Optimization Study on Adsorption of Methylene Blue from Coating Industry Wastewater by Sugarcane Bagasse Biochar

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Abstract

This study introduces an optimization investigation utilizing Response Surface Methodology (RSM) to enhance the adsorption capacity of methylene blue (MB) from wastewater generated by the coating industry. The adsorbent of interest in this research is sugarcane bagasse biochar, recognized for its potential in wastewater treatment due to its abundant availability and favourable adsorption characteristics. A Box-Behnken was used to design the experimental matrix, considering factors such as initial dilution factor (3.2 - 20 %), temperature (25 – 40 °C), and contact time (1 – 20 hours). Through the execution of the designed experiments, a predictive model was developed to correlate the significant factors and their interactions with MB removal. Furthermore, the physicochemical properties of the produced biochar were investigated through several characterization techniques. The optimization process yielded the following optimum conditions for the adsorption process: a dilution factor of 16.27%, a temperature of 40°C, and a contact time of 1 hour. An in-depth exploration of the physicochemical properties of the produced biochar was conducted, with insights into the functional groups responsible for MB adsorption provided through Fourier Transform Infrared Spectroscopy (FTIR) analysis. Additionally, Scanning Electron Microscopy (SEM) was employed to examine the surface morphology and potential binding sites on the biochar, while particle size analysis assessed the difference in particle size before and after pyrolysis of sugarcane bagasse. The results of this study contribute to the understanding of optimizing sugarcane bagasse biochar for effective removal of methylene blue from coating industry wastewater. The application of RSM allowed the determination of optimal conditions that enhance adsorption efficiency, while the characterization techniques (FTIR, SEM, and particle size analysis) reveal the properties that guide the adsorption process. This research underscores the potential of utilizing agricultural waste-derived biochar as a sustainable and efficient adsorbent for the treatment of industrial wastewater contaminated with dye pollutants.

Keywords: Response Surface Methodology, Methylene Blue, Activated carbon, Waste management

INTRODUCTION

Environmental Concerns and Methylene Blue as a Pollutant

In recent years, environmental concerns and the need for sustainable waste management practices have gained unprecedented attention across the globe. Industrial processes have produced significant quantities of effluent containing aromatic substances, heavy metals, colorants, and hazardous contaminants, posing challenges for traditional wastewater treatment techniques to effectively remove. (Elshabrawy, Elhussieny, Taha, Pal, & Fahim, 2023). Among these pollutants, Methylene Blue dye (MB) stands out as a water-soluble azo dye known for its intense color and slow rate of degradation (Rizkiana et al., 2023). When released into water, it gives rise to ammonium cations and can result in notable environmental issues even at minimal levels (C. Zhu et al., 2018). Consequently, effective treatment methods must be employed to eliminate water contamination before it enters water bodies.

Adsorption: A Key Technique for Dye Removal

The predominant methods for wastewater treatment encompass physical, biological, and chemical techniques (L.N et al., 2013). Of these techniques, the process of adsorption stands out as a viable approach for eliminating dye substances. This process employs adsorbents, small porous materials capable of attracting target contaminants (Hynes et al., 2020). Activated carbon, which is a carbon-based adsorbent, is produced from materials such as biomass, waste from biomass, or industrial waste, owing to the ready availability of source materials and the simplicity of manufacturing processes. Crucial factors that affect the adsorption capability and the interplay between Methylene Blue (MB) and activated carbon encompass parameters like surface area, pore configuration, particle dimensions, surface acidity, and the functional attributes of the activated carbon (Santoso et al., 2020). Activated carbon fabrication typically involves two stages: carbonization and activation, which can be achieved through thermal or chemical means to enhance surface functionality.

Sugarcane Bagasse: A Sustainable Solution for Methylene Blue Removal

Sugarcane bagasse (SB) pulp, a byproduct of sugar extraction from sugarcane bagasse, is abundantly available, especially as the paper industry faces a decline due to digitalization trends such as digital book and online education, and this surplus of bagasse pulp may become a waste to the environment if not properly handled (Rizkiana et al., 2023). Characterized by its cellulosic structure, availability, and cost-effectiveness, sugarcane bagasse has been explored for its potential in MB removal through adsorption. Remarkable results were reported with citrate-modified sugarcane bagasse pulp, achieving an impressive peak removal efficiency of 92.9% (Mpatani et al., 2020). Furthermore, activated carbon derived from bagasse pulp has shown promising results in MB removal (Li, Yan, Liu, & Liu, 2016). These results highlight the promise of employing sugarcane bagasse pulp and its derived materials as efficient adsorbents for Methylene Blue (MB) elimination, coupled with the added benefit of enhanced mechanical characteristics in comparison to unprocessed sugarcane bagasse.

Assessing Sugarcane Bagasse Biochar for Wastewater Treatment

This study’s primary objective is to assess the adsorption capabilities of sugarcane bagasse biochar (SBB) on coating industry wastewater. The study centers around examining how process variables impact the outcome, specifically wastewater dilution factor, adsorption contact time, and temperature, on MB adsorption capacity. The impacts of these operational factors and the enhancement of the adsorption procedure are assessed through the utilization of Design Expert software, making use of Response Surface Methodology (RSM) as a guiding tool. RSM, a statistical and mathematical technique, scrutinizes how diverse independent factors influence a dependent variable. Its value lies in diminishing the need for extensive experimentation, which includes the reduction of chemical consumption, time, expenses, and the use of costly analytical processes (Afolabi et al., 2021).

LITERATURE REVIEW

The untreated sugarcane bagasse shows limited adsorption capacity, but diverse modifications significantly enhance its ability to remove contaminants like dyes, heavy metals, and pesticides from wastewater, boosting both its adsorption capacity and efficiency (Aruna et al., 2021). Saini et al. (2022) conducted research on the utilization of biochar obtained from sugarcane bagasse for the adsorption-based elimination of phenol from an aqueous solution. They found that sugarcane bagasse pyrolyzed at 600°C showed maximum phenol removal efficiency at 74.2% and 96.1% if the biochar was treated with HNO3-. In a separate investigation, Vyavahare et al. (2018) explored the adsorption and detoxification of malachite green (MG) dye by employing sugarcane bagasse biochar produced through pyrolysis at 800°C, and the biochar exhibited the highest MG dye adsorption capacity at 3000 mg/L. Chemically altered biochar derived from sugarcane bagasse was utilized for eliminating nitrate from an aqueous solution, resulting in a maximum adsorption capacity of 28.21 mg per gram. (Divband Hafshejani et al., 2016). Meanwhile, novel ball-milled sugarcane bagasse biochar adsorbent achieved a capacity of 354mg/g of aqueous methylene blue removal (Lyu et al., 2018). Various modifications have highlighted the exceptional versatility of sugarcane bagasse as an effective adsorbent, showing how tailored treatments significantly enhance its adsorption capacity and make it a promising, sustainable, and eco-friendly solution for efficiently removing diverse contaminants from wastewater.

In this study, the adsorption of industrial wastewater rich in methylene blue was optimized using response surface methodology. In recent studies, Rizkiana et al. (2023)optimized MB removal using magnetic activated carbon derived from sugarcane bagasse through a microwave synthesis method, with ultrasound assistance. The study identified the optimum parameters for the adsorption process as follows: an ultrasonic power of 155.65 W, sonication period of 57.81 minutes and an adsorbate concentration of 89.77 mg/L,. Tran et al. (2021) studied the adsorption of their chemically modified porous carbon (CPMC) from sugarcane bagasse pyrolyzed at 600°C on MB. Their research revealed that an optimal MB concentration of 224 mg/L, a CPMC amount of 1.45 g/L, and a pH level of 9.5 resulted in an impressive 90% MB removal efficiency, demonstrating an adsorption capacity of 184.82 mg/g. An optimization study in the adsorption of methylene blue is crucial because it helps identify the most effective conditions and parameters for maximizing the efficiency of the process.

To the best of the authors' awareness, the scarcity of research dedicated to the optimization of methylene blue adsorption using modified sugarcane bagasse in authentic industrial wastewater makes this study a distinctive and valuable contribution to the field. Ezeonuegbu et al. (2021) investigated the adsorption of lead (Pb) and nickel (Ni) in untreated effluent samples from an untreated wastewater channel using sugarcane bagasse, achieving removal efficiencies of 89.31% for Pb and 96.33% for Ni. On the other hand, an adsorption study employing chitosan-modified sugarcane biochar for removing inorganic phosphate ions from real wastewater samples yielded a removal efficiency of 40.23% at pH 3 and 2.93% at pH 8 (Manyatshe et al., 2020). Multiple studies have emphasized the use of modified sugarcane bagasse for adsorbing Methylene Blue from laboratory-prepared solutions, rather than real industrial wastewater samples. (Rizkiana et al., 2023; Tran et al., 2021; Lyu et al., 2018).

METHODOLOGY

Chemicals

Methylene blue (MB) was procured from Merck (Germany). The aqueous dye solution for the calibration curve was created using methylene blue. Each solution was made with deionized water.

Preparation of Sugarcane Bagasse Biochar

The sugarcane bagasse pulp originated from a local micro-enterprise (Muar, Malaysia), which supplied it for the study. The sample was cleaned with demineralized water to remove any dirt. The bagasse was then dried under sunlight for 3 days. It was then cut into smaller sizes and then dried again in a laboratory oven to remove moisture at 70℃ for 2 hours. The samples were ground using a grinder and finely turned into powder using a cyclone mill (Retsch). The powder was transformed into sugarcane bagasse biochar (SBB) through heat treatment in a Vulcan 3-series furnace at 500℃ for a duration of 1 hour, prior to its utilization in the adsorption experiments.

Collection of Wastewater Samples

For this study, industrial wastewater rich in MB dye was generously supplied by HueCoating Resources Sdn Bhd, an outdoor sports coating company located in Seremban, Malaysia. The sample was stored at 4℃ and was used directly as stock solution. The triplicate of 50ml solutions with dilution factors of 3.2%, 11.6%, and 20% was prepared from the stock solution using the formula for serial dilution, $C1V1 = C2V2$, in volumetric flasks and were used in the adsorption experiments.

Characterization of Sugarcane Bagasse and Biochar

The morphology of SBB was investigated using a Scanning Electron Microscope (SEM) (ThermoFisher) with magnifications of 500x and 1000x. A Fourier Transform Infrared (FTIR) spectrometer from PerkinElmer was utilized for the analysis of chemical bonds and functional groups over a wavelength range spanning from 500 to 4000 cm^{-1}. Determination of particle size distributions of sugarcane bagasse powder and its biochar in aqueous suspensions were performed by using a laser diffraction particle size analyzer (Bettersize 2000).

Design Expert Software

In this study, three variables were regarded as independent factors: the wastewater dilution factor (A), contact time (B), and temperature (C). All these parameters were investigated across three different levels. The data obtained from the experimental runs were analyzed using Design Expert software version 13.0, employing the Box-Behnken Design (BBD) approach. The selection of the BBD aimed to identify the optimal, unknown point for the adsorption of MB by SBB. Table 1 outlines the scope of the independent variable values used in the experiments.

The absorbance of MB represented the system's response (dependent variable). Three-coded levels were employed for the variable process parameters.

Table 1: Three-coded levels for the variable process parameters used

Terms	Design variable	Units	Coded levels		
			-1	0	1
A	Dilution factor	%	3.2	11.6	20
B	Contact time	hour	1	10.5	20
C	Temperature	℃	25	32.5	40

Adsorption study

In the adsorption experiments, 0.9 grams of SBB were suspended in 50 mL of diluted working solutions, which ranged from 3.2% to 20%. Contact times were adjusted within the range of 1 to 20 hours, and temperatures varied from 25°C to 40°C, all within 250 mL stoppered conical flasks. These mixtures were agitated at 150 rpm using a thermostatted water bath shaker until equilibrium was reached. After each adsorption experiment, the sample underwent filtration, and the absorbance of the solution was measured at a wavelength of 665 nm using a UV-Vis Spectrophotometer (Agilent). A calibration curve was established using known concentrations of methylene blue dye, and absorbance was measured at the same wavelength. The percentage of methylene blue removal was calculated using equation 1.

$$\%removal = \frac{C_0 - C_t}{C_0} \times 100 \qquad \ldots\ldots[1]$$

Co denotes the initial concentration of the stock solution in milligrams per liter (mg/l), while C_t represents the concentration at a given time (mg/l).

DISCUSSION OF ANALYSIS AND FINDINGS

Fitting the RSM Model and Analysis of Variance (ANOVA)

From the response surface methodology (RSM), a total of 15 experiments were carried out and the quadratic model was suggested as the best fit for the study. Table 2 listed out the experimental and predicted values for the absorbance of methylene blue under three factors that affected the adsorption process. Fig. 1 depicts a graph for the experimental and predicted value of MB absorbance by UV analysis under different condition of independent variables. The absorbance prediction was derived from the experimental data provided which was expressed in terms of coded factors, as indicated by Equation (2).

$$Y = 0.3343 + 0.9003A + 0.1311B - 0.1082C + 0.2310AB - 0.0800AC + 0.2806BC + 0.6250A^2 + 0.1518B^2 - 0.0480C^2 \qquad \ldots..[2]$$

Table 2: Experimental and predicted values for the absorbance of methylene blue

Experiment No.	A	B	C	Absorbance	
				Experimental	Predicted
1	11.6	20	40	0.6854	0.7415
2	3.2	1.0	32.5	0.2425	0.3105
3	11.6	10.5	32.5	0.4044	0.3343
4	3.2	10.5	25.0	0.0511	0.0392
5	11.6	10.5	32.5	0.2539	0.3343
6	11.6	10.5	32.5	0.2731	0.3343
7	3.2	10.5	32.5	0.0508	-0.0172
8	20.0	1.0	32.5	1.66	1.65
9	11.6	10.6	32.5	0.4058	0.3343
10	20.0	10.5	32.5	1.93	2.00
11	20.0	10.5	40.0	1.61	1.62
12	20.0	20	32.5	2.44	2.37
13	11.6	1.0	25.0	0.7519	0.6957
14	3.2	20.0	32.5	0.0990	0.1109
15	11.6	20.0	25.0	0.3969	0.3969

A = Dilution factor; B = Temperature (°C); and C= Time (hour)

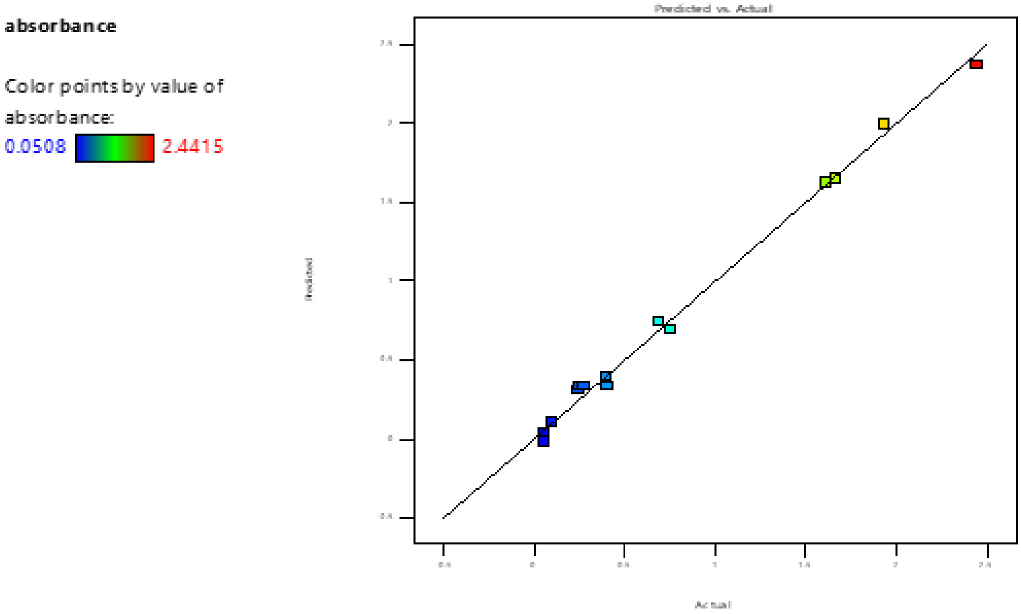


Figure 1: Predicted and experimental value of absorbance of methylene blue

Optimization Analysis

According to RSM graph, the optimum conditions for the process were identified as a dilution factor of 16.27%, a temperature of 40°C, and a contact time of one hour, resulting in an absorbance of UV at 0.44. In comparison to the absorbance of untreated wastewater (3.91), the optimum condition has successfully removed 90% of its methylene blue as displayed in Figure 2. According to study conducted by (Zhu et al. 2018), dilution factor has an impact on the adsorption of MB to biochar. In this study, the adsorption of MB to SBB increase as the initial concentration of MB in the wastewater increased (Figure 3). This is due to the increased possibility of MB molecules interacting with active sites on SBB surface and subsequently occupying all available sites. Raising the initial concentration of MB enhances the driving force, which in turn facilitates the migration of MB from the aqueous phase into the solid material. This resulting in an increased adsorption capacity.

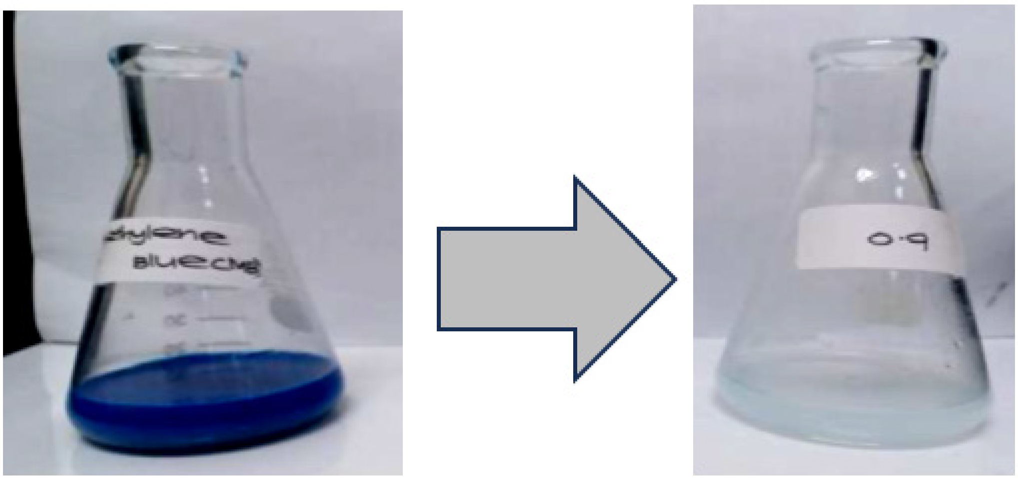


Figure 2: Colour changes of wastewater after adsorbed by SBB at optimal filtration condition

With increasing contact time, the concentration of MB was observed to rise, indicating the likelihood of competing compounds or ions within the solution competing for adsorption sites on the biochar. This phenomenon, as explored by (Lu et al., 2022) in their study, leads to a gradual displacement of methylene blue from the biochar, characterizing the process as desorption. Additionally, increasing in surrounding temperatures exert a beneficial influence on the adsorption process. This can be attributed to the amplified kinetic energy of the solution's molecules at elevated temperatures, facilitating a more pronounced interaction with the surface of the biochar. Lu et al. 2022 investigation highlights this intensified interaction, resulting in accelerated rates of adsorption.

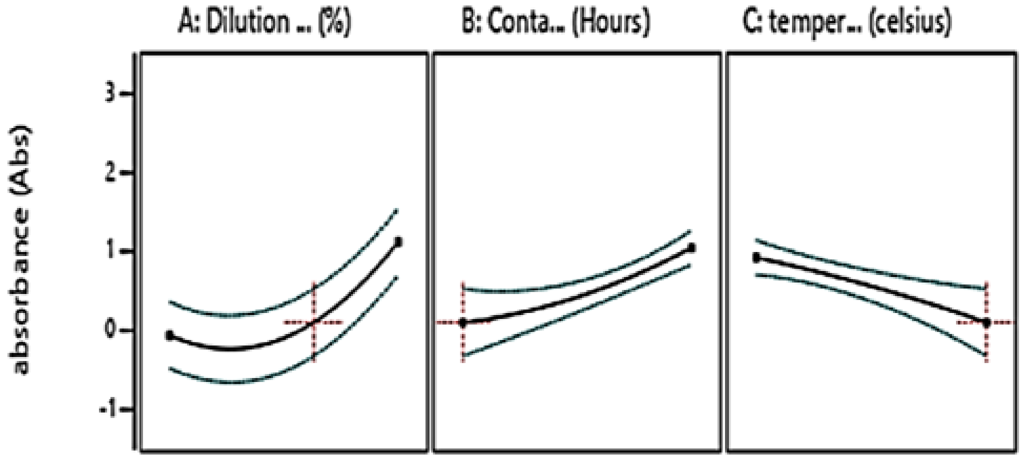
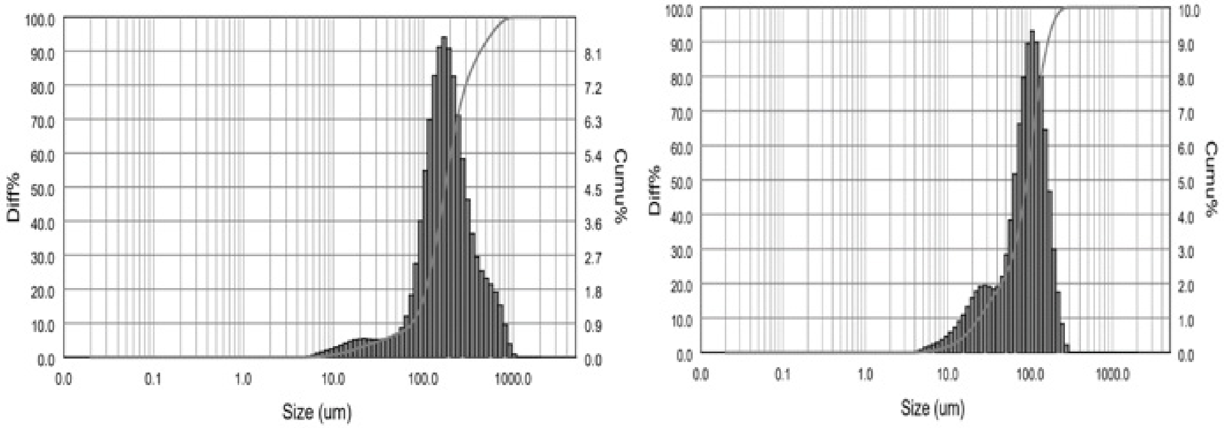


Figure 3: The effect of (A) dilution factor, (B) contact time and (C) temperature to the absorbance of methylene blue



(a) (b)

Figure 4: Particle size distribution of (a) SB and (b) SBB

Particle size distribution for SB and SBB is presented in Fig. 4. The particle size analysis of SB before pyrolysis indicates that sample sizes ranged from 10.0 µm to 1000.0 µm. The smallest observed diameter was 5 µm, constituting 0.01 % of the sample, while the largest observed diameter was 1000 µm, accounting for 16.82 %. In contrast, the particle size analysis of SBB obtained after the pyrolysis process revealed a different distribution. The smallest diameter observed was 5 µm, making up 0.09 % of the sample, and the largest diameter observed was 100 µm, comprising a significant 56.97 % of the sample. According to the data, size distribution of curves of SBB shift to lower sizes and increase in intensity simultaneously showing the size of biochar particles after pyrolysis is indeed smaller than those of the raw SB. The result was consistent with the finding reported by Ferreira et al. 2019. The primary objective of reducing the biochar size through pyrolysis is to increase its surface area. A higher surface area enhances its ability to adsorb methylene blue due to the availability of more adsorption sites (Lonappan et al. 2016).

FTIR Analysis

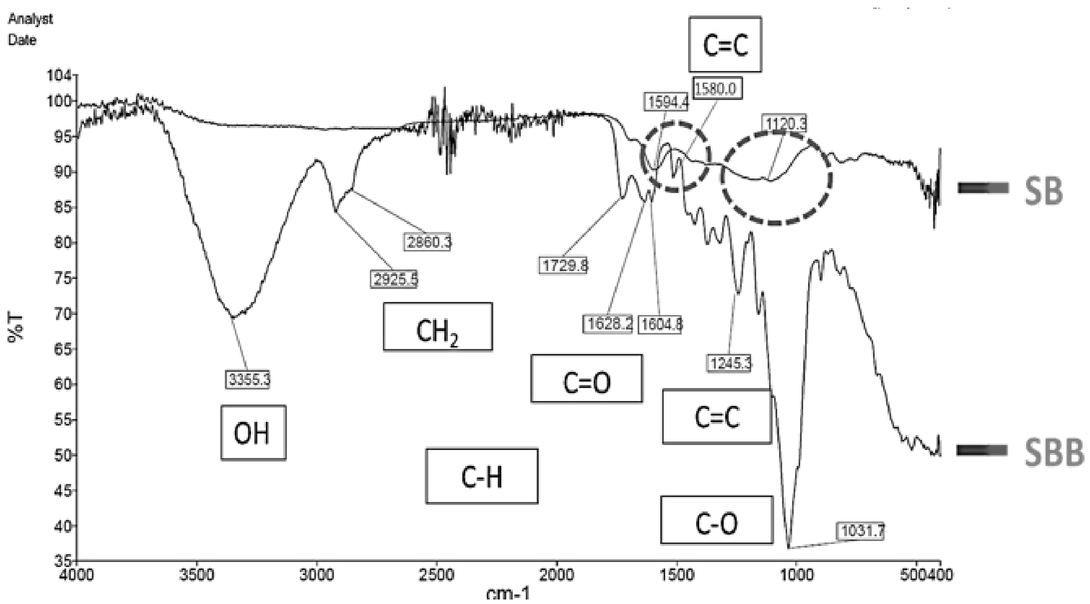
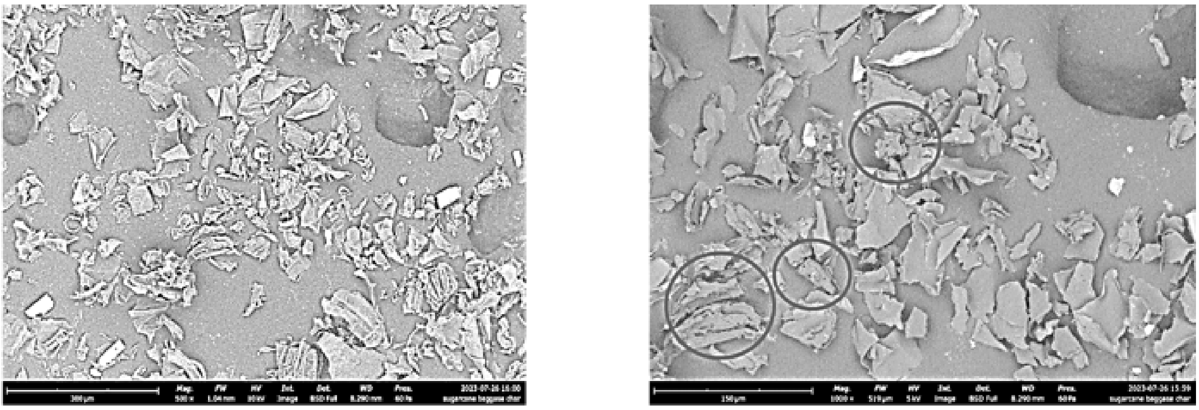


Figure 5: FTIR spectra for SB and SBB

The surface of the SB underwent an examination both pre- and post-pyrolysis, with the aim of identifying any alterations in surface characteristics, particularly in terms of the presence of functional groups. This investigation was conducted using FTIR spectroscopy. Fig. 5 illustrated the FTIR spectra of SB and SBB.

According to the findings of Sun et al. (2018), the broad peak at approximately 3355.3 cm-1 in the SB spectrum suggested the stretching vibration of hydroxyl groups (-OH) and it is reduced in SBB indicating moisture loss due to high pyrolysis temperature. Additionally, the presence of cellulose in the SB was indicated by a minor band around 2925.5 cm-1, corresponding to the CH2 stretch, which is again disappeared after pyrolysis. The peaks at 1630 to 1730 cm-1 were attributed to the C=O vibration of hemicellulose. Furthermore, the relatively weak peak at 1245.3 cm-1 was linked to C=C stretching of lignin and the high-intensity peak detected at 1030 cm-1 was associated to C-O stretching in lignin, hemicellulose, and cellulose of SB. In contrast to the original raw material, the band located at 1245 cm-1 and 1120 cm-1 were no longer visible after pyrolysis. A new band emerged at approximately 1580 cm-1 which identified as the C=C stretching vibration within the aromatic ring. The differences of the aforementioned peaks in SBB were attributed to the effective carbonization temperature, which considerably disrupted the lignocellulosic matrix.

SEM analysis



500 x

1000 x

Figure 6: SEM image of SBB under 500 and 1000 magnifications

The examination of the particle size of biochar derived from sugarcane bagasse (SBB) by using Scanning Electron Microscopy (SEM) provided insights surface structure of the biochar particles. The SEM findings indicated a varied distribution of particle sizes for SBB. The SEM images displayed distinctive traits of SBB, including irregular shapes, surface roughness, and the existence of pores within the particles, indicative of the impact of the thermal degradation and carbonization process on the original sugarcane bagasse. The appearance of larger cracks on the surface of the biochar, caused by the release of volatile organic material from the degradation of lignocellulosic matrix constituents, resulted in a better porosity due to the pyrolysis process at high temperatures (500°C). The images portrayed the conversion of sugarcane bagasse into biochar, potentially leading to changes in porosity, surface area, and overall structure, ultimately facilitating the adsorption of methylene blue.

CONCLUSION AND FUTURE RESEARCH

Based on the findings of this study, the identification of optimal adsorption conditions has proven in enhancing the efficiency of methylene blue removal from coating industry wastewater. The utilization of predictive response surface models has provided a great framework for a precise process control and optimization. These optimized conditions offer valuable insights for potential future research, highlighting the potential of sugarcane bagasse as a natural resource for mitigating the presence of methylene blue in industrial wastewater management. This highlights the significance of biochar-based adsorption processes in sustainable and eco-friendly industrial waste treatment practices.

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