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Activation of Sugarcane Bagasse Biosorbent with Potassium Hydroxide and Hydrogen Chloride to Reduce Color of Batik Wastewater

Noven Pramitasari[1]*, Faisal Basri Ramadani[1], Rovy Agustian Azis[1], Calista Safa Bezariani[1], Rohinoor Intan Berliana[1], Ririn Endah Badriani[1], Audiananti Meganandi Kartini[1], Cantika Almas Fildzah[1]

[1]Faculty of Environmental Engineering, Universitas Jember, Jl. Kalimantan No.37, Krajan Timur, Sumbersari, Sumbersari, Jember, East Java 68121, Indonesia
*E-mail: *novenpramitasari@unej.ac.id

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Abstract

The textile industry, particularly the batik sector, significantly contributes to the Gross Domestic Product (GDP) of East Java Province. However, the batik dyeing process, which uses synthetic dyes like remazol, rapid, and naphthol, causes environmental issues due to the non-biodegradable nature of these dyes, leading to environmental pollution such as an increase in color levels in water bodies. This research investigated using activated sugarcane bagasse with KOH and HCl in batch adsorption systems as an alternative method. It compared the color efficiency removal in batik wastewater with variation biosorbent mass, contact time, and activator type. The study found that the highest percentage removal of dye substances was achieved with the KOH-activated biosorbent, reaching 69.46%, and the HCl-activated biosorbent demonstrated a 60.98% removal efficiency with a mass variation of 0.4 grams and a contact time of 30 minutes. Statistical analysis using multiple linear regression showed that independent variables (biosorbent mass, contact time, and activator) significantly affect the dependent variable (color removal efficiency) simultaneously. In the partial test, the independent variables of biosorbent mass and type of activator significantly affect color removal efficiency, but contact time does not significantly affect color removal efficiency.

Keywords: Adsorption, Biosorbent, Batik Wastewater, Sugarcane Bagasse.

1. Introduction

The batik industry in Indoensia has experienced significant growth in recent years, contributing to the region's economic development. This traditional textile art, renowned for its intricate patterns and cultural significance, has evolved from small-scale family businesses to larger enterprises. This expansion has led to an increase in local employment opportunities and has also boosted tourism. The proliferation of batik workshops and retail outlets has been crucial in preserving cultural heritage, driving economic diversification, and increasing regional exports. The batik industry in East Java has created numerous job opportunities and has contributed to rural development by promoting local craftsmanship and entrepreneurship. This growth aligns with regional development plans prioritizing cultural industries as crucial economic resilience and sustainability drivers (Istiqomah et al. 2021).

According to the Geographical Industry Information System of East Java Province (2024), there are 451 small-scale batik industries in East Java. One regency with a large number of batik industries is the Jember. The data on batik industries in Jember Regency, recorded by the Geographical Industry Information System of East Java Province, consists of eight companies. These companies are Aminy Batik, Griya Batik Barata, Ngaidin's Batik, Omah Batik 78, Rumah Batik Rolla, Rubung Kuning, Singkrangkong, and Srikandi. Seven of the eight companies are located outside the industrial center. Therefore, effective waste management is crucial to prevent environmental pollution.

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The batik production process involves using dyes, which can be either natural or synthetic. According to research by Susanti (2019), the batik industry in Jember, CV Godong Mbako in Wonoasri Village, Tempurejo District, mainly uses the jati plant as a dye, with 136 plants being used, compared to other plants, such as shame plant, tarum, sonokeling, mahogany, and mango. Some other batik home industries use synthetic dyes, such as a home industry in Teleng Sari Village, Jember Regency, which uses synthetic dyes like naphthol and rapid in the batik production process, resulting in wastewater containing cadmium (Silvia, 2018).

The process of creating batik fabric involves using dyes and other chemicals, leading to the generation of wastewater that contains various pollutants such as TSS, COD, turbidity, color, and heavy metals. The levels of these pollutants in batik wastewater vary depending on the dyeing methods and types of dyes used. According to Daud et al., (2023), typical levels in batik wastewater include TSS at 72 ± 41 mg/L, COD at 867 ± 45 mg/L, and color intensity at 381 ± 29 ADMI. Additionally, other studies have reported high levels of color (4474 PtCo), lead (Pb) content in mg/L, and Total Cr ($Cr6^+$) levels reaching 940.2 mg/L (Ni'am et al., 2023). These contaminants significantly contribute to environmental pollution, impacting the quality of rivers and groundwater and posing health risks due to carcinogenic heavy metals (Panhwar et al., 2024).

Several methods have been proposed to reduce color in wastewater. Methods include microfiltration, which has a 26-56% removal efficiency, coagulation causing high sludge production, trickling filters leading to odor emission, photochemical by-product formation, and inadequate ion exchange for all dyes. Adsorption is one of the methods used to address issues related to batik wastewater. Adsorption is the process of substance accumulation at the interface of two phases. The adsorption treatment method in wastewater treatment is due to its high selectivity, minimal generation of by-products such as chemical sludge, and renewability (Zakaria et al., 2023). The effectiveness of wastewater treatment using the adsorption method is simple and cost-effective. The color removal efficiency using adsorption methods can reach 100% using modified sugarcane bagasse (Leon et al., 2020).

The adsorption process involves utilizing an adsorbent to attract and retain an adsorbate. In recent years, studies in adsorption have focused on using economical and environmentally friendly biosorbents as adsorbents. Biosorbents are materials derived from biomass, including agricultural biomass such as coffee leaves and skin, pine bark, teff straw, Pineapple Peel and sugarcane bagasse (Ma et al., 2020; Permataari et al., (2023); Pramitasari et al., 2020; Wong et al., 2020). Sugarcane bagasse is a suitable biosorbent due to its high cellulose content of 42.67%, which makes it a promising option. The cellulose content in sugarcane bagasse is reinforced by lignin compounds, strengthening the structure.

Biosorbent is more effective for removing color if it is activated. Activation is a procedure conducted on biosorbents to enlarge their surface area, expand pore volume, and degrade pollutants through the oxidation of molecules present on the biosorbent surface. There are two methods for biosorbent activation, namely physical and chemical activation. Physical activation of biosorbents involves using CO_2 or microwave heating to remove water from the pores, thereby increasing the surface area. Biosorbents are chemically activated using chemical substances to eliminate impurities and enhance their absorption capacity (Ramadhani et al., 2019).

Physical and chemical activations are commonly employed to prepare biosorbents derived from lignocellulosic biomass (Njoku et al., 2014). Chemically synthesized activated carbon typically exhibits a higher specific surface area and a greater production yield under lower operational conditions, such as treatment time and reaction temperature, than those activated through physical treatment (Brito et al., 2018). Additionally, chemical treatment can replace the -OH and phenolic groups in lignocellulosic material with negatively charged groups from the activating agents, enhancing adsorption favorability (Guarín et al., 2018). In the chemical activation procedure, the raw material is initially crushed and milled before being impregnated with selected chemical activators like sodium hydroxide (NaOH), phosphoric acid (H_3PO_4), potassium hydroxide (KOH), and zinc chloride ($ZnCl_2$). KOH is widely used among these chemical activation agents because it produces activated carbon with a high specific surface area and forms oxygen-based functional groups (Araga et al., 2017; Astuti et al., 2019). Activated carbon treated with KOH has been successfully manufactured from various lignocellulosic-based biomasses, displaying a large specific surface area and a substantial distribution of pores, making them particularly attractive as adsorbents for water treatment applications (Rashidi et al., 2016; Chomiak et al., 2017; Spessato et al., 2019). HCl is effective as an activator for biosorption. A study by Maulidiyah et al. (2021) indicated that preparing the adsorbent involved utilizing 1 M HCl as a catalyst across 10 to 70 ppm concentrations. Examination through Scanning Electron Microscopy (SEM) revealed a notable 83.26% increase in pores in the charcoal derived from salak seeds after activation.

This study aims to treat batik wastewater using biosorbents from sugarcane bagasse obtained from agricultural waste in the Jember. The research focused on knowing the effect of activator type, the amount of biosorbent used, and adsorption time on the effectiveness of color removal in batik wastewater in Jember.

2. Methodology

2.1. Materials

The materials employed in this study include batik liquid waste samples from the batik home industry in Jember, sugarcane bagasse from agricultural waste in Jember, HCL 37% (Merck, USA), KOH (Merck, USA), Kobalt (II) Klorida Heksahidrat (Pudac Scientific, Indonesia), Potassium Hexachloroplatinate (IV) (Smart Lab, Indonesia), and distilled water.

2.2. Production of Activated Sugarcane Bagasse Biosorbent

Sugarcane bagasse underwent an initial washing process. Subsequently, it was air-dried for three days to ensure the removal of water content. The dried sugarcane bagasse was then finely ground through milling, followed by a sieving process using a 100-mesh sieve to enlarge the surface area of the adsorbent. The sieved sugarcane bagasse was washed with distilled water to eliminate impurities from the previous steps. Finally, the sugarcane bagasse was oven-dried for 4 hours at 90℃ until completely dry.

The sugarcane bagasse was then activated using potassium hydroxide (KOH) and hydrogen chloride (HCl). Activation involved a ratio of 5 grams of sugarcane bagasse to 100 ml of 1M HCl and KOH. The activation lasted 90 minutes with a stirring speed of 300 rpm. The activated sugarcane bagasse was dried in an oven for 4 hours at 90℃. The dried bagasse could then be activated.

2.3. Preparation Standard Solution Pt-Co and Calibration Curve

The preparation of standard solutions follows the Indonesian National Standard (SNI) 6989:2011 on "Color Test for Water and Wastewater by Spectrophotometry." The dilution procedure for the 500 Pt-Co stock solution refers to equation 1.

$$M1 \, x \, V1 = M2 \, x \, V2 \qquad (1)$$

Remark:
M1: Concentration of the stock solution (Pt-Co)
V1: The volume of the required stock solution (mL)
M2: The concentration of the solution to be prepared (Pt-Co)
V2: The volume of the standard solution to be prepared (mL)

The standard solutions were created using equation (1), resulting in standard solutions with concentrations of 100 Pt-Co, 200 Pt-Co, 300 Pt-Co, and 400 Pt-Co. These solutions were then used to measure their absorbance values at wavelengths specified by SNI 6989:2011 within 450 nm to 465 nm. The absorbance values for the 450 nm wavelength are detailed in Figure 1.

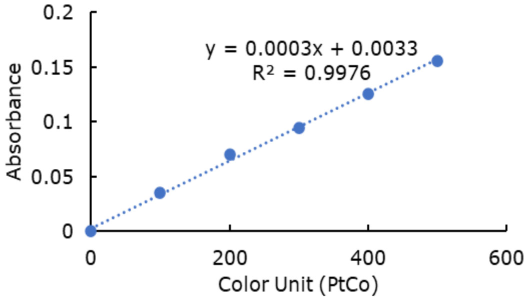

Figure 1. Absorbance Values For 450 nm Wavelength

The selected wavelength was determined based on the linear regression coefficient, which should have a value of $R^2 > 0.995$. The wavelength chosen for this study was 450 nm, with an R^2 of 0.9976.

2.4. Color Test for Batik Wastewater

The batik wastewater used in the adsorption test originated from a batik industry in Jember Regency and was a byproduct of the second washing process. Batik wastewater before and after adsorption underwent testing to determine the color removal efficiency. Color testing was conducted on batik wastewater using SNI 6989:2011 "Color Test for Water and Wastewater by Spectrophotometry."

2.5. Adsorption Process

The adsorption process was conducted in batches using sugarcane bagasse that had been activated using 1M HCl and 1M KOH. The varying masses of sugarcane bagasse added were 0.1, 0.2, 0.3, and 0.4 grams, with contact times for the adsorption process at 15 minutes, 30 minutes, 45 minutes, and 60 minutes with a magnetic stirrer stirring at 300 rpm. Following the adsorption process, the sample was analyzed to determine the final dye concentration. This process aimed to evaluate the removal efficiency of activated sugarcane bagasse treated with 1M HCl and KOH in reducing the color concentration in batik wastewater.

2.6. Statistic Test

The statistical test in this research used the Multiple Linear Regression Test with R-Studio Application. This statistical test was used to model the relationship between the independent and dependent variables. The independent variables in this research were biosorbent mass, contact time, and activator type, while the dependent variable was color removal efficiency (Tranmer et al., 2020).

3. Results and Discussion

3.1. Innitial Color Concentration of Batik Waste water

The wastewater is blue, with a pH of 8.0 to 9.0, and emits an unpleasant odor. Before conducting the adsorption test, we used a UV-Vis spectrophotometer to measure the absorbance of the batik wastewater in order to determine the initial color concentration. The measurement results revealed absorbance values of approximately 0.600 to 0.700, corresponding to around 2,000 Pt-Co.

3.2. Biosorbent Mass Effect

This research involved using different masses: 0.1, 0.2, 0.3, and 0.4 grams. The procedure entailed stirring at 300 rpm, with contact times ranging from 15 to 60 minutes. Figure 2 depicts the correlation between color removal efficiency in batik wastewater and biosorbent mass.

Based on Figure 2, the optimum adsorption results are indicated with a mass variation of 0.4. The red curve (30 minutes) shows the highest removal percentage, reaching 60.98% with a mass variation of 0.4 grams. The blue curve (15 minutes) exhibits a removal percentage of 58.20% at a mass variation of 0.4 grams. The green curve (60 minutes) indicates a removal percentage of 53.39% with a mass variation of 0.4 grams. The yellow curve (45 minutes) demonstrates a removal percentage of 53.34% at a mass variation of 0.4 grams.

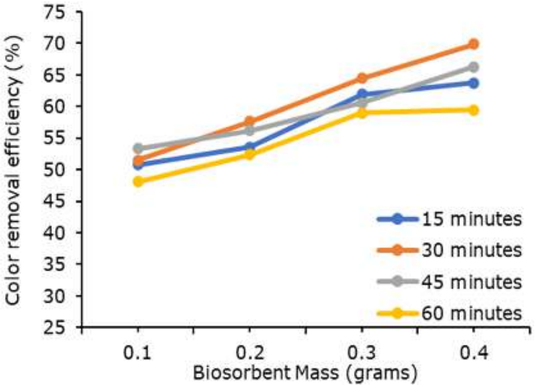

Figure 2. Biosorbent Mass Effect with HCl Activator

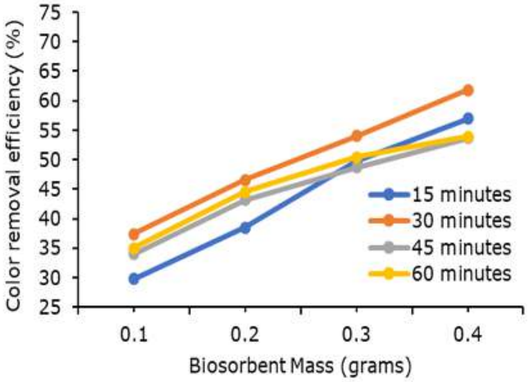

Figure 3. Biosorbent Mass Effect with KOH Activator

Based on Figure 3, the increase in mass of activated sugarcane bagasse biosorbent generally enhances the efficiency of color concentration removal in batik wastewater. For a mass variation of 0.1 gram, the color removal efficiency reaches 52.99% with a contact time of 45 minutes. At a mass of 0.2 grams, the highest efficiency is achieved at 57.46% with a contact time of 30 minutes. A mass of 0.3 grams yields the highest efficiency at 64.13% with a contact time of 30 minutes. With a mass of 0.4 grams, the removal efficiency reaches its peak at 69.46%. Increasing biosorbent mass can increase the number of free surface-active sites—the more significant number of free surface-active sites of biosorbent causes greater color removal (Djelloul et al., 2017).

3.3. Contact Time Effect

The contact time duration becomes a crucial factor in the adsorption process, influencing the amount of adsorbate that gets adsorbed. Additionally, contact time is essential to achieve adsorption equilibrium and to assess the potential use of the adsorbent in wastewater treatment (Triana, 2015). In this study, variations in contact time were applied, spanning 15, 30, 45, and 60 minutes for the adsorption process, as depicted in Figure 4.

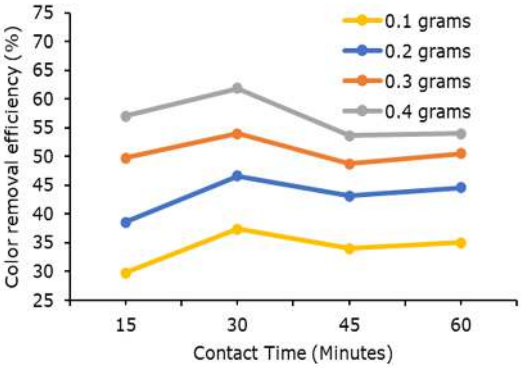

Figure 4. Contact Time Effect with HCl Activator

Based on Figure 4, the optimum contact time, as indicated by the graph, is 30 minutes. The green curve (0.4 grams mass) demonstrates a removal efficiency of 60.98% at the optimum contact time of 30 minutes. The yellow curve (0.3 grams mass) exhibits a removal efficiency of 53.51% at the optimum contact time of 30 minutes. The red curve (0.2 grams mass) shows an efficiency of 46.17% at the optimum time of 30 minutes, while the blue curve (0.1 grams mass) has a removal efficiency of 37.19% at the same optimum time. Based on these results, the optimum contact time of 30 minutes occurs because the initial adsorption process happens rapidly until this optimum time, as the pores on the biosorbent are still empty. Therefore, color molecules adhere and get adsorbed onto the empty surface, forming a layer. Subsequently, the adsorbent experiences a reduction in color absorption capability, and simultaneously, there is a release or desorption of color molecules from the biosorbent. The time after 30 minutes represents saturation, where the biosorbent undergoes no significant changes and tends to experience desorption, releasing the adsorbed color molecules due to saturation.

Based on Figure 5, various contact times were applied for 15, 30, 45, and 60 minutes in the adsorption process of batik wastewater. From the graph depicting the influence of contact time, it is observed that as the contact time increases, the amount of absorbed color also increases. At a 15-minute contact time, the color removal efficiency reaches its highest peak at 63.42% with a mass variation of 0.4 grams. At a 30-minute contact time, the highest efficiency reaches 69.45% with a mass of 0.4 grams. A 45-minute contact time achieves the highest efficiency at 65.93%. For a mass of 0.4 grams and a 60-minute contact time, the highest removal efficiency is 59.10%. The highest efficiency occurs at a biosorbent mass of 0.1 gram with a 45-minute contact time before reaching equilibrium.

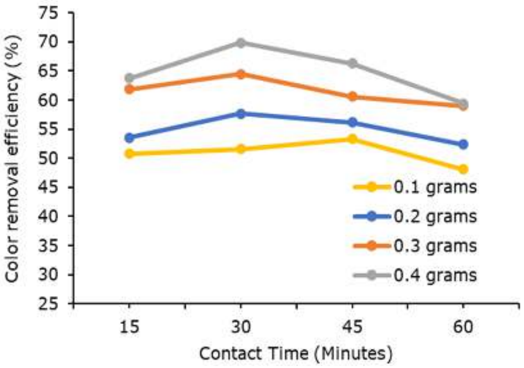

Figure 5. Contact Time Effect with KOH Activator

Equilibrium occurs when the adsorption rate equals the desorption rate, and in this study, it is achieved at a 30-minute contact time. The data indicates that the longer the contact time, the higher the amount of color adsorbed until reaching equilibrium, where the adsorbent reaches saturation.

3.4. Activator Type Effect

Adsorption using KOH as the activator exhibits the highest efficiency at a mass variation of 0.4 grams, reaching 69.46%, whereas adsorption using HCl as the activator achieves the highest efficiency at a mass variation of 0.4 grams, which is 60.98%. The results demonstrate a consistent increase in efficiency with an increase in biosorbent mass, aligning with previous studies conducted by Ahmad et al., (2018). They explain that an increase in adsorbent mass can enhance the adsorbate absorption capacity due to the availability of a larger surface area and more active sites at higher doses. This corresponds to research conducted by Ilmi et al. (2017) indicating that the specific surface area of KOH-activated adsorbent is 1.455 x 10^{-5} m^2/gram, while the specific surface area of HCl-activated adsorbent is 0.45 x 10^{-5} m^2/gram. Based on this explanation, it can be observed that KOH as the activator has a larger specific surface area compared to HCl, resulting in greater adsorption efficiency for the KOH-activated adsorbent. Applying an acid activator, specifically hydrochloric acid (HCl), augments the positive charge density on the surface, whereas the base activator, potassium hydroxide (KOH), elevates the negative charge density on the biosorbent material. This modification in surface charge properties is considerably advantageous in the adsorption of contaminants present in batik wastewater. Biosorbents enhance their negative charge and facilitate a more efficient ion exchange process, which is crucial for the sequestration of heavy metal cations such as copper (Cu), zinc (Zn), and nickel (Ni). These cations are prevalent in the synthetic dyes utilized within batik manufacturing processes. Hence, an increase in the adsorbent's negative charge directly correlates with an augmentation in the efficacy of color removal from batik wastewater, thereby representing a pivotal advancement in treating industrial effluents (Oginawati et al., 2021 and Mariana et al., 2022).

Adsorption using HCl and KOH as activators shares a similarity in the optimum contact time. The obtained optimum contact time is at the 30-minute variation. HCl activator achieves the highest removal efficiency at a mass variation of 0.4 grams, with a removal percentage of 60.98%, while KOH activator attains a removal percentage of 69.46%.

3.5. Statistical Analysis

The research conducted a multiple linear regression test to analyze the relationship between the independent variables (biosorbent mass, contact time, type of activator) and the dependent variable (color removal efficiency). The results of the multiple linear regression are presented in Table 1.

Before conducting the multiple regression analysis and hypothesis testing, several classic assumption tests were carried out to ensure that the regression model used is free from deviations in assumptions and meets the requirements for good linearity. The classic assumption tests carried out on this research data are the residual normality, multicollinearity, and heteroscedasticity. The results of the classic assumption tests are presented in Table 2 and 3.

Table 2 shows that the VIF (variance inflation factor) value is close to 1 for all independent variables. Thus, it can be concluded that in the regression between the independent variables biosorbent mass, contact time, and type of activator on color removal efficiency (y), there was no multicollinearity between the independent variables.

Table 1. Multiple linier regresion result

Coeficient	**Estimate**	**p-value**	**Signif. codes**
Intercept	19.78917	4×10^{-7}	***
Biosorbent Mass	-0.03744	2.93×10^{-10}	***
Contact Time	61.60714	0.411	***
Activator Type	12.13060	7.14×10^{-9}	***

Residual standard error: 3.55 on 24 degrees of freedom
Multiple R-squared: 0.8838, Adjusted R-squared: 0.8693
F-statistic: 60.84 on 3 and 24 DF, p-value: 2.311×10^{-11}

Table 2. Multicollinearity test

Test type	**Data Variables**	**VIF**
Multicollinearity Test	Biosorbent Mass	1.00
	Contact Time	1.061
	Activator Type	1.061

Table 3. Heteroscedasticity and normality test

Test type	**P-value**
Heteroscedasticity Test	0.6683
Normality Tes	0.5401

In this study, the heteroscedasticity test was conducted using the Breusch-Pagan-Godfrey test. Based on the results of the test, with a p-value of 0.6683, which is greater than the significance level of 0.05, we accept the null hypothesis (H0). This means that there is no heteroscedasticity, and the assumption of homogeneity of error variance is supported. The normality test for the classical assumptions of the linear regression model is based on the normality of the residual values. This test aims to determine whether the residual values in the regression equation follow a normal distribution. We conducted the residual value normality test using the Shapiro-Wilk test in our analysis. According to the Shapiro-Wilk test, the obtained p-value (0.5401) is greater than the significance level (0.05), leading to the acceptance of the null hypothesis (H0). Therefore, it can be concluded that the residual data values satisfy the normality assumption test as they are normally distributed.

Based on the classical assumption test conducted, it can be concluded that the research data meets the criteria for the classical assumption test. Therefore, the multiple linear regression model obtained is a good representation of the data in this research. The p-value (2.311×10^{-11}) is less than the ɑ (0.05), so the null hypothesis (H0) is rejected. This means there is a significant effect between the independent variables (biosorbent mass, contact time, and activator) and the dependent variable (color removal efficiency) simultaneously. In the test, each variable shows a p-value of (2.93×10^{-10}) for biosorbent mass, (0.411) for contact time, and (7.14×10^{-9}) for activator type. This implies that the independent variables of biosorbent mass and type of activator significantly affect color removal efficiency, while contact time does not significantly affect color removal efficiency in the partial test.

4. Conclusion

Based on the research results, the highest color removal efficiency value in the adsorption process using HCL-activated biosorbent was 60.98%, while KOH-activated biosorbent was 69.46%. Using biosorbent from KOH-activated sugarcane bagasse has a higher average color removal efficiency value than HCL-activated biosorbent.

Based on statistical analysis carried out using a multiple linear regression model, it can be concluded that there is a significant effect between the independent variables (biosorbent mass, contact time, and activator) and the dependent variable (color removal efficiency) simultaneously. In the partial test, the independent variables of biosorbent mass and type of activator significantly affect color removal efficiency, but contact time does not significantly affect color removal efficiency.

Further research needs to be carried out, namely characterization of the biosorbent before and after use in the adsorption process so that we can determine the relationship between biosorbent mass, contact time, and type of activator on the characteristics of the biosorbent and its effectiveness on color removal in batik wastewater.

Author contribution statement

Conceptualisation, N.P.,; Formal analysis, F.B.S.,; Investigation, R.A.A.,; Project administration, U.Y.A.,; Supervision, A.M.K.,; Writing – original draft, C.S.B.,; Writing – review and editing, R.I.B..

Data availability statement

Available on request to the corresponding Author.

Declaration of Competing Interest

The author declares that there is no conflict of interest regarding the publication of this manuscript.

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