

# **PROJECT REPORT**

**On**

**SYNTHESIS AND CHARACTERIZATION OF ACTIVATED  
CARBON FROM HUMAN HAIR WASTE AND ADSORPTION  
STUDIES USING METHYL ORANGE AS MODEL DYE**

Submitted by

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*In partial fulfilment for the award of the*

**Bachelor's Degree in Chemistry**



**DEPARTMENT OF CHEMISTRY AND CENTRE FOR RESEARCH**

**ST. TERESA'S COLLEGE (AUTONOMOUS)  
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B.Sc. CHEMISTRY PROJECT REPORT

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This is to certify that the project "SYNTHESIS AND CHARACTERIZATION OF ACTIVATED CARBON FROM HUMAN HAIR WASTE AND ADSORPTION STUDIES USING METHYL ORANGE AS MODEL DYE" is the work done by TEJA M.J.

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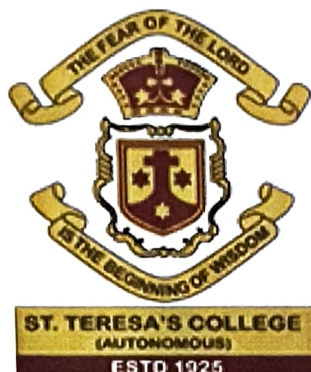
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**CERTIFICATE**

This is to certify that the project work entitled **“SYNTHESIS AND CHARACTERIZATION OF ACTIVATED CARBON FROM HUMAN HAIR WASTE AND ADSORPTION STUDIES USING METHYL ORANGE AS MODEL DYE”** is the work done by **TEJA M J** under my guidance in the partial fulfilment of the award of the Degree of Bachelor of Science in Chemistry at St. Teresa's College (Autonomous), Ernakulam affiliated to Mahatma Gandhi University, Kottayam.

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## **DECLARATION**

I hereby declare that the project work entitled “**SYNTHESIS AND CHARACTERIZATION OF ACTIVATED CARBON FROM HUMAN HAIR WASTE AND ADSORPTION STUDIES USING METHYL ORANGE AS MODEL DYE**” submitted to Department of Chemistry and Centre for Research, St. Teresa’s College (Autonomous) affiliated to Mahatma Gandhi University, Kottayam, is a record of an original work done by me under the guidance of **Dr. NISHA T. P, ASSISTANT PROFESSOR**, Department of Chemistry and Centre for Research, St. Teresa’s College (Autonomous), Ernakulam and this project work is submitted in the partial fulfilment of the requirements for the award of the Degree of Bachelor of Science in Chemistry.

  
**TEJA M J**

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Teja M J

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# Chapter 1

## INTRODUCTION

This widespread problem of water pollution is jeopardizing our health. The annual toll of unsafe water surpasses the combined casualties of war and all other forms of violence. Meanwhile, our accessible drinkable water sources remain limited, with less than 1 percent of Earth's freshwater available to humankind. The release of residual pollutants into public water resources carries significant consequences. Due to their non-biodegradable nature, a multitude of hazardous chemicals generated by industrial processes accumulate in water sediments. This contamination, particularly prevalent in textile production, where approximately 20% of global clean water pollution originates from dyeing and finishing processes, poses threats to aquatic life. Furthermore, the environmental impact extends to microplastics, with three-quarters of primary microplastics entering the environment originating from the washing of synthetic clothing. Textile wastewater discharge is a significant contributor to water pollution, containing high concentrations of pollutants detrimental to both the environment and human health, such as oil, grease, residual chlorine, chemicals, sulfur, odors, colors, as well as suspended and dissolved solids (TDS and TSS).

The main issue with textile wastewater is the presence of colored effluent. Even while we can argue that not all dyes are as harmful as others, their visual pollution has a negative aesthetic noticeable color change in the water. Highly suspended solids (SS), chemical and biochemical dyes like

vat and disperse dyes, typically show good exhaustion qualities. These colors have the ability to bind fibers and can be eliminated through physical processes like flocculation.

However, the removal of color using standard procedures is not as effective if the effluents contain water soluble or reactive dyes.

Currently, various methods are used to remove textile pollutants such as dyes using adsorbing agents like activated carbon. Adsorption, on the other hand, is among the most cost-effective techniques for removing heavy metals and dyes from aqueous solutions due to its high efficiency, straightforward procedure, and low maintenance costs. Activated Carbon (AC) which is an excellent adsorbing substance can be prepared from various substances such as agricultural waste, coconut shell, human hair (HH) etc.

Hair salons and the local barbershops consistently have HH. The global disposal of solid bio-waste from both urban and rural areas is on the rise, and the potential for converting it into materials with additional value has posed a challenge to numerous pertinent research fields. One of the main goals of the research is to turn this useless waste into useful industrial materials, like bio-based adsorbents for the removal of toxic metals from wastewater and factory effluents. This biowaste is made up of keratin, which is made up of multiple proteins with different amino acid compositions and has a typical fibrous hierarchical structure. HH is made up of 41.42% C, 13.12% N, 6.76% H, 6.5% O, and 4.24% S elements. HH was initially presented as a biosorbent for hazardous metal ions due to the presence of chelating groups like sulfur, hydroxyl, carboxyl, and amine functional groups. It was employed almost in its original form for the removal of hazardous metals from aqueous solutions [1].

Converting HH to AC is a cost-effective method because of its high carbon content, availability, abundance, environmental friendliness, continuous producibility, renewability, and very low costs. Batteries, supercapacitors, gas adsorption, and the elimination of tetracycline antibiotics from wastewater have all previously used the HH-based AC (HAC).

While encountering various methods for producing AC, it was discovered that generating AC from HH proved to be the most effective approach. This choice gained significance due to the increasing relevance of HH disposal, prompted by various factors such as the lack of awareness among laborers about proper disposal techniques. We opted for an approach in this study that proved to be the most suitable, given its cost-effectiveness, accessibility, and minimal environmental impact. Using a strong dehydrating reagent like  $\text{CaCl}_2$ , the carbonaceous material is soaked and activated in this method, which then carbonizes it into a preferred form of porous AC. Because calcium chloride has strong dehydrating and mesoporous AC production capabilities, adequate interaction between the polar molecules of water and AC in immersion calorimetry, non-corrosive behavior, and low cost, it is commonly used for the activation process of AC.  $\text{CaCl}_2$  was chosen for this investigation and applied for activation following human hair impregnation. Because of the elimination reactions and dehydration processes,  $\text{CaCl}_2$  reduces the amount of hydrogen and oxygen in the carbonaceous compounds during the activation process. The carbonization process was conducted at  $450\text{ }^\circ\text{C}$  with the use of calcium chloride as an activating agent.

In this study, we further investigated the effective adsorption capacity of HAC for the removal of Methyl Orange (MO), an azo dye widely used in the textile industry to color materials such as cotton and silk. Additionally, MO finds applications in the paper, printing, and culinary sectors. This

water-soluble azo dye serves as an acid-base indicator, exhibiting a colour change to yellow in alkaline solutions above pH 4.4, and turning orange or red below pH 3.1. Notably, MO is extensively released into industrial wastewater.

The importance of removing MO from water bodies is underscored by its prevalent use in various industries and its potential environmental impact. As a known water pollutant, MO can have adverse effects on aquatic ecosystems, disrupting the balance of aquatic life. Furthermore, the dye's release into water bodies can lead to the degradation of water quality, affecting not only the ecosystem but also posing risks to human health. Therefore, our focus on studying the effective adsorption capacity of HAC for MO removal addresses a crucial environmental concern and contributes to the development of sustainable solutions for water treatment in industrial settings.

## **1.1 IMPORTANCE OF WASTE WATER TREATMENT**

Treatment of wastewater is essential to safeguarding the environment and the health of people and animals. Wastewater can contaminate our water supplies, harm natural ecosystems, and result in dangerous diseases if it is not properly managed. Wastewater treatment plants actually do exactly what they say they will: they clean the water that flows down our drains and then release it back into the environment. More needs to be done, even with the efforts being made to put these plants all over the world. One of our most valuable resources, water, is being wasted. Wastewater can be treated in a variety of ways, and the more effective the method, the greater the proportion that can be recycled before being released into the ocean [2].

These are wastewater treatment's five primary advantages.

- Supplies filtered, safe, and clean water.
- Safeguarding your financial standing.
- Favorable to the surroundings.
- Conserving water.
- A strategy to reduce waste.

### **1.1.1 VARIOUS METHODS OF WASTE WATER TREATMENT**

The world now faces complicated challenges related to wastewater. Wastewater can be treated using a variety of techniques, including chemical, physical, biological, and combinations of these techniques. Every procedure, though, has benefits and drawbacks. Common physicochemical techniques for treating wastewater include coagulation and adsorption. Coagulation and adsorption are two very effective ways to eliminate contaminants. The following factors have a major impact on the adsorption process: pH, contact time, temperature, and adsorbent dose. The primary coagulation process parameters are pH, settling time, and coagulant dose. Although the use of chemical materials as coagulants and adsorbents has been investigated in the past, substituting chemical materials has proven to be a difficult topic lately. Natural materials have gained popularity due to their efficiency and environmentally favorable qualities, making them viable new materials in wastewater treatment. Adsorption is a process used to clean wastewater that involves moving solutes from a liquid phase to the solid phase's surface. It's regarded as an affordable and dependable technique. Adsorption has a 99.9% elimination capability for both soluble and insoluble organic contaminants. Adsorption is the process by which chemicals from liquids or gases stick to the interface between two phases,

mostly solids. Adsorption is utilized in water and used in purification to remove dissolved pollutants. Using activated carbon is the most popular method for getting rid of organic materials. However, there are also unique uses for it, such eliminating arsenic or other inorganic contaminants with the use of media other than activated carbon. Activated carbon can be applied in granular or powdered form, depending on the ultimate water quality expectations and treatment requirements. ACF, or activated carbon filtration, is mostly utilized as a polishing step in water treatment procedures and has garnered more attention recently due to its efficiency in eliminating newly identified pollutants known as “micropollutants.” If the relationships governing the adsorption processes are examined in conjunction with the various process schemes used in the water and wastewater sector, a thorough understanding of the process can be achieved [3].

## **1.2 ACTIVATED CARBON**



Fig 1.1: Activated Carbon

Activated carbon, often known as activated charcoal, is a porous substance that draws molecules out of a gas or liquid, most of which are organic. Because of how well it accomplishes this, it is the purifying agent that people use most frequently. Conversely, organic molecules originate from

the metabolism of living organisms and are primarily composed of carbon and hydrogen atom chains. All plant and animal derivatives, including petroleum and its by products, are included in this category. With its tiny pores, activated carbon acts as an adsorption medium, absorbing organic molecules. To increase its adsorption capability, it is activated using chemical or heat procedures (to make pores form). Coconut shells, coal, and other organic materials with a high carbon content are used to make activated carbon. To create a pore structure, the source material is treated with chemicals or steam. Certain poisonings can also be treated with activated carbon in an emergency. It aids in halting the body's absorption of the toxin from the stomach [4].

### **1.2.1 STRUCTURE OF ACTIVATED CARBON**

The structure of activated carbon is porous and amorphous, with pores of different sizes and forms. The pores range in diameter from 0.8 to 10 nanometers and can be irregular, rectangular, or cylindrical. Graphite is found in unrefined form as activated carbon. Its fundamental chemical structure is comparable to that of pure graphite, which is composed of layers of hexagons that have fused and are bound together by carbon atoms. There is weak van der Waals forces holding the layers together. Because of its extremely porous nature, activated carbon has a surface area of up to 1500 square meters per gram. During carbonization, the porous structure takes shape, and during activation, it continues to grow. According to recent research utilizing aberration-corrected transmission electron microscopy, activated carbons may have a structure with pentagonal and heptagonal carbon rings similar to that of fullerenes [5].

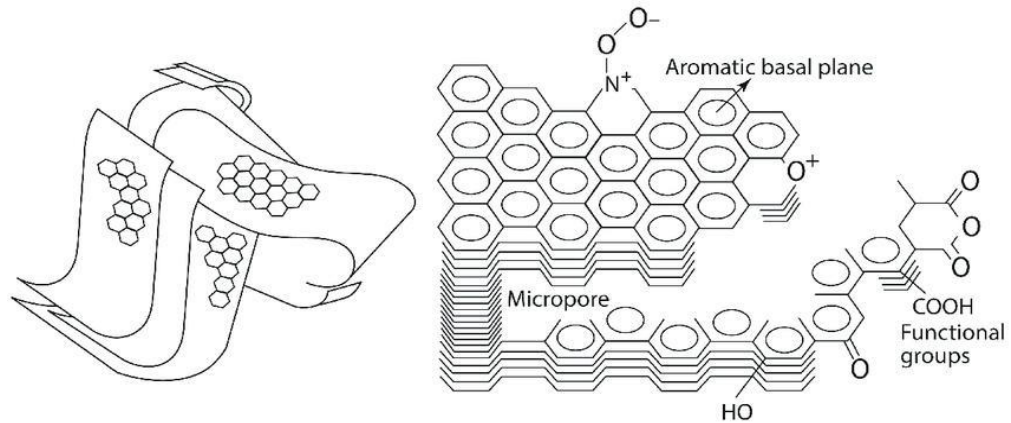


Fig 1.2: Structure of Activated Carbon

### 1.2.2 FACTORS AFFECTING ADSORPTION PROCESS OF ACTIVATED CARBON

The following elements have an impact on the activated carbon adsorption process

**Concentration:** Greater concentration results in increased carbon emissions

**Temperature:** Higher adsorption capability is associated with lower temperatures.

**Pressure:** Greater adsorption capacity is correlated with higher pressure.

**pH:** The groups on the activated carbon's surface are impacted by pH, which modifies the surface groups' charge. The adsorption process is impacted by this.

**Temperature of reaction:** The adsorption process is highly temperature-sensitive. Raising the temperature promotes the adsorption activity and quick reaction.

**Molecular structure:** The dye's molecular structures greatly influence how much of an adsorbent they will be to activated carbon.

The following are additional variables that impact activated carbon's adsorption performance:

Particle size

Porosity

Surface area

Surface chemistry: the existence of chemicals impregnated on the surface [6].

### **1.3 APPLICATIONS**

- Purification of water.

Pesticides, greases, oils, detergents, by products of disinfection, poisons, molecules that

provide color, and substances resulting from the breakdown of plants and algae as well as from animal metabolism are all retained by carbon.

Granular activated carbon (GAC) filters have been shown to be an effective way to eliminate certain pollutants from water, especially organic ones. GAC filters can also be used to get rid of substances like chlorine and hydrogen sulfide, which give water an unpleasant taste or smell like rotten eggs [7].

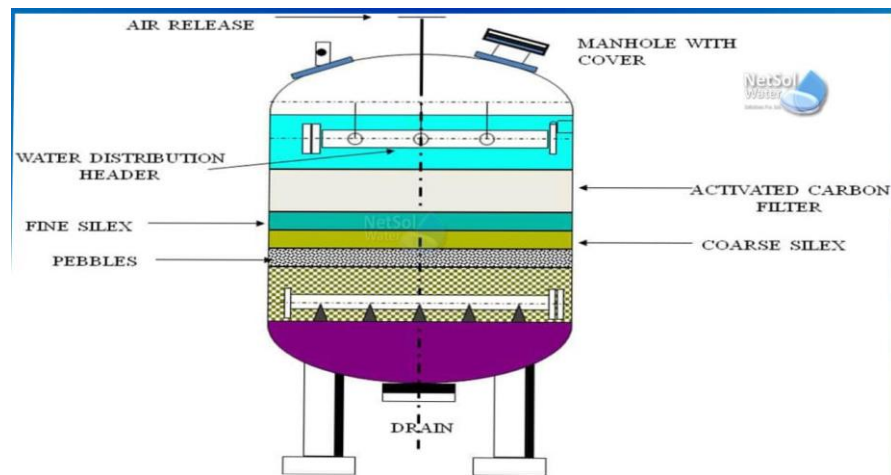


Fig 1.3: Water purification system using Activated Carbon

- Air purification and deodorization.

Cartridge respirators, public air recirculation systems, water treatment facilities, drain vents, paint application booths, and areas where organic solvents are stored or used are a few examples. Via the removal of dangerous gaseous pollutants, volatile organic compounds, and smells, activated carbon filters in air purifiers contribute to improved indoor air quality.

Living in a cleaner and healthier atmosphere is the result, particularly for those who have respiratory or allergy problems [8].

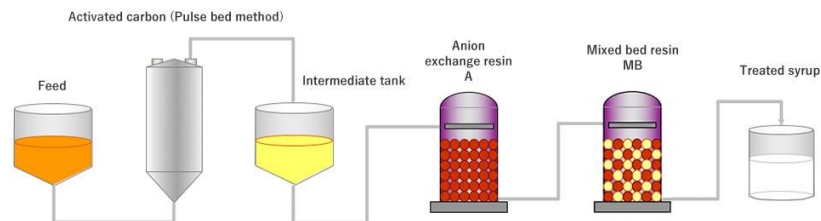
- Care for patients experiencing acute drunkenness.

Activated charcoal is used in hospitals and emergency rooms and is regarded as the “most universal antidote.”

- Refining sugar.

The main goal of this method is to keep the sugar from fermenting and going bad while retaining the color-giving proteins in the charcoal. For several decades, liquid sugar has been produced using activated carbon. In

the process of making liquid sugar, powdered activated carbon is frequently used to create translucent, colorless liquid sucrose solutions that have a neutral, sweet flavor and are free of any off flavors [9].



- Vegetable oils become dis-colored.  
(like coconut). Corn syrup and other food-grade liquids.
- Alcoholic drinks start to lose their color and smell.  
(For instance, distillates and grape wines from any region)
- Recovery of gold.

Gold is dissolved in sodium cyanide and adsorbed on activated carbon when it cannot be extracted from minerals using flotation technique. Because activated carbon can extract gold from the gold-cyanide complex, it is an essential part of the beneficiation of gold ore, making this widely used technique possible [10].

### 1.3.1 MAJOR CONSTITUENTS OF FACTORY EFFLUENTS

Numerous Industries release industrial effluents, and diverse water resources have been discovered to contain a variety of organic contaminants. Pesticides, fertilizers, hydrocarbons, phenols, plasticizers, biphenyls, detergents, oils, greases, medicines, etc. Are some of the

classes to which they belong. Typical organic components including alcohol, aldehyde, carbonic acid, wheat flour, sugar, proteins, amino acids, vitamins, hydrocarbons, fats, oils, hormones, enzymes, viruses, and bacteria are also present.

Rapid industrialization has resulted from rising demand for commodities brought on by the growing population. Consequently, as industrial setups have grown, so too has the amount of industrial waste produced. Due to their pollution of the soil, water, and air, these industrial wastes seriously harm the environment. Depending on the sector, wastewater can have a variety of contents, including biodegradable materials like paper, leather, and wool as well as non-biodegradable waste such heavy metals, pesticides, and plastic. Wastewater from industries may be combustible, reactive, poisonous, or carcinogenic. Therefore, the discharge of garbage into water bodies can have disastrous repercussions on the environment and human health if adequate treatment and management procedures are not followed.

Dye effluents are another major problem if not adequately treated, dyes found in water effluents can be hazardous and damaging to both the environment and living organisms.

Aquatic life can be killed by wastewater dyes, which can also lower the stream's dissolved oxygen level.

The following are a few ways that water effluent dyes may affect the environment:

**Color:** Wastewater can have intense colors due to dyes and textile colors.

**Transparency:** Water bodies' quality and transparency can be impacted by even minute concentrations of dyes, which can make them quite visible.

**Light:** Synthetic dyes that have not been treated may reduce the amount of visible light that reaches the photic ozone.

Azo dyes have the potential to be hazardous, particularly mutagenic and carcinogenic.

Synthetic dyes that are left untreated can have harmful effects on humans, animals, and plants. They are also carcinogenic and mutagenic.

There are now several wastewater treatment techniques in use that have been developed to lessen the environmental impact of dyes. Among these techniques are:

Membrane separation: This technique filters wastewater that has been dyed using membrane micropores.

Adsorbents: These consist of activated charcoal, silica gel, zeolites, and alumina. Natural adsorbent clay called zeolite has grown in favor as a less expensive substitute for activated carbons (AC) in wastewater treatment [11].

### **1.3.2 METHYL ORANGE IN FACTORY EFFLUENTS**

Titration frequently uses methyl orange, a pH indicator. It's an organic substance that, in acidic aqueous solutions, turns from red to yellow. It functions well as an indicator in aqueous solutions between pH 3.1 and pH 4.4. The weak base methyl orange is yellow when it is unionized. The medium turns bright red when it becomes acidic. The solution turns red when methyl orange is employed as an acid indicator. Methyl orange turns yellow when combined with a base. Ingesting methyl orange can be dangerous. It could irritate the gastrointestinal tract and result in diarrhea, vomiting, and nausea. Respiratory tract discomfort may result from inhalation.

Water-soluble azo dyes like methyl orange (MO) are widely utilized in the food, paper, printing, and textile industries. It is a significant pollutant that is dumped into wastewater, which can contaminate water and make wastewater processing challenging [12].

Even at extremely low doses, MO is hazardous and can lead to human cancer. Due to its stability, limited biodegradability, and solubility in water, it is challenging to extract from aqueous solutions using standard techniques for water treatment or purification.

Several methods available for eliminating color from wastewater containing dyes, for example:

- Adsorption
- Fluctuation and coagulation
- Oxidation by chemicals (chlorination, ozonation, etc.)
- Either electrocoagulation or electro oxidation
- Degradation by photosynthesis

## **1.4 CHARACTERIZATION**

### **X-ray Diffraction:**

A potent analytical method for examining a material's crystalline structure is by analyzing X-ray diffractograms (XRD). Determining the arrangement of atoms within a crystalline substance yields important information about the material's crystallography, crystal size, orientation, and phase composition. It is employed in many scientific and industrial domains. An X-ray tube is used to create X-rays, which are then focused on the crystalline sample. X-rays interact with the crystal lattice of the sample, resulting in scattering and constructive interference in various

directions. A detector records the diffraction pattern that is produced as a result of this. By analyzing the XRD pattern, scientists can ascertain the material's crystal structure, including its atom arrangement, unit cell diameters, and lattice characteristics. Applications for X-ray diffraction (XRD) include quality control, geology, chemistry, pharmaceuticals, and material characterization. It is a vital tool in many fields and a non-destructive method that gives comprehensive information about the interior structure of materials [13].

### **Fourier Transform Infrared Spectroscopy:**

The Fourier Transform infrared spectroscopy (FTIR) is a commonly used analytical method for determining and characterizing a substance's chemical makeup. It gauges how infrared radiation, which is located in the electromagnetic spectrum slightly past visible light, interacts with materials. When infrared light travels through a sample, some of it is absorbed by the sample. This results in a plot of the sample's absorbance as a function of the frequency of the infrared light, which is the fundamental idea behind Fourier transform infrared spectroscopy (FTIR). This absorption, which is connected to molecular vibrations, can reveal details about the sample's functional groups and chemical bonds.

The sample may consist of a liquid, solid, or gas. An interferometer modulates the infrared light in a typical FTIR spectrometer, and a detector records the interference pattern that results. The interferogram is then subjected to a Fourier transformation by a computer to produce the infrared spectrum, which displays peaks at particular wavelengths linked to the chemical components of the sample.

The near-infrared (NIR), mid-infrared (MIR), and far-infrared (FIR) are the three primary regions that make up the infrared portion of the

electromagnetic spectrum. The mid-infrared range, which is typically between 4000 and 400  $\text{cm}^{-1}$  (wavenumbers), is the main focus of FTIR spectroscopy. To sum up, FTIR spectroscopy is a strong and adaptable analytical instrument utilized for material characterization, quality assurance, and research in many scientific and industrial domains. It is a helpful tool in many fields since it offers important information about the chemical composition and structure of a wide range of substances [13].

**UV Visible Absorption spectrophotometer:**

UV-Visible spectroscopy a popular analytical method for examining how molecules and atoms in a sample absorb in the ultraviolet and visible light region of electromagnetic spectrum. It offers useful details regarding the concentration, chemical makeup, and electronic structure of many compounds. The idea behind UV-Vis Spectroscopy is that when the energy of the incoming photons equals the energy difference between the ground state and an excited state, molecules and atoms can absorb light at particular wavelengths. A UV-Vis spectrophotometer, which consists of a light source, a monochromator, a sample holding or cuvette, a detector, and a computer for data analysis, is the main tool used for this approach. Usually, the material is dissolved in an appropriate solvent before being tested in a cuvette for absorbance. The absorbance (A) of the sample is determined by measuring the intensity of incident and transmitted light using a spectrophotometer and calculating the difference in intensity.

An absorption spectrum, or a plot of absorbance (A) against the wavelength (or frequency) of the incident light, is created using the absorbance data. Both qualitative and quantitative analysis can be performed with UV-vis spectroscopy, which makes it possible to ascertain the amount of a specific material present in the sample. The fields of biology and biochemistry,

chemical kinetics, quantitative and qualitative analysis, environmental analysis, and pharmaceuticals are among the applications of UV-vis spectroscopy [13].

**Scanning Electron microscopy:**

A concentrated stream of high-energy electrons is employed in scanning electron microscopy to produce a range of signals at the surface of solid specimens. The majority of SEM microscopy applications involve gathering data over a predetermined portion of the sample's surface to create a two-dimensional image that shows spatial changes in characteristics including material orientation, texture, and chemical characterization. Analysis of specific sample point locations can also be done with the SEM. This method works particularly well for determining crystalline structure, crystal orientations, and chemical compositions in a semi-quantitative or qualitative manner. The EDS system software analyses the energy spectrum to ascertain the abundance of particular elements. The EDS detector divides the distinctive X-rays of various elements into an energy spectrum. An example of an EDS spectrum is a plot of X-ray counts against energy (in KeV). The different elements in the sample are represented by energy peaks. With the use of energy dispersive X-ray spectroscopy, one may map the element composition over a far larger raster area and determine the chemical composition of materials down to a spot size of a few microns. When combined, these abilities offer basic compositional data for a large range of materials, such as metals and polymers [13].

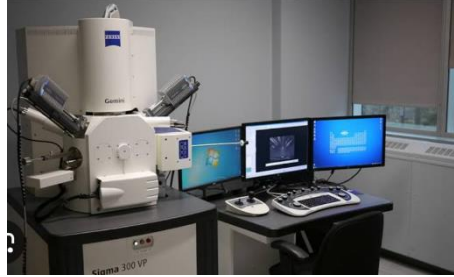


Fig 1.4: Scanning electron microscope

# Chapter 2

## MATERIALS AND METHODS

### 2.1 INTRODUCTION

Activated carbon is an excellent adsorbent due to its greater surface area, microporous ability, and chemical complexity of its area. Activated carbon can be prepared from various substances such as coconut shell, agricultural waste, human hair etc. Here we are synthesizing activated carbon from human hair waste, using  $\text{CaCl}_2$  as activating agent.  $\text{CaCl}_2$  is a strong dehydrating agent which reduces the amount of oxygen and hydrogen in the carbon compound during activation process. This chapter deals with the synthesis and characterization of activated carbon from human hair. The characterization techniques used are FTIR spectroscopy, SEM, CHNS analysis, and XRD. The synthesized activated carbon is used in the adsorption of methyl orange.

#### 2.1.1 MATERIALS REQUIRED

- Human hair waste collected from a local saloon
- Distilled water
- 25% calcium chloride solution (Nice Chemicals Pvt Ltd, Kerala).

### **2.1.2 APPARATUS REQUIRED**

- Magnetic stirrer
- Muffle furnace
- Air oven
- Laboratory mortar and pestle

### **2.2 SYNTHESIS OF HUMAN HAIR ACTIVATED CARBON**

Human hair was cleaned with hot distilled water for 15 minutes after being washed twice with warm tap water for 30 minutes. During three hours at room temperature, the 25% calcium chloride solution was gently combined with the cleaned hair. The mixture was transferred to a silica crucible and closed with an airtight lid. After being moved to a silica crucible, the mixture was sealed with an airtight covering. The process of activation and carbonization starts.

The system was gradually heated to 75–80°C for two hours, 110°C for three hours, and 450 °C for forty minutes. The product was then dried at 105 °C in an oven after being cleaned with distilled water on a magnetic stirrer at 25 °C and filtered until no chlorine remained in the filtrate. In order to get a fine and uniform powder, human hair activated carbon was lastly pulverized using a lab mortar and pestle [1].



fig 2. 1 Human hair

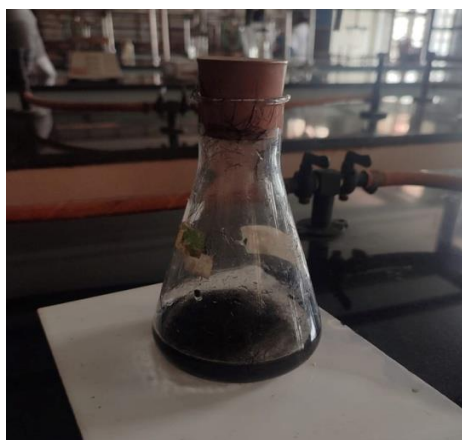


fig 2. 2 Hair mixed with  $\text{CaCl}_2$  solution

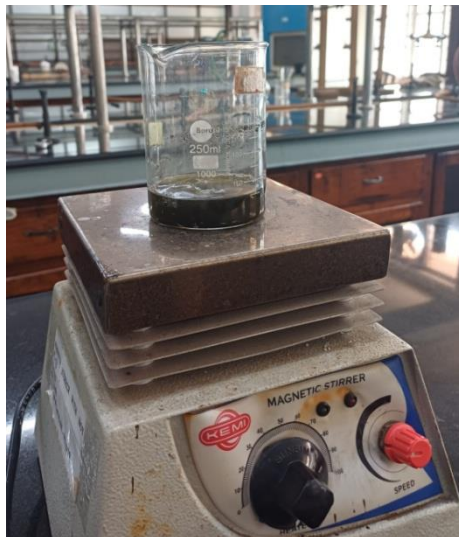


fig 2. 3 Activated-carbon mixed with distilled water on magnetic stirrer



fig 2. 4 Powdered-carbon

### **2.3 CHARACTERIZATION OF HUMAN HAIR ACTIVATED CARBON**

Fourier Transform Infrared Spectroscopy was used to determine the functional group of activated carbon. The FTIR analysis method uses infrared light to scan test samples and observe chemical properties. The FTIR spectra of samples were recorded in the transmittance mode in the range of 400 - 4,000  $\text{cm}^{-1}$ . IR spectrum was recorded using Fourier Transform Infra-Red spectrometer, Thermo-Nicolet iS50.

Scanning Electron Microscopy (SEM) is a test process that scans a sample with an electron beam to produce a magnified image for analysis. The method is also known as SEM analysis and SEM microscopy, and is used very effectively in microanalysis, SEM was performed from DST SAIF Cochin.

CHNS elemental analysis provide a means for the rapid determination of carbon, hydrogen, nitrogen and sulfur in organic matrices and other types of materials. It was performed in DST SAIF Cochin.

X-Ray diffraction analysis (XRD) is a non-destructive technique that provides detailed information about the crystallographic structure, chemical composition. XRD works by irradiating a material with incident X-rays and then measuring the intensities and scattering angles of x rays on the material. X ray pattern was recorded using Burker D8 Advance at DST SAIF Cochin.

### **2.4 ADSORPTION STUDIES USING METHYL ORANGE AS MODEL DYE**

About 0.01 g of methyl orange was taken and made up-to 100 ml using distilled water. 10 ml of this solution was pipetted out into a beaker. The

solution was made up-to 100 ml using distilled water. To 20 ml of the above solution 0.3 g of powdered activated carbon was added.

The solution was mixed well by magnetic stirring for time intervals of 30, 20 and 10 min and was centrifuged at 1100 rpm for 20 min. The absorbance spectrum of the supernatant solutions mixed for various time intervals, as well as the methyl orange solution before adding activated carbon was measured using UV-Visible spectrometer.

# Chapter 3

## RESULTS AND DISCUSSION

### 3.1 CHARACTERIZATION OF ACTIVATED CARBON

#### 3.1.1 FTIR SPECTROSCOPY

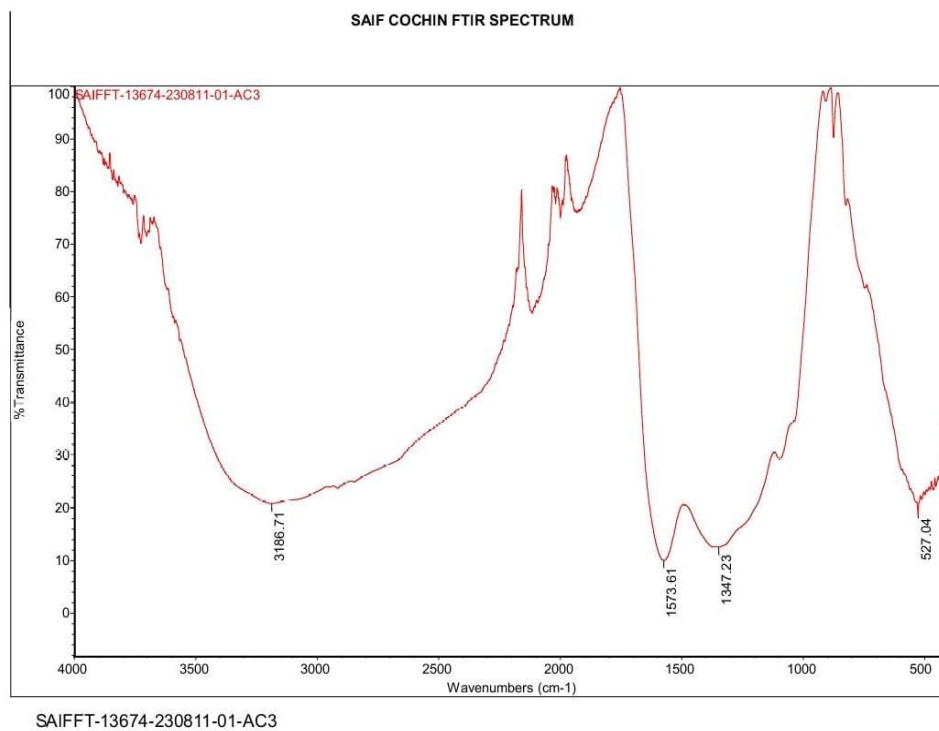


Fig 3.1 shows the FTIR spectra of activated carbon. The structural changes before and after chemical treatment were determined through the analysis

of the FTIR spectra of the samples. In the FTIR spectra of the activated carbon prepared, covering the range of  $500\text{--}4000\text{ cm}^{-1}$ , crucial information about its functional groups is revealed. Notably, a relatively broad peak at  $3186.71\text{ cm}^{-1}$  is observed, indicating the presence of water molecules on the sample and stretching vibration of  $\text{CH}_3$  is overlapped with the stretching vibration of  $\text{H}_2\text{O}$  molecules. Another significant peak at  $1573.61\text{ cm}^{-1}$  corresponds to the stretching vibration of aromatic  $\text{C}=\text{C}$ , while the peak at  $1347.23\text{ cm}^{-1}$  is attributed to the bending vibration of  $\text{C-H}$  bond. Additionally, the absorption peak at  $527.04\text{ cm}^{-1}$  is indicative of a characteristic fingerprint region in the spectrum. These detailed insights from the FTIR spectra provides a comprehensive understanding of the structural characteristics and functional groups present in the activated carbon sample [1].

### 3.1.2 SCANNING ELECTRON MICROSCOPE (SEM)

#### □ SEM

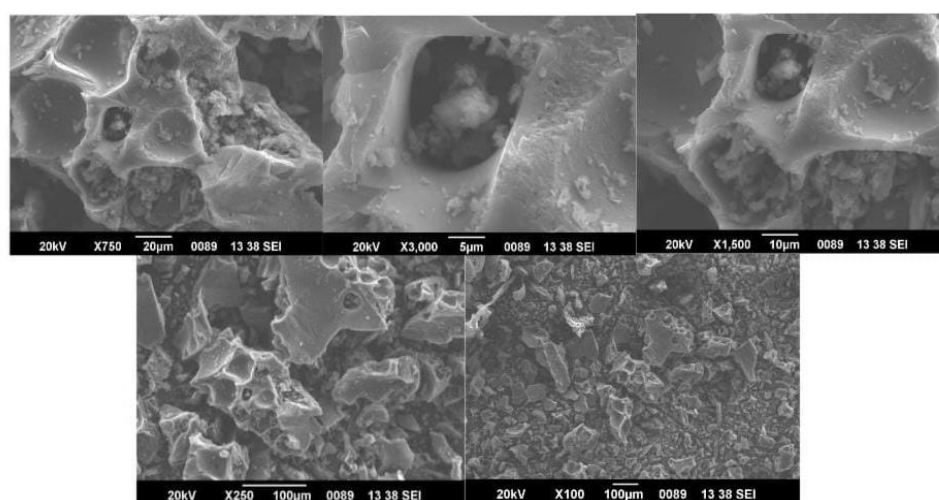


Fig 3.2 shows SEM images of hair based activated carbon. Scanning electron microscopy is a highly functional technique used to obtain high-resolution images and detailed surface information of samples. The surface morphologies of prepared activated carbon at 3,000x and 10,000x magnifications are shown in fig 3.2[1].

### 3.1.3 CHNS ANALYZER

CHNS elementary analyzers provide a way for the rapid determination of carbon, hydrogen, nitrogen and sulfur in organic matrices and other types of materials [1].

#### □ CHNS

	%C	%H	%N	%S
Activated Carbon	54.58	0.65	11.37	0.32

### 3.1.4 XRD

The powder X-ray diffraction (XRD) patterns were carried out on Bruker AXS DB advance X-ray diffractometer. The prepared activated carbon from human hair were characterized by X-ray diffraction. The peak was observed at  $2\theta$  values of 29.417. The observed sharp and intense peak shows the highly crystalline nature of the prepared sample. Average crystalline size was calculated using Debye-Scherrer formula  $D = \frac{k\lambda}{\beta \cos\theta}$ , where  $D$  is the crystalline size,  $\lambda$  is the wavelength of incident X-ray,  $\beta$  is the full width of half maximum (FWHM) of a diffraction peak,  $\theta$  is the diffraction angle and  $K$  is the Scherrer's constant. Average

crystalline size was calculated using Scherrer formula and was found to be 40.3312nm [1].

X-Ray Diffractogram - SAIF Kochi  
AC4 (Coupled TwoTheta/Theta)

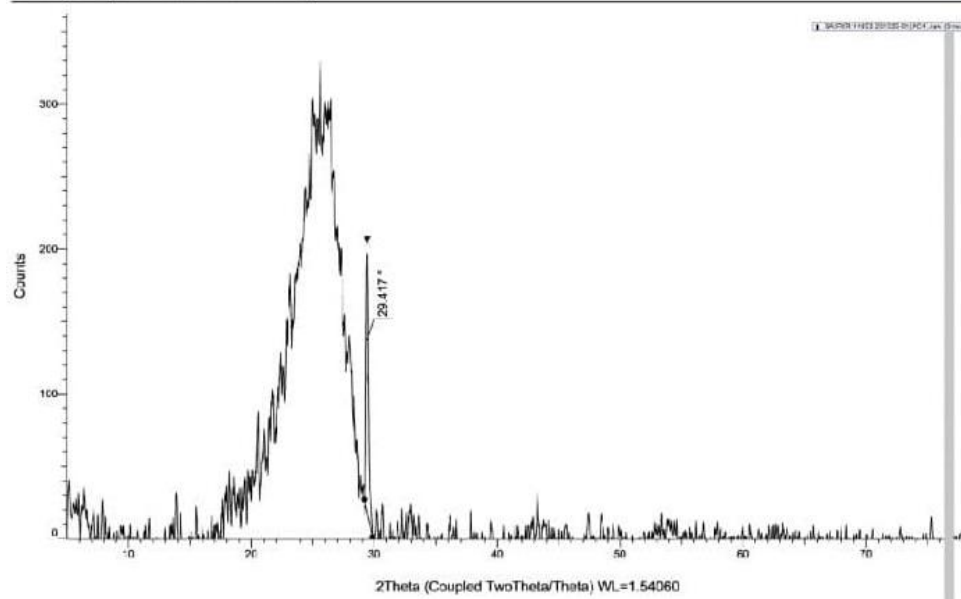


Fig 3.3 shows XRD pattern of human hair and its activated carbo

$$D = K d / \beta \cos \theta$$

$$2\theta = 29.417$$

$$2\theta = 29.417 \times \pi / 180$$

$$2\theta = 0.5132 \text{ radians}$$

$$\theta = 0.2566 \text{ radians}$$

$$\beta = 0.196$$

$$\beta = 0.196 \times \pi / 180$$

$$\beta = 0.0034$$

$$D = 0.89 \times 1.5406 \times 10^{-10} / 0.0034 \times \cos(0.2566)$$

$$D = 40.3312 \text{ nm}$$

## **3.2 APPLICATION**

### **3.2.1 ADSORPTION OF METHYL ORANGE**

The fig3.4 gives the photograph of methyl orange solution and Fig 3.5, Fig 3.6, Fig 3.7 gives the photographs of mixture of methyl orange and the synthesized activated carbon at different time intervals. The UV spectrum of the solution at different time intervals is given by fig. Table 3.2.1 shows the absorbance of methyl orange by activated carbon stirred for different time intervals. The spectrum shows a decrease in absorbance after adding activated carbon [15].



Fig 3.4 Methyl orange solution



Fig 3.5 Methyl orange solution after adsorption by activated carbon for 30 min.



Fig 3.6 Methyl orange solution after adsorption by activated carbon for 20 min



Fig 3.7 Methyl orange solution after adsorption by activated carbon for 10 min

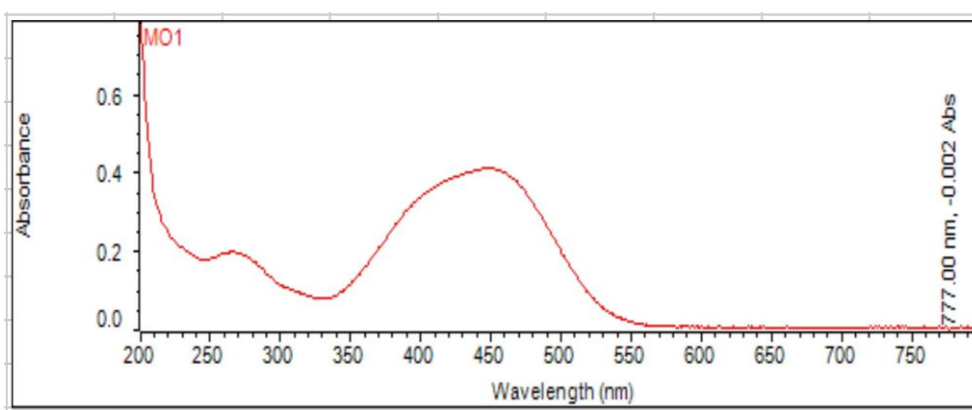


Fig 3.8 Adsorption spectrum of methyl orange before adsorption.

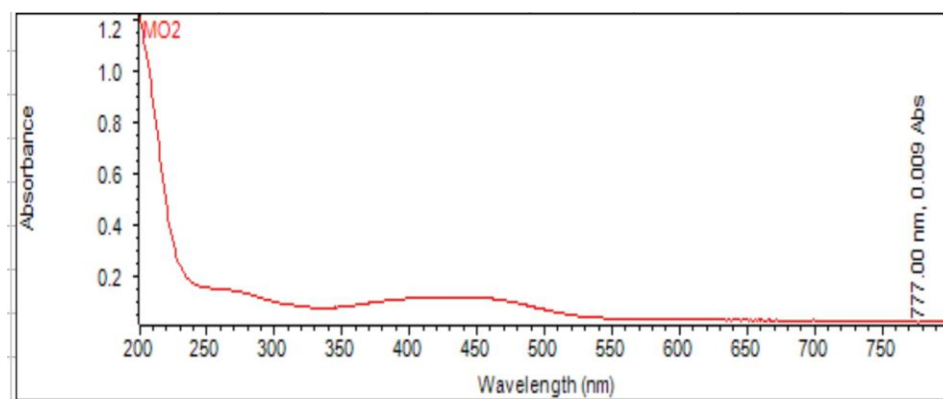


Fig 3.9 Adsorption spectrum of methyl orange after adsorption by activated carbon for 30 min.

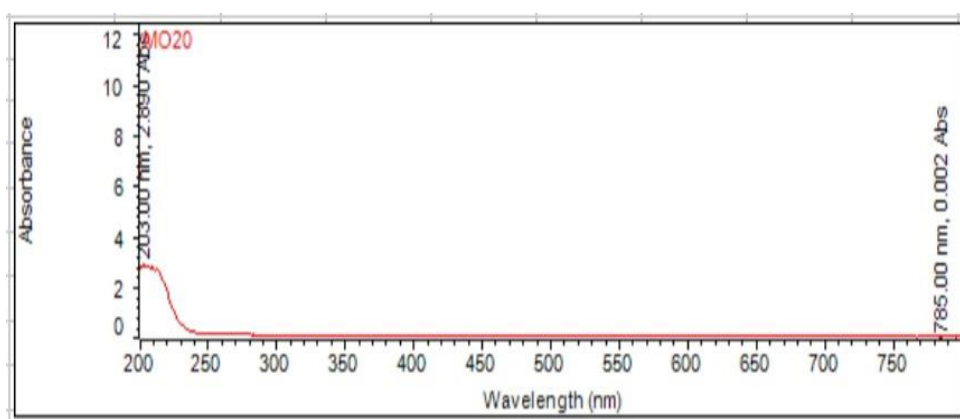


Fig 3.10 Adsorption spectrum of methyl orange after adsorption by activated carbon for 20 min.

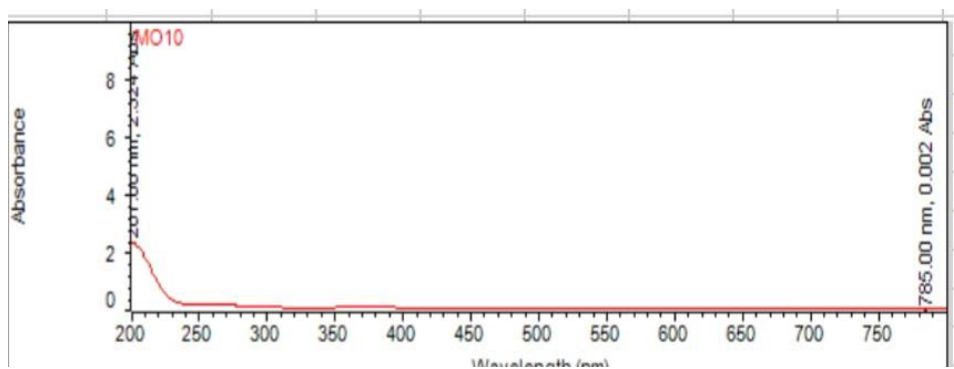


Fig 3.11 Adsorption spectrum of methyl orange after adsorption by activated carbon for 10 min.

Mixing time (min)	Absorbance at 451 nm
0	0.40993
10	0.33992
20	0.30871
30	0.10644

Table 3.1.1

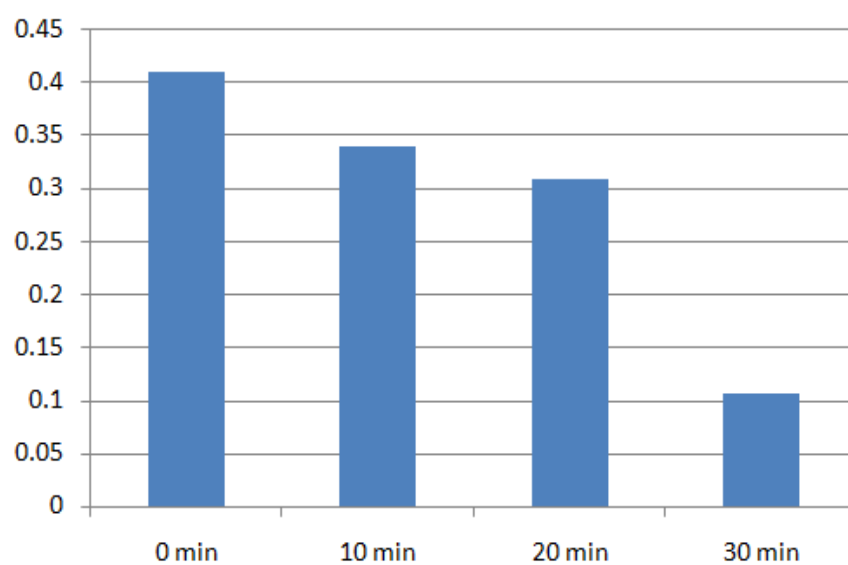


Fig 3.12

# Chapter 4

## CONCLUSIONS

In conclusion, this study has effectively demonstrated the synthesis of activated carbon derived from human hair waste, utilizing calcium hydroxide as an activating agent. The methodology employed underscores its environmentally friendly nature, repurposing human hair waste for the creation of activated carbon. This approach not only addresses environmental concerns but also proves to be economically viable, presenting an affordable alternative in various applications. The characterization of the synthesized samples through FTIR, XRD, SEM, and CHNS analysis has provided valuable insights into their structural and compositional properties. Moreover, the efficacy of the prepared activated carbon was evaluated through the adsorption of methyl orange dye, revealing its remarkable adsorption capabilities over different time intervals.

Looking ahead, the implications of this research extend beyond the scope of this study. The utilization of human hair waste for the synthesis of activated carbon opens avenues for sustainable waste management practices. Furthermore, the potential applications of activated carbon in dye degradation and water treatment highlight its significance in addressing environmental challenges. As we move forward, future research endeavors could explore optimization techniques to enhance the adsorption efficiency of activated carbon derived from human hair waste. Additionally,

investigating the feasibility of scaling up production processes and evaluating its performance in real-world scenarios will contribute to its practical applications. In essence, this study not only signifies a significant step towards sustainable waste utilization but also underscores the potential of activated carbon derived from human hair waste in mitigating environmental pollution and promoting green chemistry practices.

1. S. R. Mousavi-Qeydari, A. Samimi, D. Mohebbi-Kalhor, and E. Ahmadi, "A novel activated carbon from human hair waste: Synthesis, characterization and utilization thereof as an efficient, reusable Ni (II) adsorbent," *Current Research in Green and Sustainable Chemistry*, vol. 4, p. 100141, 2021, doi: <https://doi.org/10.1016/j.crgsc.2021.100141>.
2. R. R. Karri, G. Ravindran, and M. H. Dehghani, "Chapter 1 - Wastewater—Sources, Toxicity, and Their Consequences to Human Health," in *Soft Computing Techniques in Solid Waste and Wastewater Management*, R. R. Karri, G. Ravindran, and M. H. Dehghani, Eds., Elsevier, 2021, pp. 3–33. doi: <https://doi.org/10.1016/B978-0-12-824463-0.00001-X>.
3. R. Sathya, M. V. Arasu, N. A. Al-Dhabi, P. Vijayaraghavan, S. Ilavenil, and T. S. Rejiniemon, "Towards sustainable wastewater treatment by biological methods – A challenges and advantages of recent technologies," *Urban Clim*, vol. 47, p. 101378, 2023, doi: <https://doi.org/10.1016/j.uclim.2022.101378>.
4. S. Bubanale and M. Shivashankar, "History, Method of Production, Structure and Applications of Activated Carbon." [Online]. Available: [www.ijert.org](http://www.ijert.org)
5. H. Demiral, I. Demiral, F. Tmsek, and B. Karabacakoęlu, "Pore structure of activated carbon prepared from hazelnut bagasse by chemical activation," in *Surface and Interface Analysis*, Mar. 2008, pp. 616–619. doi: 10.1002/sia.2631.

6. M. Gayathiri, T. Pulingam, K. T. Lee, and K. Sudesh, “Activated carbon from biomass waste precursors: Factors affecting production and adsorption mechanism,” *Chemosphere*, vol. 294, p. 133764, 2022, doi: <https://doi.org/10.1016/j.chemosphere.2022.133764>.
7. H. R. Lotfy and H. Roubík, “Water purification using activated carbon prepared from agriculture waste — overview of a recent development,” *Biomass Convers Biorefin*, vol. 13, no. 17, pp. 15577–15590, Nov. 2023, doi: [10.1007/s13399-021-01618-3](https://doi.org/10.1007/s13399-021-01618-3).
8. J. Roegiers and S. Denys, “Development of a novel type activated carbon fiber filter for indoor air purification,” *Chemical Engineering Journal*, vol. 417, p. 128109, 2021, doi: <https://doi.org/10.1016/j.cej.2020.128109>.
9. E. W. Harris, “Activated Carbon in Sugar Refining,” *Ind Eng Chem*, vol. 34, no. 9, pp. 1057–1060, Sep. 1942, doi: [10.1021/ie50393a009](https://doi.org/10.1021/ie50393a009).
10. K. L. Rees and J. S. J. van Deventer, “The mechanism of enhanced gold extraction from ores in the presence of activated carbon,” *Hydrometallurgy*, vol. 58, no. 2, pp. 151–167, 2000, doi: [https://doi.org/10.1016/S0304-386X\(00\)00130-4](https://doi.org/10.1016/S0304-386X(00)00130-4).
11. D. A. Yaseen and M. Scholz, “Textile dye wastewater characteristics and constituents of synthetic effluents: a critical review,” *International Journal of Environmental Science and Technology*, vol. 16, no. 2, pp. 1193–1226, Feb. 2019, doi: [10.1007/s13762-018-2130-z](https://doi.org/10.1007/s13762-018-2130-z).
12. K. El Hassani, B. H. Beakou, D. Kalnina, E. Oukani, and A. Anouar, “Effect of morphological properties of layered double hydroxides on adsorption of azo dye Methyl Orange: A comparative study,” *Appl Clay Sci*, vol. 140, pp. 124–131, May 2017, doi: [10.1016/j.clay.2017.02.010](https://doi.org/10.1016/j.clay.2017.02.010).

13. S. and K. K. K. Sinha Prerna and Banerjee, “Characteristics of Activated Carbon,” in Handbook of Nanocomposite Supercapacitor Materials I: Characteristics, K. K. Kar, Ed., Cham: Springer International Publishing, 2020, pp. 125–154. doi: 10.1007/978-3-030-43009-2\_4.
14. A. Mittal, A. Malviya, D. Kaur, J. Mittal, and L. Kurup, “Studies on the adsorption kinetics and isotherms for the removal and recovery of Methyl Orange from wastewaters using waste materials,” J Hazard Mater, vol. 148, no. 1–2, pp. 229–240, Sep. 2007, doi: 10.1016/j.jhazmat.2007.02.028.
15. Y. Yao, H. Bing, X. Feifei, and C. Xiaofeng, “Equilibrium and kinetic studies of methyl orange adsorption on multiwalled carbon nanotubes,” Chemical Engineering Journal, vol. 170, no. 1, pp. 82–89, May 2011, doi: 10.1016/j.cej.2011.03.031.
16. Anastopoulos, Ioannis, and George Z. Kyzas. 2014. “Agricultural Peels for Dye Adsorption: A Review of Recent Literature.” Journal of Molecular Liquids 200:381–89. doi: <https://doi.org/10.1016/j.molliq.2014.11.006>
17. Bhatnagar, Amit, William Hogland, Marcia Marques, and Mika Sillanpää. 2013. “An Overview of the Modification Methods of Activated Carbon for Its Water Treatment Applications.” Chemical Engineering Journal 219:499–511. doi: <https://doi.org/10.1016/j.cej.2012.12.038>.
18. Bubanale, Sadashiv, and M. Shivashankar. n.d. History, Method of Production, Structure and Applications of Activated Carbon.
19. Ciaccio, L. L., and R. V RUHE. 1973. “Water and Water Pollution Handbook.” Soil Science 116(6).

- 
20. Darwish, A. A. A., M. Rashad, and Hatem A. AL-Aoh. 2019. "Methyl Orange Adsorption Comparison on Nanoparticles: Isotherm, Kinetics, and Thermodynamic Studies." *Dyes and Pigments* 160:563–71. doi: <https://doi.org/10.1016/j.dyepig.2018.08.045>.
21. Erickson, David R. 1995. "Chapter 12 - Bleaching/Adsorption Treatment." Pp. 203–17 in *Practical Handbook of Soybean Processing and Utilization*, edited by D. R. Erickson. AOCS Press.
22. Foo, K. Y., and B. H. Hameed. 2010. "An Overview of Dye Removal via Activated Carbon Adsorption Process." *Desalination and Water Treatment* 19(1–3):255–74. doi: 10.5004/dwt.2010.1214.
23. Halder, Joshua, and Nazrul Islam. 2015. "Water Pollution and Its Impact on the Human Health." *Journal of Environment and Human* 2(1):36–46. doi: 10.15764/EH.2015.01005.
24. Heidarinejad, Zoha, Mohammad Hadi Dehghani, Mohsen Heidari, Gholamali Javedan, Imran Ali, and Mika Sillanpää. 2020. "Methods for Preparation and Activation of Activated Carbon: A Review." *Environmental Chemistry Letters* 18(2):393–415.
25. Iwuozor, Kingsley O., Joshua O. Ighalo, Ebuka Chizitere Emenike, Lawal Adewale Ogunfowora, and Chinenye Adaobi Igwegbe. 2021a. "Adsorption of Methyl Orange: A Review on Adsorbent Performance." *Current Research in Green and Sustainable Chemistry* 4:100179. doi: <https://doi.org/10.1016/j.crgsc.2021.100179>.
26. Kamali, Mohammadreza, D. P. Suhas, Maria Elisabete Costa, Isabel Capela, and Tejraj M. Aminabhavi. 2019. "Sustainability Considerations in Membrane-Based Technologies for Industrial

- Effluents Treatment.” *Chemical Engineering Journal* 368:474–94. doi: <https://doi.org/10.1016/j.cej.2019.02.075>.
27. Lin, Li, Haoran Yang, and Xiaocang Xu. 2022. “Effects of Water Pollution on Human Health and Disease Heterogeneity: A Review.” *Frontiers in Environmental Science* 10.
28. Malkoc, Emine. 2006. “Ni(II) Removal from Aqueous Solutions Using Cone Biomass of *Thuja Orientalis*.” *Journal of Hazardous Materials* 137(2):899–908. doi: <https://doi.org/10.1016/j.jhazmat.2006.03.004>.
29. Mekonnen, Mesfin M., and Arjen Y. Hoekstra. 2015. “Global Gray Water Footprint and Water Pollution Levels Related to Anthropogenic Nitrogen Loads to Fresh Water.” *Environmental Science & Technology* 49(21):12860–68. doi: [10.1021/acs.est.5b03191](https://doi.org/10.1021/acs.est.5b03191).
30. Mittal, Alok, Arti Malviya, Dipika Kaur, Jyoti Mittal, and Lisha Kurup. 2007. “Studies on the Adsorption Kinetics and Isotherms for the Removal and Recovery of Methyl Orange from Wastewaters Using Waste Materials.” *Journal of Hazardous Materials* 148(1):229–40. doi: <https://doi.org/10.1016/j.jhazmat.2007.02.028>.
31. Montazer-Rahmati, Mohammad Mehdi, Parisa Rabbani, Atefeh Abdolali, and Ali Reza Keshtkar. 2011. “Kinetics and Equilibrium Studies on Biosorption of Cadmium, Lead, and Nickel Ions from Aqueous Solutions by Intact and Chemically Modified Brown Algae.” *Journal of Hazardous Materials* 185(1):401–7. doi: <https://doi.org/10.1016/j.jhazmat.2010.09.047>.
32. Moss, Brian. 2008. “Water Pollution by Agriculture.” *Philosophical Transactions of the Royal Society B: Biological Sciences* 363(1491):659–

33. Yao, Yunjin, He Bing, Xu Feifei, and Chen Xiaofeng. 2011. "Equilibrium and Kinetic Studies of Methyl Orange Adsorption on Multiwalled Carbon Nanotubes." *Chemical Engineering Journal* 170(1):82–89. doi: <https://doi.org/10.1016/j.cej.2011.03.031>
34. Sinha Prerna and Banerjee, Soma and Kar Kamal K. 2020. "Characteristics of Activated Carbon." Pp. 125–54 in *Handbook of Nanocomposite Supercapacitor Materials I: Characteristics*, edited by K. K. Kar. Cham: Springer International Publishing.
35. Vakili, Mohammadtaghi, Shubo Deng, Giovanni Cagnetta, Wei Wang, Pingping Meng, Dengchao Liu, and Gang Yu. 2019. "Regeneration of Chitosan-Based Adsorbents Used in Heavy Metal Adsorption: A Review." *Separation and Purification Technology* 224:373–87. doi: <https://doi.org/10.1016/j.seppur.2019.05.040>.
36. Yagub, Mustafa T., Tushar Kanti Sen, Sharmeen Afroze, and H. M. Ang. 2014. "Dye and Its Removal from Aqueous Solution by Adsorption: A Review." *Advances in Colloid and Interface Science* 209:172–84. doi: <https://doi.org/10.1016/j.cis.2014.04.002>