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Green Synthesis of Copper Nanoparticles using Turmeric (*Curcuma longa*) Mixed with Activated Carbon for Adsorption of Remazol Brilliant Blue R Dye from Wastewater

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ABSTRACT

Industrialization has surged alongside economic growth, leading to severe wastewater pollution, particularly from dye disposal, which poses risks to water consumers. This study investigates an innovative method for dye removal from wastewater. Previous research has examined the use of nanoparticles mixed with activated carbon to remove dyes such as methyl orange and reactive red 81. However, no studies have specifically addressed the removal of Remazol Brilliant Blue R (RBBR) dye using a combination of copper nanoparticles and activated carbon. RBBR is frequently found in textile industry wastewater. In this research, activated carbon (AC) derived from banana peels was combined with copper nanoparticles (CuNPs) synthesized using a green synthesis method with Curcuma longa extract. The primary goal was to evaluate the adsorption efficiency of this copper nanoparticle-activated carbon (CuNPsAC) composite for the removal of RBBR dye. The synthesis process involved mixing turmeric extract with NaOH and copper sulfate pentahydrate with AC, followed by combining both mixtures. Samples were characterized using UV-Vis spectrometry, Fourier-transform infrared spectroscopy (FTIR) and Differential Scanning Calorimeter (DSC). The findings revealed that CuNPsAC demonstrated significantly higher adsorption efficiency for RBBR dye (90%) compared to AC alone (65.8%) when using 1g of the adsorbent. Other factors, such as adsorbent mass, contact time, dosage, and solution pH, were optimized, with the best results achieved at 1g CuNPsAC, 160 minutes of contact time, 30 mg adsorbent dosage, and pH 9. This study underscores the effectiveness of the CuNPsAC composite in removing dye pollutants from wastewater, offering a sustainable solution for dye wastewater treatment. The green synthesis method using Curcuma longa and banana peels not only boosts dye removal efficiency but also presents an eco-friendly alternative to traditional approaches. These findings suggest a promising strategy for reducing the environmental impact of textile dye effluents.

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1. Introduction

The growing population has brought increased attention to wastewater treatment in recent years. Consequently, the demand for wastewater reclamation has surged. According to the UN's Sustainable Development Goals Report 2019; the countries in Northern Africa, as well as Western, Central, and Southern Asia, are currently experiencing medium to high levels of water stress [1]. As a result, non-traditional water sources are receiving greater focus to help alleviate the pressure on natural resources. Recycled wastewater, particularly from municipal wastewater treatment plant effluents, has the potential to become a net producer of renewable energy by converting the chemically bonded energy in raw organic contaminants into usable energy carriers, while also generating recyclable and renewable resources [2]. In addition to this energy potential, treating wastewater can reduce environmental pollution, preventing toxic impurities and harmful industrial by-products from contaminating water sources, which are vital to the survival of many living organisms. A variety of methods can be used for wastewater treatment, including chemical, biological, and physical processes.

This research focuses on physical treatment due to its simplicity, ease of operation, and effectiveness in color removal, as noted by Adesanmi *et al*., [3]. Unlike chemical treatments, which can produce hazardous by-products, or biological treatments, which are often time-consuming, physical methods like adsorption, coagulation/flocculation, and filtration are faster and more straightforward. Adsorption was chosen for this study because it is quick, efficient, and produces fewer hazardous by-products, as confirmed by Poerio *et al*., [4]. Wastewater from the textile industry is typically characterized by high pH, elevated concentrations of suspended particles, chlorides, nitrates, metals (such as manganese, sodium, lead, copper, chromium, and iron), high chemical oxygen demand (COD), biochemical oxygen demand (BOD), and significant dye content. Proper treatment is essential before releasing this wastewater into nearby water sources. This research highlights the use of dye adsorption as a key component of wastewater treatment prior to environmental discharge. Several physical and chemical methods, such as filtration, flotation, adsorption, and photocatalysis, can be employed to remove dye from industrial effluents. Adsorption has emerged as a standard method due to its simplicity, scalability, and effectiveness in removing trace pollutants from water.

In this study, adsorption was carried out using copper nanoparticles (CuNPs) combined with activated carbon (AC). CuNPs have shown to be effective in organic pollutant degradation through reductive or oxidative processes and photocatalytic degradation, according to Rafique *et al*., [5]. Copper is a widely used and cost-effective material, making CuNPs particularly appealing due to its abundance in nature. These nanoparticles can be synthesized and characterized through various techniques, and their stability and reactivity are critical for their potential use in nano-electronic devices. However, CuNPs are prone to oxidation at atmospheric pressure, so efforts have been made to develop more stable Cu-based NPs with complex structures like core/shell CuNPs, as discussed by Manoj et al. [6]. CuNPs have broad applications, including antimicrobial agents, nano-metal lubricant additives, capacitor materials, EMI shielding, heat sinks, and thermally conductive materials. To prevent the aggregation of CuNPs during wastewater treatment, various additives, such as zeolites, silica gel, activated carbon, and polymeric adsorbents, have been incorporated in previous research [7-11].

In this study, activated carbon was chosen due to its widespread use in water treatment and its ability to remove organic substances, including dyes effectively. Mixing AC with CuNPs is expected to enhance adsorption efficiency and reduce nanoparticle aggregation. As Beketov *et al*., [12] noted, a thin carbon shell can prevent metal nanoparticle aggregation and oxidation. AC, which is highly porous and has a large surface area, is commonly derived from carbonized materials and is widely used for adsorption in both environmental and industrial applications [13]. Activated carbon's high porosity and surface area make it an effective adsorbent, as up to 90% of AC products contain carbon. The main functional groups responsible for pollutant adsorption, such as carboxyl, carbonyl, phenol, lactone, and quinone, are crucial in determining AC's adsorbing properties. The specific raw materials and activation processes used to produce AC also impact its performance. In this study, banana peels were used as the raw material for AC production. The primary focus of this research was to investigate the adsorption capacity of the CuNPs-AC composite for dye removal, specifically targeting Remazol Brilliant Blue R (RBBR), a widely used dye in the textile industry known for its vivid color and stability. RBBR is often found in high concentrations in textile wastewater, with only around 50% fixing during the dyeing process, as reported by Sonal *et al.*, [14]. The green synthesis method was used to produce CuNPs from turmeric (Curcuma longa) extract, which was then combined with activated carbon. This study emphasizes the potential of this environmentally friendly approach in enhancing dye adsorption from textile wastewater.

2. Methodology

2.1 Curcuma Longa (turmeric) Extraction Solution

Curcuma longa, approximately 63 g, was washed several times with distilled water to remove impurities and dust particles. After the washing process was completed, it was cut into smaller pieces and boiled in a 500 mL glass beaker with 250 mL of ionized water for 30 minutes. The sample was then filtered using Whatman 40 mm filter paper to obtain a pure extract, free of impurities.

2.2 Green Synthesis of CuNPs

The previously extracted turmeric solution was mixed with a $CuSO_4$ solution at a ratio of 4:1. The mixture was stirred for 30 minutes at room temperature. The color change from blue to sea green indicated the formation of CuNPs. Further analysis was conducted to confirm the formation of the CuNP compound. UV-Vis spectrometry was used to determine the adsorption of CuNPs, while FTIR spectra were utilized to study the functional groups in the prepared CuNPs. Other portions of the CuNPs were combined with AC, which was prepared afterward. Both samples were analyzed for UV-Vis absorption spectra to confirm the formation of CuNPs and CuNPsAC. Note that different masses of AC (0.25 g, 0.5 g, 0.75 g, 1 g, and AC only) were used as one of the manipulated variables in testing dye adsorption efficiency.

2.3 Activated Carbon Production from Banana Peels

For the production of AC, banana peels (BP) were washed several times and then dried in an oven at 100 °C. The mass of the banana peels was measured both before and after drying. The difference in mass was essential to confirm that the drying process was complete. The mass after drying needed to remain constant, indicating that all the water had been removed from the peels. The peels were kept dry and weighed repeatedly to ensure the mass remained consistent. Once dried, the peels were crushed using a pestle and mortar. The resulting powder was calcined at 600 °C, forming a finer powder in the furnace after 1 hour. This powder was then activated through chemical reduction, where 5 g of the adsorbent powder was mixed with 250 mL of 1M KOH and stirred for 12 hours on a magnetic stirrer at room temperature (25 °C). Afterward, the powder was washed several times with distilled water until the pH became neutral. The adsorbent was then filtered using filter paper and underwent another drying process in an oven at 50 °C. AC samples with masses of 0.25 g, 0.5 g, 0.75 g, and 1 g were subsequently mixed with the CuNPs solution to study the relationship between AC mass and adsorbent efficiency.

2.4 Preparation of CuNPsAC

4 mL of 3 mM $CuSO_4$ was mixed with the synthesized AC at different masses (0.25 g, 0.5 g, 0.75 g, and 1 g) for 10 minutes on a magnetic stirrer at room temperature (25 °C). Simultaneously, 20 mL of turmeric extract was mixed continuously for 10 minutes with 1 mL of 0.1 M NaOH on a magnetic stirrer at room temperature (25 °C). Once both mixtures were prepared and allowed to stand for 10 minutes, they were combined and stirred on a magnetic stirrer for 30 minutes at 50 °C. After mixing, the mixture was centrifuged to separate the AC solids from the liquid. The solid was then dried in the microwave for 24 hours before the CuNPsAC was tested for dye adsorption.

2.5 Adsorption of Dyes

The RBBR dye was diluted to 10 mg/L for the adsorption procedure. Adsorption was carried out using a magnetic stirrer at a constant speed of 300 rpm. To evaluate the adsorption capacity, the initial concentration of the RBBR dye solution and the mixture of CuNPsAC with the RBBR dye were collected. For each experiment, 50 mL of 10 mg/L RBBR dye was mixed with 10 mg of CuNPsAC at masses of 0.25 g, 0.5 g, 0.75 g, or 1 g, as well as AC alone, to examine the effects of BPAC mass on dye adsorption. The adsorption efficiency using UV-Vis spectrometric data was calculated. Additional parameters in this research included varying adsorption contact times (20 min, 40 min, 60 min, 80 min, 100 min, 120 min, 140 min, and 160 min), adsorbent dosages (10 mg, 20 mg, 30 mg, and 40 mg), and pH levels (pH 3, pH 7, pH 9, and pH 10), as referred to in Mohamad *et al*., [15] with some modifications to suit the conditions of this research.

3. Results

3.1 UV-Vis Spectrometry Analysis

Figure 1 shows the absorbance spectra over a wavelength range from 200 nm to 800 nm, with significant absorbance observed between 200 nm and 350 nm. Specifically, the major absorbance peaks are noted around 227 nm and 231 nm. For the CuNPs, the peak occurs at 227 nm with an absorbance of 8.168. This peak is indicative of copper nanoparticles [16], which typically exhibit strong absorbance in the UV range due to their electronic transitions. In contrast, the CuNPsAC, has a major peak at 231 nm with an absorbance of 7.945. This slight shift in the peak position suggests an interaction between the copper nanoparticles and the activated carbon matrix, possibly altering the electronic environment of the nanoparticles. When comparing the spectra, it is evident that the absorbance intensity is slightly higher for CuNPs compared to CuNPsAC, implying that the addition of activated carbon might slightly reduce overall absorbance. The minor shift from 227 nm to 231 nm further indicates some interaction between the copper nanoparticles and the activated carbon. Additionally, both spectra show a series of smaller peaks between 200 nm and 300 nm, which could be attributed to various transitions within the material, potentially due to the presence of multiple components or different states of the nanoparticles. The slight decrease in absorbance intensity in CuNPsAC suggests that the activated carbon might be providing additional surface area or porosity, thereby enhancing the adsorption properties. Activated carbon derived from banana peel is known for its high surface area and porosity, which can significantly enhance the adsorption capacity for dyes like RBBR. The combination with copper nanoparticles can offer a synergistic effect, where the activated carbon adsorbs the dye molecules, and the nanoparticles facilitate their degradation through photocatalysis.

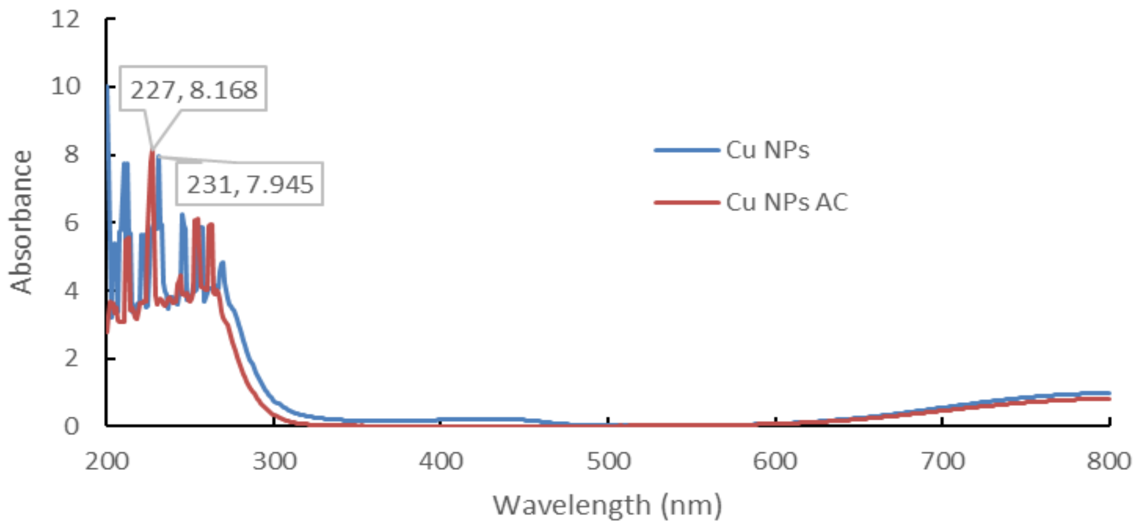

Fig. 1. UV-Vis spectra of CuNPs and CuNPsAC

3.2 FTIR Spectra Analysis

The formation of CuNPs and CuNPsAC can be further confirmed through FTIR analysis by examining their functional groups. Figure 2 shows several peaks that confirm the synthesized CuNPs and CuNPsAC. Amaliyah *et al.*, [17] state that the peaks between wavelengths 1600 - 1700 cm^{-1} can be ascribed to O-H bending. Additionally, N-H is stretching located at the peak of 3271 cm^{-1} for CuNPs, while for CuNPsAC, O-H stretching can be found at the peak of 3375 cm^{-1}. According to previous research, the peaks in this range are related to the functional groups found in extract compounds that serve as capping agents during the creation of CuNPs. Other peaks, such as C-H stretching, can be found at 2917 cm^{-1} for CuNPs. Several peaks for CuNPs at 1393 cm^{-1}, 1448 cm^{-1}, and 1638 cm^{-1} represent C-OH stretching vibrations, C=C aromatic ring stretching, and C=C stretching, respectively. Meanwhile, peaks for CuNPsAC at 1663 cm^{-1} represent C=C stretching. However, the peaks obtained for CuNPsAC only show some of the expected peaks. According to research by Rengga *et al.*, [18], BPAC should also have peaks representing bands such as O-H bond and C-H stretching. The obtained CuNPsAC does not show these peaks, possibly because the CuNPs and AC were not well-mixed. The mixing process only involved stirring, not sonification, which is necessary to obtain the purest version of CuNPsAC. This fact may have disturbed the reading. However, according to previous work by Mohamed [19], all these peaks can be found in both synthesized CuNPs and CuNPsAC samples.

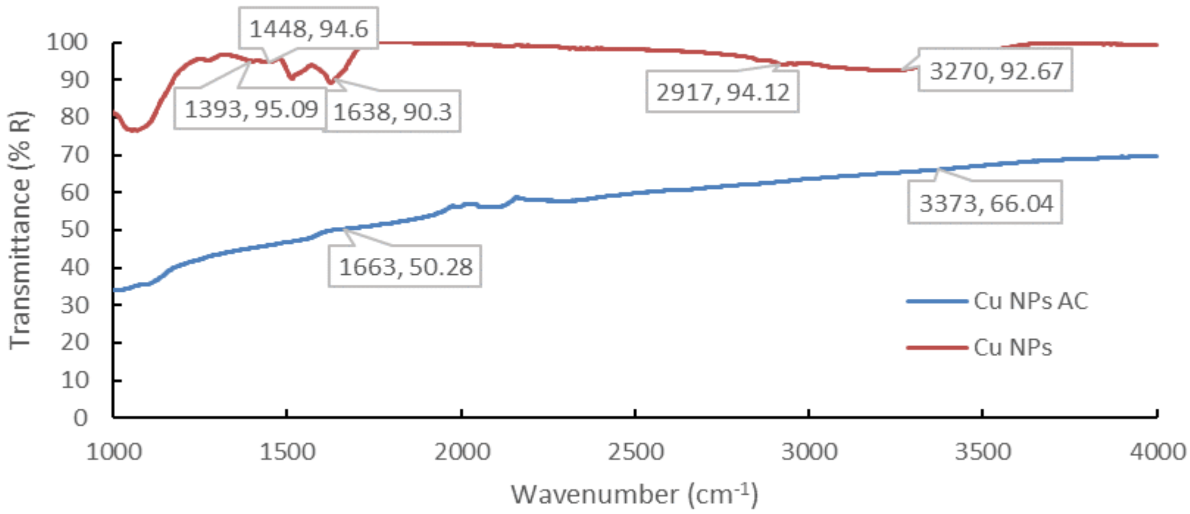

Fig. 2. FTIR spectra of CuNPs and CuNPsAC samples

3.3 Morphology Analysis

Field Emission Scanning Electron Microscopy (FESEM) analysis was performed to examine the morphology of CuNPs and CuNPsAC samples. Figure 3 (a) shows the image of the synthesized CuNPs. The FESEM micrograph demonstrates that the CuNPs are randomly distributed with a size range between 100 and 200 nm, indicating that the nanoparticles are agglomerated. These agglomerated CuNPs resemble other nanoparticles reported in the literature by Nasollahzadeh *et al*., [20]. The high-resolution FESEM image confirms that the particle size is approximately 5 to 20 nm. This finding is significant because it shows that using BPAC not only aids in the adsorption process but also helps prevent CuNPs from agglomerating. Figure 3 (b) displays the morphology of the CuNPsAC, which also shows a particle size range of 100 to 200 nm. According to the same research by Nasollahzadeh *et al*., [20], the expected CuNPs distribution histogram can be visualized using Figure 4, indicating that the nanoparticle size ranged between 5 to 20 nm. This result is consistent with previous research by Rajesh *et al*., [21], in which the FESEM images of the synthesized CuNPs had an average size of around 20 nm.

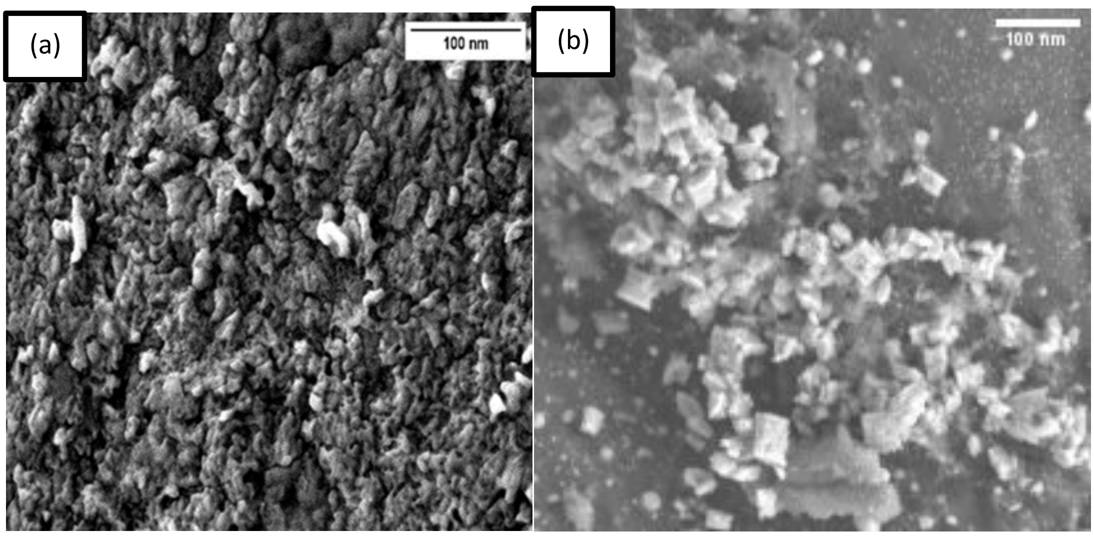

Fig. 3. SEM images of (a) CuNPs and (b) CuNPsAC synthesized

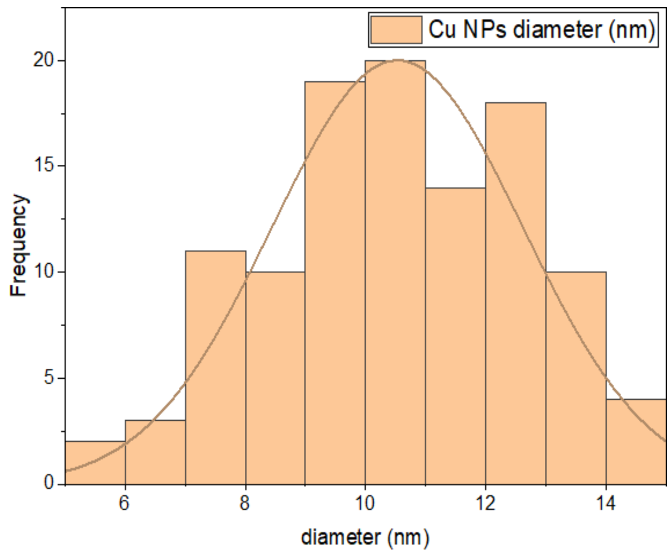

Fig. 4. Histogram of CuNPs size distribution

3.3 Thermal Analysis

In this research, Differential Scanning Calorimetry (DSC) was used to analyze the temperature and changes in the physical characteristics of the samples over time. Figure 5 presents the DSC graphs of CuNPs. The T_m peak, T_m onset, and T_m endset of CuNPs were 163.93 °C, 142.49 °C, and 179.50 °C, respectively. Based on these T_m values, two distinct exothermic peaks were observed for the CuNPs. These peaks appeared in the temperature ranges of 141.89 °C to 164.77 °C and 214.43 °C to 254.74 °C, indicating water absorption and crystallization. According to Ismail *et al*., [22], physically adsorbed water molecules and the desorption of biomolecules responsible for stabilization and CuNP reduction contribute to the overall weight loss observed. Research by Su *et al*., [23] indicates that the crystallization temperature of copper nanoparticles is approximately 379.05 °C to 433.2 °C. The difference in the obtained temperature may be due to the synthesis method of the CuNPs.

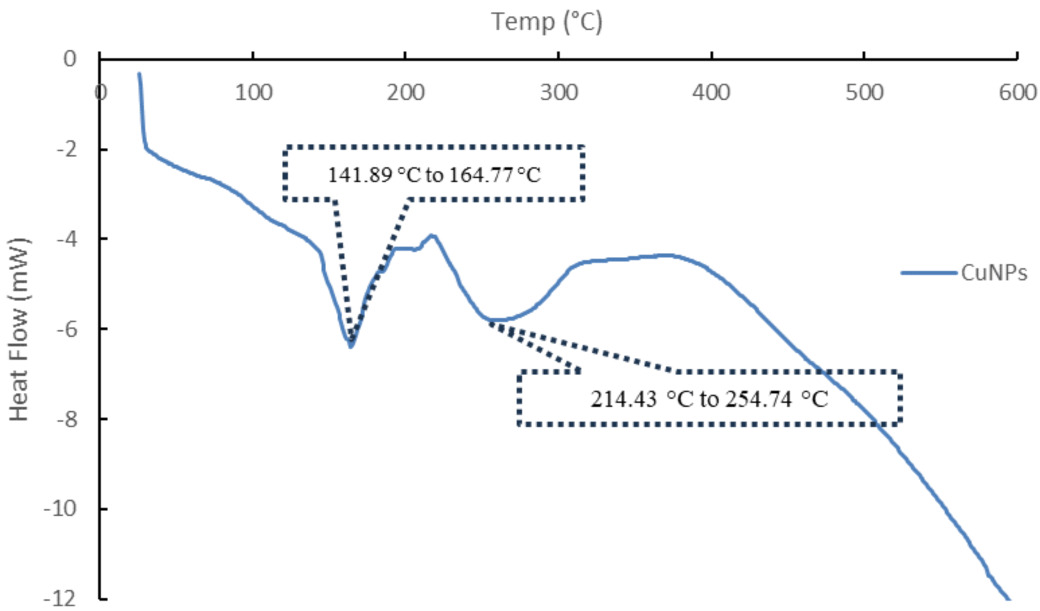

Fig. 5. DSC analysis of CuNPs

3.3 Dye Adsorption Applications
3.3.1 Effect of different AC mass

The prepared adsorbents, designated as 0.25SAC, 0.5SAC, 0.75SAC, and 1SAC, were utilized in this study as shown in Table 1. Pure AC was also used for comparison to evaluate the RBBR adsorption efficiency between CuNPsAC and pure AC. The mass was standardized to 30 mg for all samples (AC only, 0.25SAC, 0.5SAC, 0.75SAC, and 1SAC) in the RBBR dye adsorption efficiency test. Results show that, compared to the AC-only scenario (65.76%), CuNPsAC with masses below 0.75 g exhibited lower RBBR dye adsorption efficiencies, with 0.25 g CuNPsAC and 0.5 g CuNPsAC showing efficiencies of 7.71% and 41.04%, respectively. This observation indicates that AC remains effective in removing RBBR dye in this study.

Table 1
Result of adsorption efficiency at different CuNPsAC masses in comparison to AC only

	AC only	CuNPsAC (30 mg)			
	30mg	0.25SAC	0.5SAC	0.75SAC	1SAC
Adsorption (%)	65.76	7.71	41.04	87.76	90.02

3.3.2 Effect of adsorption contact times

Generally, adsorption capacity and dye removal efficiency increase with extended contact time. Initially, the amount of dye adsorbed onto the carbon surface rises rapidly. Over time, this process slows down and eventually reaches a plateau, as noted by Hameed *et al*., [24]. A dynamic equilibrium is established between the quantity of dye adsorbed onto the AC and the amount of dye desorbed from the adsorbent. The balanced time refers to the duration required to reach this equilibrium. After 80 minutes of adsorption, the RBBR dye adsorption efficiency slowed down, reaching around 95% or higher. This indicates that the equilibrium point of RBBR dye adsorption was being approached gradually. As the contact time between the CuNPsAC surface and the RBBR dye increases, the process eventually reaches its equilibrium point, where the RBBR dye removal from wastewater remains constant or experiences only minor changes. From an industrial perspective, operating the adsorption process beyond 160 minutes is unnecessary and uneconomical due to the increased utility and energy consumption associated with prolonged treatment. According to the results of this study, the adsorption process can be effectively stopped between 100 to 120 minutes, achieving a dye removal efficiency of 95.59% to 99.47% as shown in Table 2.

Table 2
Result of adsorption efficiency at different contact times

	Contact times (min)							
	20	40	60	80	100	120	140	160
Adsorption (%)	64.84	79.24	87.77	95.98	98.59	99.47	99.61	99.82

3.3.3 Effect of adsorbent dosages

Due to the need to establish the decolorization degree and the forecast the adsorbent cost for per unit of treated solution, the effect of adsorbent dosage is especially the most crucial factor to consider according to researchers Foo and Hameed [25]. According to the results, CuNPsAC with the dosages of 30 mg and 40 mg have a better and high RBBR dye adsorption efficiency, about 97.97% and 91.20%, respectively when compared to the dosages of 10 mg (5.04%) and 20 mg (57.61%) as shown in Table 3. Although CuNPsAC at 40 mg showed > 90% of RBBR dye adsorption, CuNPsAC at 30 mg demonstrated the highest RBBR dye adsorption at 97.97%. Therefore, 30 mg of CuNPsAC was chosen as the best condition for RBBR dye adsorption in this study, which can also minimize the usage of adsorbent and economically solve this dye pollution issue toward a better environment.

Table 3
Result of adsorption efficiency at different CuNPsAC

	Dosage (mg)			
	10	20	30	40
Adsorption (%)	5.04	57.61	97.97	91.20

3.3.4 Effect of pH

The final parameter analysed in this study was the solution pH. To understand its effect on RBBR adsorption efficiency, the pH of the RBBR dye solution was adjusted to 3, 7, 9, and 10. As shown in Table 4, the highest RBBR dye adsorption efficiency was observed at pH 9 with a value of 99.73%, followed by pH 7 with approximately 99.31%.The dye solution under acidic conditions had the lowest adsorption efficiency at 88.24%. This indicates that the pH of the dye solution significantly affects adsorption efficiency. According to Okareh *et al.*, [26], increasing the pH reduces the concentration of H+, thereby decreasing the competition between protons and metal ions for adsorption sites on the adsorbent surface. In other words, adsorption efficiency increases with a higher solution pH. This observation demonstrates that acidity inhibits the adsorption of positively charged dye cations due to electrostatic repulsion. At the same time, alkalinity enhances the adsorption of electropositive compounds, as noted by Foo and Hameed [24]. Akmil-Basar *et al.*, [27] found that excess H+ ions at acidic pH levels compete with dye cations for adsorption sites, leading to lower cationic dye adsorption. Table 4 summarizes the RBBR dye adsorption efficiency of the prepared CuNPsAC at different solution pH levels, showing that acidic conditions had the lowest adsorption efficiency compared to neutral and alkaline conditions.

Table 4
Result of adsorption efficiency at different pH

	pH			
	3	7	9	10
Adsorption (%)	88.24	99.31	99.73	98.18

4. Conclusions

This study focused on the green synthesis of copper nanoparticles (CuNPs) and the production of activated carbon (AC) from banana peels, which were then combined to form CuNPsAC. The optimization of CuNPsAC was achieved by blending two mixtures: one with varying amounts of AC (0.25 g, 0.5 g, 0.75 g, and 1 g) in 4 mL of 3 mM $CuSO_4$, and the other containing 20 mL of turmeric extract and 1 mL of 0.1 M NaOH. UV-Vis spectrometry confirmed the successful synthesis of CuNPs and CuNPsAC, showing peaks at 231 nm and 227 nm, respectively. FTIR analysis identified the functional groups in CuNPsAC with peaks at specific wavelengths, while the nanoparticle size was determined to be between 5 and 20 nm. Differential Scanning Calorimetry (DSC) analysis revealed exothermic peaks corresponding to crystallization at various temperature ranges, from 30.7 °C to 597.6 °C, for both CuNPs and CuNPsAC. The RBBR dye adsorption efficiency was evaluated, showing

90.0% for 1 g of CuNPsAC and 65.8% for 1 g of AC. The study also examined various parameters affecting dye adsorption, such as AC mass, contact time, adsorbent dosage, and solution pH. The highest adsorption efficiency was observed with 1 g of CuNPsAC, 160 minutes of contact time, 30 mg of adsorbent, and a solution pH of 9. The RBBR dye's blue color gradually faded as contact time increased.

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