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Physicochemical Characterization and Adsorptive Performance of Agro-based Adsorbent for Methylene Blue Removal from Aqueous Solution

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Abstract—The discharge of synthetic dyes into aquatic systems poses a persistent environmental challenge because of their recalcitrance and potential impacts on water quality and human health. In this study, jujube seed-derived adsorbent (JJS) was developed and evaluated for the removal of methylene blue (MB) from aqueous media. The adsorbent exhibited favorable physicochemical properties, including a carbon yield of 72.42 %, bulk density of 0.59g/cm^{-3}, pH of 8.61, iodine number of 556.89 mg/g-1, moisture content of 7.62 %, ash content of 1.40 % volatile matter of 0.60, and a point of zero charge (pH$_{pzc}$) of 7.0. Brunauer-Emmett-Teller (BET) analysis revealed a high specific of 940.650 m^2/g^{-1}, indicating adsorptive surface. Fourier-Transform Infrared spectroscopy (FT-IR) identified a pronounced hydroxyl (-OH) band at 3417.98 cm^{-1}, suggesting the contribution of oxygen containing surface functionalities of MB uptake. Scanning Electron Microscopy (SEM) revealed a high porous morphology, while Energy Dispersive Xay Spectroscopy (EDX) confirmed carbon (83.08 %), oxygen (15.66 %), and calcium (0.69 %) as the major detected elements. The JJS adsorbent demonstrated consistently high MB removal efficiencies over three treatment cycles, achieving 96.04 %, 97.60 % and 99.84 % removal in cycles 1, 2, and 3 respectively. These results demonstrate that jujube seed-derived biomass possesses favorable physicochemical and structural characteristics for dye adsorption and can serve as a promising low-cost and environmentally sustainable precursor for the development of adsorbents for MB-contaminated aqueous effluents, particularly wastewater associated with tie-and-dye activities.

Keywords—Adsorbent, Dye, Jujube seed, Effluent, Methylene blue

I. INTRODUCTION

In textile industry, large amount of effluent is discharged from dyeing and finishing processes whereby 10–15 % of dyes are estimated to be lost in the effluent (Godwin *et al.*, 2022). The requirement to minimize residual dyes in the effluent becomes a major environmental concern due to the stringent restriction and regulation has been established on industrial effluents. Although wastewater undergoes conventional wastewater treatment before being discharged into aquatic systems, a low concentration of dyes remaining in the effluent water along with other toxic chemical substances can affect the aesthetic and transparency of water stream (Luo *et al.*, 2019).

Besides, dyes discharged into the aquatic system not only reduce the dissolved oxygen concentration but also affect the aquatic ecosystem resulted from the toxicological properties of dyes, since most of the dyes are known to be toxic, carcinogenic, teratogenic and mutagenic toward the aquatic life. In addition to that, it can also lead to a decrease in sunlight (Mohamed and Ahmed 2017). With the prediction of freshwater shortages in the future due to climate change, treated effluent becomes second water source for non-potable usage in industrial, businesses and household sectors (Muktar *et al.,* 2021). The colour removal process thus becomes one of the crucial steps to ensure the quality of the reclaimed water, but additional procedures in the textile effluent treatment inevitably increase the cost of overall treatment and cause harmful secondary pollution (Saini, 2017).

The discharge of effluents to the various water bodies are one of the potential sources of environmental contamination and pollution as it is reported that there are over 100,000 commercially available dyes with a production of over 7×10^5 metric tons per year (Ugbe and Abdus-Salam, 2020; Kuang *et al.,* 2020). Obvious, the growth of textile industry in Nigeria contributes a lot of industrial wastewaters generated in the country (Elmorsi *et al.,* 2019). The consequences of dyes in water bodies are huge. However, 1.0 mg/l of dye present in drinking water can significantly affect the color and make the water unhealthy for human consumption (Borah, 2015). Therefore, to avoid these hazardous effects, textile wastewater must be adequately treated before they can be discharged into receiving water bodies.

Therefore, there is serious need and attention to make efforts to reduce the wastewater generation in order to provide clean water that meets our teeming population needs as well as sustainable development goals. However, this research has revealed the analytical secrets of this suitable novel adsorbent Jujube Seeds (*Ziziphus mauritiana*) for removal of methylene blue (MB) in aqueous solution through physicochemical characterization and adsorptive performance.

II. MATERIALS AND METHODS

Study area

Maiduguri urban with a land mass of 137.356Sq km (NPC, 2016), is located between latitude N 11°46'18" to N 11°53'21" and longitude E 13°03'23" to E 13°14'19" (Google Earth, 2026). The area lies within the lake Chad Basin formation, which is an area formed as a result of down-warping during the Pleistocene period (Waziri, 2012). According to the 2006 population census, the population of Maiduguri was estimated at 1.275 million people, comprising mainly the Kanuri, Shuwa, Babur-Bura and other indigenous and non-indigenous ethnic groups. This population live in about 10,334,46 households (NPC, 2016). Maiduguri climate is characterized by dry and wet seasons. Four distinct seasons are identified by natives of the city. These are: cool dry (harmattan season) from December to February; hot dry season from march to late may; raining season from June to September; and humid dry transitional period between September and November (Mukhtar *et al.,* 2021). The daily average temperature are high throughout the year ranging between 25 ºC to 44 ºC, where lower temperature are recorded in January and the highest in April (Mukhtar *et al.,* 2019).

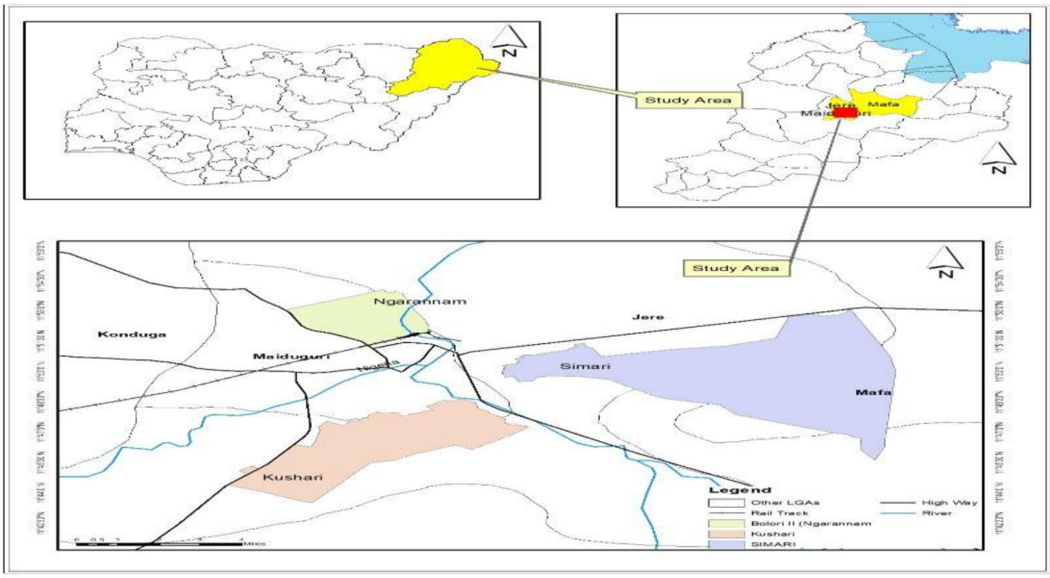

Fig. 1: Map of the Sample collected Area

Sample Collection and Preparation

The Jujube sample were purchased from custom market, Maiduguri and the seeds were wash thoroughly with deionized water and carbonized at 400 ºC on a pyrolyzer under nitrogen condition in the Department of Chemistry Research laboratory, Kashim Ibrahim University, KIU.

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Fig. 2: Jujube (*Ziziphus mauritiana*) seeds

Physicochemical Analysis

The activated sample was characterized by determining some physico-chemical parameters such as ash content, bulk density, volatile matter, iodine number, carbon yield, moisture content, pH_{pzc}, and pH using standard analytical methods according to slightly adopted method from (Abdus-Salam and Ikudayisi, 2017; Abdullahi *et al.,* 2026).

Instrumental Characterization Analysis

The phase composition of the Jujube (*Ziziphus mauritiana*) seeds samples were analyzed using instruments/equipment for various characterization analyses are; Scanning Electron Microscopy (SEM) to observe the morphology and particle size of the adsorbents, Energy Dispersive X-ray Spectroscopy (EDX) to determine the elemental composition of the adsorbents, Brunartt Emmett Teller (BET) for surface area analysis and pore size, X-ray Diffraction (XRD) use to determine the phase, the degree of crystallinity and Fourier Transmission Infra-Red (FTIR) for investigation of the surface functional group of the adsorbent. These instrumental analyses were achieved according to standard methods (Bundi *et al.,* 2026).

III. RESULTS AND DISCUSSION

The Jujube seeds particles synthesized is fine in texture and insoluble in water but readily soluble in acids. It is weakly magnetic and leaves a black stain on fabrics (Alkali and Fatimah, 2023). consequently, other physico-chemical characteristics of the Jujube seed which give comprehensive information and insights into the adsorption behavior of the activated carbon includes; the percentage yield, bulk density, iodine number, moisture content, ash content, organic matter and surface area which are summarized, recorded and presented in Table 1.

Table 1: Summary of Physico-chemical characteristics of the Adsorbent

Sample Code	Carbon Yield (%)	Bulk Density (g/ml)	pH	pH_{pzc}	Iodine Number (mg/g)	Moisture Content (%)	Ash Content (%)	Volatile Matter (%)
JJS	72.42	0.59	8.61	7.0	556.89	7.62	1.4	0.6

Physicochemical Analysis

Point of Zero Charge

The point of zero charge (pH_{pzc}) is the pH at which the total number of positive and negative charges on the surface of the adsorbents becomes zero (Abdus-Salam and Adekola, 2005). This parameter is very important in the adsorption phenomena, especially when electrostatic forces are involved in the mechanisms (Sulejmanović *et al*., 2019). The pH_{pzc} is the point where the curve of pH final versus pH initial intersects (Nguyen *et al*., 2019). The point of zero charge for JJS was found to be 7.0 as shown in Fig. 2. The zero-point charge of JJS is below the solution pH (pH = 7.8) and hence the negative charge density on the surface of the JJS increased, which favors the adsorption processes (Aliabadi *et al*., 2018). The results from Table 1 shows that at pH > pH_{pzc} the surface is negatively charged which favors the adsorption of positively charged species (Nwosu *et al*., 2018).

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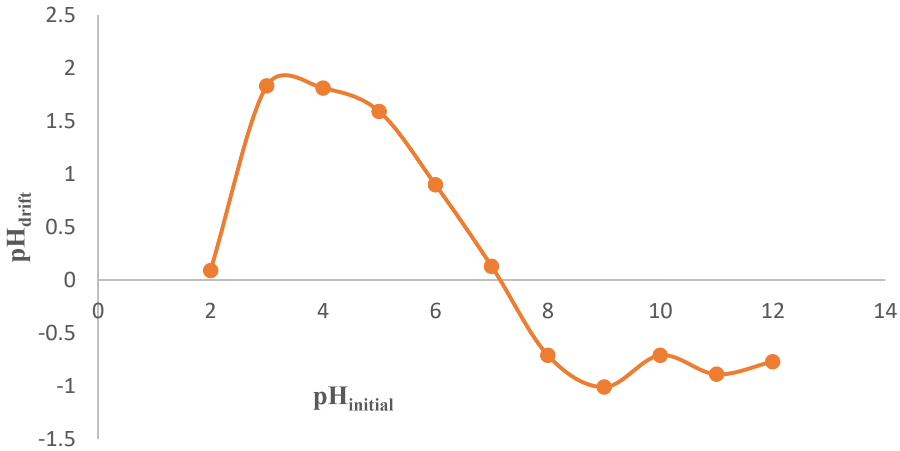

Fig. 3: Graph of pH_{pzc} of JJS

Bulk Density

Bulk density is defined as weight of fiber per unit volume, often expressed as g/mL. A high bulk density is very important in packaging and transportation and is desirable as it can significantly reduce cost (Adebayo *et al*., 2016). The result obtained was 0.59 g/mL as shown in Table 1. However, the values obtained were higher than 0.2 to 0.3 mg/L range reported by Adebayo *et al*., (2016); lower than 0.8 to 1.2 g/mL by Adegoke *et al*., (2014) and lower than 1.03 to 1.86 g/mL by Nwosu *et al*., (2018).

Iodine number

Iodine number is a widely used parameter for activated carbon testing for its simplicity and a rapid assessment of adsorbent quality. It gives an estimate of its surface area and porosity (Ekpete and Horsfall, 2011). The results of iodine number as shown in Table 1 was 556.89 mg/g. The value obtained was below 822.2 mg/g reported by Nwosu *et al*., (2017).

Moisture Content

Moisture content of the sample was determined and it was observed that the moisture content of JJS was 7.62 %. The ability of a sample to absorb moisture when placed in humid environment suggests good applicability of the sample to adsorption processes (Falah *et al*., 2015). The values obtained were higher than 3.88 % of Saygili and Guzel, (2017). The low moisture content shows that the particle density is relatively low and the material could be best for adsorption process (Buhari *et al*., 2020).

Carbon Yield

It can also be seen from Table 1 that the carbon yield was 72.42 %. This could be attributed to the release of volatiles during carbonization to eliminate non-carbon species (Gao *et al*., 2013). The values obtained in this characterization were higher than 48.83 % result reported by Saygili and Guzel, (2017).

Ash Content

It is observed that the ash content analysis in carbonization process is simply the burning away of organic and volatile contents, leaving the inorganic minerals (Viena and Nizar, 2019). The ash content generally gives insight on overall activity of activated carbon, the lower the ash content, therefore, the better the performance of the adsorbent material (Hesas *et al*., 2013). The values of the ash content obtained from Table 1. 1.4 % which was lower than 2.89 % obtained by (Abdus-Salam and Buhari, 2016).

Volatile Matter

Volatile matter is one of the most common parameters measured in adsorbent samples. It is measured as a weight percent of gas emissions from a sample that is released during heating to 950 °C in an oxygen free environment (Viena and Nizar, 2019). The volatile matter revealed 0.6 % as shown in Table 1. The value obtained was lower than the value reported by Ekpete *et al*., (2017). The lower values of ash content and volatile matter may predict the effectiveness of the adsorbent in adsorption process.

Instrumental Characterization Analysis

The SEM micrograph of JJS was shown in Fig. 4. The JJS image revealed that surfaces contained an abundance of large numerous pores which enhanced its potential in adsorption studies and may facilitate ion binding active sites (Kuang *et al.,* 2020). The structure obtained was in concordance with the result by Nwosu *et al*., (2019).

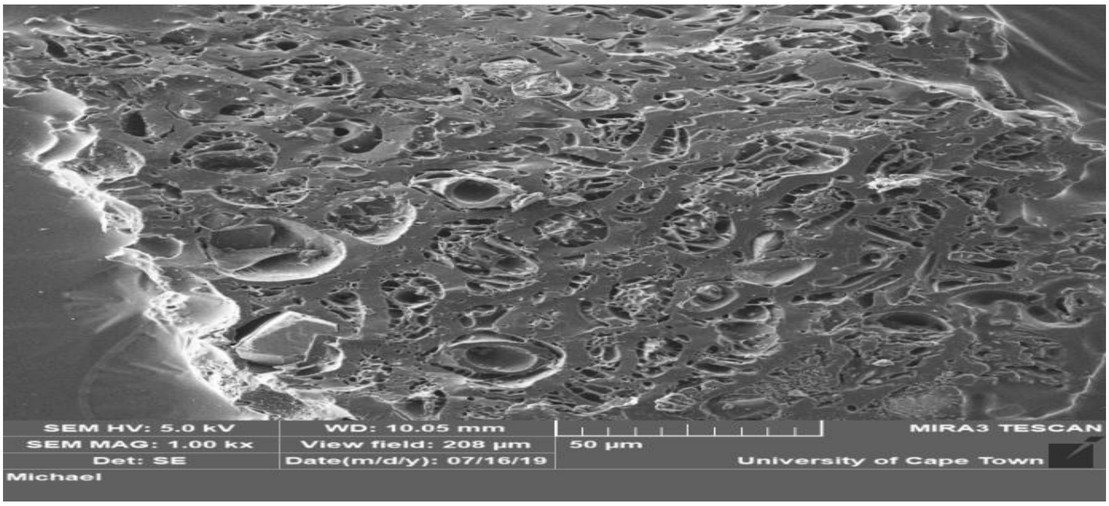

Fig. 4: SEM Micrograph of JJS

Table 2: Summary of Elemental Composition of the Adsorbent (JJS)

Elements	C	O	Ca	K	S	P	Total (%)
Values (%)	83.08	15.66	0.69	0.33	0.15	0.09	100

The major elements confirmed by the EDX analysis are carbon, oxygen, Calcium, Potassium, Sulphur and Phosphorous are 83.08 %, 15.66 %, 0.69 %, 0.33 %, 0.15 % and 0.09 % respectively. The spectrum for this result was presented in Fig 5. The oxygen atom in activated carbon can have strong effects on the adsorption of contaminants, especially when it presents to an edge of carbon surface (Alam *et al*., 2020). Many studies showed that ion adsorption onto activated carbon occurs due to the ion exchange with protons in oxygen functional groups (Nieto-Márquez *et al*., 2017).

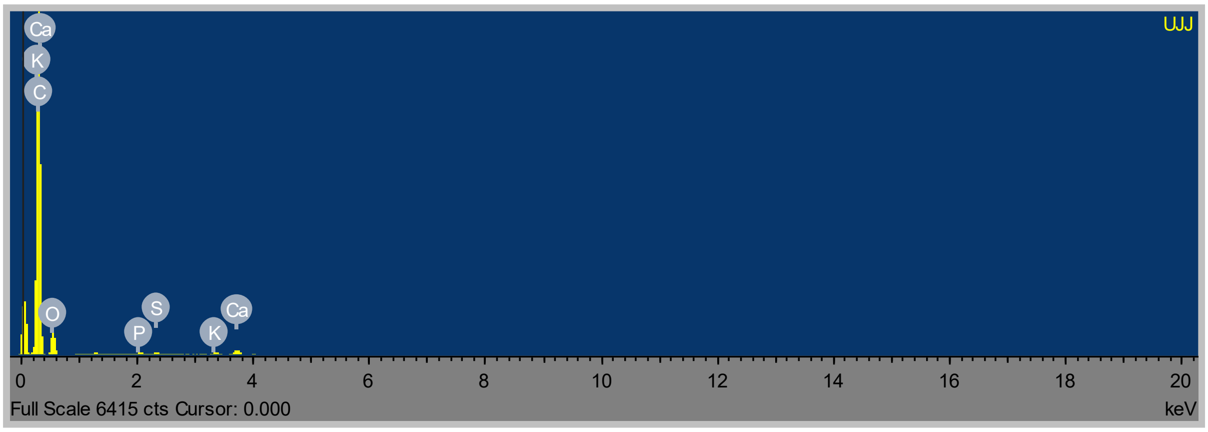

Fig. 5: EDX Spectrum of JJS

Table 3: Summary of the Assignment of some major peaks on the Adsorbent

Functional Groups	-OH stretching vib.	C=O stretching vib.	Ref.
Frequencies	3417.98	1614.47	Alkali and Kyari, 2026

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Surface chemistry is the main parameter for determining the adsorption capacity of activated carbon (Labied *et al*., 2018). As such, the FTIR spectrum of the JJS was presented in Fig. 6. The absorption peak of JJS at 3417.98 and 1614.47 cm^{-1} corresponded to the O-H groups of phenols or alcohols and C=O carboxylic acids contained in cellulose, pectin and lignin respectively (Šabanović *et al*., 2020). The results obtained are with conformity with the composite, vibrational peaks at about 3421.83 cm^{-1}, 1618.33 cm^{-1} ascribed by (Alkali and Kyari, 2026).

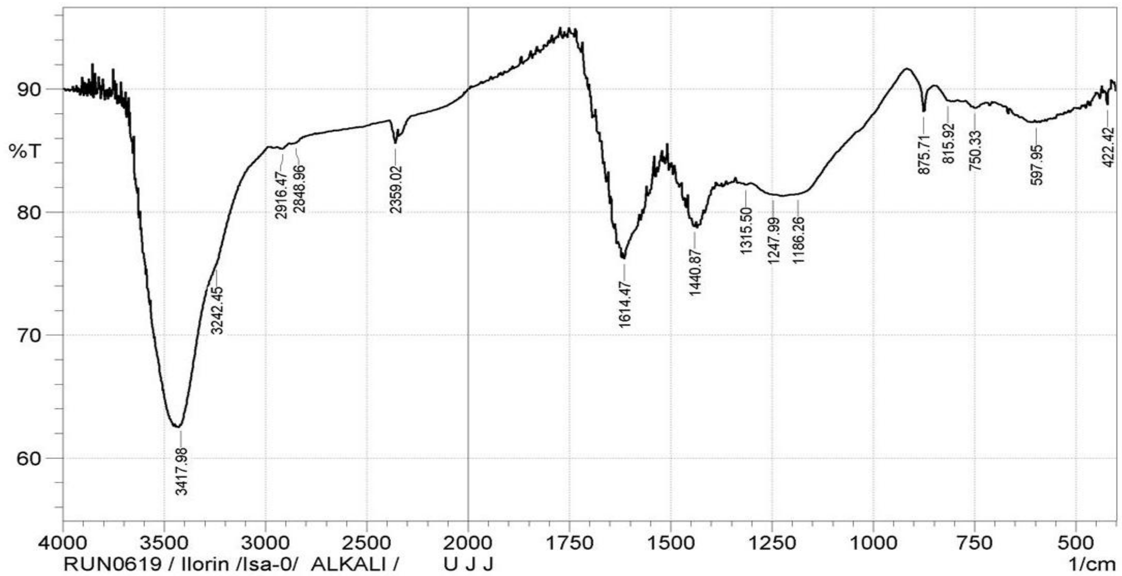

Fig. 6: FT-IR spectrum of JJS

BET

Table 4: Summary of BET Analysis of the Adsorbent

BET Parameter	Results
Surface Area (m^2/g)	
Single point surface area at P/P_o	940.650
t-Plot micro-pore area	998.90
t-Plot external surface area	670.520
Langmuir Surface Area	670.520
BJH Adsorption cumulative surface area of pores	870.240
BJH Desorption cumulative surface area of pores	640.250
Pore volume (cm^3/g)	
Single point adsorption total pores volume	0.604460
t-Plot micro-pore volume	0.150050
BJH Desorption cumulative volume of pores	0.451203
Pore size (nm)	
Adsorption average pore width (4V/A by BET)	3.25350
Desorption average pore width (4V/A by BET)	3.26750
BJH Adsorption average pore width (4 V/A)	4.4450
BJH Desorption average pore width (4V/A)L	5.0855

BET = Brunauer-Emmett-Teller, Surface Area Analysis BJH = Barrett-Joyner-Halenda, Pore Size and Volume Analysis

The N_2 physiorption isotherm of the JJS was displayed in Fig. 4. The summary of the BET surface area results were presented in Table 4. It can be observed from Table 4 that BET surface area of JJS was 940.650 m^2/g. The surface area results obtained are higher compared with 246 m^2/g value report for Jujube pit adsorbent by Gao *et al*., (2020). These values are within the minimum range of 500 to 1500 m^2/g needed for industrial application and removal of small molecules from aqueous solution (Nwosu *et al*., 2019). Similarly, the N_2 adsorption-desorption isotherms results showed that all the adsorbents

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belong to type IV isotherms with values in the range of 2 to 5 nm according to IUPAC classification of adsorption isotherm (Ayob *et al*., 2021). The pore volume and pore area distributions of the adsorbents determined from the BET were shown in Table 4 indicating mesoporous nanomaterial with capillary condensation (Ayob *et al*., 2021).

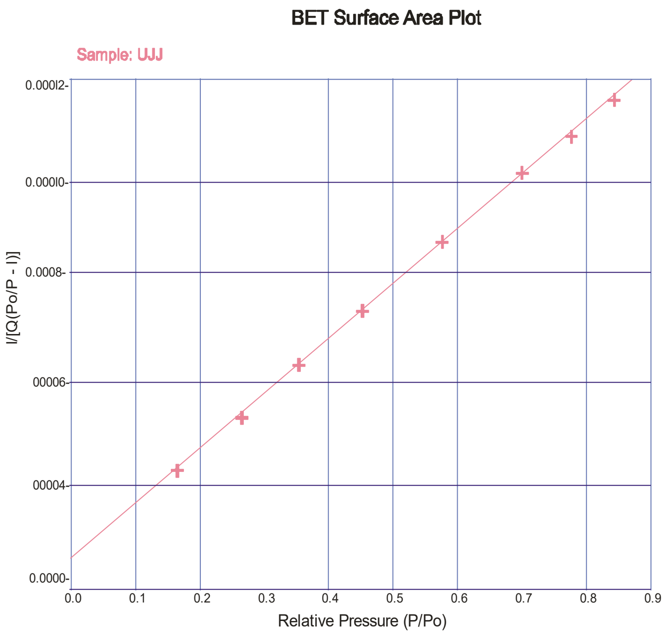

Fig. 7: BET plot of JJS

XRD Analysis

Table 5: List of scattering angles of Adsorbent

Adsorbent Code	Observed scattering angles, 2θ°
JJS	15.96°, 18.80°, 24.26°, 30.78°, 40.84°, 50.45°, 52.98°, 65.02°, 72.86°, 78.84°

Adsorption reaction may lead to changes in molecular and crystalline structure of the adsorbent, hence XRD will provide plausible explanation on the proportion of each crystalline phase of the adsorbent. An overlay of the diffractograms for the JJS was shown in Figs. 5. Similarly, the list of scattering angles of 2θ° for the adsorbent was also presented in Table 5.

It can be observed from the information of XRD that the diffraction peaks at 2θ = 40.84° is related to graphene structure of the Jujube Seeds (JJS) and the value obtained was in agreement with literature reported by (Altalhi *et al*., 2021; Nemeth *et al*., 2018). The prominent diffraction peaks in the adsorbent were in the range of 20° < 2θ < 80° which is common to all activated carbon powders, and corresponds to the sp^3 lattice reflections (Iijima, 1991). The positions of all maxima coincided with the peaks characteristics of crystalline structure of cellulose and all the peaks obtained were in agreement with literature report of (Ayob *et al*., 2021; Bundi *et al.,* 2026). Obviously, the peaks obtained are in accordance with the International Centre for Diffraction Database (ICDD) and Joint Committee on Powder Diffraction System (JCPD) respectively.

Effluent Treatment Analysis Results

The results obtained for the effluent analysis of MB unto the JJS was presented in Table 6.

Table 6: Level of MB from the Effluent

Adsorbent	Raw Conc. (mg/L)	Conc. after adsorption (mg/L)	Amount removed (mg/L)	Permissible limit (EPA, 2021) (mg/L)	% Removal

Cycle 1	0.924	0.008	0.916	0.2	91.34
Cycle 2		0.005	0.919		99.46
Cycle 3		0.001	0.923		99.89

It could be observed from the Table that, the raw wastewater from tie and dye has value of MB value: 0.924 mg/L. The raw wastewater value obtained were higher than the maximum permissible limit of MB concentrations in industrial effluent recommended by US/EPA (0.2 mg/L) (2021). As such, it could be major contribution to many life-threatening diseases to microorganism in the soil, and their bioaccumulation by aquatic organisms. Some these effects include; severe damage to kidney, brain, liver, reproductive and nervous systems as well as oxidative stress and impaired respiratory infections in human body (Wang *et al*., 2019) Therefore, there is need for continuously protection of the environment, human health and the aquatic life through the use adsorbents for the removal of toxic dyes from wastewater (Hani *et al*., 2019).

However, after the adsorption, the percentage removal of the MB in three (3) cycles from the wastewater were presented as follows: cycle 1 = 96.04 %, cycle 2 = 97.6 % and cycle 3 = 99.84 % respectively. These novel results confirmed the great potentials of this adsorbent (JJS) in the treatment of wastewater from the tie and dye. Consequently, the results obtained were in conformity with literature reports (Ugbe and Abdus-Salam, 2020).

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Disclosure Statement
No potential conflict of interest was reported by the authors.

Conflicts of Interest:
The authors declare no conflict of interest.

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