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The Influence of Solution Concentration and pH for Removal Indigo Carmine using Banana Stem as a Biosorbent

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Abstract
The textile industry is one sector that generates effluent containing a significant quantity of pollutants, including dyes, and thus has an effect on the environment. Synthetic dyes, which are toxic and difficult to degrade, are utilized extensively in the textile industry. Therefore, a new study utilizes banana stems as a cost-effective biosorbent to remove indigo carmine dye. This research aims to investigate the use of banana stem waste as a bisorobent in batch method to adsorption indigo carmine dye. The optimal concentration and pH values are 2 and 900 mg/L, respectively. The interaction between the indigo carmine dye and the banana stems is suggested by the FTIR analysis. The solution's indigo carmine dye may be adsorbed by banana stem.

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INTRODUCTION

Indonesia's rapidly expanding textile sector generates a substantial amount of waste. The textile industry generates trash through fabric melting processes, which have a detrimental impact on the aquatic environment mostly because of the utilization of textile dyes (Chong et al., 2023). Textile dyes are manufactured chemical compounds with an aromatic structure that are resistant to degradation. The effluent produced by this textile dye poses a hazardous threat to both human health and the environment. Indigo carmine dyes have significant toxicity, even when present in low concentrations (Hevira *et al*., 2020; Samchetshabam *et al*., 2017).

Indigo carmine is extensively utilized as a dye for jeans. The wastewater resulting from the production of polyester fiber and denim dyes contains a significant amount of indigo carmine dyes. The concentration of dye in the wastewater varies from 30%, without undergoing any treatment, and is subsequently disposed of in the garbage (Arenas et al., 2017). The presence of the indigo carmine dye leads to a substantial increase in the COD value of the water, hence disturbing the ecology within the water body (Sulistyowati et al., 2018). The structure of indigo carmine dye consists of four benzene rings with double bonds and two negatively charged sulphonated clusters. The application of these dyes can result in skin and eye irritation, permanent corneal damage, and gastrointestinal discomfort characterized by symptoms such as nausea, vomiting, and diarrhea (Kesraoui et al., 2017).

Various techniques have been employed to remove dyes from wastewater, including electrocoagulation (Secula et al., 2011), degradation (Ariguna et al., 2014), and membrane separation (Ahlawat et al., 2022). Biosorption is an alternate technique that utilizes agricultural waste as a biosorbent. The utilization of agricultural waste is prevalent due to its cost-effectiveness, eco-friendliness, accessibility, significant dye absorption capability, and absence of waste generation (Zein et al., 2020).

Several studies have utilized waste materials as biosorbents for the purpose of dye adsorption. For instance, tarp seeds were employed to remove Rhodamine B dye (Lim et al., 2017), pensi shell were used to remove Metanil Yellow dye (Zein et al., 2019), shrimp shell waste was utilized to adsorption Metanil Yellow dye (Ramadhani et al., 2020), chicken eggshell wastes were employed for the adsorption of Indigo Carmine dye (Hevira, *et al*., 2020), and Carbon baggase for Adsorption Metilen Blue (Hatimah et al., 2022). The mentioned biosorbent is a limited source of waste. Indonesia possesses a varied range of plentiful agricultural waste that has the potential to serve as a biosorbent for color absorption. An example of such a plant is the banana plant, which yields a staggering nine million tons per year (BPS, 2022).

Approximately 60% of the waste from banana plants consists of banana stems, which have the potential to be utilized as biosorbents. Banana stems contain cellulose (43.3%), hemicellulose (20.6%), and lignin (27.8%) (Misran et al., 2022). This chemical has functional groups like hydroxyl, carbonyl, and others that are what make it work to absorb cation and anion color pollutants (Zein, *et al.,* 2022). Prior studies have established that bananas comprise metal oxides as well as function groups, including hydroxyl, carboxyl, carbonyl, and others, which are capable of interacting with cationic dye molecules (Zein et al., 2023). Hence, the present study employed a batch method to investigate the capacity of banana stems as biosorbents for the adsorption of anionic dye (indigo carmine) Banana stem was activated using HNO_3 and optimization was done by studying parameters in the adsorption process such as pH and the initial concentration of the indigo carmine dyes.

METHOD

Chemical and Apparatus

Banana stem (BP) was collected from Padang, West Sumatera, Indonesia, Indigo Carmine, Aquades, HNO_3 p.a (Merck), NaOH technical (Merck), Citrate Acid ($C_6H_8O_7$) (Merck), Hydrated Sodium Citrate ($C_6H_5Na_3O_7.2H_2O$) (Merck), Acetate Acids (CH_3COOH) (Merck), Natrium acetate (CH_3COONa) (Merck), Pospat monobasic sodium (NaH_2PO_4) (Merck), Dibasic Pospat Natrium (Na_2HPO_4)(Merck) and shell paper.

The apparatus were an analytical balance (Kern & Sohn GmbH), pH meter (Metrohm), rotary shaker (Edmun Buhler 7400 Tubingen), oven (Memmert, Germany), crusher (Fritsch, Germany), mortar grinding (Fritsch, Germany) and UV-Vis spectrophotometer (Genesys 20 Thermo Scientific. Several analytical instruments were used for characterization of BS biosorbent before and after adsorption such as Fourier transform infrared (FTIR) spectrophotometer (Unican Mattson Mod 7000 FTIR, United States), Thermogravimetric analysis (TGA, DTG-60 Simultaneous DTA-TG Apparatus Shimadzu, Japan), and X-Ray fluorescence spectroscopy (XRF, PANalytical Epsilon 3, Netherlands).

Biosorbent Preparation

Banana Stems (BP) are washed with water, dried at room temperature, peeled. Then stirred ($\leq 36 \mu$m) and obtained banana stem powder. 25 g of banana stem powder is soaked with HNO_3 0.01 M for 3 hours. After that it is washed with aquades to neutral pH and then dried (Zein et al., 2023).

Preparation of Indigo Carmine Solution

Indigo Carmine dye is prepared by weighing 0.1000 g of the indigo carmine dye dissolved in 100 mL of aquades using 100 mL of pumpkins measure. Indigo carmine dye is diluted into several concentration which is then used as a standard solution with varying concentration (10 – 1000 mg/L) (Hevira, et al., 2020)

Determination of pH point of zero charge (pH_{pzc})

To determine pH_{pzc}, the initial step involves altering the pH of a solution containing 0.1 M KCl within the pH range of 2.0 to 9.0 (pH_i). This adjustment is achieved by adding either 0.1 M HNO_3 or NaOH. After that, 0.1 g of the biosorbent BP was put into 50 mL of each solution, and the resulting mixture was agitated on a shaker for a duration of 24 hours. Following that, the pH_f of the supernatant was determined. The plot was created to show the relationship between the change in pH (ΔpH) and the initial pH. The pH_{pzc} value of the biosorbent was determined by finding the point of intersection (at ΔpH=0) on this curve (Ramadhani et al., 2020).

Batch biosorption studies

The biosorption examination was conducted using a batch system, with various parameters including pH (from 1 to 5), and initial concentration (from 100 to 1000 mg L^{-1}). The BP was utilised in batch experiments conducted in 100 mL Erlenmeyer conical flasks containing 10 mL of a known concentration indigo carmine dye solution. The flasks were rotary shaken at 100 rpm for a specific amount of time, after which the mixture underwent filtration. The dye concentration upon adsorption was measured using a spectrophotometer at a wavelength of 609 nm (Hevira et al., 2021). The equilibrium adsorption capacity qe (mg g-1) was determined using equation (1).

$$q = \frac{(C_0 - C_e)V}{m} \quad (1)$$

To determine the percentage of dye removal, the following equation was used:

$$\%R = \frac{(C_0 - C_e)}{(C_e)}\ x\ 100 \quad (2)$$

where C_0 and C_e were initial and equilibrium dyes concentration in solutions (mg L^{-1}), respectively; V was volume of the solution (L); m was the amount of biomass (g) (Zein et al., 2022)

Isotherm studies

The experimental data was obtained to investigate the Langmuir and Freundlich isotherm models at the initial concentration parameter. Equations 3 and 4 introduced the linearized Langmuir and Freundlich isotherm models, respectively.

$$\frac{1}{q_e} = \frac{1}{k_L q_m C_e} + \frac{1}{q_m} \quad (3)$$

$$\log q_e = \log K_f + \frac{1}{n} \log C_e \quad (4)$$

The variables in the equation are as follows: q_m represents the maximum monolayer adsorption capacity of the adsorbent (as measured in mg g^{-1}), K_L is the Langmuir adsorption constant (measured in L mg^{-1}), q_e represents the concentration of adsorbate on the adsorbent at equilibrium (measured in mg g^{-1}), C_e reflects the concentration of adsorbate in the solution at equilibrium (measured in mg L^{-1}), and K_f and n are the Freundlich constants (Kekes & Tzia, 2020; Singh et al., 2022).

RESULTS AND DISCUSSION

Characterization of Biosorbent

FTIR (Fourier Transform Infrared)

The functional group characterization of BP was determined through FTIR (Fourier Transform Infrared) in the range of 4000 - 400 cm^{-1} In Figure 1.

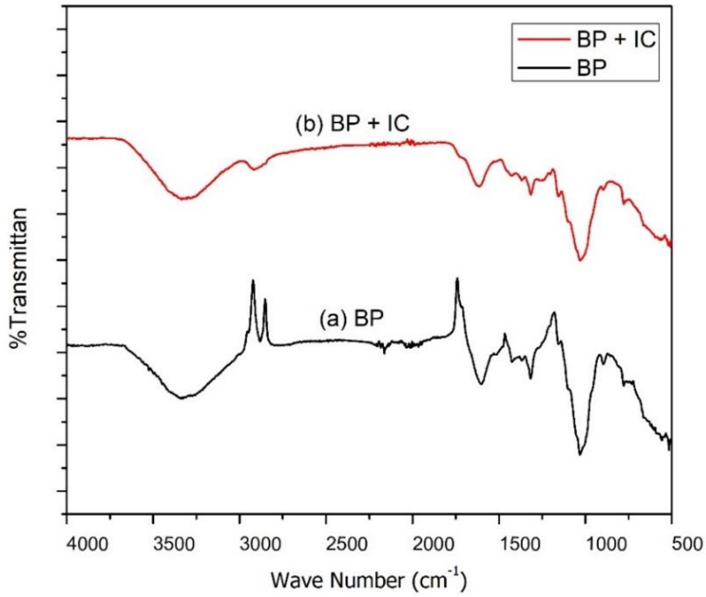


Figure 1. FTIR Spectrum of BP before and after adsorption IC dye

Figure 1 provides data about the spectrum of several functional groups at related wave numbers. These include the O-H functional group at 3327 cm^{-1}, the C-H functional group at 2881 cm^{-1}, the C=C functional group at 1600 cm^{-1}, the C-H functional group of CH_3 at 1425 cm^{-1}, the C-N functional group at 1317 cm^{-1}, and the C-O functional groups at 1031 cm^{-1}. The existence of many peaks correlating to distinct functional groups implies the intricate characteristics of the biosorbent BP. The O-H group is obtained from organic substances, such as cellulose, hemicellulose, and lignin, which are present in the BP. Following the absorption of indigo colourant by carmine BP, there is a change in the wavelength of the functional groups. This indicates a modification in the binding energy of the functional cluster due to the interaction between the adsorbent and the adsorbent. In addition, there is a newly detected peak that signifies the existence of indigo carmine dye on the surface of BP (Dahiru et al., 2018; Zein et al., 2023).

Figure 1 illustrates the shifting of certain functional groups following the adsorption process of indigo carmine dye. The O-H functional group had a slight shift from 3327 cm^{-1} to 3334 cm^{-1}. This suggests that there is an interaction between BP and the indigo carmine dye. The C-H frequencies transition from 2881 cm^{-1} to 2916 cm^{-1}, indicating the occurrence of curved vibration in the CH_3 group. The presence of indigo carmine dye is indicated by the appearance of the S=O functional group at the wave number of 1155 cm^{-1} (Agorku *et al*., 2015; Hevira, *et al*., 2020).

X-ray Fluorescene (XRF) analysis of BP

XRF analysis is performed to determine the chemical composition of biosorbents both before and after the adsorption of indigo carmine dye. The purpose of this study is to classify the element type and ascertain the composition of the material observed in Table 1.

Table 1. Chemical composition of BP before and after adsorption process of IC dyes

Compounds	Before biosorption (%)	After Biosorption (%)
Mg	0.12	0.05
Si	0.71	1.34
Ca	6.6	15.23
S	0.07	0.14

Table 1 demonstrates that BP primarily consists of calcium (Ca) and calcium oxide (CaO). Following the adsorption process of indigo carmine dye on the biosorbent BP, it is evident that there is a notable increase in the percentage of certain elements, including Si, Ca, and S. This outcome suggests that there is an assimilation process occuring, leading to higher percentages of the element S, which is a constituent of the indigo carmine dye's structure. The adsorption of indigo carmine dye using BP takes place at a pH of 2, with BP being considered to carry a positive charge. This assumption is predicated on the pH value being greater than the pH at the point of zero charge (pH_{pzc}), in which case the boiling point (BP) would be positive. The absorption of basic metal oxides like MgO would decrease as a result of ion exchange between H+ ions introduced by the addition of acid to the solution and Mg^{+2} ions of BP (Hevira, *et al*., 2020; Musić *et al*., 2011).

Analysis of point of zero charge (pH_{pzc})

The pH value where the total charge on the biosorbent surface is zero and the total charge on the positive surface site is equal to the negative charge is known as the pH point of zero charge, or pH_{pzc}. At pH values greater than the point of zero charge (pH_{pzc}) $pH>pH_{pzc}$, the surface carries a negative charge, while at pH values lower than pH_{pzc} ($pH<pH_{pzc}$), the surface carries a positive charge (Kragović et al., 2019). Figure 2 displays the outcomes of the BP pHpzc measurement.

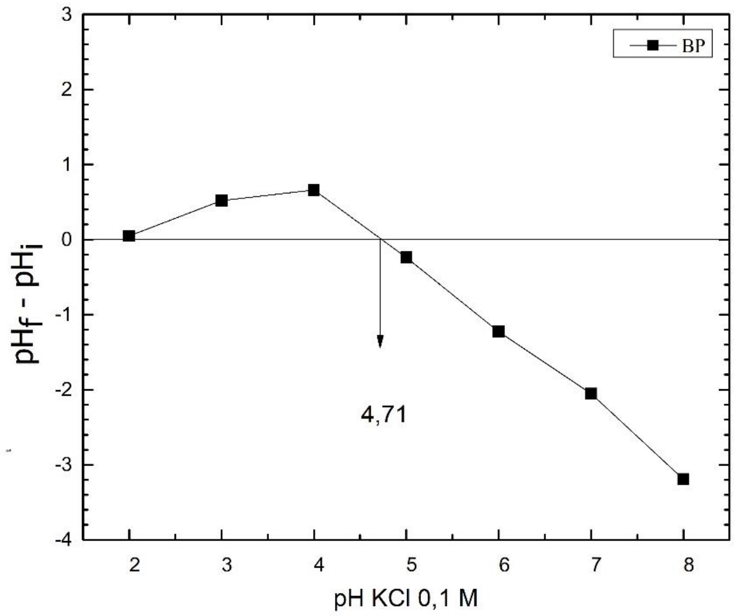


Figure 2. pHpzc for BP

Figure 2 illustrates that the BP has a pH_{pzc} (point of zero charge) value of 4.71. At this certain value, the BP surface demonstrates a state of neutrality, lacking either positive or negative charges. The adsorption of indigo carmine dye becomes difficult when the pH is above the point of zero charge (pH_{pzc}). The existence of a negative charge on the biosorbent material's surface results in an electrostatic hindrance between the biosorbent surface and the indigo carmine dye molecule. Below the pH_{pzc}, the surface undergoes protonation, resulting in a positive charge. This positive charge allows the surface to efficiently adsorb anionic dye. Given these conditions, the adsorption process occurs with optimal effectiveness because of the positively charged surface of the banana stem. This leads to a strong electrostatic attraction between the surface of biosorbent and the indigo carmine dye (Basharat et al., 2020).. Moreover, the presence of sulphonate groups in indigo carmine dye indicates the anionic properties of indigo carmine dye in acidic conditions. The pH_{pzc} result determines the adsorption process of indigo carmine dye utilising the biosorbent BP. The process can be conducted under acidic conditions, namely within the pH range of 1-5, to determine the optimal pH state for the process. In a study conducted by Stavrinou (2018), banana leather residues were investigated as biosorbents for optimal adsorption of the Orange G colouring ingredient at a pH lower than the pHpzc (6.54). According to Stavrinou et al. (2018), the study determined that the most favourable pH level for adsorption was pH 2 (Stavrinou et al., 2018).

Effect of pH on adsorption capacity of IC dye

The adsorption of indigo carmine dye is significantly influenced by the pH level. The degree of ionization of the dye molecule, the electrostatic interaction between the surface of the biomolecule and the indigo carmine dye, and the characteristics of the biosorbent surface are all determined by the pH of the solution. The biosorbent surface may absorb indigo carmine dye optimally because it is positively charged at pH<pHpc, as indicated by a pHpzc of 4.71 of BP (Zein et al., 2019; Zein, et al., 2023). The pH range of 1 to 5, as shown in Figure 3, was examined in this work to determine how the solution's pH affected the indigo carmine dye adsorption process.

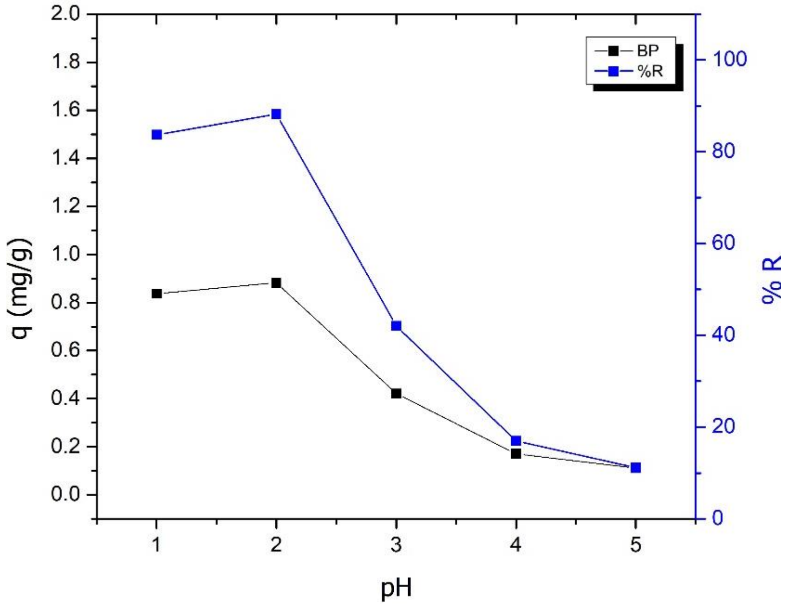


Figure 3. Effect of pH on the adsorption capacity of IC dye (C_0: 10 mg L^{-1} ; biosorbent mass: 0,1 gram; contact time: 60 minutes ; adsorption temperature: 25 ℃ ; volume: 10 mL; particle size: 36 μm ; and 100 rpm)

As may be observed from Figure 3, the capacity for adsorption is at pH 2. The adsorption capacity result rises from 0.8372 to 0.8818 mg/g under pH 1–2 conditions. The adsorption capacity dropped from 0.4203 to 0.1116 mg/g at pH 3 to 5 at higher pH levels (pH>2). This is due to the fact that the electrostatic interaction between the dye molecules and the biosorbent surface is significantly influenced by the pH of the solution. The adsorption capacity rises as a result of the increased electrostatic attraction between H^+ ions and IC anions at low pH levels. Higher pH levels cause a rise in OH^- ions, which causes OH^- ions and indigo carmine dye to compete with one another for the biosorbent's active site. As a result, the absorption capacity decreases due to an electrostatic attraction resistance between the BP biosorbent and the indigo carmine dye on the biosorbent's surface (Febriani et al., 2022; Zein et al., 2019).

The optimum pH value that was attained was 2, and this pH value matches the pH_{pzc} value (4.71). There are more proton ions available to help H^+ ions become positively charged on the biosorbent's surface in BP at pH <pH_{pzc}, which can enhance the electrostatic interaction between the negatively charged indigo carmine dye and the positively charged biosorbent site. Moreover, two aromatic rings and two sulfonic acid groups give indigo carmine dye its great removal effectiveness at low pH (Kuete et al., 2022). Harrache (2019) reported the same results in a study that used activated carbon to find the best pH 2 adsorption of indigo carmine dye (Harrache et al., 2019).

Effect of the initial concentration of the solution on the adsorption capacity of the indigo carmine dye and equilibrium studies

The initial concentration of indigo carmine dye is a crucial factor in influencing the adsorption capacity of carmine indigo dye since it is linked to the solid-liquid adsorption process. The concentration of indigo carmine dye regulates the interaction between adsorbents and adsorbed substances by influencing the transfer of color molecules between liquid and solid phases (Almeida et al., 2017). The effect of the initial concentration of indigo carmine dye was carried out at a concentration of 20 – 1100 mg/L at pH 2. The result of the determination can be seen in Figure 4.

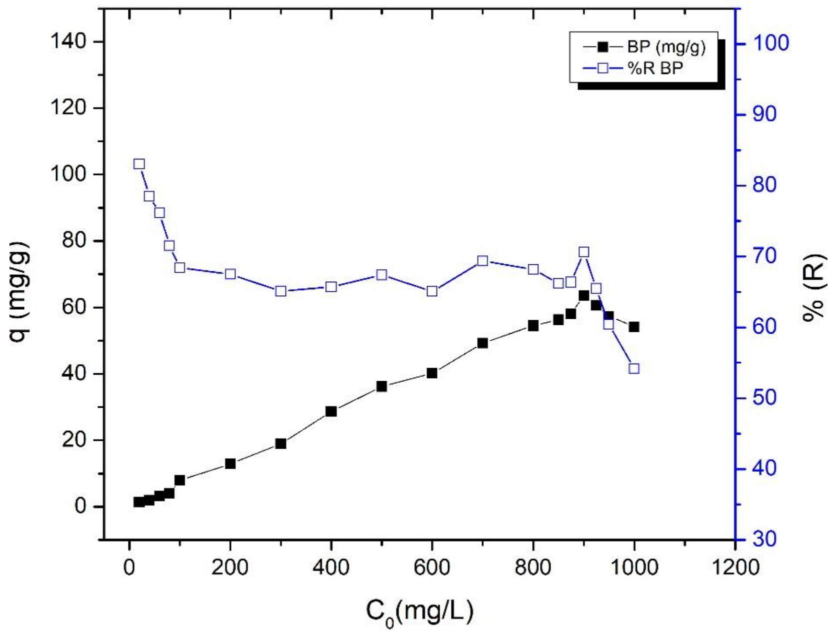
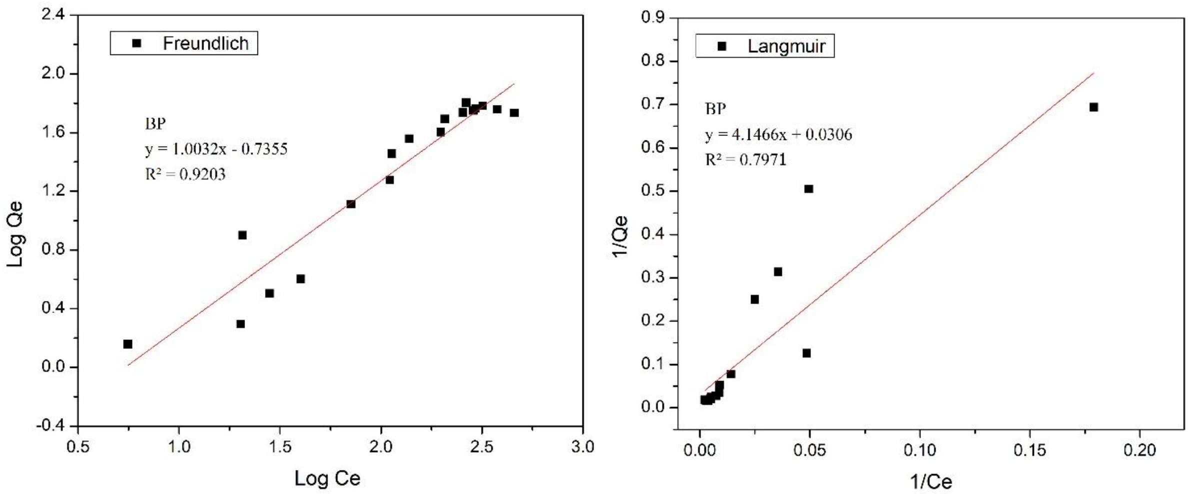


Figure 5. Effect of IC dye initial concentration on adsorption capacity (pH: 2 ; biosorbent mass: 0.1 gram ; contact time: 60 minutes ; adsorption temperature: 25 ℃ ; volume: 10 mL ; particle size: 36 μm ; and 100 rpm)

Figure 6 demonstrates that the optimal concentration of indigo carmine dye for the BP biosorbent is between 100 mg/L and 900 mg/L. At this concentration range, the absorption capacity of the biosorbent increases from 7.94 mg/g to 63.54 mg/g, with a corresponding removal rate of 70.60%. This demonstrates that augmenting the initial concentration of indigo carmine dye can enhance the absorption capability of the BP biosorbent for indigo carmine dye until it reaches the point of equilibrium. As the quantity of indigo carmine dye increases, more active sites on the biosorbent surface will become fully occupied by indigo carmine dye molecules. The loss of binding ability of indigo carmine dye in the BP biosorbent occurs when the active surface of the biosorbent can no longer bind further molecules of the dye. At concentrations above the optimal level, the absorption capacity of indigo carmine dye fell from 63.54 mg/g to 54.19 mg/g when the initial concentration was 1000 mg/L. Under these circumstances, the affinity between the indigo carmine dye and the BP biosorbent will be hindered due to the saturation of active sites on the biosorbent's surface. Consequently, the biosorbent becomes incapable of accommodating the indigo carmine dye molecules (Batu et al., 2023; Hevira, Zilfa, et al., 2020; Maghri et al., 2012).

Concentration is influenced by the abundance of active sites on the biosorbent's surface, as well as the rising concentration of the indigo carmine dye solution. Furthermore, the determination of concentration is necessary for establishing the adsorption isotherm in investigations on adsorption (Kumar & Raju, 2020). The isotherm model of IC dye adsorption was evaluated using the initial concentration impact. How adsorbent and adsorbate interact may be explained using the adsorption isotherm model. In this study, the Freundlich and Langmuir isotherms were employed. The equilibrium between the adsorbent and the adsorbate, where a monolayer developed at homogenous locations throughout the adsorption process, was described by the Langmuir isotherm model. According to the Freundlich isotherm model, a molecule interaction followed a multilayer adsorption mechanism with a heterogeneous energy distribution from the active site (GULYUZ, 2021) The results were displayed in Figure 6



(a) (b)

Figure 6. Isotherm model graph Freundlich (a) Langmuir (b) of BS-QEW

The Freundlich isotherm model was observed to be closely matched with the IC dye adsorption using BS-QEW, as shown in Figure S2 (R^2 = 0.9731). Adsorption occurred during the formation of multilayers, as predicted by the Freundlich isotherm model. The Freundlich isotherm model predicted that the energy distribution at the active site on the biosorbent surface was heterogeneous Öztürk e*t al*., 2020).

CONCLUSION

As an adsorbent, banana stems were effective at removing indigo carmine dye from effluent. It was a cost-effective and environmentally benign adsorbent. The maximum adsorption capacity was 63.54 mg g^{-1}. The adsorption procedure complied to the Freundlich models of isotherms. This demonstrated that banana stems could be produced easily, affordably, and sustainably as an adsorbent. The research findings demonstrated that banana stems can be utilized as biosorbents for the adsorption of anionic dyes. This investigation aims to contribute to the body of knowledge and serve as a benchmark for other studies concerning the adsorption of pollutant waste from the textile industry.

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