

Isotherm Study of Methylene Blue (Mb) Adsorption on Activated Carbon Synthesized from Wheat Straw Biomass

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ORIGINAL RESEARCH

Abstract—This research aims to investigate the adsorption behavior of methylene blue (MB) dye onto activated carbon (AC) synthesized from wheat stover. Activated carbon was prepared through a two-step process, involving carbonization of wheat stover followed by chemical activation. The synthesized activated carbon was characterized using various analytical techniques such as Fourier-transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM) done at 1500x and 179µm, 1000x and 269µm 500x and 537µm and at 1000x and 269µm Magnification for both the raw wheat stover and activated carbon. The adsorption process was studied through batch experiments, where the effect of various parameters such as initial MB concentration, pH, contact time, and temperature was evaluated. The Langmuir and Freundlich isotherm models were applied to analyze the equilibrium data and determine the adsorption capacity and intensity of the adsorbent calculations obtained for both models. For the Freundlich model, it was 0.9956 while for the Langmuir model was 0.9853. Having the best fit for the experiment to be Freundlich because it is closest to 1. Overall, this study provides valuable insights into the adsorption behavior of methylene blue onto activated carbon synthesized from wheat stover. The findings contribute to the understanding of agricultural waste valorization for the production of effective adsorbents for water treatment applications, specifically in the removal of cationic dyes.

Keywords— Biomass, Adsorption, Activated Carbon, Adsorbent, Characterization.

1 INTRODUCTION

The effect of industrial activities on rivers contributes greatly in effluent which affect human, plant and aquatic animals. In the textile industry, whose waste product comes through water, its liquid waste from the industry consists of hazardous chemical which need to be treated properly before introduced to the water bodies. Over the years, several physicochemical methods have been used to remove dye (methylene blue) and recovering of colored material from wastewater (examples are reverse osmosis, advanced oxidation processes with the powerful oxidants, etc.), which have proven not satisfactory because of its high properties in composition, costly for midrange companies and basically time consuming in a large scope or wide range (Godswill, 2017.)

Adsorption techniques have proven otherwise over the years and are prevalent in many fields in which its method was made to improve efficiency, its easily regenerated, reduce cost and perform effectively. Adsorbent consist of high internal surface area that enables adsorption process, also for a successful

adsorption process, the choice of adsorbent is one of the important key factor. (Rahmawati et al., 2021.) serve as adsorbent in the removal of methyl blue dye from waste water. Wheat Stover also known as wheat straw is in form of leftover of harvested wheat grain which serves as fuel, biomass for fermentation etc. This research would also show the graphical representation (adsorption isotherm) of the surface adsorbent material and the adsorption nature, when the adsorption process is equal to the desorption rate been in a state of equilibrium using the Freundlich and Langmuir model assumptions.

Textile industries contributes significantly in the pollution of water and has become a serious environmental issue in recent years. Hazardous and mutagenic compounds in this industry's wastewater are dangerous to life and consumption in any field. Textile companies utilize about 10 million kg of dye per year, with 90% of it ending up in fabric, and this industry releases 10,000 tons of dye into the water stream each year (Carmen and Daniela, 2014).

The adsorption technique has long been a popular approach for color removal and wastewater treatment. It is the most efficient methodology of all because of its ease of design and operation, low cost of evaluation, and dependability. Because of its excellent dye and material adsorption ability, this approach has become a stepping stone in dye waste water treatment. Although commercial activated carbon is expensive, so agricultural waste material such as wheat stover is used to create activated carbon because of its availability and low cost. During this process, we will use wheat stover as our adsorbent, with low cost as the primary criteria for selection. Wheat stover

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Section D- MATERIAL AND METALLURGICAL/CHEMICAL ENGINEERING AND RELATED SCIENCES

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has the properties of adsorption, is biodegradable and its can also serve as fuel. Having this approach in place, it is beneficial in providing the environment a safe and harmless river. Its efficiency also leads to creating of job for different sector of industries which can specialize in the treatment of wastewater from industries before disposal and also solving one of the world's problems.

2 MATERIALS AND METHODOLOGY

2.1 MATERIALS

The list of materials and equipment used for this experiment are as follows: methyl blue, leathers for samples, zinc chloride, wheat stover, nitrogen gas cylinder, muffle furnace, oven dryer, shaker, litmus paper, crucibles, stirrers, bottles (120ml), uv spectrophotometer, mechanical scale, beakers, measuring cylinders

2.2 METHODOLOGY

Wheat stover used in this research was gotten from a farm at Bayero University Kano, this material was thoroughly washed and cleansed with distilled water, then it was dried in a laboratory dryer at 106°C for 24 hours. The dried wheat stover was grounded and sieved into different sizes about 1-2cm in size, the product obtained was stored in an impermeable container for the next step. (Ebrahimian et al., 2014). The Preparation of Activated Carbon Using Wheat stover was carried out using the method below

2.2.1 IMPREGNATION

In this stage pretreated raw wheat stover was mixed with zinc chloride in specific ratios from (1:0.5 to 1:3)(having the wheat stover at constant ratio and zinc chloride in varying ratios), then it was stirred uniformly. The impregnated samples were introduced into the crucibles for the next step of carbonization. This procedure was done at room temperature

2.2.2 CARBONIZATION

The impregnated sample from ratio (1:0.5 to 1:3) was placed in crucibles and then introduced into a muffle furnace. The furnace was heated to 600°C at the rate of 10°C/min for 2hr. Nitrogen gas (N₂) was passed into the muffle furnace throughout to prevent oxidation. Once the activation process was completed, the activated carbon was cooled and washed with distilled water to remove any remaining activating agent residuals until the pH of the samples solutions were neutral, and excess water was drained off.

The following equation was used to calculate the activated carbon yield.

$$\text{Yield (\%)} = \frac{M_{ac}}{M_o} \times 100. (\text{Craison et al., 2021}) \quad (1)$$



Fig. 1, Muffle Furnace



Fig. 2, Grinded Samples.



Fig. 3, Heated samples



Fig. 4, Cleaning Process



Fig. 5, Activated Carbon produced.

2.3. BATCH ADSORPTION STUDIES.

2.3.1 BATCH ISOTHERM STUDIES.

The batch adsorption experiment was done according to the procedures of (Anokhaa et al., 2020) was adopted and modified.

The adsorption capacity of the activated carbon was evaluated using MB. MB came from the market. By combining MB crystal with distillate water, different concentrations of MB solution from 5 to 25 ppm were prepared. Then, 20ml of the ready-to-use MB solution received 0.1g of the produced activated carbon. For 30 minutes, the solution was maintained in a shaking incubator at 30°C with a 150rpm rotating speed. The final concentration of the methylene blue at 664 nm wavelength was measured using a spectrophotometer.

The adsorption capacities (mg/g) at equilibrium adsorption capacities (q_e) at different concentrations were determined by:

$$q_e = \frac{(C_o - C_e)V}{W} \quad (2)$$

where C_o and C_e (mg/L) are the liquid-phase concentrations of dye at initial and equilibrium, respectively. V is the volume of the solution (L) and W is the mass of dry adsorbent used (g)

2.3.2 BATCH KINETIC STUDIES.

The procedure of kinetic experiments was identical to the equilibrium tests. The aqueous samples was taken at different time intervals (30, 60, 90, 120, 150, 180, 210mins) and the concentrations of dye was similarly measured.

The amount of adsorption at time t, q_t (mg/g), was calculated by:

$$q_t = \frac{(C_o - C_t)V}{W} \quad (3)$$

Where C_o , C_t and C_e (mg/L) are the liquid phase concentrations of dye at initial and at time t respectively. V is the volume of the solution (ml) and W is the mass of dry adsorbent used (g).

2. 4.CHARACTERIZATION.

The characterization was done according to the procedures of (Ebrahimian et al., 2014) and was adopted and modified.This process was carried out in other to know the effectiveness of the activation processes.

2.4.1. SCANNING ELECTRON MICROSCOPY ANALYSIS (SEM) .

Using the Phenom Prox Desktop Scanning Electron Microscope, Scanning Electron Microscopy (SEM) was carried out to study the physical structure change of samples. The sample was mounted on double adhesive having been attached to a sample stub, and 5nm of gold was sputtered onto it using a Quorum Technologies model Q150R sputter coater. It was then brought into the SEM machine's chamber, where it was adjusted and focused using NaVCaM before being switched to SEM mode., It's focus, brightness and contrast was automatically adjusted. Afterward the morphologies of different magnification was stored in a drive.

2.4.2. FOURIER TRANSFORM INFRARED ANALYSIS (FTIR).

Fourier Transform Infrared Analysis (FTIR) is a powerful analytical technique used to identify and characterize chemical compounds based on their infrared absorption spectra. It provides information about the functional groups and molecular structure of a sample by measuring the interaction of infrared light with the sample.

Principle of FTIR Analysis: Infrared light consists of a range of wavelengths that correspond to the vibrational energy levels of different chemical bonds within a molecule. Regarding the analysis , When infrared light was passes through a sample, certain wavelengths was absorbed by the sample, while others were transmitted. The absorbed wavelengths are characteristic of the chemical bonds present in the sample. The transmitted and absorbed wavelengths was measured and the FTIR spectrometer generated a spectrum that represents the unique fingerprint of the sample.

3.0 RESULT AND DISCUSSION.

3.1 RESULT

3.1.1 CALIBRATION DATA.

Table 1.0 explained why the results of the MB concentration increases alongside the amount of MB adsorbed per unit mass of activated carbon. Since the initial concentration of MB solution increases. This is mostly caused by an increase in the concentration gradient's driving force, and the graph of Concentration vs Absorbance shown in figure 1.0 below, which is linear with a slope of 7.5391, serves as an illustration of the result. Additional, the slope of this graph was used to determine all concentrations at equilibrium (C_e) values over the course of the experiment.

Table 1.0: Absorbance from a concentration of methylene blue (mb).

S/N	Absorbance	Concentration
1	0	0ppm
2	0.858	5ppm
3	1.845	10ppm
4	2.061	15ppm
5	2.676	20ppm
6	2.848	25ppm

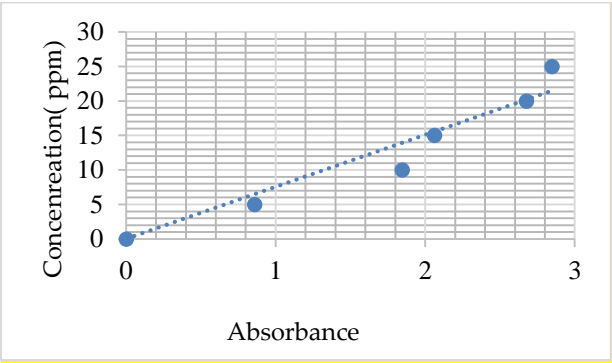


Figure 1.0: A graph of Concentration vs Absorbance given us a slope of 7.5391 . $y = 7.5391x$

3.1.2 ADSORPTION OF METHYLENE BLUE (MB) AT VARYING LOADING RATIO.

The results of an absorption experiment with a constant weight of activated carbon (AC) of 0.10g, a constant volume of concentration of Methylene Blue (MB) of 20 ml, and a variable loading ratio are displayed in Table1.1 – Table 1.5. It is possible to say with the increase in concentration, the percentage removal will also increase, and our optimum point was gotten at 1:1.30 which was further used for the Characterization process.

The reason for having various ratios is to have an optimum point gotten from th use of litmus paper reaction which would be used in carrying out the experiment after checking various ratios (1:0.5,1:1,1:1.5,1:2,1:2.5,1:3)

Table 1.1 Adsorption study using 1:0.5

concentration	Absorbance	Final Concentration	Initial Concentration	% Removal	Absorbance capacity
5ppm	0.822	6.19714	6.46854	4.1958	0.05
10pmm	1.184	8.92629	13.9096	35.826	0.99
15ppm	1.167	8.79813	15.536	43.377	1.34
20ppm	1.445	10.894	20.174	46.001	1.85
25ppm	1.581	11.9193	21.471	44.487	1.91

Table 1.2 Adsorption study using 1:1.5

Concen tration	Absor bance	Final Concen tration	Initial Conce ntratio n	% Remo val	Abso rbanc e capac ity
5ppm	0.853	6.43085	6.4685	0.5827	0.007
10ppmm	1.403	10.5773	13.909	23.956	0.666
15ppm	1.345	10.1400	15.538	34.740	1.079
20ppm	1.444	10.8864	20.174	46.038	1.857
25ppm	1.414	10.6602	21.471	50.356	55.76

S/N	Time (mins)	Concentration at equilibrium	Adsorption capacity (q _e)
1	30	1.492742	3.995723
2	60	1.34196	4.025879
3	90	1.364577	4.021356
4	120	1.145943	4.065083
5	150	1.025318	4.089208
6	180	0.934848	4.107302
7	210	0.904692	4.113333

Table 1.3. Adsorption study using 1:3.0 (optimum).

concent ration	Absor bance	Final Concen tration	Initial Concentr ation	% Rem oval	Abso rbanc e capac ity
5ppm	0.855	6.44593	6.4685	0.3496	1.219
10ppmm	1.253	9.44649	13.909	32.086	4.528
15ppm	1.247	9.40125	15.538	39.495	6.018
20ppm	1.341	10.1099	20.174	49.887	7.955
25ppm	1.393	10.5019	21.471	51.088	8.117

Table 1.4 Adsorption study on natural wheat Stover.

concen tration	Absor bance	Final Concen tration	Initial Conce ntratio n	% Remo val	Absor bance capaci ty
5ppm	1.6666	12.564	6.46854	94.240	1.219
10ppmm	1.550	11.6856	13.909	15.989	0.4445
15ppm	1.3444	10.132	15.5380	34.792	4.9321
20ppm	1.5486	11.675	20.1746	42.130	6.0910
25ppm	1.2444	9.382	21.4713	56.304	2.417

3.1.3 EFFECT OF CONTACT TIME ON ADSORPTION.

The MB dye was used to calculate the contact time using a dosage of 0.10g and 25ppm Which was the solution where the highest percentage removal of the methylene blue was gotten . The shaker was set at 150rpm and the activated carbon was mixed with the methylene blue at different time interval to round up a total of 3.5hrs.(30, 60, 90, 120, 150, 180, and 210mins).So from the Table 2.0 shows that the concentration at equilibrium (Ce) is decrease with increase of time and the adsorption capacity by activated carbon (AC) is increase with increase in time. Figure 2.0 is a graph of adsorption capacity by AC (q_e) against time (T) and it proved that adsorption capacity by activated carbon increase with increase in time and figure 3.0 is the graph of concentration at equilibrium (Ce) against time(T), which also proved that concentration at equilibrium (Ce) decrease with increase in time.

Table 2.0 Adsorption at constant concentration and weight (25ppm and 0.1g) at different time.

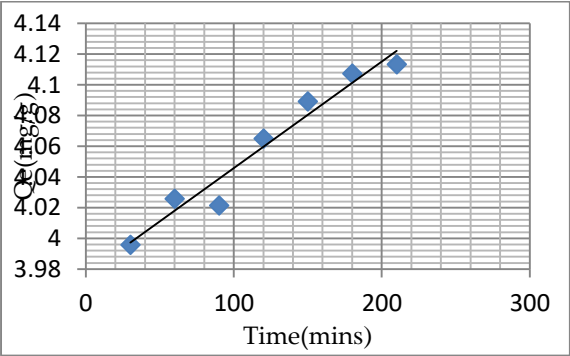


Figure 2.0: Graph of Adsorption Capacity (q_e) against Time (t) .y=0.0007x +3.9763.

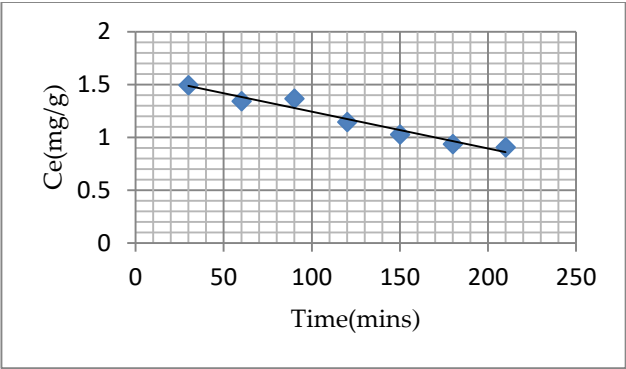


Fig 3.0: Graph of Concentration (ce) against Time (t) .y= - 0.0035x +1.5897.

3.1.4 ABSORPTION ISOTHERMS.

3.1.4.1 LANGMUIR ISOTHERM MODEL.

$$q_e = \frac{(C_o - C_e)V}{W}$$

using this equation above qe would be determined to know the best fit.

From the data gotten ,

Co= 21.47136

Ce=0.6634411

W=0.1g

V=0.02mg/l

$$q_e = \frac{(21.47136 - 0.6634411)0.02}{0.1} = 4.161583.$$

Table 3.0. Values of ce, qe and $\frac{C_e}{q_e}$.

S/N	Concentration at equilibrium	Qe	Ce/qe
1	1.492742	3.995723	2.676768
2	1.34196	4.025879	3
3	1.364577	4.021356	2.946961
4	1.145943	4.065083	3.547368
5	1.025318	4.089208	3.988235
6	0.934848	4.107302	4.393548
7	0.904692	4.113333	4.546667

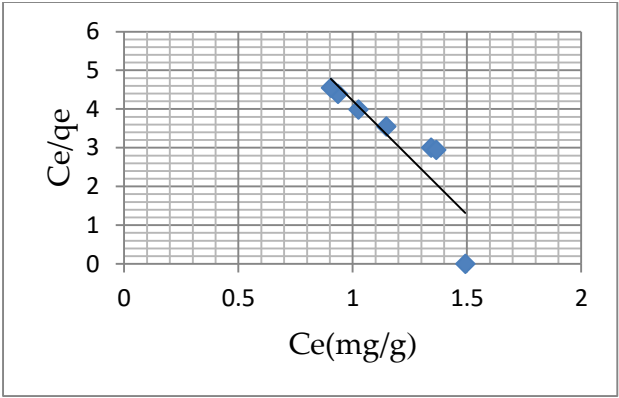


Fig 4.0 : Graph of Ce/qe against Ce. y = -3.202x + 7.3412.R² = 0.9853.

Regression value (R²) is 0.9853

Slope = 1/qo = 3.202

qo = 1/-3.302 = -0.31.

intercept = 1/bqe = 1/b* 4.161583 b = 0.032

3.1.4.2 FREUNDLICH ISOTHERM MODEL.

Linearized Freundlich isotherm model expression:

Log qe= Log kf + ($\frac{1}{n}$) Log Ce

Ce= slope x absorbance

Kf = Adsorption capacity

n = Adsorption intensity

A graph of log qe vs logCe was plotted.

Table 4.0 values of Ce,Qe, log qe and log ce from optimum sample.

S/N	Concentration at equilibrium	Qe	Log qe	Log Ce
1	1.492742	3.995723	0.6016	0.17398
2	1.34196	4.025879	0.6043	0.12773
3	1.364577	4.021356	0.6044	0.13500
4	1.145943	4.065083	0.60906	0.05916
5	1.025318	4.089208	0.61163	0.01085
6	0.934848	4.107302	0.61355	- 0.02925
7	0.904692	4.113333	0.6142	-0.0434

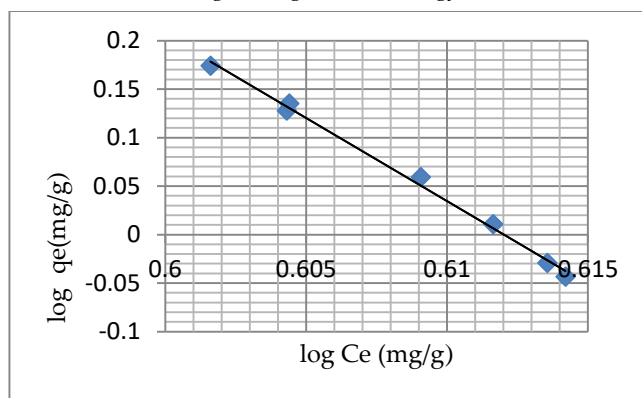


Fig 5.0 A graph of log Q_e vs log C_e .

$$y = -17.133x + 10.485$$

$$R^2 = 0.9956$$

From the graph drawn, It explains the Freundlich Isotherm relationship, the information from the graph given below.

Regression value = 0.9956.

Intercept value = 10.485

Slope = -17.133.

3.2 DISCUSSION.

Therefore we can say the best fit between the isotherms was compared by calculating the regression values for isotherms based on the calculations obtained for both models. For the Freundlich model, it was 0.9956 while for the Langmuir model was 0.9853. Therefore the best fit for the experiment is Freundlich because it is closest to 1.

3.2.1. SEM

Scanning Electron Microscopy (SEM) Activated carbon active sites which was used in the adsorption process, have been identified as shown in Figure 5.0 which shows the morphology of the adsorbent.

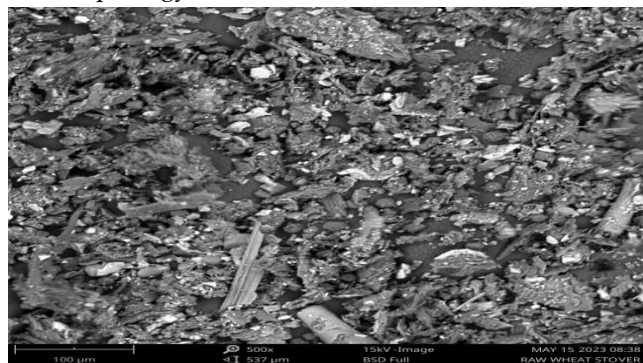


Fig 5.0. Scanning Electron Microscopy (Sem) at 500x and 537μm Magnification (Activated Carbon).

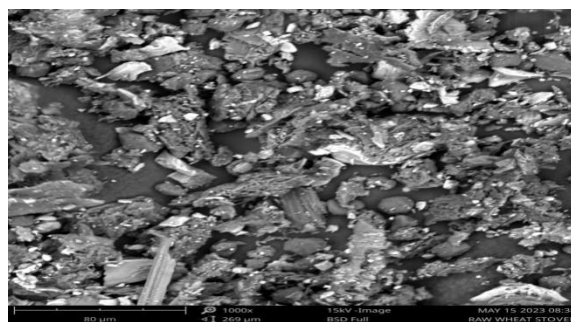


Fig 6.0. Scanning Electron Microscopy (Sem) at 1000x and 269μm Magnification (Activated Carbon).

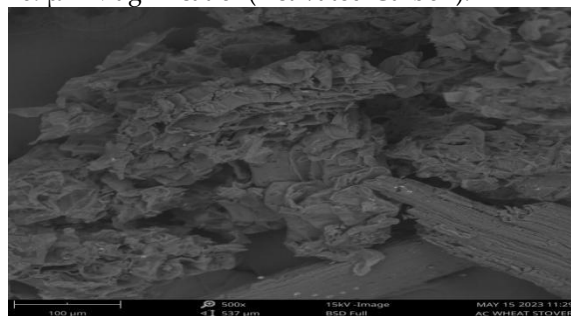


Fig 7.0. Scanning Electron Microscopy (Sem) at 500x and 537μm Magnification (Raw wheat stover).

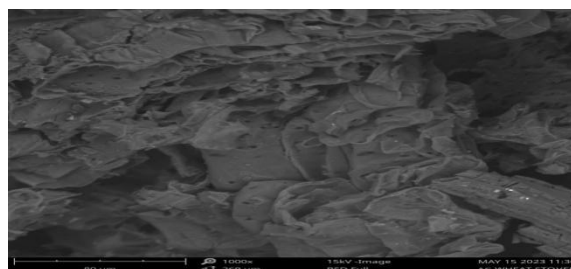


Fig 8.0. Scanning Electron Microscopy (Sem) at 1000x and 269μm Magnification (Raw wheat stover).

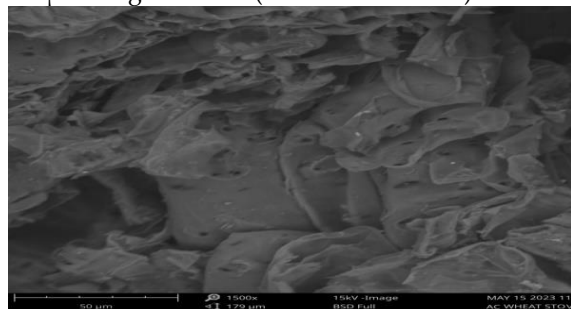


Fig 9.0. Scanning Electron Microscopy (Sem) at 1500x and 179μm Magnification (Raw wheat stover)

3.2.2 FOURIER TRANSFORM INFRARED SPECTROSCOPY (FTIR).

FTIR analysis was used to examine the structural changes that occur during the activation process and the natural wheat Stover, providing information about its chemical composition and functional groups.

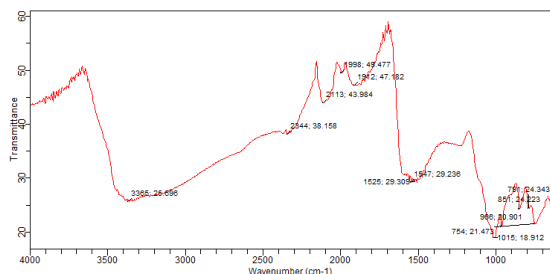


Fig 10.0.FTIR analysis On Activated Carbon.

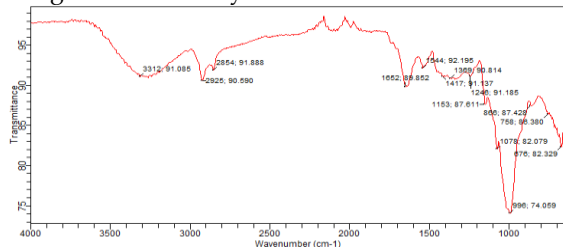


Fig 11.0.FTIR analysis on Natural Wheat Stover

4.0 CONCLUSION.

In conclusion, the Isotherm Study Of Methylene Blue Adsorption on Activated Carbon Synthesized from Wheat Stover Biomass has provided valuable insights into the Adsorption behavior and potential applications of this sustainable material. The analysis of the Isotherm data has revealed important information regarding the Adsorption Capacity, Equilibrium, And Surface properties of the Activated Carbon. The results indicate that the Activated Carbon derived from Wheat Stover has demonstrated significant potential as an effective Adsorbent for the removal of Methylene Blue Dye from Aqueous Solutions. The study has shown the Adsorption process follows the Freundlinch Isotherm Model, Adsorption process involves Heterogeneous surfaces and multilayer Adsorption. The Freundlich Isotherm is commonly used to describe Adsorption on heterogeneous surfaces, where the Adsorbate molecules interact with different Adsorption sites on the adsorbent material. It assumes that the adsorption capacity of the material increases continuously with the Adsorbate concentration.

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