], necessitating proper waste treatment, especially when dealing with hazardous substances. Untreated dye waste can cause severe harm to the ecosystem, environment, and living organisms.

In Putat Jaya Batik House's context, two dyes commonly used are Remazol Blue and Indigosol Yellow. Remazol Blue, a reactive dye, imparts a vibrant blue color to textiles, is resistant to fading, and is widely employed in dyeing and printing processes [2]. Despite its beneficial properties, Remazol Blue is toxic to aquatic ecosystems and poses carcinogenic risks to humans. On the other hand, Indigosol Yellow belongs to the Indigosol dye class, known for its stability and fade-resistant characteristics, resulting in soft or pastel colors. These dyes are used in dyeing and dabbing processes.

To address the issue of dye waste, adsorption has emerged as a cost-effective, straightforward, and efficient method for removing pollutants from water, particularly dye waste [3]. Various active agents have been explored to enhance adsorption capacity [4], and while activated carbon is commonly used in industries, its expense and difficulty in renewal, especially in powder form, pose challenges. The use of Bidara Arab leaves as a bio-adsorbent offers a promising alternative due to their affordability, accessibility, and renewability. Previous studies have also explored other bio-adsorbent materials, such as apple peels [5], olive seeds [6], palm kernel shells [7], peach and apricot seeds [8], corncobs [9], coffee grounds [10], tea dregs [11], coconut shells [12], and biopolymers like chitosan [13] and alginate [14]. The utilization of Bidara Arab leaves in this context holds potential for further development and application in addressing dye waste issues.

Utilizing bio-adsorbents presents a favorable alternative to reduce the excessive reliance on industrial activated carbon. Bidara Arab leaves *(Ziziphus spina-christi)* offer a promising solution as bio-adsorbents due to their cost-effectiveness, eco-friendliness, and renewability [15]. Previous studies have demonstrated successful adsorption applications of carbon derived from Bidara Arab leaves, including the removal of Mn(II) ions, with maximum adsorption capacity of manganese calculated from Langmuir isotherm was around 172 mg/g [16], Hg(II) ions with maximum adsorption removal up to 91.2% [17], triphenylmethane dye with maximum adsorption removal up to 95% [18], and malachite green dye with maximum adsorption capacity up to 329.49 mg/g [19]. In this research, carbon from Bidara Arab leaves was employed as a bio-adsorbent to capture mixed dyes of Remazol Blue and Indigosol Yellow, aiming to minimize or eliminate the dye concentration in wastewater. The study focused on crucial parameters such as contact time, adsorbent mass, initial concentration, and pH [20]. This research has the potential to be developed for wastewater treatment in batik production at the Putat Jaya Batik House and other textile industries that utilize Remazol Blue and Indigosol Yellow dyes, ensuring safer discharge of wastewater into the environment, thus benefiting human health and the ecosystem.

2. EXPERIMENTAL METHODS

2.1. Tools and Materials

The tools used in this research are watch glass, spatula, glass stirrer, analytical balance, volumetric flask of 100 mL; 250 mL; and 1000 mL, beaker volume of 50 mL; 100 mL; and 250 mL, measuring pipette 1 mL; 5 mL; and 10 mL, pro pipette, magnetic stirrer, Whatman filter paper, funnel, pH meter, crucible 250 mL, oven Thermo scientific, furnace Nabertherm, mortar, pestle, 25 µm sieve, UV-Vis spectrophotometer Genesys 10S, cuvette, FTIR Shimadzu, SEM Bruker, XRD PHILIPS-binary.

The materials used in this research are Bidara Arab leaves *(Ziziphus spina-christi)*, aqua demineralized (DM), Remazol Blue dye, Indigosol Yellow dye, NaOH (Merck KGaA), and 37% HCl (Smart-Lab).

2.2. Adsorbent Preparation

Bio-adsorbents from the Bidara Arab leaves *(Ziziphus spina-christi)* were firstly prepared by drying under sunlight. Afterwards, the dried leaves were washed with aqua DM and placed in a 250 mL crucible for further drying at 80°C for 24 hours. Once completely dried, the leaves were carbonized in a muffle furnace Nabertherm at a temperature of 700°C for 3 hours. The resulting Bidara Arab leaf carbon was then finely ground into a powder using a mortar and pestle and sieved through a 25 µm sieve, rendering it ready for use as a bio-adsorbent [20]. The prepared Bidara Arab leaves adsorbent underwent characterization using FTIR Spectrophotometer Shimadzu, Scanning Electron Microscope (SEM) Bruker, and X-ray Diffraction (XRD) PHILIPS-binary.

2.3. Maximum Wavelength Determination

The dye solution (Remazol Blue and Indigosol Yellow) 60 ppm was tested using a UV-Vis spectrophotometer Genesys 10S at a wavelength of 400-800 nm to measure the maximum absorbance. Aqua DM is used as a blank solution [21].

2.4. Determination of Optimum Adsorption Condition

The Bidara Arab leaf carbon adsorbent was accurately weighed using an analytical scale of 40 mg and then transferred into a 100 mL beaker glass. Subsequently, Remazol Blue and Indigosol Yellow solutions, each with a concentration of 60 ppm and 5 mL volume, were added to the beaker glass containing the Bidara Arab leaves carbon. The mixture of adsorbent and mixed dyes was stirred using a magnetic stirrer at 450 rpm for 10 minutes. The mixture was filtered using filter paper to separate the solution from the adsorbent, yielding the filtrate for further analysis. The absorbance of each dye in the mixture was measured using a UV-Vis spectrophotometer. Aqua DM served as a blank for measuring the absorbance of Remazol Blue and Indigosol Yellow. This treatment process was repeated three times to obtain triplicate data. Similarly, the treatment process was repeated with variations in contact time (10, 20, 30, 40, 45, and 50 minutes) [21], adsorbent dose (30, 35, 40, 45, 50, and 55 mg) [22], initial concentration (22, 24, 26, 28, 30, and 32 ppm) [22], and pH levels (3, 4, 6, 8, 10, and 12) [22]. Adsorption capacity and efficiency can be calculated using the formula:

Capacity:

$$Q = \left(\frac{C_0 - C_e}{m}\right) xV \tag{1}$$

Efficiency:

$$\%E = \left(\frac{C_0 - C_e}{C_0}\right) x100\% \tag{2}$$

Where:

Q : Adsorption capacity per molecular weight (mg/g)
C_0 : Initial concentration of the solution (mg/L)
C_e : Final concentration of the solution (mg/L)
V : Solution volume (mL)
m : Adsorbent mass (g)
$\%E$: Adsorption efficiency

3. RESULTS AND DISCUSSIONS

3.1. Characterization Results

The FTIR spectra of Bidara Arab leaf carbon displayed characteristic peaks at various wavenumbers, as shown in Figure 1(a). The peak at 3391 cm^{-1} indicated the presence of -OH stretching, while the absorption band at 1427 cm^{-1} suggested forming an oxygen functional group, particularly the C=O conjugate bond in the carboxylate group. Additionally, the peak observed at 874 cm^{-1} indicated the presence of C=C bending, and the peak at 712 cm^{-1} pointed to the presence of S=O stretching. A peak at a wavenumber around 1000 cm^{-1} indicates the presence of C-O [21].

Figure 1(b) presented the XRD diffractogram of Bidara Arab leaves carbon, revealing a distinctive peak at 2θ=29.4074, indicating activated carbon's presence. The presence of this peak also indicated the crystallinity of the activated carbon. The peak obtained is following previous research conducted by Abshirini et al. (2019). The carbon diffraction peaks are similar, with a slight shift in intensity when compared to pure graphite (JCPDS) Number 75-1621 [21].

The surface morphology of Bidara Arab leaf carbon was captured in Figure 1(c); according to research conducted by Abshirini et al. (2019), the SEM images revealed numerous bumps and cavities, indicative of a high specific surface area for the carbon [21]. Moreover, the presence of huge pores on the carbon surface made it suitable for adsorbing pollutants, such as the mixed dyes in this study [22].

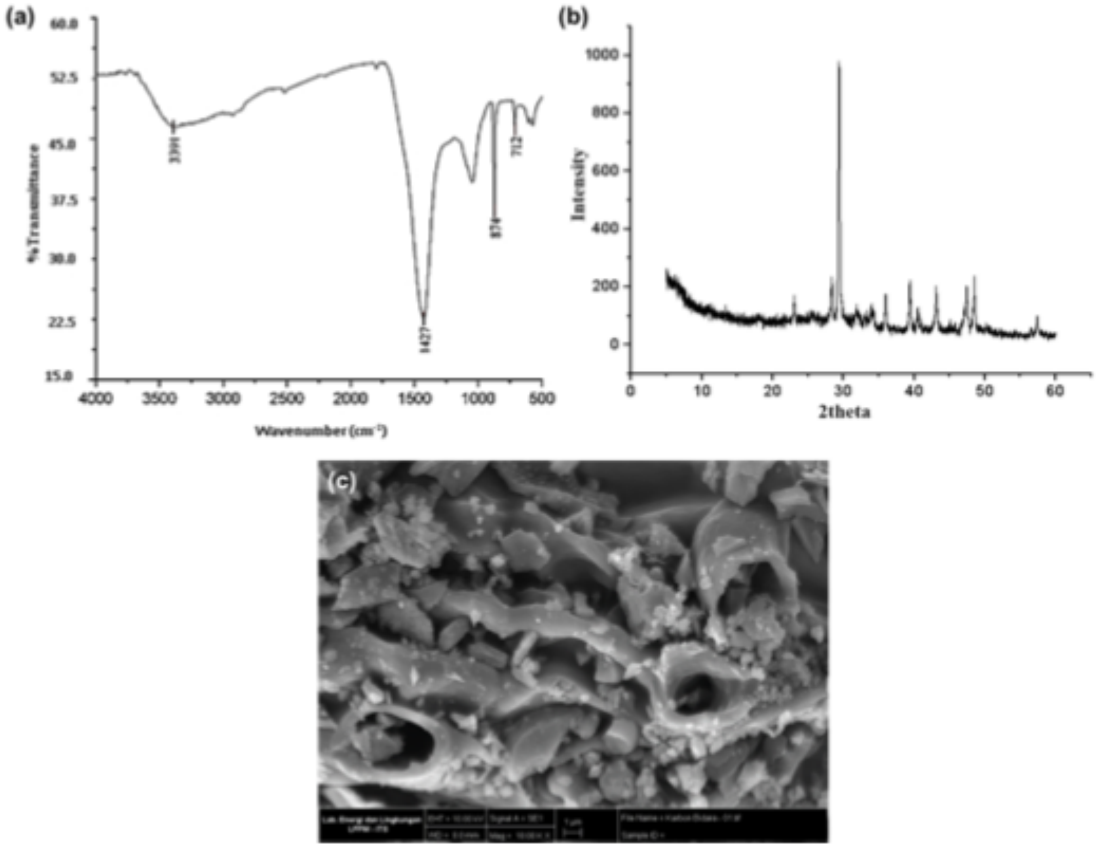

Figure 1. Characterization results of Bidara Arab leaf carbon using (a) FTIR, (b) XRD, and (c) SEM.

3.2. Maximum Wavelength Determination

Based on the curves shown in Figure 2(a) and (b), the maximum wavelength for Remazol Blue is 595 nm and for Indigosol Yellow is 465 nm. The maximum wavelength graph from Remazol Blue and Indigosol Yellow shows a different peak shape in Figure 2(a) Remazol Blue maximum wavelength curve shows a wide peak; the same peak shape was obtained in the study conducted by Sathishkumar et al. (2012) [23]. The Indigosol Yellow peak in Figure 2(b) shows a different shape in the form of two sharp peaks; a similar peak shape was obtained in a study conducted by Ni'mah et al. (2020) [24].

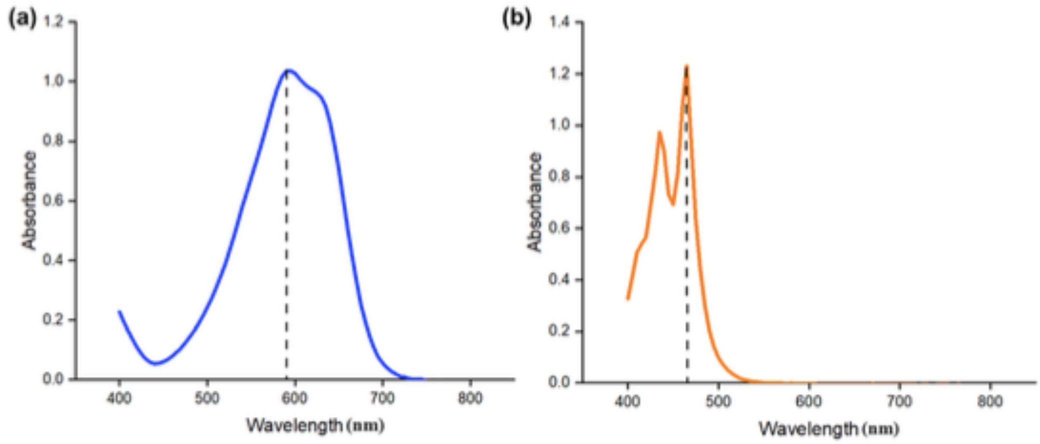

Figure 2. Maximum wavelength curve (a) Remazol Blue 60 ppm and (b) Indigosol Yellow 60 ppm.

Remazol Blue and Indigosol Yellow 60 ppm, each taken as much as 10 mL, then mixed. Figure 3 shows curves with peaks from the dye mixture. The curve's peaks followed the maximum wavelength of each dye, which is 595 nm for Remazol Blue and 465 nm for Indigosol Yellow.

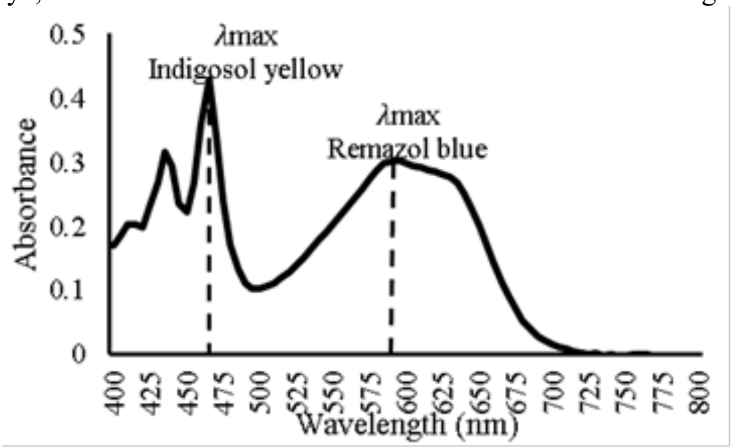

Figure 3. Maximum wavelength curve of mixed dyes.

3.3. Calibration Curve

As shown in Figure 4, the correlation coefficient (R2) is the degree of correlation between the absorbance value and the concentration value. The calibration curve obtained from the Remazol Blue solution has a linear regression of y = 0.0115x + 0.0208 with a correlation coefficient (R^2) of 0.9981. In Indigosol, Yellow obtained linear regression y = 0.0138x + 0.034 with a correlation coefficient (R^2) of 0.9958. A correlation coefficient value close to one (1) indicates that the calibration curve has promising results with a small error rate [25].

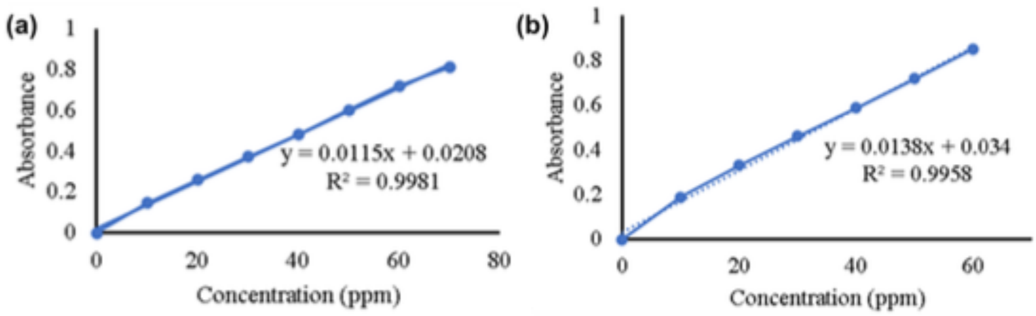

Figure 4. Calibration curve from standard solution of (a) Remazol Blue and (b) Indigosol Yellow.

3.4. Adsorption Studies

As shown in Figure 5(a), the %Removal of the mixed dyes increased from minute 10 to 45 and then decreased at minute 50. The increase in %Removal occurs because the carbon-derived Bidara Arab leaves (adsorbent) used still have many active sites, so the adsorbent will adsorb more adsorbate with the more time given for adsorption [26]. As shown in the graph, a decrease in %Removal at minute 50 can occur because all the active sides of the adsorbent have been saturated, causing a decrease in adsorption and desorption or re-release of the adsorbate that has been adsorbed. Desorption can occur because the adsorbent that has been saturated cannot absorb the adsorbate [27]. The results showed that the optimum contact time required for adsorption of the dye mixture was 45 minutes with a percent removal of 91.92% ± 0.59% for Remazol Blue and 70.77% ± 0.72% for Indigosol Yellow.

As shown in Figure 5(b), the %Removal of the mixed dyes increased from 30 mg to 50 mg, then decreased to 55 mg. The increase in %Removal of adsorbent mass indicates that adding adsorbent mass may improve absorption. The increase occurs because the more adsorbent mass added, the surface area of the adsorbent will increase; it affects the active side of the adsorbent, whose role is to bind and absorb the adsorbate so that the absorption of the adsorbate performs optimally [28]. This adsorption is classified as chemisorption due to the chemical bond between the adsorbent and adsorbate [29]. The graph in Figure 5(b) shows a decrease in %Removal at 55 mg. The decrease in %Removal at higher adsorbent mass occurs due to partial aggregation or clumping so that the surface area of the adsorbent decreases. A decrease in surface area also results in a decrease in the adsorption efficiency [30]. The optimum adsorbent mass required for the adsorption of Remazol Blue and Indigosol Yellow mixed dyes is 45 mg with a percent removal of 87.28% ± 0.95% for Remazol Blue and 66.18% ± 1.58% for Indigosol Yellow.

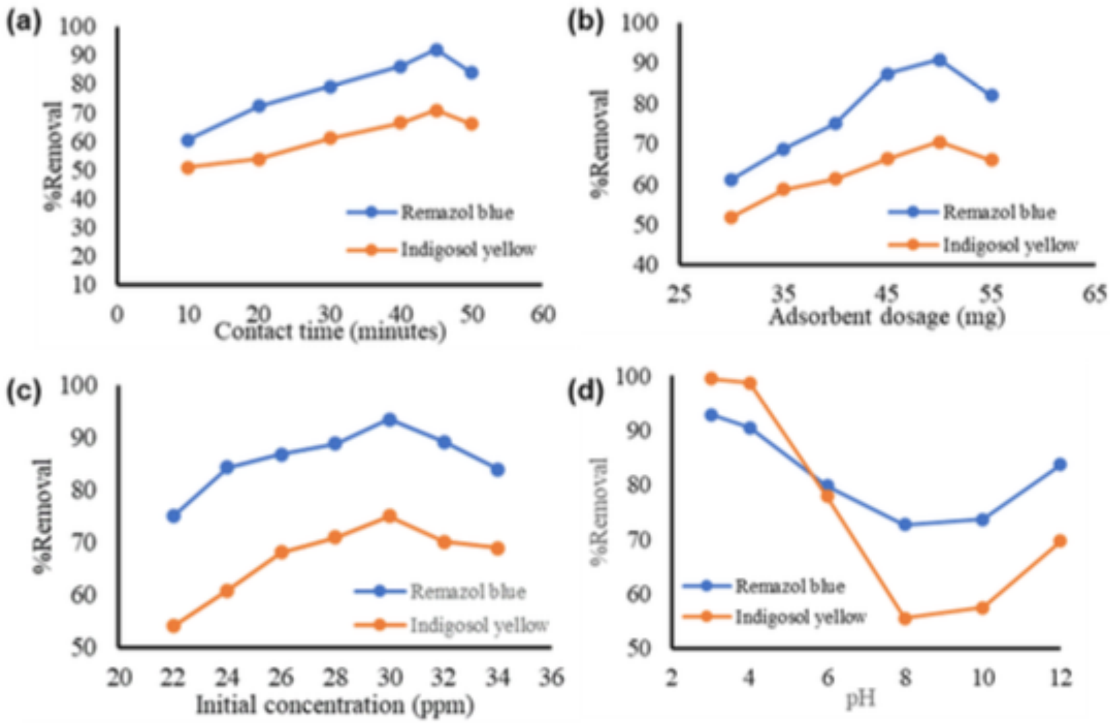

Figure 5. Removal graph for mixed dyes adsorption through variation of (a) contact time, (b) adsorbent dosage, (c) initial concentration, and (d) pH.

Figure 5(c) shows that the %Removal of the mixed dyes increased at 22 ppm to 30 ppm, then decreased at 32 and 34 ppm. The increase in %Removal due to the initial concentration indicates that the active site on the adsorbent surface is not yet saturated; the higher the initial concentration, the higher the dye absorbed by the adsorbent [31]. The decrease in %Removal to the initial concentration that occurred at 32 and 34 ppm indicates that the adsorbent has been saturated, so it can no longer bind the dye molecules that are still contained in the solution. Results showed that the optimum initial concentration required for adsorption of Remazol Blue and Indigosol Yellow mixed dyes was using an initial concentration of 30 ppm with a percent removal of 93.47% ± 0.49% for Remazol Blue and 74.96% ± 0.91% for Indigosol Yellow. As shown in Figure 5(d), the %Removal of Remazol Blue and Indigosol Yellow mixed dyes is high at the pH of strong acid and strong base; the highest %Removal is reached at pH 3, then decreased to pH 8 and increased again at pH 8 to pH 12. At acidic conditions, the adsorption mechanism in the dye mixture begins with deprotonation of the dye mixture. Protonation of the adsorbent will occur, and adsorption will occur when the dye mixture reaches the surface of the protonated adsorbent. Protonation on the adsorbent's surface can increase the adsorption activity. The lower pH of the adsorbate causes the adsorption process to run optimally because of the interaction between the negative charge on the mixed dyes and the positive charge on the adsorbent [32]. At strong base conditions, adsorption can occur, but the %Removal value is lower than in strong acids because, in alkaline conditions, the adsorbent becomes negatively charged and will oppose the dye; thus, the amount of adsorbed substance is lower. Results showed that the optimum pH required for adsorption of Remazol Blue and Indigosol Yellow mixed dyes was at pH 3 with a percent removal of 92.89% ± 1.07% for Remazol Blue and 99.50% ± 0.25% for Indigosol Yellow. The maximum adsorption capacity of each dye was 6.89 mg/g for Remazol Blue and 6.63 mg/g for Indigosol Yellow.

TABLE 1. Comparison with the previous work.

Source of Bio-activated carbon	**Parameter**				**Uptake (mg/g)**	**Efficiency (%)**	**Ref.**
	Adsorbent dosage (g)	**Dye concentration (g/l)**	**Contact time(min)**	**pH**			
Ilalang weeds	0.075	-	-	2-4	13.42	-	[33]
Monotheca buxifolia waste seeds	0.200	0.030-0.100	180	1-3	96.34	-	[34]
Acacia sawdust	-	1.000	-	-	-	81.14	[35]
Citrus reticulata	1.000	-	30	-	-	96.00	[36]
Pinang Frond	0.050	0.050	180	-	-	87.60	[37]
Durian seeds	-	-	-	2	-	95.17	[38]
Coconut Shells	3.000	0.200	60	2	-	65.00	[39]
Jatropha curcas pods	0.200	0.050	120	3	-	95.00	[40]
Pine cone	0.100	0.150	20	1,5	-	98.70	[41]
Rambutan Peels	0.300	0.100	-	8	-	78.38	[42]
Mangosteen Peels	1.500	0.100`	-	-	-	80.35	[43]
Pomegranate peels	0.200	0.025	1440	-	-	81.35	[44]
Coconut coir	0.040	0.005	180	1-3	-	96.00	[45]
Coconut shells	5.000	0.001	-	2	-	98.70	[46]
Watermelon rinds	0.500	0.400	1320	2	-	97.20	[47]
Bidara Arab leaves	0.450	0.030	45	2	-	99.50	This work

4. CONCLUSIONS

Research on the Adsorption of Remazol Blue and Indigosol Yellow Mixed Dyes using Bidara Arab Leaves (Ziziphus spina-christi) Carbon has been done. Based on the results, it can be concluded that Bidara Arab leaves carbon is an adsorbent that can be used to reduce the concentration of Remazol Blue and Indigosol Yellow mixed dyes contained in batik processing waste. The results showed the optimum conditions at 45 minutes, adsorbent mass of 45 mg, initial concentration of 30 ppm, and at pH 3 with %Removal each 91.92%; 90.76%, 93.47%, and 92.89%, for Remazol Blue and 70.77%, 70.37%, 74.96%, and 99.50% for Indigosol Yellow. The maximum adsorption capacity of each dye was 6.89 mg/g for Remazol Blue and 6.63 mg/g for Indigosol Yellow.

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