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Advancing Environmental Sustainability: Optimizing the Application of Activated Carbon from Sugarcane Trash and Cotton Seed Waste for Methylene Blue Adsorption

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Keywords:

Adsorbent; Adsorption; Biomass waste; Chemical activation; Concentration; Dye; Environmental sustainability.

Highlights:

- Characteristics of feedstock (sugarcane trash and cotton seed waste) were analyzed.
- Activated carbon was prepared from sugarcane trash and cotton seed waste in presence of activation chemicals.
- Prepared activated carbon investigated for adsorption of methylene blue.

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Abstract: The adsorption of Methylene blue onto activated carbons produced from a blend of Sugarcane and Cotton Seed Waste (biomass waste) was investigated in this study. Potassium hydroxide (KOH) and Sulphuric acid (H_2SO_4) served as activating agents in producing the activated carbons. Firstly, the characterization of biomass was performed using proximate and elemental analysis subsequently; the characterization of the activated carbons was achieved using Fourier transform infrared spectroscopy, scanning electron microscopy (for surface morphology) and Brunauer–Emmett–Teller (BET) (for surface area determination). Experimental results showed that an increase in temperature and the ratio decreased the yield of activated carbon. This indicated that temperature and ratio had an inverse relationship with the yield of activated carbon, as observed from the results. Also, an optimal yield of 45% at 400°C and IR2, denoting the impregnation ratio for KOH-activated carbon was achieved. By increasing the temperature from 400-500°C, the BET surface area increased, and by increasing the temperature above 500°C, the surface area decreased due to intense heat effects at 600°C, causing the evolution of volatile matter and surface area decreased at 600°C as a result of shrinkage and closure of produced pores at higher temperature. It was observed from the BET analysis and the optimal conditions that the AC surface area of 780.4 m²/g at 500°C for KOH-activated carbon was achieved. FTIR spectra indicated absorption peaks, which suggested the presence of C-C, C-H, and C-O bonds, as well as an O-H bond. The adsorption studies were conducted under different adsorption conditions using various adsorption parameters such as effect of Adsorbent dosage, contact time, pH, initial dye concentration which indicated that KOH Activated carbon from cotton seed waste showed the best MB Adsorption and optimal condition for adsorption were at basic medium [PH12], initial dye concentration 10mg/l, contact time 140min and adsorbent dosage 0.1g. The existing study concluded that cottonseed waste may be employed as a low-cost adsorbent for the elimination of primary dye.

تعزيز الاستدامة البيئية: تحسين تطبيق الكربون المنشط من مخلفات قصب السكر ونفايات بذور القطن لامتزاز أزرق الميثيلين

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الخلاصة

تم في هذه الدراسة استقصاء امتزاز أزرق الميثيلين على كربونات مُنشَّطة مُنتَجة من خليط من مخلفات قصب السكر وبذور القطن (مخلفات الكتلة الحيوية). استخدم هيدروكسيد البوتاسيوم (KOH) وحامض الكبريتيك (H_2SO_4) كمعامل مُنشَّطة في إنتاج الكربون المنشط. أولاً، تم توصيف الكتلة الحيوية باستخدام التحليل التقريبي والتحليل العنصري؛ ثم تم توصيف الكربون المنشط باستخدام مطيافية الأشعة تحت الحمراء بتحويل فورييه، والمجهود الإلكتروني الماسح (لتقييم المورفولوجيا السطحية)، وطريقة بروناور-إيميت-تيلر (BET) (لتحديد المساحة السطحية). أظهرت النتائج التجريبية أن زيادة درجة الحرارة ونسبة التنشيط أدت إلى انخفاض إنتاجية الكربون المنشط. وهذا يشير إلى وجود علاقة عكسية بين درجة الحرارة ونسبة التنشيط وبين إنتاجية الكربون المنشط، كما هو ملاحظ من النتائج. كما تم تحقيق عائد أمثل بلغ ٤٥٪ عند درجة حرارة ٤٠٠ درجة مئوية ونسبة تنشيط (IR2) للكربون المنشط بالبوتاسيوم. وبزيادة درجة الحرارة من ٤٠٠ إلى ٥٠٠ درجة مئوية، زادت المساحة السطحية (BET)، بينما أدت زيادة درجة الحرارة فوق ٥٠٠ درجة مئوية إلى انخفاض المساحة السطحية بسبب التأثير الشديد للحرارة عند ٦٠٠ درجة مئوية، مما تسبب في تحرر المواد الطيارة وانكماش المسام وإغلاقها عند درجة الحرارة المرتفعة، فانخفضت المساحة السطحية عند ٦٠٠ درجة مئوية. وقد أظهر تحليل BET وفي الظروف المثلى تحقيق مساحة سطحية للكربون المنشط بلغت ٧٨٠.٤ م²/جم عند ٥٠٠ درجة مئوية للكربون المنشط بالبوتاسيوم. أشارت أطيف الأشعة تحت الحمراء (FTIR) إلى وجود قمم امتصاصية تدل على وجود روابط C-C، C-H، وC-O، وكذلك رابطة O-H. وأجريت دراسات الامتزاز تحت ظروف امتزاز مختلفة باستخدام معايير امتزاز متنوعة مثل تأثير جرعة المادة المازة، وزمن التلامس، ودرجة الحموضة (pH)، والتركيز الابتدائي للصبغ. وأشارت النتائج إلى أن الكربون المنشط بالبوتاسيوم والمنتج من مخلفات بذور القطن أظهر أفضل أداء لامتزاز أزرق الميثيلين، وكانت الظروف المثلى للامتزاز في الوسط القلوي (pH12)، وتركيز ابتدائي للصبغ مقداره ١٠ ملغ/لتر، وزمن تلامس ١٤٠ دقيقة، وجرعة مادة مازة مقدارها ٠.١ غرام. وخلصت الدراسة الحالية إلى أنه يمكن استخدام مخلفات بذور القطن كمادة مازة منخفضة التكلفة لإزالة الصبغة الأساسية من المحاليل المائية.

الكلمات الدالة: مادة مازة؛ امتزاز؛ مخلفات الكتلة الحيوية؛ التنشيط الكيميائي؛ التركيز؛ الصبغة؛ الاستدامة البيئية.

1. INTRODUCTION

From a global perspective, an alarming situation has elevated environmental protection over the years. Approximately 15% of dyes are lost in the general world production of dyeing techniques and released as liquid effluent; these wastes are causing soreness, also hindering mild dispersion and disturbance of the environment. Moreover, these wastes are toxic to creatures. Some examples of synthetic dyes include aniline blue, methylene blue, crystal violet, toluidine blue, basic fuchsin, and Congo red. Methylene blue is one of the synthetic dyes that are utilized extensively as a colorant for papers, in wool, silk, and cotton. However, if not addressed appropriately in an environmentally friendly manner, it is fatal, and the presence of this chemical compound has been reported to cause teratogenicity and embryo toxicity. Moreover, MB generally has a high molar absorption coefficient, causing attenuation of sunlight transmittance; therefore, the presence of MB can negatively affect the photosynthesis process, which leads to an impact on the entire water ecosystem. All these associated MB toxicities and adverse health and environmental impacts leave no choice but to various technologies for the treatment of MB-loaded wastewater. Methylene blue has applications in natural science, chemistry, health science, and the tinting industries. Its interaction causes sickness, anemia, hypertension, and vomiting [1]. Techniques for treating wastewater that includes dyes is chemical oxidation, liquid-liquid extraction, and adsorption. These techniques have some restrictions, which consist of low effectiveness and a limited scope

[2]. Adsorption is a pleasant method amongst others due to its simplicity in design, operation, and suitability. The range of adsorption depends on certain situations, the character of the adsorbent, its surface vicinity, and porosity. In keeping with this; numerous adsorbents have been developed with exclusive properties. Activated carbon has been a widely used adsorbent with Good-sized particles [3]. Activated carbon is a lively, trendy adsorbent of an extensive range of medications and chemical substances, inhibiting their digestive absorption [4]. They contain a wide variety of altered surface agencies, for water sanitization, and recapture of substances, and many others. Floor modification can motivate governing adsorption forces at the bottom of activated carbon [5]. The activated carbon surface can be made to apply these commanding forces through activation. In advance research, active carbon was improved to enhance its adsorptive properties for impurities. The adsorption properties of activated carbons are typically tested using colored particles, in addition to methylene blue (MB; $C_{16}H_{18}ClN_3S$), which is extensively utilized in industry [6-8]. For the characterization of activated carbon, the BET (Brunauer, Emmett and Teller) approach, FTIR (Fourier transform infrared spectroscopy), and SEM (Scanning electron microscopy) analyses can be employed [5, 6, 9-14]. In a large part of the world, wastewater from textile industries is a threat to environment. The composition of wastewater will depend on the different chemicals, compounds, and dyes used in the industry. In textile primary pollutants of wastewater produces from finishing and dying

steps that involved during the dying of the natural or manmade fibers to desired color and processing of those into the final products, different type of Sizing agents is used to yarn before production of fabric to ensure secure and fast weaving process and are removed later from the woven fabrics in wet process [2, 3, 15-18]. Due to increasing concerns about portable water for industrial use, wastewater treatment becomes more important for reuse and removal of other contaminants. There are four different methods to treat wastewater, which are: biological method, physicochemical method, electrical, acoustical, and electromagnetic processes, as well as nuclear treatment. The other techniques are percolation, ultra filtration, reverse osmosis, dialysis, ion exchange, oxidation using chlorine, solvent extraction, ozone, chlorine dioxide, hydrogen peroxide adsorption, evaporation, coagulation, photochemical reactions, foam flotation, activated sludge, aerobic, anaerobic treatment, bacterial treatment, microbial reduction, irradiation due to nuclear radiations, electrolysis, electro-dialysis, magnetic separation and ultrasonic treatment [19-23]. The objective of this study was to prepare activated carbon from the biomass waste using sugarcane trash and cotton seed waste in activation chemicals KOH and H_2SO_4 in muffle furnace at carbonization temperature from 400-600°C, activated carbon was used for adsorption application of methylene blue, Characterization of feed stock was performed using proximate, element analysis, BET, FTIR, and SEM analysis was performed for characterization of prepared activated carbon.

2. EXPERIMENTAL PROCEDURE

2.1. Materials

Sugarcane trash, also known as cane trash, emerges as a valuable biomass resource in sugar-producing nations globally, showcasing its potential for sustainability. With a calorific value similar to that of bagasse, it offers the added advantage of lower moisture content, enabling quicker drying. Sourced from the environmentally conscious Chillianwala agriculture farm, this biomass resource emphasizes the benefits of adopting sustainable practices. Moreover, the significance of cotton, a pivotal agricultural commodity in Pakistan, extends beyond its contribution to various textile products; it exemplifies the potential for both sustainable and economically advantageous utilization of natural resources in the region. Cotton seed waste samples are obtained from the Bilal Cotton factory in Rehme-shah tehsil, Tandliwala district Faisalabad. Two chemical activation agents, i) 1M KOH with specifications including a density of 2.12 g/cm³ at 25°C, a melting point of 360°C, and a boiling point of 1327°C, and ii) 40 wt. % H_2SO_4 with specifications including a boiling

point of 611 K, a specific gravity of 1.84 at 298 K, and a melting point of 10.31 °C, were sourced from the Research and Development department of National Fertilizer Corporation (R&D NFC) in Faisalabad, Pakistan. These materials were employed in synthesizing activated carbons from primary biomass materials. Structure of the raw material is a significant element to determine the selection of biomass for AC production by using proximate Analysis by ASTM, D1762-84 (Reapproved 2001), and proximate analysis is defined as: (1) moisture, (2) volatile matter, (3) fixed carbon, and (4) Ash. The final elemental evaluation is done using a CHNS analyzer.

2.2. Preparation of Activated Carbon

50g of each biomass sample is impregnated with H_2SO_4 and KOH solution with two different activation ratios (1:2, 1:3) for KOH and (1:0.5, 1:1) for H_2SO_4 . Then, stirring is done using a magnetic stirrer at 85°C to facilitate activation. After that, to increase the activation rate, the sample is left to stir occasionally at room temperature for 24 hours. Then, the impregnated biomass filtrated to remove excess chemicals and dried in the oven at 105°C. The mixtures are then taken in a crucible for further carbonization. The carbonization process is made in a muffle furnace at three different temperatures (400, 500, and 600°C), and the temperature is reached at a rate of 10°C/min. After that, to prevent oxidation and volatile matter from reaching the heating zone, nitrogen gas is used to provide an inert atmosphere. The yield is calculated as the weight loss during the carbonization process. After that, the product was dissolved in a 0.1M HCl solution to remove contamination and excess chemicals from the pores and surface of the AC, The AC is then washed with hot distilled water to achieve a neutral pH. The AC product is dried in an oven at 105°C for 24hrs. Finally, the product is grounded and packed in closed plastic bags for further use.

2.3. Characterization of Activated Carbon

The pore size and Surface area of the produced activated carbon are obtained using an analysis technique of net surface area through N_2 gas adsorption at a constant temperature. The technique was developed by three people: Edward Teller, Paul Emmett, and Stephen Brunauer in 1938. This method measures pore size and surface area based on gas adsorption on solid porous substances. It calculates the surface area using the adsorption and desorption data. From the data, the amount of gas needed to formulate a monolayer on the upper area of the material can be calculated. These experiments will be carried out at the Central laboratory of Lahore University of Management Sciences (LUMS) using a BET analyzer. The scanning electron microscope

(SEM) is used to estimate the surface structure of activated carbon, and the SEM images of AC are investigated. The carbon tape was split into minor pieces and secured on a disc to get an SEM image. At that point, the outer coat of the disc is removed, and a very small quantity of carbon is applied to carbon (black) tape, which is then tightly fixed by pressing it on to a clean surface of silicon (Rawal et al., 2018). FTIR is an adaptable technique for determining surface chemistry and chemical bonding present in samples, these experiments will be carried out at the central Hi-Tech lab of Government College University Faisalabad (GCUF) using a scanning electron microscope.

2.4.Characterization of Activated Carbon

By batch adsorption experimentation using a typical MB ($C+H_{18}N_3SCL$, AR grade) mixer as an experimental dye molecule, the adsorptive properties can be obtained. Many factors, for example, primary dye concentration, adsorbent dosage, pH, agitation speed, contact time, and temperature, have a substantial impact on the adsorption tendency (Gonawala & Mehta, 2014). To inspect the effect of those variables, adsorption experiments were conducted in a batch trial. A mixture of 100 mL MB dye at the chosen concentration is arranged in a conical flask of 250 mL by apt thinning of the stock mixture and adjusting its anticipated pH. At that point, add the acknowledged quantity of AC and the obtained mixture shook using a magnetic mixer for a predefined time. Afterwards, centrifuge the suspension and examine the supernatant for the dye removal percentage using UV-spectroscopy at 664nm. The final concentration of balanced MB obtained from the calibration arch. The different types of activated carbon testers are stable and differentiate the level of MB uptake (q_e ; mg/g) estimated by means of subsequent expression:

$$\text{Percentage removal} = \left[\frac{C_0 - C_e}{C_0} * 100 \right] \quad (1)$$

$$\text{Adsorption capacity} = \left[\frac{C_0 - C_e}{W} * V \right] \quad (2)$$

Where the initial MB concentration is represented by C_0 (mg/L) the equilibrium concentration of MB is mentioned as C_e , the volume of the MB mixture is written as V (L), and the mass of activated carbon as W (g), which is used in the trial. Plotting a standard curve is important for calculating the final concentration of MB from absorbance, which in turn is important for determining the percentage of color removal and adsorption capacity. For this reason, different MB concentrations (from 0-10mg L⁻¹) were prepared and their respective absorbance was measured using a UV-spectrophotometer. Lastly, the linear association of absorbance contrasted with the MB concentration design

and equation of final MB concentration is initiated from the graph slope.

$$\text{Absorbance} = [\text{slope} * C_e + y - \text{intercept}] \quad (3)$$

The optimum value of adsorbent dosage for maximum removal of methylene blue dye is estimated by conducting a batch adsorption test using an AC sample with the highest surface area. A MB (100 ml) solution with 10 mg /L concentration is added to five conical flasks (250 ml), each holding different masses of AC taster (0.01, 0.03, 0.05, 0.07, and 0.1g). Finally, the adsorbent dosage with the maximum adsorption capacity can be considered as the optimum value for the adsorption experiment. To analyze the effect of time on the adsorption procedure, an experiment is carried out by varying the duration (20, 40, 60, 80, 100, and 120 min). A 0.1 g of AC sample is taken into five different conical flasks (250 ml) and 100ml of prepared standard MB solution having a PH of 10 is added to every conical flask. Finally, the percentage removal and adsorption capacity are calculated, accordingly, to select the best duration for maximum MB adsorption. To determine optimum inception concentration of MB, standard MB solution of 100 ml at a concentration rate of (10, 20, 30, 40 and 50 mg/L) are added into a sequence of (250 ml) conical flasks each holding 0.1g activated carbon. Finally, percentage removal and adsorption capacity are determined from the relation mentioned above so that tester with highest adsorption capacity can be selected as the best condition for adsorption of MB. For maximum removal of the dye, the optimum pH value was determined using a batch-type adsorption experiments against pH (range: 3-12), with the addition of 1M HCl or NaOH solution. 0.1 g of Activated carbon was stirred with 100 ml of a standard solution of methylene blue dye in flasks at room temperature for 140min and a pH of 12 was used for measurements. Finally, the percentage of dye removal is determined.

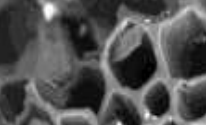
3.RESULTS AND DISCUSSION

3.1.Elemental Composition and Surface Morphology

The results of the proximate analysis of biomass waste, i.e., sugarcane trash and cottonseed waste, are given in Table 1, which shows the presence of volatile matter 56.7%, moisture contents 20.3%, ash contents 9% and fixed carbon 14% for sugarcane trash waste. For cottonseed waste, the volatile matter is 62.5%, the moisture content is 11.5%, the ash content is 8% and the fixed carbon is 18%. This biomass has lower ash and higher volatile matter values, which means it is a good starting material for activated carbon synthesis. Also, biomass with low ash content would be a good raw material for AC formations, as it would help minimize the effect of inorganic contaminants on pore development during the activation process.


Proximate Analysis	Sugarcane Trash Waste	Cotton Seed Waste
Fixed carbon	14	18
Moisture content	20.3	11.5
Volatile matter	56.7	62.5
Ash	9	8

Elements	Calibration stage Percentage (wt. %)	Validation stage Percentage (wt. %)
Carbon	47.57	51.7
Hydrogen	6.13	7.94
Oxygen	43.99	37.89
Sulfur	0.19	0.06
Nitrogen	2.12	2.41



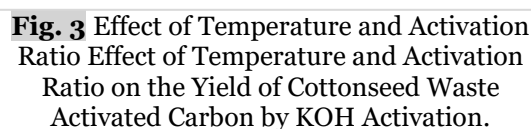
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The effect of temperature and activation ratio on the yield of activated carbon from KOH activation at impregnation ratios (IR2, IR3) and temperature ranges (400–600°C) is shown in Fig. 2 for sugarcane trash waste, which indicates that temperature and activation ratio have inverse effect on yield of activated carbon as shown in graph yield decreases by increasing temperature and ratio. The reason behind this is that, by increasing the temperature, a continuous degradation of biomass components occurs, At an IR3 ratio, there is more degradation of biomass components and more evolution of volatile matters as compared to IR2. Therefore, we have a higher value of yield of 37% at 400°C and IR2.



The effect of sulfuric acid activated carbon from both biomasses are shown in Figs. 4, 5, which indicates that by increasing the temperature and activation ratio decrease the yield of activated carbon primarily due to the effect of sulfuric acid as a dehydrating agent, which facilitates the prevention of tar formation and other unwanted reaction byproducts throughout activation. For sugarcane trash activated carbon using sulfuric acid, the maximum yield is 31% at 400°C and IR 0.5. For activated carbon from cottonseed waste using sulfuric acid, the maximum yield is 38 % at 400°C, and IR1. According to the above characterization study, it is summarized that the optimal value of yield is obtained from cottonseed activated carbon using KOH activation.

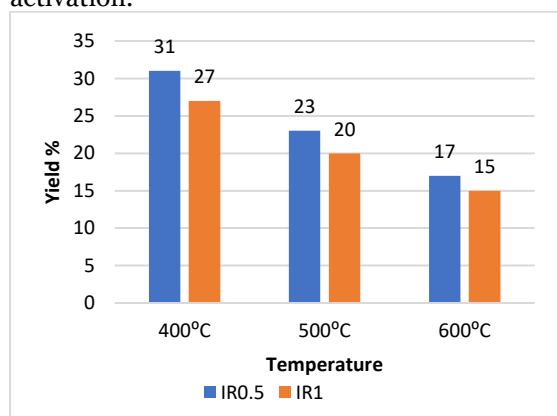


Fig. 4 Effect of Temperature and Impregnation Ratio on the Yield of Activated Carbon from Sugarcane Trash Carbon by H_2SO_4 .

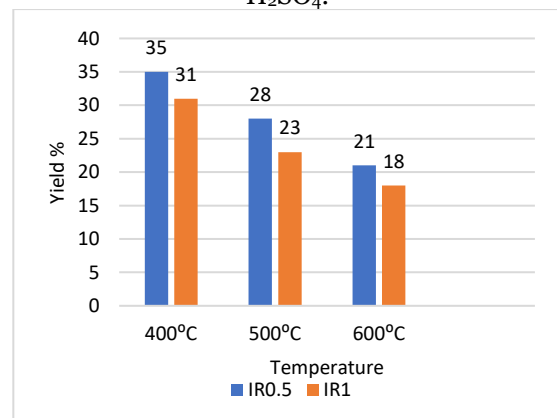


Fig. 5 Effect of Temperature and Activation Ratio on the Yield of Activated Carbon from Cottonseed Waste by KOH Activation.

3.3. Scanning Electron Microscopic Analysis

Characterization of activated carbon is performed using SEM, BET, and FTIR analysis, respectively. To examine the prepared AC, SEM is used to investigate surface properties. SEM micrographs of the chemically activated carbons using H_2SO_4 , and KOH have been provided in Figs. 6, 7. SEM images show a nicely developed porous floor transformed into

a well-defined structure at a better resolution. SEM photographs have pore diameters in the μm range, which are referred to as channels for micro-pore surfaces. From the analysis, it can be observed that each of the adsorbents has a complex texture.

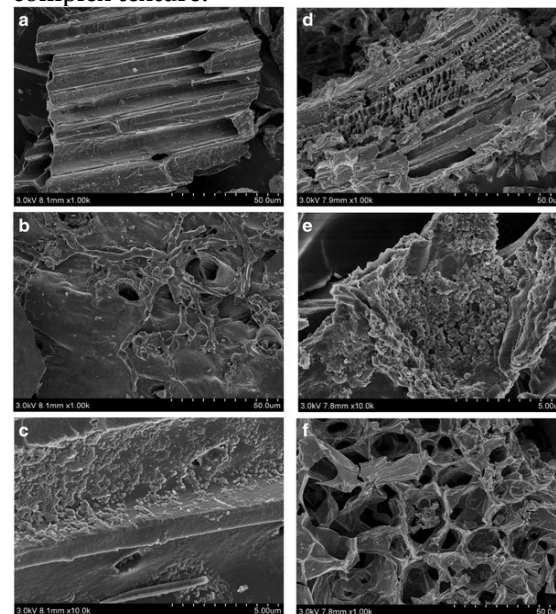


Fig. 6 SEM Images of the Prepared ACs from Sugarcane Trash.

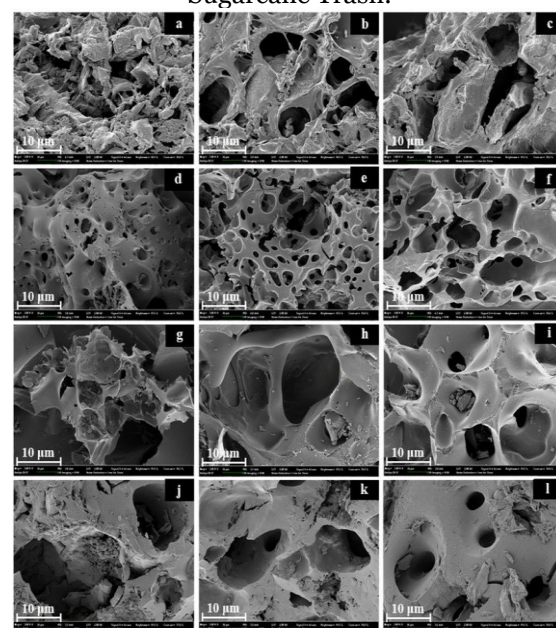


Fig. 7 SEM Images of the Prepared ACs from Cotton Seed Waste.

3.4. Fourier Transform Infrared Spectroscopy (FTIR)

Fig. 8 showed FTIR spectra for the activated carbon made from sugarcane waste and cottonseed waste. Absorption peaks have been found at 1580 and 879.95 cm^{-1} , at 1707.62 and 3637.93 cm^{-1} , at 1101 cm^{-1} which suggest the presence of C-C, C-H and C-O and O-H respectively. The identification of useful components at the surface of the ACs is essential because it could also assist in describing the sort and extent of sorption

happening on the surface of the hydrocarbon with a specific substrate.

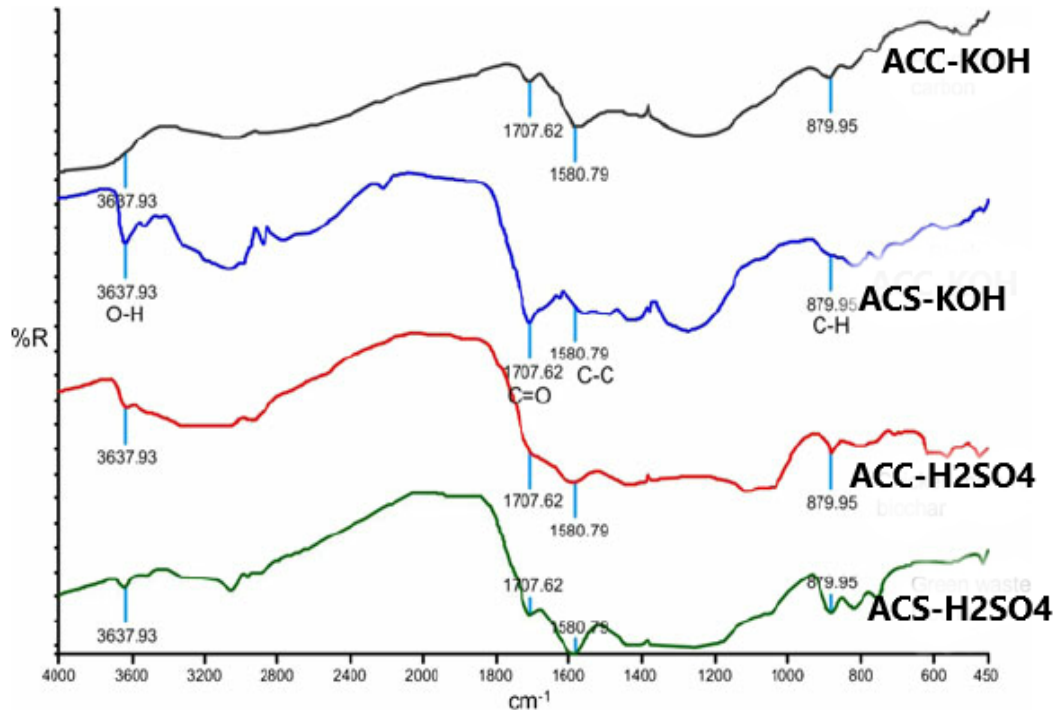


Fig. 8 FTIR Spectra of Prepared Activated Carbon.

3.5. Brunauer, Emmett, and Teller (BET)

The effect of temperature on the ACS surface area is observed from 400 to 600 °C. Table 3 and 4 and Fig. 9 showed that the surface area of KOH activated AC increases from 645.8 to 710.5 m²/g from 400-500 °C due to intense heat effects at 500 °C caused evolution of volatile matter and creates more pore caused an increase in surface area and surface area decreases at 600 °C as result of shrinkage and closure of produced pores at higher temperature. The surface area of H₂SO₄ activated ACs increases from 495.9 to 546.7 m²/g from 400 to 600 °C due to intense heat effects at 600 °C, causing the evolution of

volatile matter and surface area decreases at 600 °C as a result of shrinkage and closure of produced pores at higher temperature. Also here, sulfuric acid acts as a dehydrating agent.

Table 3 Surface Area and Pore Features for Sugarcane Trash Activated Carbon.

Samples	S _{BET} (m ² /g)	Micropore Area (m ² /g)	Micropore Volume (cm ³ /g)
KOH-ACS-400°C	645.8	657.32	0.131
KOH-ACS-500°C	710.5	728.81	0.151
KOH-ACS-600°C	520.6	563.5	0.125
H ₂ SO ₄ -ACS-400°C	495.9	512.98	0.135
H ₂ SO ₄ -ACS-500°C	546.7	553.34	0.153
H ₂ SO ₄ -ACS-600°C	413.4	456.3	0.118

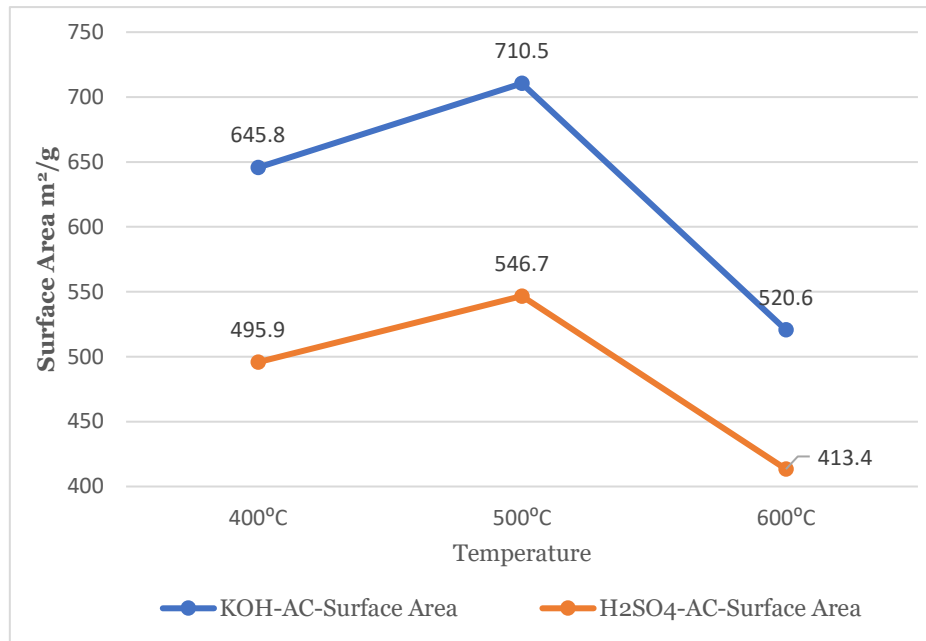


Fig. 9 Effect of Temperature on Surface Area of Sugarcane Trash Waste Activated Carbon.

Table 4 Surface Area and Pore Characteristics for Cotton Seed Waste Activated Carbon & Variation of Absorbance with MB Concentration.

Samples	S_{BET} (m ² /g)	Micropore Area(m ² /g)	Micropore Volume(cm ³ /g)
KOH-ACC-400°C	725.9	733.32	0.29
KOH-ACC-500°C	780.4	798.93	0.35
KOH-ACC-600°C	634.2	689.4	0.21
H2SO4-ACC-400°C	575.6	623.56	0.32
H2SO4-ACC-500°C	699.5	702.89	0.38
H2SO4-ACC-600°C	528.2	574.5	0.29
Initial MB Concentration (mg/l)	%Absorbance		
0	0		
2	0.243		
4	0.475		
6	0.698		
8	0.965		
10	1.198		

Fig. 10 shows the effect of cottonseed waste AC, which follows same trend as sugarcane waste activated carbon. The surface area of KOH-activated ACC increases from 725.9 to 780.4 m²/g from 400 to 500°C and decreases at 600°C, but a higher value of AC surface area as compared to sugarcane waste AC. Therefore, we obtained an optimal value of AC surface area of 780.4m²/g at 500°C. The calibration curve is important to find the final concentration

directly from UV spectrophotometer. Therefore, a calibration for MB at a wavelength of 664nm was obtained from the adsorption experimentation of MB using biomass-derived activated carbon with a higher surface area. The result is as follows: The slope from Fig. 11 is 0.119, so that at any time, the final equilibrium concentration is equal to the rate of to that value of slope. $C_e = \text{absorbance} / 0.119$.

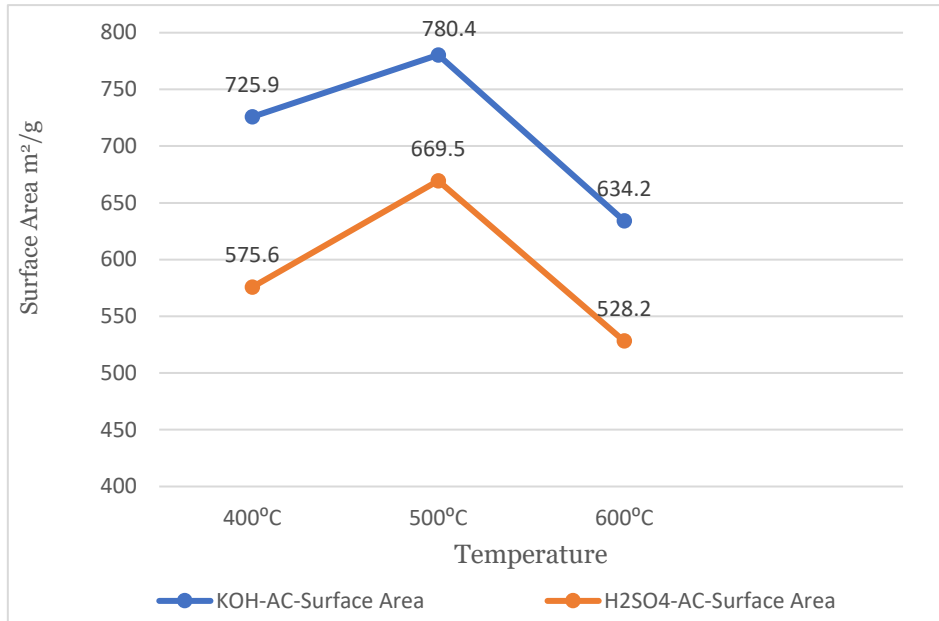


Fig. 10 Effect of Temperature on Surface Area of Cotton Seed Waste Activated Carbon.

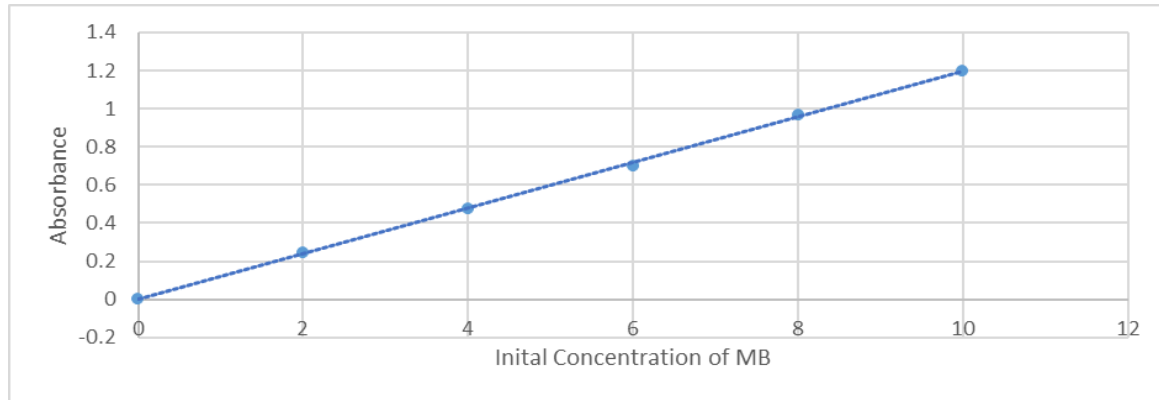


Fig. 11 Calibration Curve for Equilibrium Dye Concentration C_e .

3.6. Effect of Adsorbent Dosage

The activated carbon percentage's removing capacity is primarily due to its porous composition and pore size, as well as the polarization, solubility, and molecular size of the adsorbate [24]. Fig. 12 and Table 5 illustrate the effect of adsorbent dosage on the removal percentage of MB. This is demonstrated by increasing the dosage up to 0.01g, where the

percentage of dye removal increases. This is due to the increased number of adsorbent active sites. Therefore, it was investigated that 0.01g/100 ml was the optimum adsorbent dosage for Mb adsorption. It seemed that the adsorbent dosage (0.01g/100ml) resulted in the percentage removal of MB of 83.5% for ACS and 94.3% for ACC. Here, the optimal condition is 0.1gdosage of cottonseed waste activated.

Table 5 Effect of Adsorbent Dosage on MB Removal.

Adsorbent Dosage (g)	Absorbance-ACS	Ce-ACS (mg/l)	Absorbance-ACC	Ce-ACC (mg/l)	% Dye Removal-ACS	% Dye Removal-ACC
0.01	0.641	5.39	0.506	4.25	46.1%	57.5%
0.03	0.508	4.27	0.368	3.09	57.3%	69.2%
0.05	0.378	3.18	0.271	2.28	68.2%	77.2%
0.07	0.338	2.48	0.160	1.35	75.2%	86.5%
0.1	0.196	1.65	0.063	0.58	83.5%	94.2%

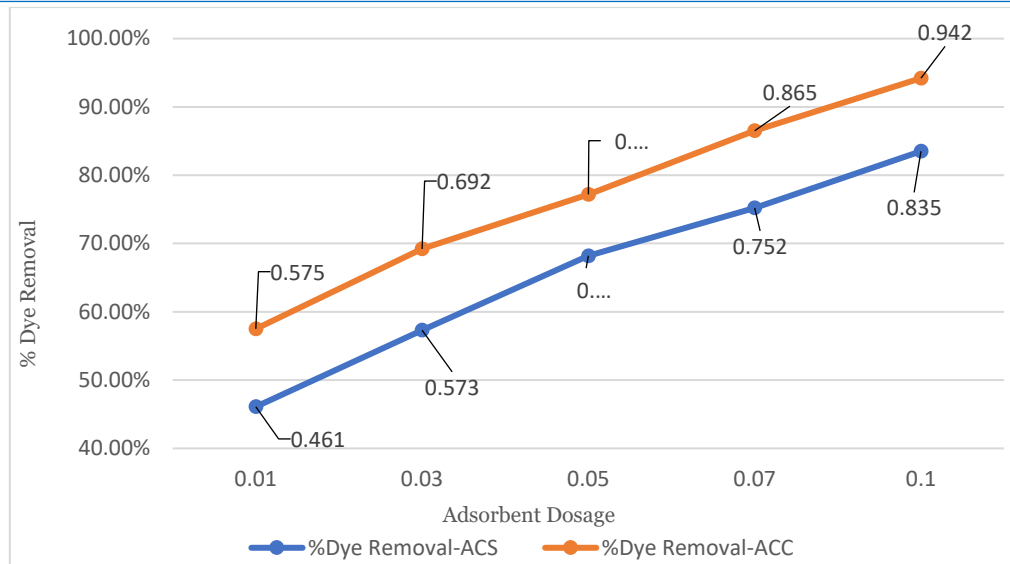


Fig. 12 Effect of Adsorbent Dosage on % Dye Removal.

3.7. Effect of Adsorbent Dosage

By varying the pH from 3 to 12 under constant adsorption parameters, the effect of the initial pH of the MB solution on percentage removal is determined. The results from the experimentation are illustrated in Table 6 and Fig. 13. Showing that by increasing pH, the %dye removal increases. The removal percentage of MB increases from 36.5% to 83.5.9% and from 49.1% to 94.2% as PH varies from 3 to 12, respectively. The reason behind this is that the AC surface is to be negatively charged. At low pH, due to acidic medium, there is an increase in hydrogen ion concentration which would then neutralize the negatively charged carbon surface thereby decreasing the adsorption of the positively charged dye cation and the %dye removal is minimal. As from Fig. 13 pH is optimal pH for removal percentage of MB was 92.4%. A similar effect of initial dye pH was reported by [25].

3.8. Effect of Contact Time

From Fig. 14 and Table 7, the effect of contact time on %dye removal is given below. In the present research, the constant parameters used were 10 mg/l of MB, 0.1 g of AC, and a pH of 12. The dye removal increases with an increasing contact time, From 20 to 140 minutes, as shown in the Fig. 14. The % dye removals are increased

from 36% to 81.3% for ACS and 47% to 94.4% for ACC. Therefore, 140min were considered the optimum contact time for conducting the adsorption experiment in this study using cottonseed waste activated carbon. A similar result has been reported by [13]. The present study focused on the preparation of activated carbon from coconut leaves using FeCl_3 and its application for methylene blue removal.

3.9. Effect of Initial Dye Concentration

Due to the binding active sites found on the adsorbent surface, the initial MB concentration affects the adsorption process. These experiments are done by varying initial dye concentration in the range of (10-60 mg/ l) at constant adsorption parameters (0.01 g, PH 12, and 140 min) to find the effect of initial dye concentration on %dye removal. Results are given below. From Fig. 15 and Table 8, it was observed that the maximum removal of MB, at 83.5% for ACS and 94.2% for ACC, occurred at the minimum initial concentration of dye (10 mg/l). After that, the dye removal decreases by increasing the initial dye concentration. So the optimal conditions for dye removal are 10mg/l at initial dye concentration against cotton seed waste activated carbon [14].

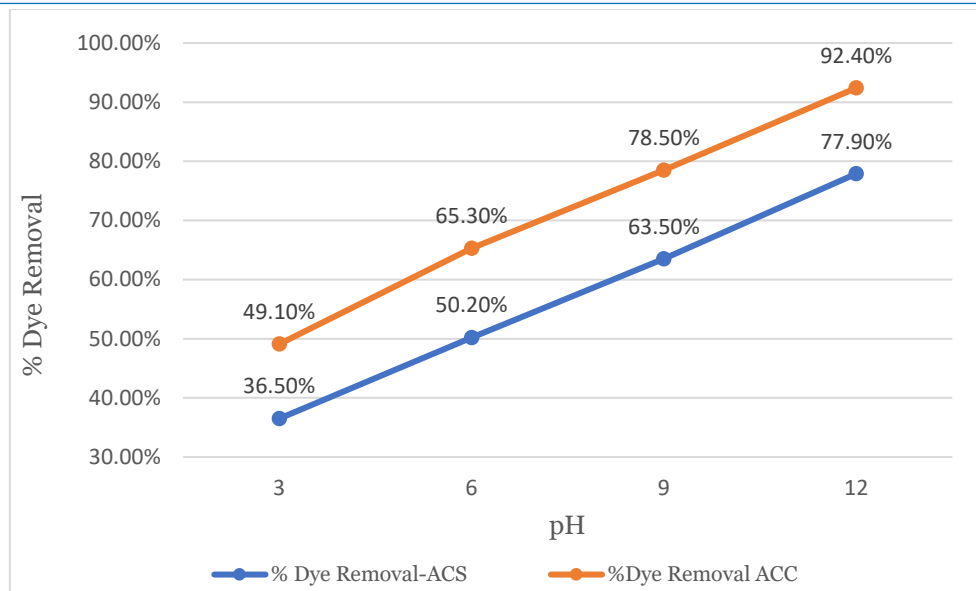


Fig. 13 Effect of pH on % Dye Removal.

Table 6 Effect of Initial pH on MB Removal.

pH	Absorbance-ACS	Ce-ACS (mg/l)	Absorbance-ACC	Ce-ACC (mg/l)	% Dye Removal-ACS	% Dye Removal-ACC
3	0.756	6.35	0.606	5.09	36.5%	49.1%
6	0.593	4.98	0.413	3.47	50.2%	65.3%
9	0.434	3.65	0.256	2.15	63.5%	78.5%
12	0.263	2.21	0.090	0.76	77.9%	92.4%

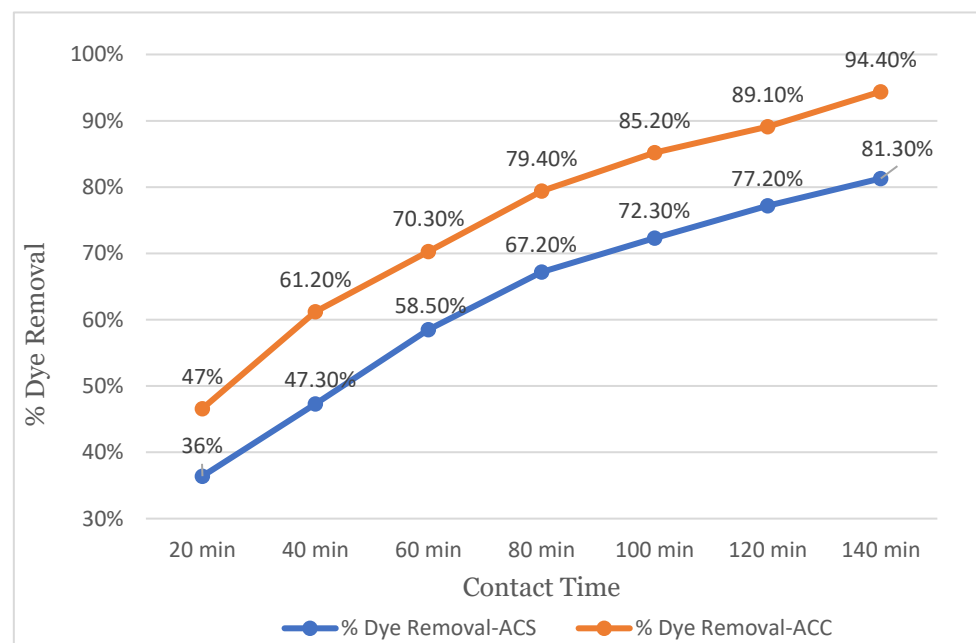


Fig. 14 Effect of Contact Time on % Dye Removal.

Table 7 Effect of Contact Time.

Contact Time	Absorbance-ACS	Ce-ACS (mg/l)	Absorbance-ACC	Ce-ACC (mg/l)	% Dye Removal-ACS	% Dye Removal-ACC
20	0.761	6.40	0.631	5.30	36%	47%
40	0.627	5.27	0.462	3.88	47.3%	61.2%
60	0.494	4.15	0.353	2.97	58.5%	70.3%
80	0.390	3.28	0.245	2.06	67.25	79.4%
100	0.330	2.77	0.176	1.48	72.3%	85.2%
120	0.271	2.28	0.130	1.09	77.2%	89.1%
140	0.223	1.87	0.067	0.56	81.3%	94.4%

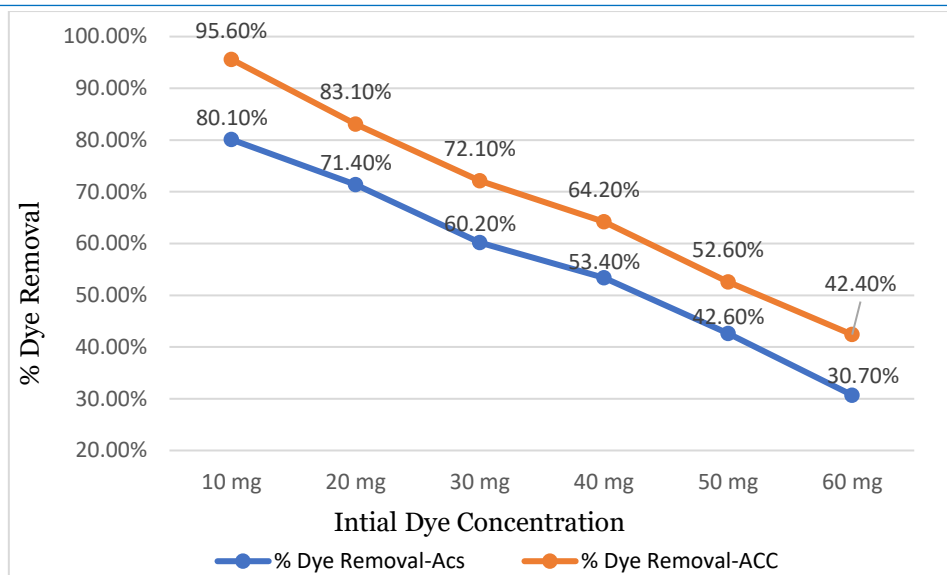


Fig. 15 Effect of Initial Dye Concentration.

Table 8 Effect of Contact Time.

Initial Dye Conc. (mg/l)	Absorbance-ACS	Ce-ACS (mg/l)	Absorbance-ACC	Ce-ACC (mg/l)	% Dye Removal-ACS	% Dye Removal-ACC
10	0.237	1.99	0.052	0.44	80.1%	95.6%
20	0.681	5.72	0.402	3.38	71.4%	83.1%
30	1.418	11.92	0.994	8.35	60.2%	72.1%
40	2.205	18.53	1.701	14.3	53.4%	64.3%
50	3.434	28.86	2.820	23.7	42.6%	52.6%
60	4.953	41.62	4.088	34.35	30.7%	42.65

4. CONCLUSIONS

The main conclusions of the present study can be summarized as follows: This work examined the synthesis and characterization of activated carbon from biomass waste, namely sugarcane trash and cottonseed waste, using KOH and H₂SO₄ as activating agents. Firstly, characterization of biomass using proximate and elemental analysis indicated the presence of more volatile matter and less ash content, which meant it, was a good starting material for AC production, as it had low ash content, which would facilitate the minimum effect of inorganic impurities on pore development during the activation process. The elemental analysis indicated that our biomass was mainly composed of carbon, hydrogen, and oxygen, making it a cellulosic material, which is a better choice as a raw material for AC production. The yield of the AC produced indicated the effect of carbonization temperature and activation ratio [26]. Results concluded that by increasing the temperature and ratio, the yield of activated carbon decrease which meant that temperature and ratio had an inverse relation with the yield of activated-carbon. Also, it was concluded that the optimal value of yield was 45% at 400°C and IR2 for KOH activated carbon, while for sugarcane trash waste; the yield was 37% under similar conditions. Sulfuric acid activation resulted in decreased yields due to its dehydrating effect, with optimal yields obtained at 400°C and specific activation ratios for each biomass (31% for sugarcane trash and 38% for

cotton seed waste). The BET surface characterization of AC revealed that the surface area of AC prepared from biomass waste, including sugarcane trash and cottonseed waste, was affected by the carbonization temperature. The surface characterization by BET showed a trend of increasing surface area up to 500°C, followed by a reduction at higher temperatures due to pore closure. There were clear distinctions between KOH and H₂SO₄ activation in this process. In particular, KOH activation exhibited a greater surface area than H₂SO₄ activation. In the 400–500°C temperature range, the surface area of KOH-activated AC increased from 645.8 to 710.5m²/g, whereas the surface area of H₂SO₄ activated AC increased from 495.9 to 546.7m²/g in the same temperature range. The increase in surface area observed by elevating the temperature from 400 to 500°C was attributed to enhanced pore development. However, beyond 500°C, a decline in surface area occurred due to intense heat effects, leading to the evolution of volatile matter and subsequent pore shrinkage at 600°C. Notably, the BET analysis identified the optimal conditions for the AC surface area to be 780.4m²/g, achieved at 500°C for KOH-activated carbon. FTIR spectra for the activated carbon made from sugarcane waste and cottonseed waste indicated the Absorption peaks, which suggested the presence of C-C, C-H. And C-O and O-H, respectively. The identification of useful components at the

surface of the ACs was essential because it could also assist in describing the sort and extent of sorption occurring on the surface of the hydrocarbon with a specific substrate. The adsorption studies tested against MB adsorption through using different adsorption conditions using various adsorption parameters, such as the effect of adsorbent dosage, contact time, pH, initial dye concentration which indicated that KOH Activated carbon from cotton seed waste showed the best MB adsorption and optimal conditions for adsorption were at pH 12, initial dye concentration of 10mg/l, contact time of 140 min and adsorbent dosage of 0.1g. Adsorption studies and characterization studies showed that KOH-activated carbon from cotton seed waste had optimal results compared to sugar cane waste activated carbon. Additionally, this study demonstrated that produced AC could be effectively utilized for waste water treatment and the treatment of different effluents, thereby helping to reduce the disposal biomass waste to the environment [27].

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ABBREVIATIONS

<i>A</i>	Area, m ²
AC	Activated Carbon
<i>C_p</i>	Specific heat capacity, J/(kg °C)
<i>D</i>	Cylinder diameter, m
<i>g</i>	Gravitational constant, m/s ²
<i>h</i>	Heat transfer coefficient, W/(m ² °C)
<i>I</i>	Current, A
<i>K</i>	Thermal conductivity, W/ (m°C)
<i>L</i>	Fin length, m
MB	Methylene Blue
<i>N</i>	Fins number
<i>Nu</i>	Nusselt number
<i>Q</i>	Input heat, W
<i>R</i>	Fin radius, m
<i>Ra</i>	Rayleigh number
<i>R_{th}</i>	Thermal resistance, °C/W
<i>t</i>	Fin thickness, m
<i>T</i>	Temperature, °C
Greek symbols	
α	Thermal diffusivity, m ² /s
β	Expansion coefficient, 1/K
μ	Dynamic viscosity, kg/ (m s)
ν	Kinematic viscosity, m ² /s
ρ	Density, kg/m ³
ε	Emissivity
Φ	Input voltage, V
Subscripts	
<i>B</i>	base
<i>In</i>	input
∞	Ambient

REFERENCES

- [1] Chanzu HA, Onyari JM, Shiundu PM. **Biosorption of Malachite Green from Aqueous Solutions onto Polylactide/Spent Brewery Grains Films: Kinetic and Equilibrium Studies.** *Journal of Polymers and the Environment* 2012; **20**(3): 665-672.
- [2] Chaudhari A. **Adsorption Capacity of Activated Carbon Prepared by Chemical Activation of Lignin for the Removal of Methylene Blue Dye.** *International Journal of Advanced Research in Chemical Science* 2015; **2**(8): 1-13.
- [3] Crini G, Lichtfouse E. **Advantages and Disadvantages of Techniques Used for Wastewater Treatment.** *Environmental Chemistry Letters* 2019; **17**(1): 145-155.
- [4] Donkadokula NY, Kola AK, Naz I, Saroj D. **A Review on Advanced Physico-Chemical and Biological Textile Dye Wastewater Treatment Techniques.** *Reviews in Environmental Science and Bio/Technology* 2020; **19**(3): 543-560.
- [5] El-Shafey EI, Ali SNF, Al-Busafi S, Al-Lawati HA. **Preparation and Characterization of Surface Functionalized Activated Carbons from Date Palm Leaflets and Application for Methylene Blue Removal.** *Journal of Environmental Chemical Engineering* 2016; **4**(3): 2713-2724.
- [6] Feng P, Li J, Wang H, Xu Z. **Biomass-Based Activated Carbon and Activators: Preparation of Activated Carbon from Corncob by Chemical Activation with Biomass Pyrolysis Liquids.** *ACS Omega* 2020; **5**(37): 24064-24072.
- [7] Foo KY, Hameed BH. **Porous Structure and Adsorptive Properties of Pineapple Peel Based Activated Carbons Prepared via Microwave Assisted KOH and K₂CO₃ Activation.** *Microporous and Mesoporous Materials* 2012; **148**(1): 191-195.
- [8] Ghaedi M, Mazaheri H, Khodadoust S, Hajati S, Purkait MK. **Application of Central Composite Design for Simultaneous Removal of Methylene Blue and Pb²⁺ Ions by Walnut Wood Activated Carbon.** *Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy* 2015; **135**: 479-490.
- [9] Gonawala K, Mehta MJ. **Removal of Color from Different Dye Wastewater by Using Ferric Oxide as an Adsorbent.** *International Journal*

- of *Engineering Research and Applications* 2014; **4**(5): 102-109.
- [10] Hadi P, Xu M, Ning C, Sze Ki Lin C, McKay G. **A Critical Review on Preparation, Characterization and Utilization of Sludge-Derived Activated Carbons for Wastewater Treatment.** *Chemical Engineering Journal* 2015; **260**: 895-906.
- [11] Husien S, El-taweel RM, Salim AI, Fahim IS, Said LA, Radwan AG. **Review of Activated Carbon Adsorbent Material for Textile Dyes Removal: Preparation, and Modeling.** *Current Research in Green and Sustainable Chemistry* 2022; **5**: 100325.
- [12] Nasrullah A, Saad B, Bhat AH, Khan AS, Danish M, Isa MH. **Mangosteen Peel Waste as a Sustainable Precursor for High Surface Area Mesoporous Activated Carbon: Characterization and Application for Methylene Blue Removal.** *Journal of Cleaner Production* 2019; **211**: 1190-1200.
- [13] Ogungbenro AE, Quang DV, Al-Ali K, Vega LF, Abu-Zahra MRM. **Physical Synthesis and Characterization of Activated Carbon from Date Seeds for CO₂ Capture.** *Journal of Environmental Chemical Engineering* 2018; **6**(4): 4245-4252.
- [14] Pallares J, González-Cencerrado A, Arauzo I. **Production and Characterization of Activated Carbon from Barley Straw by Physical Activation with Carbon Dioxide and Steam.** *Biomass and Bioenergy* 2018; **115**: 64-73.
- [15] Raper E, Stephenson T, Anderson DR, Fisher R, Soares A. **Industrial Wastewater Treatment Through Bioaugmentation.** *Process Safety and Environmental Protection* 2018; **118**: 178-187.
- [16] Rashid R, Shafiq I, Akhter P, Iqbal MJ, Hussain M. **A State-of-the-Art Review on Wastewater Treatment Techniques, the Effectiveness of Adsorption Method.** *Environmental Science and Pollution Research* 2021; **28**(8): 9050-9066.
- [17] Rashid RA, Jawad AH, Ishak MAM, Kasim NN. **FeCl₃-Activated Carbon Developed from Coconut Leaves: Characterization and Application for Methylene Blue Removal.** *Sains Malaysiana* 2018; **47**(3): 603-610.
- [18] Rawal S, Joshi B, Kumar Y. **Synthesis and Characterization of Activated Carbon from the Biomass of Saccharum Bengalense for Electrochemical Supercapacitors.** *Journal of Energy Storage* 2018; **20**: 418-426.
- [19] Shah A, Shah M. **Characterization and Bioremediation of Wastewater: A Review Exploring Bioremediation as a Sustainable Technique for Pharmaceutical Wastewater.** *Groundwater for Sustainable Development* 2020; **11**: 100383.
- [20] Shrestha R. **Characterization of Activated Carbons Prepared from a Locally Available Material by Iodine Number.** *Journal of the Institute of Engineering* 2017; **13**(1): 139-144.
- [21] Shrestha R, Ban S, Devkota S, Sharma S, Joshi R, Tiwari AP. **Technological Trends in Heavy Metals Removal from Industrial Wastewater: A Review.** *Journal of Environmental Chemical Engineering* 2021; **9**(4): 105688.
- [22] Thompson KA, Shimabuku KK, Kearns JP, Knappe DRU, Summers RS, Cook SM. **Environmental Comparison of Biochar and Activated Carbon for Tertiary Wastewater Treatment.** *Environmental Science & Technology* 2016; **50**(20): 11253-11262.
- [23] Wang H, Xu J, Liu X, Sheng L. **Preparation of Straw Activated Carbon and Its Application in Wastewater Treatment: A Review.** *Journal of Cleaner Production* 2021; **283**: 124671.
- [24] Wang WL, Hu HY, Liu X, Shi HX, Zhou TH, Wang C. **Combination of Catalytic Ozonation by Regenerated Granular Activated Carbon (rGAC) and Biological Activated Carbon in the Advanced Treatment of Textile Wastewater for Reclamation.** *Chemosphere* 2019; **231**: 369-377.
- [25] Xu J, Chen L, Qu H, Jiao Y, Xie J, Xing G. **Preparation and Characterization of Activated Carbon from Reedy Grass Leaves by Chemical Activation with H₃PO₄.** *Applied Surface Science* 2014; **320**: 674-680.
- [26] Zhang F, Ge Z, Grimaud J, Hurst J, He Z. **Long-Term Performance of Liter-Scale Microbial Fuel Cells Treating Primary Effluent Installed in a Municipal Wastewater Treatment Facility.** *Environmental Science & Technology* 2013; **47**(9): 4941-4948.
- [27] Zhang G, Yang H, Jiang M, Zhang Q. **Preparation and Characterization of Activated Carbon Derived from Deashing Coal Slime with ZnCl₂ Activation.** *Colloids and Surfaces A: Physicochemical and Engineering Aspects* 2022; **641**: 128512.