International Journal of Research in Engineering, Science and Management
Volume 7, Issue 7, July 2024
https://www.ijresm.com | ISSN (Online): 2581-5792

Removal of Methylene Blue from Waste Water using Yam Peel Adsorbent

Ojo Francis Kolawole[1*], Ogunyemi T. Babatunde[2], Balogun Nifemi[3]
[1]Lecturer, Department of Chemical Sciences, Bingham University, Karu, Nasarawa, Nigeria
[2]Lecturer, Department of Chemistry, Federal University Otuoke, Bayelsa, Nigeria
[3]Student, Department of Chemical Sciences, Bingham University, Karu, Nasarawa, Nigeria

***Abstract*: The removal of methylene blue from waste water using yam peel as adsorbent was studied. The SEM and FTIR were used to characterize the adsorbent before and after adsorption. The effects of various experimental parameters, such as the contact time, methylene blue dye concentration, adsorbent dosage, PH and temperature on the adsorption process were investigated. The FTIR showed that the OH and C=O groups were the major sites for methylene blue uptake onto the yam peel adsorbent. SEM analysis revealed the porous and irregular structure of the adsorbent, thus contributing to the diffusion of methylene blue ions to the adsorbent surface. Isotherm modeling showed that the Langmuir model best fitted the adsorption data (R2 > 0.995). In addition, the separation parameter for the dye was less than unity indicating that yam peels waste biomass was an excellent adsorbent for methylene blue dye. Kinetics was well described by the pseudo-first order model. Thermodynamics revealed a physical, endothermic adsorption of methylene blue dye onto the adsorbent. The thermodynamics of the MB-YP system indicate that the adsorption process is spontaneous.**

***Keywords*: adsorption, methylene blue, yam peel, Langmuir isotherm.**

1. Introduction

Since the dawn of technology and man's pursuit for industrialization, pollution has been a troubling experience for humanity. The cosmetics, textile, food, plastic, carpet, printing, and leather sectors are among the largest users of dyes (Ge et al., 2019). The textile industry, with a global use of roughly 700,000 tons of dye per year, is the greatest consumer of dyes among these industries (Aguayo-Villarreal et al., 2020). As a result, a considerable amount of colors are released into the environment from textile manufacturing wastewaters. This results in serious water pollution, as well as severe negative effects on the ecosystem, which is cause for concern (Hizkeal et al., 2021). Water dyes are difficult to remove.

Due to their high stability, which enhances resistance to biological and photo-degradation, dyes are difficult to remove from water (El-Gamal et al., 2015). They also restrict light penetration into water bodies, inhibiting aquatic plants' photosynthetic activity (An et al., 2020a; Chen et al., 2018). Furthermore, their breakdown products in water can be mutagenic, carcinogenic, and harm the skin, brain, liver, and kidneys, as well as the central neurological and reproductive systems (Vasanth, and Kumar, 2005).

Solvent extraction, ion exchange, precipitation, oxidation, reduction, photocatalytic degradation, filtration, coagulation, flocculation, and adsorption are commonly used to remove dyes and other contaminants from industrial effluent (Ge et al., 2019).

Chemical precipitation, solvent extraction, membrane separation, ion exchange, electrolytic techniques, coagulation/ flotation, sedimentation, filtration, membrane process, biological processes, chemical oxidation or reduction, and adsorption have all been used to remove dyes and pollutants from industrial waste water over the years (El-Zahhar et al., 2014).

The majority of these approaches, on the other hand, are capital costly, difficult, inefficient, and require specialized employees. Because of its simplicity, cheap cost, effectiveness, biodegradability, and reusability, the adsorption of pollutants onto bio-materials, known as biosorption, is the preferred method (An et al., 2019a; Crini et al., 2019; Dil et al., 2016; Kocaman, 2020). However, the majority of adsorption experiments have been conducted using activated carbon, which has the disadvantage of being expensive.

As a result, today's scientists are focusing their efforts on the use of low-cost adsorbents for the uptake of pollutants like methylene blue dye. Microorganisms, yam peel, rice husk, orange peels, corncob, groundnut shell, cotton wastes, and sugarcane bagasse are just a few of the low-cost biomass adsorbents that have been employed (Das and Mondal, 2011; Amer et al., 2010; Hegazi, 2013). Yam peel (YP) is one of these biosorbents that has shown a high capacity for absorbing various types of pollutants (Akpomie and Conradie, 2020). Furthermore, after cassava, yam is the world's second largest tuber crop, grown in various parts of the world, particularly West Africa, resulting in massive amounts of peel bio-waste that can be easily harnessed (Pius, 2006).

However, this commonly accessible, low-cost, and effective bio-waste has yet to be used to remediate methylene blue dye-contaminated industrial waste water.

The purpose of this study is to look into the usage of yam peel as a cheap and effective adsorbent for removing methylene blue from industrial waste water.

*Corresponding author: fktdavid2@gmail.com

2. Materials and Method

A. Chemicals and Materials

Methylene blue dye, zinc sulphate (ZnSO4), sodium hydroxide (NaOH), sodium chloride (NaCl), nitric acid (HNO3), and hydrochloric acid were employed in this investigation (HCl). The chemicals were used simply as they have been obtained, with no further purification. Masaka market in Karu, Nasarawa state, Nigeria, was where the yam was purchased.

1) Preparation of yam peel adsorbent

The peels of the purchased yam were carefully removed and washed using tap water to remove any contaminants on the surface. After that, they peels were chopped and left to dry for 48 hours prior to getting oven dried for 24 hours (Labcon model). After that, the dried peels were pulverized into powder form with a pestle and mortar, afterwards sieved through a 100-m mesh sieve, and the finely ground yam peel was labeled as YP.

2) Adsorbent characterisation

The surface characteristics of the produced YP sorbent were determined to see if they affected the methylene blue dye absorption from solution. Fourier transform infrared (FTIR) spectroscopy was used to analyze the surface functional groups on the adsorbent materials (FTIR; Bruker Tensor 27 model). The morphology and particle size of the adsorbents were analyzed using field emission scanning electron microscopy (FE-SEM; JEOL model).

3) Adsorption experiments

In a 250 mL volumetric flask, an adequate amount (25.0 mg) of methylene blue dye was mixed with distilled water to have a stock solution with a concentration of 100 mg/L. Dilution was used to make concentrations of 5–25 mg/L from the stock. 0.1 M HCl or 0.1 M NaOH were used to modify the pH of these solutions to a range of 2.0–9.0. The batch approach was used to perform biosorption, with 0.05 g of adsorbent mixed with 10 mL of 25 mg/L methylene blue contaminated solution and agitated for 5 minutes. Variations in pH (3.0–9.0), contact time (30–270 min), methylene blue concentration (5–25 mg/L), temperature (303–313 K), and adsorbent dosage (0.01–0.05g) were investigated. The studied parameter was changed while the others were kept constant. For quality assurance, each experiment was repeated twice and the average was recorded. The filtrate was then tested for residual methylene blue using a UV spectrophotometer at a wavelength of 663 nm after centrifugation at 4000 rpm for 15 minutes.

The following formulae were used to calculate the uptake capacity (qe, mg/g) and percentage removal (R %) (Ezekoye et al., 2020)

- R(%) = (Co – Ce)/Co100 2
- Qe = (Co – Ce)v/m 1
- Qe = (Co – Ce)v/m 2
- Co and Ce in mg/L are the starting and equilibrium methylene blue dye concentrations, accordingly, where m (g) is the adsorbent weight and v (L) is the volume of solution.

The trials were carried out twice and the average values were reported.

3. Result and Discussion

A. Effect of Contact Time

The uptake capacity increased from 0.605 to 3.41 mg/g as the contact time increased from 30 to 270 minutes. The material had a corresponding increase in percentage MB removal from 12.1 to 68.2 %. At 240 minutes, equilibrium MB adsorption was achieved. The initial increase in MB adsorption was attributed to the abundance of active sites on the adsorbents, which were eventually depleted and saturated, resulting in equilibrium.

Table 1
Effect of contact time at pH 8.0, dye conc. 25 mg/L, Temp 303 K, dosage 0.05 g on methylene blue uptake onto YAM PEEL

Time (min)	Uptake capacity (q_e) (mg/g)	Removal (%)
30	0.605	12.1
60	1.020	20.4
90	1.510	30.2
120	2.265	45.3
150	2.720	54.4
180	3.055	61.1
210	3.310	66.2
240	3.410	68.2
270	3.410	68.2

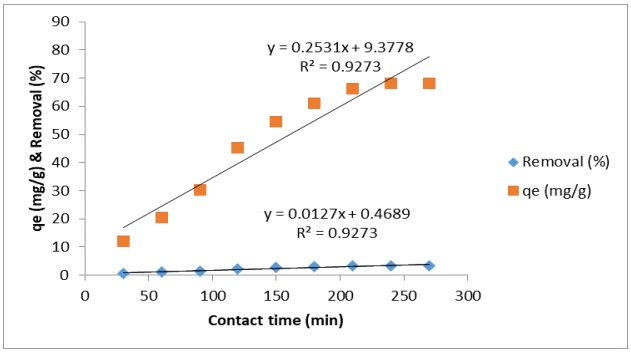

Fig. 1. Effect of contact time on methylene blue removal and uptake unto YAM PEEL at pH 8.0, dye conc. 25 mg/L, Temp 303 K, dosage 0.05 g

B. Effect of Concentration of MB on Adsorption

The uptake capacity (q_e) for YP increased from 0.803 to 3.265 mg/g when the MB concentration was increased from 5 to 25 mg/g, as shown. Higher dye concentrations result in greater interaction of MB dye with active groups on the surface of adsorbent materials, resulting in higher uptake (Dehmani et al., 2020). On the other hand, we detected a drop in total percentage biosorption from 80.3 to 65.3 percent at the same time. This could be because the active sites on the surfaces of the adsorbents can easily absorb MB from solution at low dye concentrations, but become saturated at higher concentrations, leaving more MB in solution.

Table 2
Effect of Dye concentration on methylene blue onto YAM PEEL at at pH 8.0, time 240 mins, Temp 303 K, dosage 0.05 g

Dye conc. (mg/L)	Uptake capacity (q_e) (mg/g)	Removal (%)
5	0.803	80.3
10	1.532	76.6
15	2.174	72.5
20	2.776	69.4
25	3.265	65.3

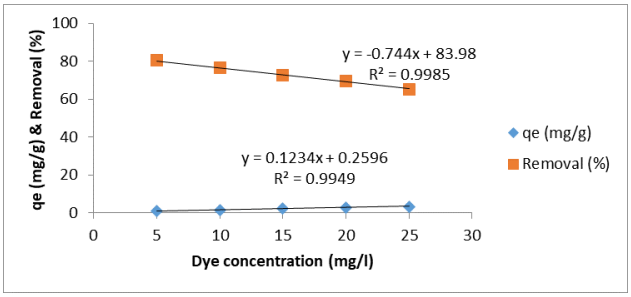

Fig. 2. Effect of contact time on methylene blue removal and uptake unto YAM PEEL at pH 8.0, dye conc. 25 mg/L, Temp 303 K, dosage 0.05 g

C. *Effect of Adsorbent Dosage*

Figure 3 shows the removal of MB dye from solution onto YP as a function of biosorbent material dose. With an increase in biosorbent dosage from 0.01 to 0.05g, the adsorption uptake capacity of MB reduces dramatically from 7.53 to 3.36 mg/g. This is due to the availability of more active sites as dose increases, resulting in less dye molecules being adsorbed onto individual active sites for the removal of the same dye concentration (25 mg/L) of MB. The decrease in uptake capacity is also owing to increasing collision between biosorbent particles, leading to aggregation of the active sites as the biosorbent dosage is raised (Li et al., 2004). As a result, at larger biosorbent dosages, maximum utilization of the active sites was not reached. Conversely, at larger dosages, the presence of more active sites resulted in a greater overall clearance of MB molecules from solution. The removal of MB rose from 30.2 to 67.2 % when the biosorbent dosage was increased from 0.01 to 0.05 g. This demonstrates that the uptake capacity reflects the adsorbent material's properties, whereas the percentage removal is linked to the adsorbate or pollutant in solution (Ge et al., 2019).

Table 3
Effect of Adsorbent dosage on methylene blue onto YAM PEEL at pH 8.0, time 240 mins, Temp 303 K, dye conc. 25 mg/L

Adsorbent dosage (g)	Uptake capacity (q_e) (mg/g)	Removal (%)
0.01	7.550	30.2
0.02	4.425	35.4
0.03	4.175	50.1
0.04	3.881	62.1
0.05	3.360	67.2

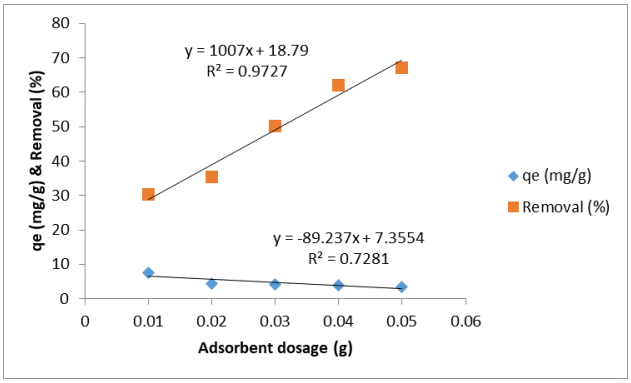

Fig. 3. Effect of contact time on methylene blue removal and uptake unto YAM PEEL at pH 8.0, dye conc. 25 mg/L, Temp 303 K, dosage 0.05 g

D. *Effect of Solution pH*

Because the pH determines the surface properties of the adsorbent, as well as the ionization and solubility of dyes, dye adsorption is greatly influenced by the pH of the effluent. Figures 7 and 8 show the dependency of MB adsorption onto YP as a function of solution pH. When the pH is raised from 3.0 to 9.0, the absorption of MB for YP increases from 1.86 to 3.5 mg/g. An increase of 37.2 percent to 70.1 percent in removal. Due to the cationic nature of the dye, a higher pH of the solution was beneficial for MB adsorption (Vasanth and Kumar, 2005). In literature, a consistent decline in BPB adsorption with decreasing pH was also observed (Kocaman, 2020). Electrostatic repulsion between the cationic MB molecules in solution and the increased positive charge on YP could explain the reduced MB removal at lower pH. Higher pH, on the other hand, resulted in electrostatic attraction between MB and the materials' negative surface sites (Kocaman, 2020). Electrostatic interaction must have played a key role in MB uptake onto the adsorbents, according to the results.

Table 4
Effect of pH on methylene blue onto YAM PEEL at dosage 0.05 g, time 240 mins, Temp 303 K, dye conc. 25 mg/L

Effect of pH	Uptake capacity (q_e) (mg/g)	Removal (%)
3	1.86	37.2
4	2.155	43.1
5	2.76	55.2
6	3.12	62.4
7	3.405	68.1
8	3.505	70.1
9	3.505	70.1

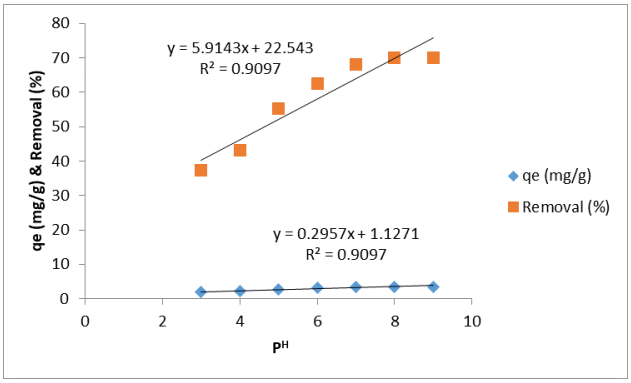

Fig. 4. Effect of contact time on methylene blue removal and uptake unto YAM PEEL at pH 8.0, dye conc. 25 mg/L, Temp 303 K, dosage 0.05 g

E. *Effect of Temperature*

Table 5
Effect of temperature on methylene blue on to YAM PEEL at dosage 0.05 g, time 240 mins, pH 8.0, dye conc. 25 mg/L

Effect of Temperature (K)	Uptake capacity (q_e) (mg/g)	Removal (%)
303	3.41	68.2
308	3.61	72.1
313	3.87	77.4

Increased solution temperature from 303 to 313 K resulted in a small rise in MB adsorption from 3.41 to 3.87 mg/g onto YP. In addition, the adsorbent showed an improvement in percentage removal from 68.2 to 77.4 %. Other dyes have also shown a comparable increase in adsorption onto YP adsorbents

as temperature rises (Fan et al., 2017)

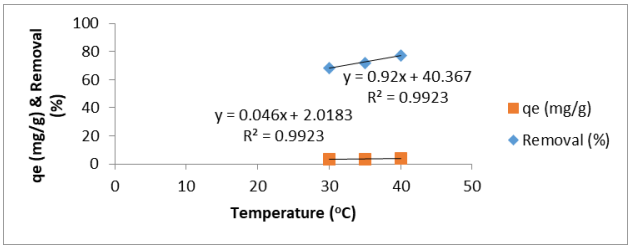

Fig. 5. Effect of contact time on methylene blue removal and uptake unto YAM PEEL at pH 8.0, dye conc. 25 mg/L, Temp 303 K, dosage 0.05 g

F. Isotherm Modelling

The Freundlich and Langmuir isotherm models were used to determine the biosorption isotherm, with the following equations (Foo and Hameed, 2010):

$$\text{Log } q_e = \log k_f + [1/n] \log C_e$$

$$Ce/q_e = 1/ql.K_l + C_e/q_l$$

Figures 6 and 7 show the isotherm model fittings for methylene blue uptake onto YP, whereas Table 6 shows the model parameters that were determined. As evidenced by the highest R2 value, the Langmuir model provided the best match to the dye concentration data for YP adsorbent. The separation factor parameter, SF, as employed by Kannan and Sundaram, was utilized to examine the favorability of dye ion adsorption on yam peels waste biomass (2001). The following relationship defines the separation factor SF:

$$S_f = 1/1+K_lC_o$$

Where K_L = Langmuir isotherm constant
Co = initial dye concentration of 25mg/L.
The parameter indicates the shape of the isotherm as follows:
$S_F > 1$ unfavourable isotherm
$S_F = 1$ linear isotherm
$S_F = 0$ irreversible isotherm
$0 < S_f < 1$ favourable isotherm
The methylene blue dye separation parameter is less than unity, suggesting that yam peels waste biomass is indeed an excellent adsorbent.

Table 6
Equilibrium isotherm modelling of methylene blue adsorption onto YAM PEEL

Isotherm	Yam Peel (YAM PEEL)
Langmuir	
K_l (L/mg)	0.17
q_l (mg/g)	5.46
R_l	0.19
R^2	0.995
Freundlich	
N	1.541
K_f (L/g)	0.844
R^2	0.994

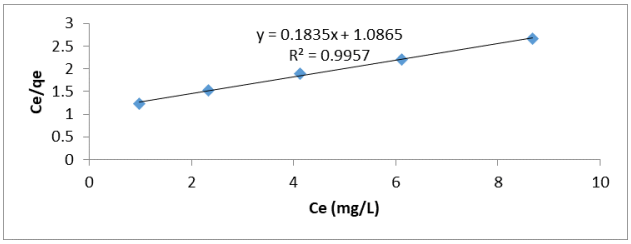

Fig. 6. Langmuir isotherm fittings for the adsorption of methylene blue adsorption onto YAM PEEL

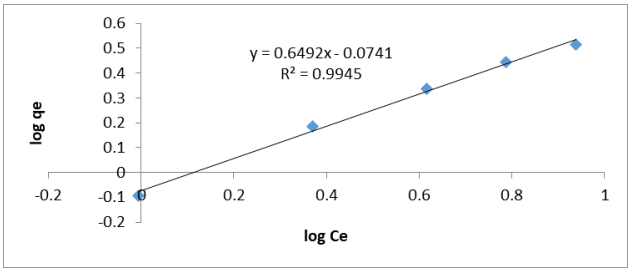

Fig. 7. Freundlich isotherm fittings for the adsorption of methylene blue adsorption onto YAM PEEL

G. Adsorption Kinetics

The pseudo-first order (PF) and pseudo-second order (PS) kinetic models were used to model kinetic data, with the following equations (Ezekoye et al., 2020):

$$\text{Log } (q_e - q_t) = \log q_e - (K_1 t/2.303)$$

$$t/q_t = 1/K_2q_e^2 + t/q_e$$

The rate constants for the PS and PF kinetic data models, respectively, are K1 and K2. The uptake capacity at time (mg/g) is represented by the qt (mg/g) (t).

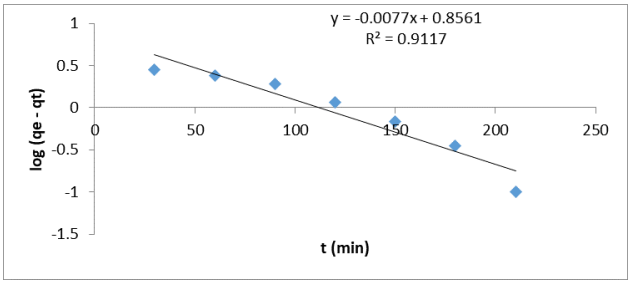

Fig. 8. Pseudo-first order kinetic fittings for the adsorption of methylene blue onto YAM PEEL

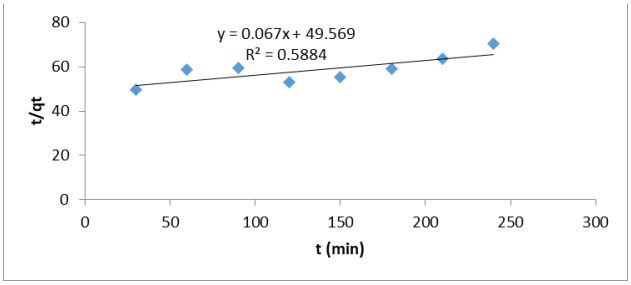

Fig. 9. Pseudo-second order kinetic fittings for the adsorption of methylene blue onto YAM PEEL

Figures 8 and 9 illustrate the kinetic model fittings for

methylene blue adsorption, while Table 7 shows the calculated kinetic parameters. The PF kinetic model had a higher R2 than the PS kinetic model, indicating that the PF model better predicts MB absorption on YP and that chemisorption is not the rate determining mechanism (Fan et al., 2017). For practical application in industrial wastewater treatment, faster equilibrium uptake is desirable in adsorption systems, as time is saved in the process of treatment.

Table 7
Kinetic modelling of methylene blue adsorption onto YAM PEEL

Kinetic model	Yam Peel (YAM PEEL)
Pseudo-first order	
K (min^{-1})	0.016
R^2	0.911
Pseudo-second order	
K_2 (g/mg min)	0.0000905
R^2	0.588

H. Adsorption Thermodynamics

The thermodynamic aspect of adsorption was investigated by using the following equations to calculate the changes in free energy (Go), enthalpy (Ho), and entropy (So) during the adsorption process:

Ln Kc = So/R - Ho/RT Go = -RT ln Kc 8 Ln Kc = So/R - Ho/RT

Where Ca (mg/ L) = Co – Ce is the concentration of metylene blue adsorbed from solution onto the biosorbent at equilibrium, T (K) is the sorption temperature, and R (J/molK) is the universal constant, and Kc (Ca/Ce) is the equilibrium constant. Table 8 shows the thermodynamic characteristics of methylene blue adsorption onto yam peel. Negative Go values suggested a possible and spontaneous uptake of methylene blue dye onto biosorbent materials (Fathy et al., 2019). The weak forces of attraction between methylene blue and the surface functionality of YP must have played a key part in the total adsorption process, according to this physisorption mechanism (Chen et al., 2018). Because of the physical nature of interactions between the methylene blue pollutant and the adsorbents, easy regeneration of our produced materials is likely.

Table 8
Thermodynamic analysis of methylene blue adsorption onto YAM PEEL

T1 (K)	K_c	ΔG° (KJ/mol)	ΔH° (KJ/mol)	ΔSo (J/mol K)	R^2
303	3.1447	2.886	26.887	98.105	0.979
308	3.5842	3.268			
313	4.4248	3.870			

I. Adsorption Mechanism

The FTIR examination of the adsorbents before and after the adsorption of BPB revealed the mechanism of methylene blue dye (MBD) adsorption onto YP. This is essential because alterations in the materials' adsorption bands after adsorption aid in identifying the surface functional groups essential for the dye's adsorption (Hizkeal et al., 2021). Figure 1 shows the FTIR spectra of YP before and after MBD uptake. After adsorption, significant shifts in the OH bands from 3327 to 3354 cm^{-1}, the C=O bands from 1608 to 1625 cm^{-1}, and the C-O stretching from 1053 to 1031 cm-1 were found, while the C-H and C=C largely remained constant. This suggests that YP's OH, C=O, and C-O functional groups were responsible for MBD adsorption through interactions with MBD molecules in solution. Shifts in the absorption bands of YP from 3336 to 3295 cm^{-1}, 1624 to 1621 cm^{-1}, and 1645 to 1640 cm^{-1} for OH, C=O, and C-O, respectively, demonstrated that these functional moieties were responsible for the uptake of MBD onto YP, as shown in Fig. 9b. The OH, NH, and C=O functional groups are primarily responsible for the adsorption of contaminants onto biosorbents, as previously indicated. The presence of hydroxyl and carboxyl functionalities suggests that H-bonding and hydrophobic as well as electrostatic interactions may be involved in the uptake of MBD onto the adsorbent (Putro et al., 2019).

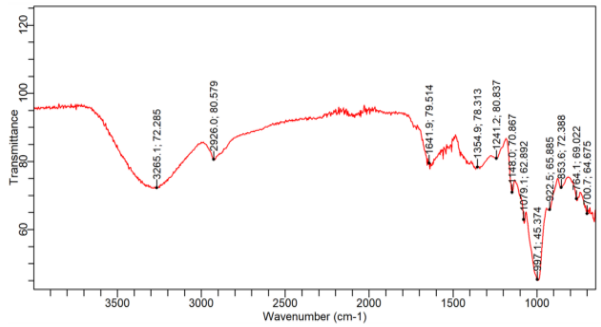

Fig. 10. Fourier transform infrared spectra of biosorbent (YAM PEEL) before adsorption

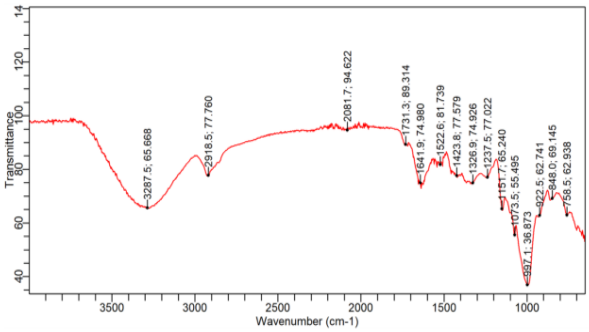

Fig. 11. Fourier transform infrared spectra of biosorbent (YAM PEEL) after adsorption

J. Scanning Electron Microscopy (SEM) Analysis

The surface morphology of the adsorbents was examined using scanning electron microscopy (SEM). Figures 12 and 13 show SEM images of the adsorbents before and after adsorption, accordingly. The porous and uneven structure of the adsorbent was revealed in the image of the adsorbent before adssorption. It revealed a large number of heterogeneous pores, an uneven surface, and particle aggregation in a range of shapes and sizes. The availability of pores aids in methylene blue ion movement to the adsorbent surface (Gautam et al., 2014). The porous structure also suggests that physical adsorption is important in removing methylene blue ions from solution (Vafakhah et al., 2014). The surface of the adsorbent after adsorption appears to be more regular, with similar particle shapes and sizes, yet the surface is mainly heterogeneous, with

a large number of pores, implying possible adsorbent reuse.

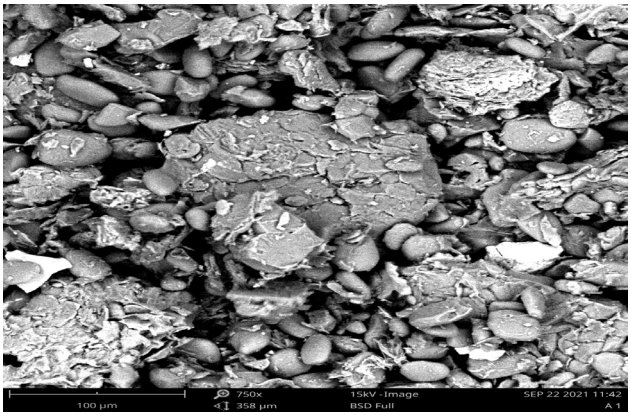

Fig. 12. Scanned electron micrographs (SEM) of the adsorbents before adsorption

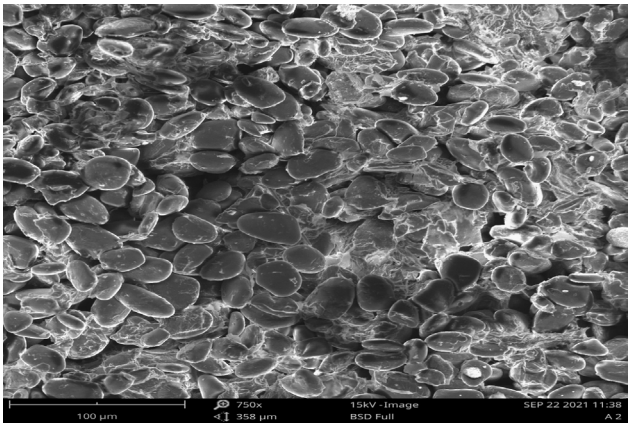

Fig. 13. Scanned electron micrographs (SEM) of the adsorbent after adsorption

4. Conclusion

According to the findings, ACFs can be employed as an adsorbent to remove methylene blue from waste fluids.

The current study reveals that yam peel, a low-cost biomass that is abundant, may be employed as a sorbent to remove methylene blue dye from aqueous solutions.

Initial pH, adsorbent dosage, methylene blue concentration, and contact time all influenced the amount of dye adsorbed.

The sorption equilibrium data were found to fit the Longmuir isotherm, indicating monolayer adsorption on a homogenous surface.

The adsorption kinetics can be predicted using a pseudo-first-order model.

The yam peel's affinity for the methylene blue dye is corroborated by the negative value of free energy change, indicating that sorption is spontaneous.

A. Recommendation

More research should be done on the modification of yam peel, notably by impregnation with zinc oxide nano particles, as a way of enhancing yam peel's adsorption capacity for methylene blue adsorption.

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