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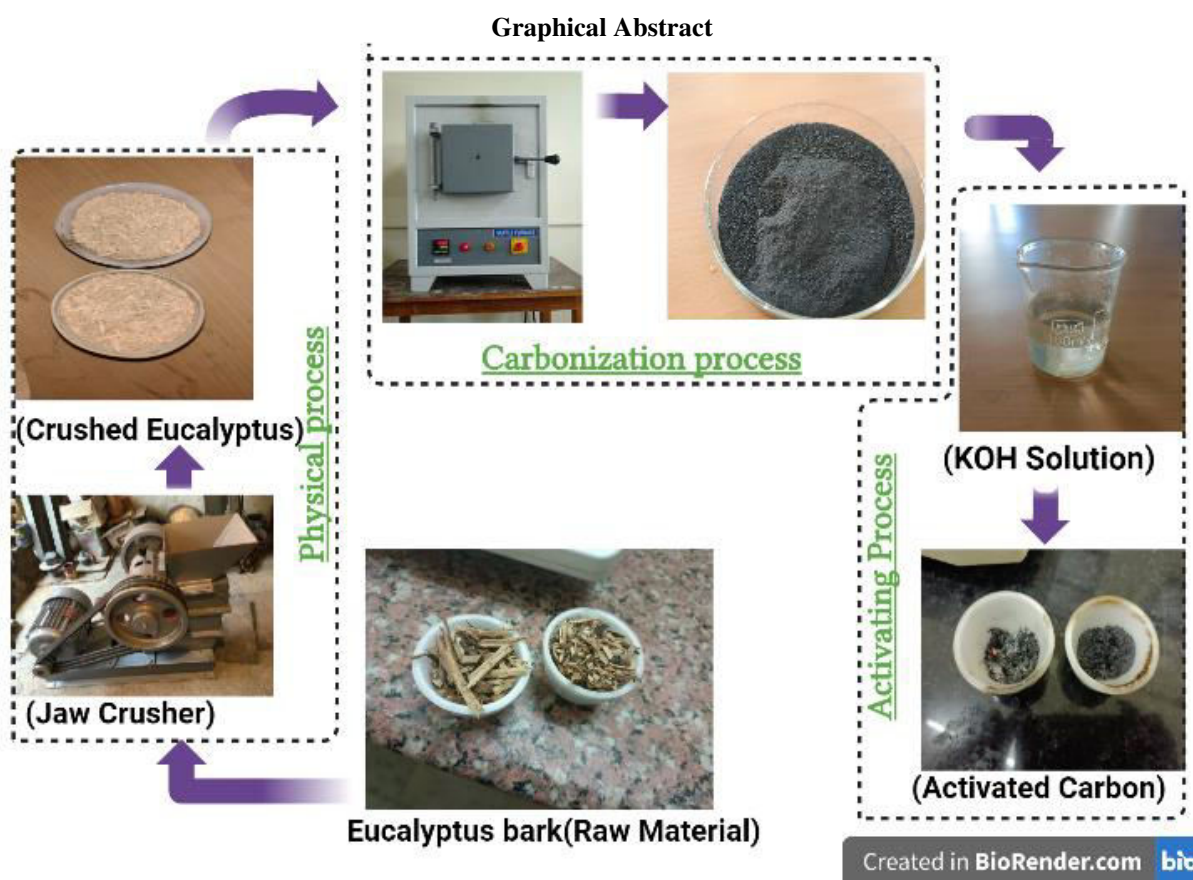
Synthesis of Adsorbent from Eucalyptus Bark

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ABSTRACT: A heterocyclic aromatic chemical called methylene blue (MB) is employed as a drug or a synthetic textile dye. Researchers are looking into how the adsorption process can mitigate its ecotoxicity. This project's goal is to evaluate a carbon-based adsorbent's effectiveness for MB dye uptake based on its highest adsorption capacity. The adsorbent is a new and promising carbon material for dye adsorption for the industrial waste produced by textile industries due to the combination of high surface area and large pore volume. By carbonising the affordable Eucalyptus Bark and then activating it with KOH, it was possible to successfully create nitrogen-enriched carbon adsorbents with large surface areas. Techniques for SEM characterization were employed to figure out the structure. The optimized activated sample was discovered to have a remarkable surface area and a high absorption capacity. Over 500 studies on MB adsorption have been published (within the past 5 years were analyzed). Utilizing UV spectroscopy, it has been determined how effective Eucalyptus bark powder is as a cheap adsorbent for removing the cationic dye methylene blue (MB) from the aqueous solution.

KEYWORDS: Eucalyptus Bark, KOH activation, Methylene blue, UV- Vis spectroscopy.



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I. INTRODUCTION

The most challenging environmental problems of the twenty-first century are water scarcity and pollution, which rank right up there with climate change. The demand for textile products is rising proportionally with population growth. Colors coupled with different contaminants in varied amounts are found in textile effluent[1]. Textile businesses are to blame for one of the worst environmental pollution issues in the world because they produce unwanted dye effluents. As a result, environmental legislation generally mandates that textile factories purify these effluents before releasing them into receiving watercourses. For processing, the textile industries need a lot of colourants, water, and other chemicals. Dispersible, difficult to manage, high volume and hazardous and toxic waste are the four basic classifications for textile waste[2]. Additionally, there is a different class of compounds, called dyes or colourants. The majority of dyes used by the textile industry today are synthetic. Due to their high stability, dyes can linger in water bodies for an extended period of time before being deadly to aquatic life[3]. These colouring compounds or dyes directly interfere with light and prevent it from penetrating to the bottom of the water, which causes aquatic plants to produce less oxygen and negatively impacts the ecosystem as a whole. In addition, a lot of colours are known to have mutagenesis consequences, skin irritation, infections, and other disorders[4].

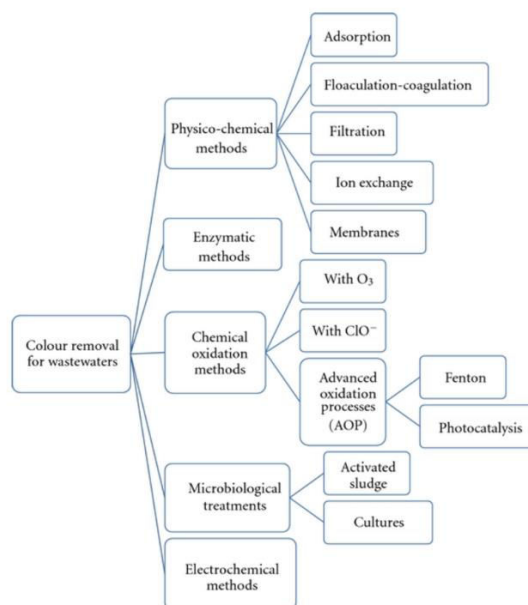


Figure 1: treatment method for textile wastewater¹

There are various conventional methods of removing dyes including coagulation and flocculation [5], oxidation or ozonation [6] and membrane separation [7]. However, these methods are not widely used due to their high cost and economic disadvantage. In contrast, an adsorption technique is by far the most versatile and widely used. The most common adsorbent materials are: alumina silica [5], metal hydroxides [8] and activated carbon [9]. As proved by many researchers [10], removal of dyes by activated carbon is economically favorable and technically easier.

Activated charcoal can also be made from Eucalyptus wood. Ash, total extractives, lignin, and suberin made up the Eucalyptus bark's average chemical make-up in the order: 18.5%, 10.7%, 20.0%, and 1.9%. A carbon-rich, stable variety of charcoal called activated charcoal can be applied to agricultural land for agronomic or environmental goals, for the adsorption of heavy metals from dyes, or even in the pharmaceutical and cosmetic sectors. All types of biomass can be carbonised in a pyrolysis reactor with little to no oxygen to produce activated charcoal. Any organic waste can be converted into an extremely stable form of carbon that can be used to create activated carbons through the pyrolysis process. The use of activated charcoal in various fields are shown in Figures 2.



Figure 2: application of Activated charcoal [4].

It is possible to accomplish pyrolysis under several sorts of reaction circumstances. The primary constituents of biomass are long polymeric chains of cellulose, hemicellulose, and pectin, along with some additional substances like volatiles. The amount of each product varies depending on the temperature, residence duration, heating rate, pressure, the kind of precursors we're utilising, and the configuration and architecture of the reactor. Dehydration, drying, or the elimination of moisture happens at temperatures below 200°C. Hemicellulose begins to break down between 200 and 280 °C, which produces some syngas and a negligible amount of bio-oil. The cellulose degrades in a wide range of 240 to 350 °C, and when cellulose degrades it again creates some syngas, some additional bio-oil, and some biochar, which is the solid residue that will be left out. Temperatures above 350 °C and up to 400 to 500 °C cause lignin to break down primarily into charcoal and a small quantity of bio-oil.

Muñoz et al., 2003 [12] used to activate pine sawdust with phosphoric and boric acid. It was discovered that pine sawdust was combined with the activating agent solution in a ratio of 1.5 solids to 1 solution for 72 hours in the phosphoric acid solution and for 13 hours in the boric acid solution. Ismadji et al., 2005 [13] generated activated carbon from vacuum pyrolysis-derived Eucalyptus sawdust char. It was observed that the char's maximum interfacial area was reached at 850 °C and began to decline as the temperature rose. Açıkyıldız et al., 2014 [14] used three different forms of precursors to make activated carbon, including cornel seed (*Cornus mas L.*) and pine (*Pinus sylvestris*) sawdust. In samples manufactured from pine, rose seed, and cornel seed, it was discovered that zinc chloride produced the optimum adsorptive properties at 400–500 °C, an impregnation ratio of 1.5, and an impregnation time of 1-2 h.

Eucalyptus Bark will be turned into carbonised material for this project, which will then be further processed and turned into activated carbon with the right activating chemicals (chemical activation). Additional research on the stability, adsorption potential, and other qualitative and quantitative aspects of activated carbon will be conducted. The surface of the adsorbent needs to have a lot of holes so that dye particles can attach. Microporous pores are those that are less than 2 nm, mesoporous pores that are between 2 and 50 nm, and macroporous pores that are larger than 50 nm. The pore size that is generated on the surface of the adsorbent will also be evaluated; ideally, the pore size should be large enough to trap the dye molecules; if the pore is too small, efficient adsorption cannot occur. An appropriate activating agent and impregnation ratio can provide the desired pore size. The impregnation ratio is calculated by dividing the mass of the organic material to be converted into activated carbon or non-activated porous carbon by the mass of the activating agent (NaOH, KOH, etc.).

II. METHODS AND METHODOLOGY

Raw material

Eucalyptus bark(EB) was used for this experiment which was received from a farm in Hoshiarpur district of Punjab , where the cutting down of Eucalyptus cultivation was taking place. Eucalyptus bark is an essential bark that contains volatile active compounds. To slow down the process of releasing such volatile compounds, a binding agent such as a polymer matrix is needed. 97% pure KOH was used which was bought from Loba Chemie Pvt. Ltd. Methylene blue(Microscopical stain) was used and it was bought from Isocheime Laboratories Pvt. Ltd.

Preparation of carbonized sample

Initially the bark was derived from the farmlands in pretty raw form which were supposed to crushed into finer particle for further process. The bark brought up since they had materialistic impurities of sand and dust they were washed thoroughly first using the tap water water under high pressure water supply then gently washed in distilled water for removal of tap water based impurities due to high turbidity and high TDS value of tap water used initially.

Then using ball mill the further crushing was progressed where the bigger structured was procured into finer particle. The finer particles produced were again washed in distilled water. Then kept in a tray dryer for removing the overall moisture from the sample due high moisture it was kept for natural drying initially as well.

This crushed material was further taken for carbonization where the sample was placed in the muffle furnace using a crucible which is made up of high heat resistive substance. The temperature of the muffle furnace was kept around 450 degree Celsius and it was ensured that the oxygen supply to the furnace is nullified using a jacket made up of iron the material of the furnace and a cotton covering over it. This avoidance of oxygen will stop the ash formation of our sample.

Activation of carbonized sample

The carbonized sample was then activated with two different methods to analyze the better adsorption capacity. The distinguished sample were prepared using as AC-01 and AC-02.AC-01 was prepared using KOH as an activating agent at an imperial impregnation ratio of 3:4. Initially KOH solution was prepared by dissolving 3.9 gram of KOH in one liter of solvent(distilled water). Later the KOH solution was mixed in the carbonized sample simultaneously agitation was achieved using a magnetic stirrer in order to get the uniform composition of KOH-carbonized slurry. Then this slurry was transferred in the crucible (which is made up of high heat resistive substance) and kept in the muffle furnace under the ambient temperature of 250 degree Celsius for the time period of 1 hour. The activated carbon AC-01 so produced had basic Ph level which was then neutralized by acid wash(distilled water). This process was reluctant in nature since a lot of distilled water was consumed in neutralizing the activated carbon sample AC-01.

AC-02 was prepared with a distinguish method in which the slow pyrolysis wasn't carried out in the muffle furnace. the KOH solution was mixed in the carbonized sample simultaneously agitation was achieved using a magnetic stirrer in order to get uniform composition of KOH-carbonized slurry. The activated carbon AC-02 so produced had a basic Ph level which was then neutralized by acid wash(distilled water). This process was reluctant in nature since a lot of distilled water was consumed in neutralizing the activated carbon sample AC-02.

CHARACTERIZATION

UV spectrometry

UV spectrometry (also known as UV spectroscopy) is a technique used to measure the absorbance or transmission of ultraviolet (UV) radiation by a sample. It involves passing a beam of UV light through a sample and measuring the amount of light that is absorbed by the sample as a function of the wavelength of the light.

SEM (scanning electron microscope)

A scanning electron microscope (SEM) is an instrument used to analyze the surface structure of materials at high magnification. The SEM works by using a focused beam of electrons to scan the surface of a sample and detecting the electrons that are scattered or emitted from the sample to generate an image.

XRD (X Ray Diffraction)

XRD peaks provide valuable information about the structure and properties of a carbon sample. Here are some key insights that can be obtained from XRD i.e. Crystal Structure, Phase Identification, Crystallite Size and Strain, . Degree of Graphitization and Preferred Orientation.

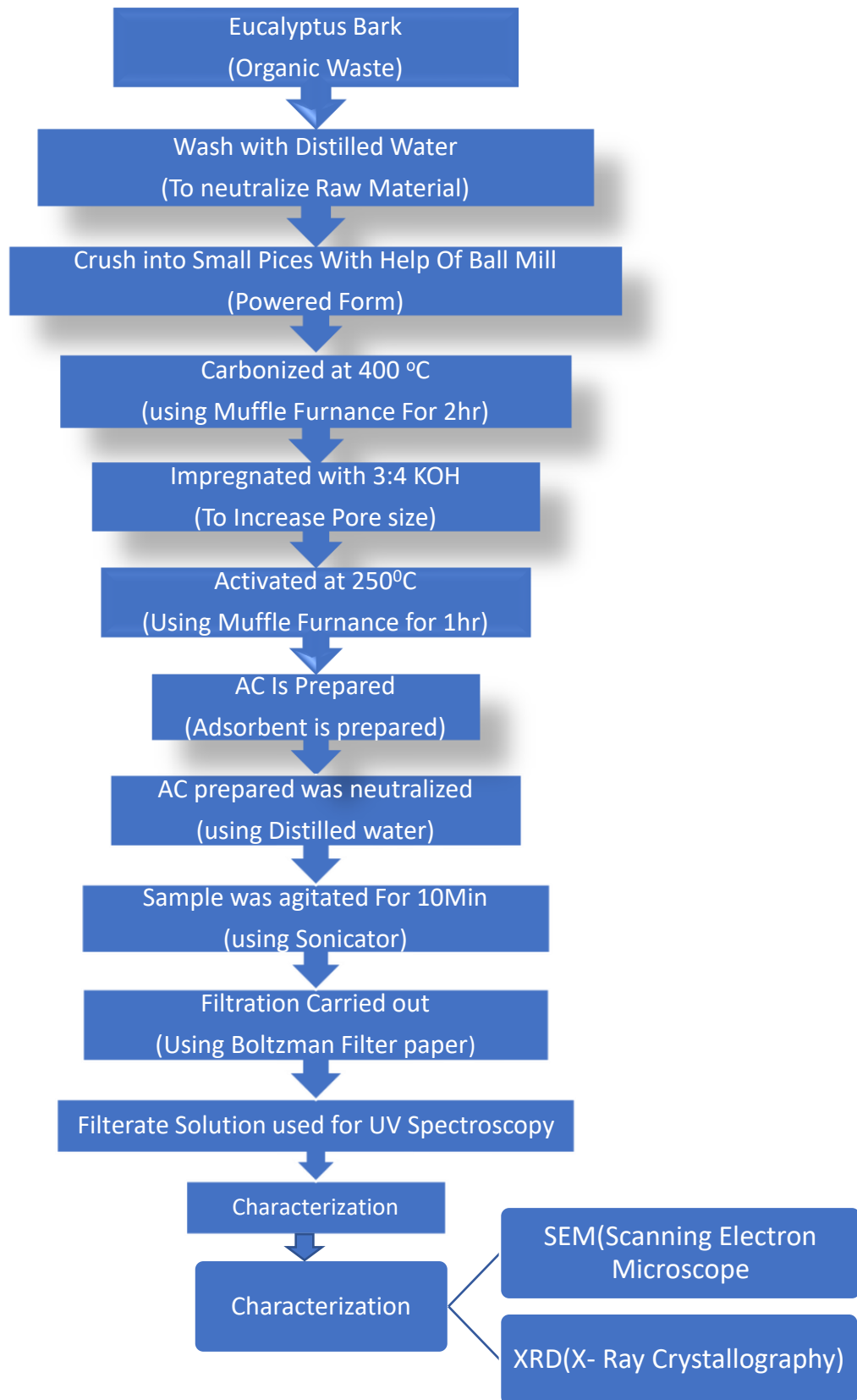


Fig 3 : Flow diagram of experimental procedure

III. RESULT AND CONCLUSION

RESULT:

SEM CHARACTERIZATION

SEM images are very useful to obtain accurate details about pore structure of carbonized sample and activated carbon and the comparison between carbonized sample and activated carbon. The surface morphology of carbonized sample, activated sample, and activated sample without slow pyrolysis have been shown using the SEM analyzer. The SEM images revealed noteworthy changes in the surface morphology of carbonized was seen after activation in both cases. The surface of activated carbon shows an irregular structure, which is loaded with crystalline salt on inorganic matter such as sulfur, nitrogen, and oxygen. However, it is evident from the Figure that carbonization at 450 °C causes the elimination of volatile matter from the sample resulting in a rough and partially porous surface of the carbonized sample. Subsequently, a highly porous surface is observed on the activated carbon in the presence of KOH after carbonization at 450 °C produced as shown in figure12(a,b).

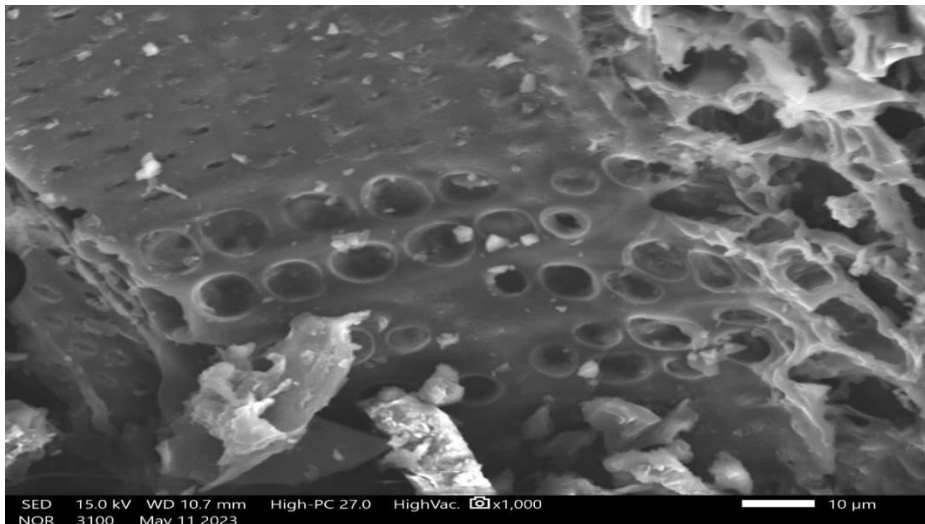


Fig 5: Sem Image of Carbonized Sample

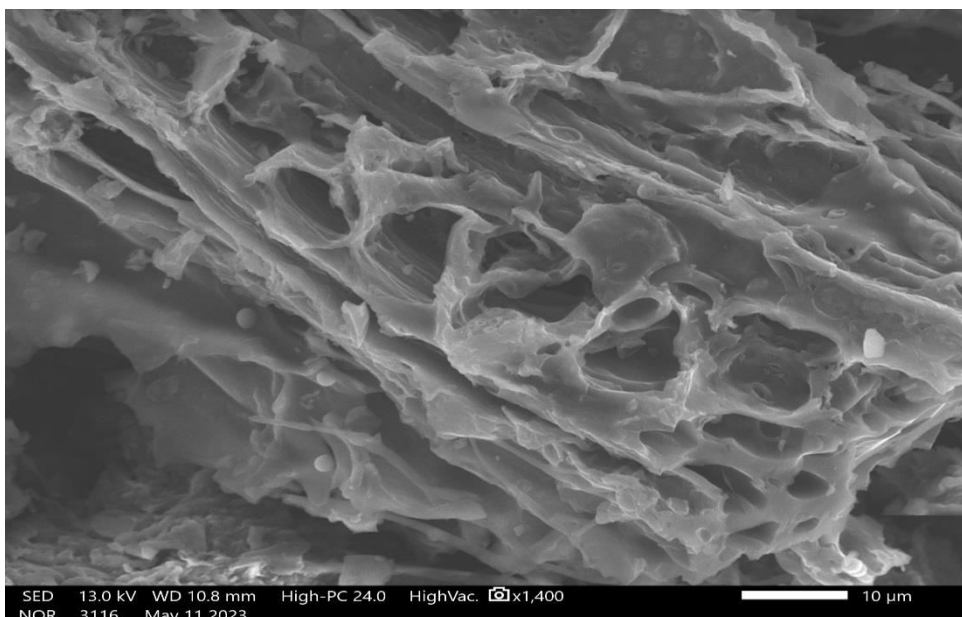


Fig 6(a): Sem Image of activated carbon using Muffle Furnace

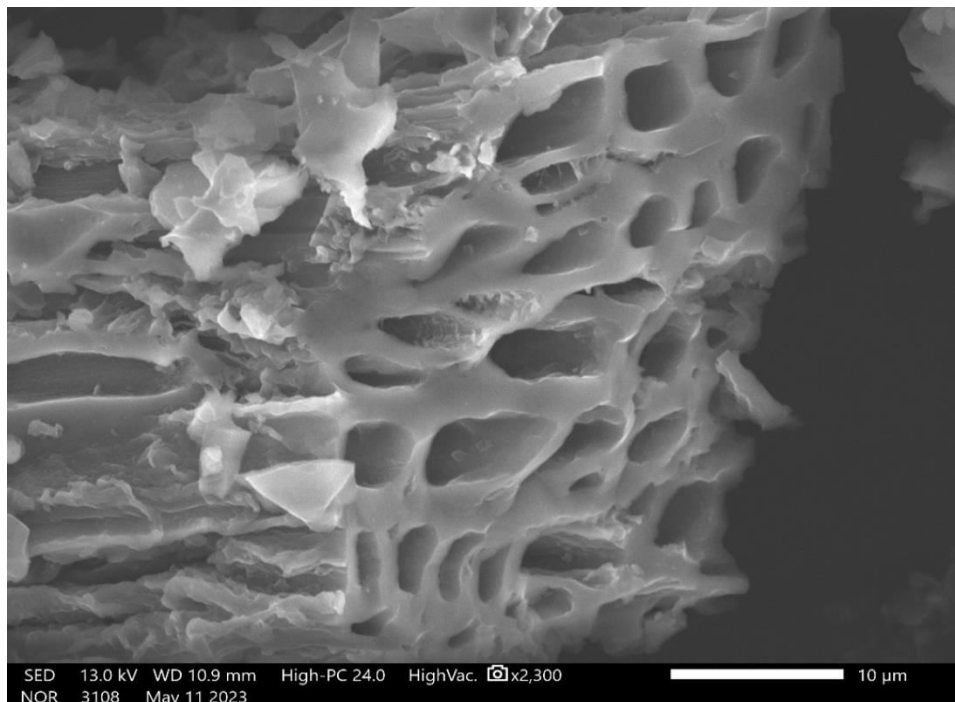


Fig 6(b): Sem Image of activated carbon without using Muffle Furnace

UV SPECTROMETRY

An analytical technique called UV-vis spectroscopy determines how much of various visible and ultraviolet light wavelengths a sample transmits or absorbs. The methylene blue absorption curve was plotted in the first stage. To determine the wavelength that the methylene blue solution absorbs, a full wavelength scan was carried out. The maximal absorbance was measured at 655 nm [Fig 14], thus samples will be further examined at this wavelength. Three samples of 4, 8, and 12 ppm of methylene blue were created from a stock solution with 1000 ppm of the dye for the purpose of plotting curves. In a UV spectrophotometer, the diluted samples were examined [fig 15], and the corresponding absorbance was noted as depicted in Figure 6.

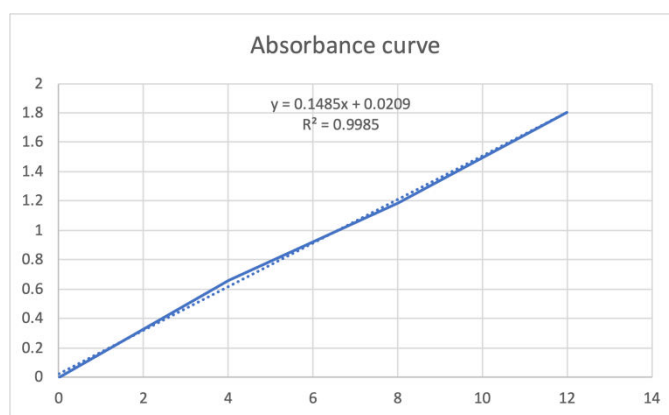


Fig 4 : Standard Absorbance Curve

The 1000 ppm stock solution was first used to make various ppm solutions. 25ml of 200, 300, and 350 ppm methylene blue solutions were prepared in sets of two using a sonicator to continuously mix the activated carbon and inactive carbon for 30 minutes. The filtrate from the subsequent filtering of the left overall solution was used for UV-spectrometry. Thus, by using tables 1,2 and 3, we indicated the methylene blue absorbance in activated and unactivated carbon. After comparing the absorbances of both cases, we discovered that the methylene blue absorbance in the activated carbon sample was slightly higher than that of the carbonised sample when activated using muffle furnace. The absorbance curves developed in UV spectrometry are shown in figures [17-19]. With this rise, the SEM results are



Sr No.	Initial concentration (in ppm)	Absorbance After adsorption (l/ppm/cm)	Concentration after Adsorption(in ppm)	Dye Adsorbed (mg of dye/g of adsorbent)
1	150	1.376	9.1252	17.609
2	200	0.980	6.458	22.733

Table 1: Adsorption capacity of Un-activated carbon

likewise satisfactory because they both demonstrate that the surface area has grown in terms of pore size and porosity. When the concentration of the methylene blue solution was raised, we could observe that the absorbance was constant, allowing us to determine the material's absorbance capacity for both the activated sample and the carbonized sample.

Sr No.	Initial concentration (in ppm)	Absorbance after adsorption (l/ppm/cm)	Concentration after adsorption(in ppm)	Dye Adsorbed (mg of dye/g of adsorbent)
1	150	1.354	8.977	17.627
2	200	2.714	18.135	24.192

Table 2: Adsorption capacity of Activated carbon (With Muffle)

Sr No.	Initial concentration (in ppm)	Absorbance After adsorption (l/ppm/cm)	Concentration after Adsorption(in ppm)	Dye Adsorbed (mg of dye/g of adsorbent)
1	150	1.506	10.006	17.499
2	200	2.416	16.1286	22.983

Table 3: Adsorption capacity of Activated carbon (Without Muffle)

4.1.3 Conclusion

An activated carbon was prepared using a carbonized sample which was activated using KOH.

The well-developed pore structure of carbon resulted in increased specific surface area and changes in surface chemistry. The textural structure and surface chemistry of activated Carbon could be controlled by adjusting the activation conditions, such as activation temperature and impregnation ratio. The optimum activation condition was determined to be 3:4 impregnation ratio and 450°C activation temperature. The activated carbon had the large external surface area and pore volume which is exhibited by SEM images which depicted high porosity as pore got higher enlargement.

Activated carbon exhibited the good adsorption capacity for Methylene Blue of (20.908mg/g), which was far greater than that of untreated Carbonized sample. The maximum adsorption capacity of Methylene Blue onto activated carbon after being stripped of was also very good (24.192 mg/g).

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