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Utilization of Biomass-Derived Bottom Ash for the Removal of Textile Azo Reactive Dye from Aqueous Solutions

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Abstract

Bottom ash (BA) generated from the combustion of a specific biomass mixture consisting of Gliricidia sepium and Hevea spp. (Rubber wood) in a boiler was investigated for the removal of JAKOFIX RED ECON (JRE), a commercial azo reactive dye, from aqueous solutions. The effects of adsorbent dosage, shaking time, settling time, and pH of the initial solution on the adsorption efficiency were evaluated. Batch experiments were conducted using 100 ml of 20 ppm JRE solution, shaken at 150 rpm at 298 K. The optimum adsorbent dosage was 0.400 g, and the adsorption process was found to be most effective in acidic conditions (pH 2.5). Equilibrium was achieved after 35 minutes of shaking, and there was no effect on the settling time, indicating a rapid adsorption process. The findings demonstrate that bottom ash derived from biomass is an efficient and cost-effective adsorbent for the removal of JRE (azo reactive dye) from aqueous solutions under optimized conditions.

Keywords: Bottom ash, Azo reactive dye, Adsorption, Efficient

1. Introduction

Azo dyes are the most widely used class of dyes, accounting for over 60% of all dyes and about 70% of those used in industry, and they are known for being highly water soluble (Leechart, Nakbanpote and Thiravetyan, 2009) (Tupare, 2024). They are organic compounds that have the functional group R-N=N-R', where R and R' are usually aryl groups. The chemical structure of azo dyes contains a backbone, chromophoric groups, auxochrome groups, and solubilizing groups. The color of the azo dye is determined by azo bonds, associated auxochrome, and chromophore (Tupare, 2024). Reactive dye in wastewater has low biodegradation in aerobic conditions, and most of the azo dyes produce carcinogenic aromatic amines in anaerobic conditions (Leechart, Nakbanpote and Thiravetyan, 2009). The azo bond, the most unstable part of azo dye structures, undergoes enzymatic reduction by azo reductase in various mammalian organs, producing aromatic amines. These metabolites, absorbed through the intestine and excreted in urine, are associated with

toxic, mutagenic, and carcinogenic effects (Pinheiro, Touraud and Thomas, 2004). Dyes are widely used to color products in various industries, including textiles, tanneries, paper and pulp mills, and the food industry, and these industries generate dye-bearing wastewater as a result of their operations (Gupta *et al.*, 2005). These wastewaters must undergo effective pretreatment to remove color and reduce pollutant load before being discharged into the Central Wastewater Treatment Plant (CWTP) to ensure compliance with environmental regulations and protect aquatic ecosystems.

Several conventional treatment methods have been developed for the removal of dyes from wastewater, including coagulation, flocculation, and membrane technologies (Mokif, 2019). The adsorption process is a better alternative, as it is effective and produces high-quality effluents that do not produce toxic substances such as free radicals, which are produced under the photo-degradation using UV radiation, and do not consume chemicals as in the coagulation and flocculation methods (Gupta *et al.*, 2005). Additionally, adsorption is a desirable technique if the adsorbent is inexpensive, easily accessible, and doesn't need an expensive pretreatment step (Dinçer *et al.*, 2007). Carbon-based adsorbents are highly effective in removing synthetic dyes. However, the production of commercially available carbon-based products, such as activated carbon, is highly energy-intensive and requires large quantities for treatment, which limits their large-scale application (Forgacs, Cserháti and Oros, 2004). Therefore, scientists pay attention to developing low-cost, effective, and readily available adsorption materials to reduce the adsorbent dose and the disposal problems of dye-bearing wastewater (Leechart, Nakbanpote and Thiravetyan, 2009). These include calcined mussel shells (Haddad *et al.*, 2014), *Mangifera indica* seeds (Alencar *et al.*, 2012), cupuassu shells (Cardoso *et al.*, 2011), calcined eggshells (Slimani *et al.*, 2013), and tree ferns (Ho, Chiang and Hsueh, 2005). Boilers are crucial in industrial sectors, providing the heat and steam necessary for production processes. They are used in the chemical manufacturing industry, the food and beverage industry, textile and leather production, paper mills, etc. (Payel *et al.*, 2023). Biomass boilers generate a large amount of ash as a waste product, which poses significant challenges in terms of storage, disposal, and utilization (James *et al.*, 2012).

Although bottom ash has previously been investigated as an adsorbent for dye removal, its adsorption behaviour may vary depending on the biomass feedstock from which it is generated. Therefore, the adsorption performance reported for one type of bottom ash cannot necessarily be directly extrapolated to bottom ash derived from other biomass sources. In the present study, the bottom ash was generated from the combustion of a specific biomass mixture consisting of *Gliricidia sepium* and *Hevea*

spp. (rubber wood) in a boiler. To the best of our knowledge, no previous study has specifically investigated bottom ash derived from this biomass mixture for dye removal. Therefore, this study aims to investigate the adsorption of azo reactive dye (JRE) using bottom ash, a waste product generated from boiler operations in a fabric dyeing and printing industry. In this study, the effects of dosage, pH, shaking time, and settling time were investigated using batch experiments for the synthetic dye solution.

2. Material and Method

2.1. Preparation of Adsorbent

Bottom Ash (BA) was obtained from the combustion of a sustainable biomass mixture consisting of *Gliricidia sepium* and *Hevea spp*. (rubber wood) in a boiler at a fabric dyeing and printing facility in Sri Lanka, which generate as waste. BA was water-treated following the procedure reported by Leechart, Nakbanpote and Thiravetyan, (2009) to develop the surface area, surface charge, and porosity. The 100 g of BA was added to 1 L of distilled water, and the solution was shaken for 1 hr in an orbital shaker (LabGenius) at 150 rpm. Then, BA was washed with an excess of distilled water until the pH of the solution was stable at ~13. Next, the adsorbent was dried for 4 hr at 105°C and sieved to obtain particles of size <75 µm (Leechart, Nakbanpote and Thiravetyan, 2009).

2.2. Preparation of Adsorbate

JAKOFIX RED ECON (JRE), produced by Jay Chemical Industries Ltd., was selected for this study. Chemically, this is an azo reactive dye. Its aqueous solution has a maximum absorbance range of 460-580 nm, and the solubility is 100 g/L in water. A 1000 ppm JRE dye solution was prepared as a stock solution, and experimental solutions were prepared by diluting the stock solution using distilled water. The pH of the solutions was adjusted using 0.1 M NaOH and 0.1 M HCl, and the pH measurements were obtained using a HANNA pH meter.

2.3. Batch Experiment

Adsorption experiments were performed in 250 ml beakers with 100 ml of 20 ppm JRE dye solution and shaken at 150 rpm. The effects of dosage (0.100 g – 0.700 g), shaking time (10 min – 45 min), settling time (5 min to 40 min), and pH (2.0-12) were investigated to determine the efficiency of the adsorbent. All chemicals used were of analytical grade. All experiments were conducted in triplicate, and the results were expressed as mean ± standard deviation (SD). The residual dye concentration was

measured using a UV-Visible spectrophotometer (Yildirim and Acay, 2025). The removal percentage of JRE dye was calculated using the following equation:

$$Removal\ Percentage = \frac{(C_i - C_r)}{C_i} \times 100\ \% \qquad (1)$$

Where C_i and C_r are the initial dye concentration and residual dye concentration (mg L^{-1}), respectively (Yildirim and Acay, 2025).

3. Result and Discussion

3.1. Effect of Adsorbent Dosage on JRE Dye Adsorption

The adsorbent dosage is a critical parameter influencing adsorption capacity. To investigate this factor, the dosage of BA was varied while maintaining all other experimental conditions constant (100 ml of 20 ppm JRE dye solution, 1 hr shaking time, 1 hr settling time, ambient pH ~9, and 150 rpm). The results indicated that dye removal efficiency increased from 51.55% ± 1.28 to 97.95% ± 0.35 as the adsorbent dosage was raised from 0.100 g to 0.400 g. This trend can be attributed to the increase in surface area and the corresponding rise in the number of available active sites for adsorption with the increase in adsorbent dosage (Jayaram *et al.*, 2009). However, beyond this point, the removal efficiency reached an equilibrium, which may be explained by the decrease in the concentration gradient between the dye solution and the adsorbent surface (Yildirim and Acay, 2025). Therefore, 0.400 g was selected as the optimum adsorbent dosage for further experiments.

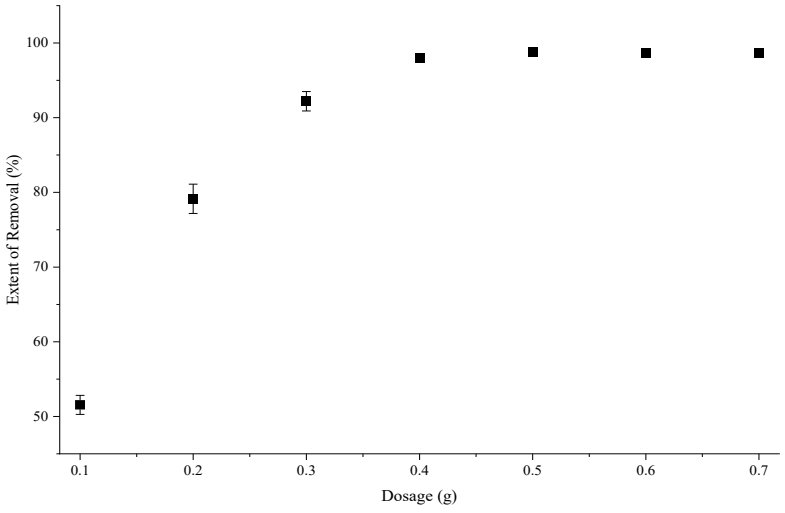

Figure 1: Extent of removal of JRE by BA particles at different dosages (100 ml of 20 mg/L JRE dye solution, 1 hour shaking time, 1 hour settling time, ambient pH ~9, 150 rpm agitation)

3.2. Effect of Shaking and Settling Time on JRE Dye Adsorption

The effect of contact time on the adsorption of the JRE Azo reactive dye was evaluated to get an idea about the equilibrium time (Yildirim and Acay, 2025). As illustrated in Figure 2, adsorption experiments were conducted using 100 ml of 20 ppm JRE azo dye solution with shaking times ranging from 10 to 45 minutes, 0.400 g of adsorbent, agitation at 150 rpm, a settling period of 1 hour, and ambient pH conditions of approximately 9. The removal efficiency increased from 81.95% ± 0.62 to 98.74% ± 0.11 as the shaking time was extended from 10 to 45 minutes. A rapid rise in adsorption was observed within the first 10 minutes, which can be attributed to the availability of abundant vacant adsorption sites at the initial stage. After 35 minutes, the removal percentage reached equilibrium as the adsorption sites became saturated. Hence, the optimum shaking time was determined to be 35 minutes (Gupta *et al.*, 2005).

The system may involve different mass transfer or diffusion processes that occur at a relatively slow rate. Hence, it is important to evaluate the removal of JRE after the optimum settling time to confirm that complete equilibrium has been achieved (Chieng, Lim and Priyantha, 2014). Hence, adsorption of JRE using BA was evaluated at different settling times, ranging from 5 minutes to 40 minutes, while keeping other parameters constant (100 ml of 20 ppm JRE dye solution, 35 shaking time, ambient pH ~9, and 150 rpm). As illustrated in Figure 3, there is no effect on the settling time.

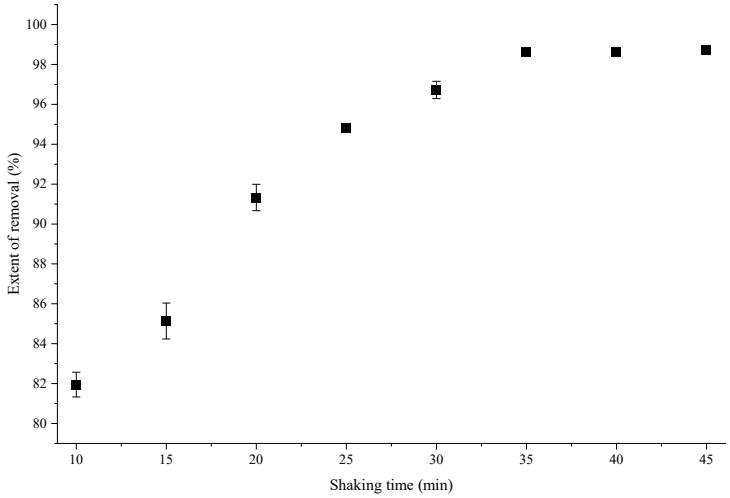

Figure 2: Extent of removal of JRE by BA particles at different shaking time (100 ml of 20 mg/L JRE dye solution, 0.400 g dosage,1 hour settling time, ambient pH ~9, 150 rpm agitation)

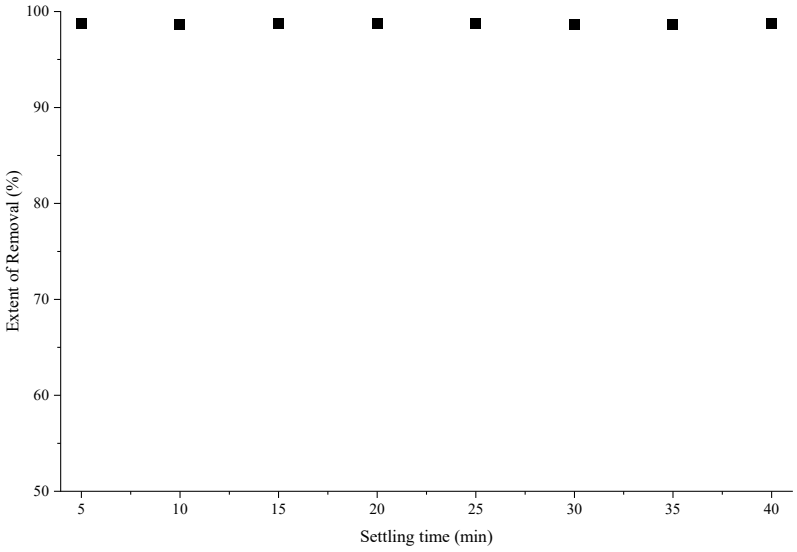

Figure 3: Extent of removal of JRE by BA particles at different settling time (100 ml of 20 mg/L JRE dye solution, 0.400 g dosage, 35 min shaking time, ambient pH ~9, 150 rpm agitation)

3.3. Effect of pH on the JRE Dye Adsorption

The pH of the initial dye solution is a critical parameter influencing dye adsorption, as it directly affects the surface charge characteristics of the biosorbent (Haddad *et al.*, 2014). Figure 4 illustrates the influence of pH of the initial solution (ranging from 2 to 12) on the adsorption of JRE onto BA, while other experimental parameters were kept constant (100 ml of 20 ppm JRE solution, 0.400 g dosage, 35 minutes shaking time). The results show (Figure 4) a gradual decline in dye removal efficiency, decreasing from 98.76% ± 0.09 at pH 2.0 to 90.61% ± 0.18 at pH 12. The effect of the initial pH of JRE was explained with amphoteric character. Since, depending on the pH of the system, the surface of the adsorbent may be negatively or positively charged, which has a direct effect on the electrostatic interactions. The JRE is a reactive dye. Reactive dyes are anionic dyes that contain sulphonate groups and carboxylic groups (D-SO_3Na, D-COONa) that are dissociated and produce anionic dye ions as shown in Eqs. 2 and 3 (Leechart, Nakbanpote and Thiravetyan, 2009) (Periyasamy, Dutta and Tehrani-Bagha, 2024). According to the results (Figure 4), the removal percentage was high at a lower pH. At lower pH, the surface of the BA may develop positively charged due to H_3O^+ ions in the solution, which enhances the adsorption of negatively charged dye anions (Dinçer *et al.*, 2007). The extent of removal of JRE was constant below 2.5 pH. Therefore, the optimum pH for adsorption of JRE onto BA was selected as 2.5 with a 98.73% ± 0.18 removal percentage.

$$D\text{-}SO_3Na \blacktriangledown D\text{-}SO_3^- + Na^+ \quad (2)$$

$$D\text{-}COONa \blacktriangledown D\text{-}COO^- + Na^+ \quad (3)$$

Although the highest removal efficiency (98.73% ± 0.18) was observed at pH 2.5, considerable JRE removal (90.61% ± 0.18) was also achieved at pH 12. This indicates that the BA retained substantial adsorption performance under alkaline conditions, suggesting potential for practical application without necessarily requiring the adjustment of industrial wastewater to a strongly acidic pH.

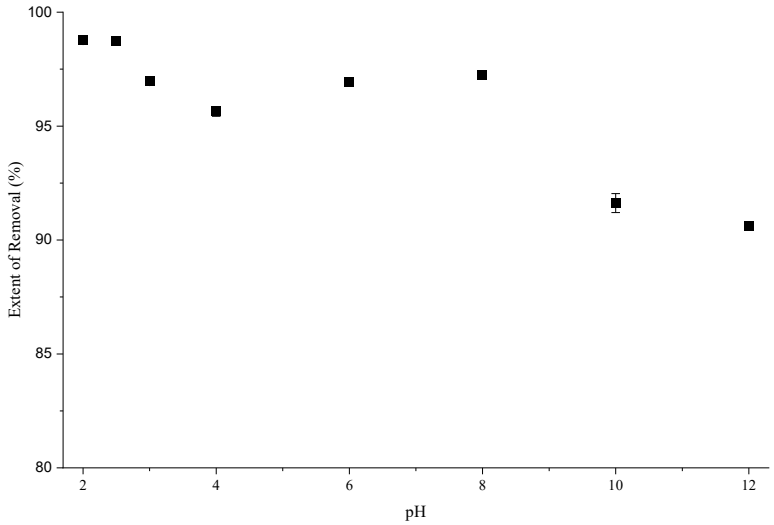

Figure 4: Extent of removal of JRE by BA particles at different initial pH (100 ml of 20 mg/L JRE dye solution, 0.400 g dosage, 35 min shaking time, 150 rpm agitation)

Further studies should include detailed physicochemical characterization of the biomass-derived bottom ash, including surface functional groups, surface morphology, elemental and mineralogical composition, surface area, and point of zero charge. Such characterization would provide a more comprehensive understanding of the properties of the biomass-derived bottom ash. In addition, adsorption isotherm and kinetic modelling should be investigated in greater detail to provide deeper insight into the adsorption mechanism.

4. Conclusions

This study demonstrates that bottom ash generated from combustion of wood (BA) can serve as an effective biosorbent for the removal of JRE (reactive azo dye) from aqueous solutions. Batch adsorption experiments revealed that BA exhibited significant de-coloration ability under optimized conditions. The optimum adsorption performance was achieved using a 0.400 g dosage, a shaking time of 35 minutes, and an initial solution pH of 2.5, with 100 ml of 20 ppm dye solution agitated at 150 rpm. The results also indicated that settling time had no significant effect on the adsorption efficiency. These findings highlight the potential application of BA as a low-cost and sustainable adsorbent for treating JRE (Azo reactive dye) dye from aqueous solution.

Competing interests

The authors have no competing interests to declare.

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