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Physical Activation Process and Characterization of Bioactive Carbon, with a Focus on its Applications

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Abstract

Activated carbon is a form of carbon that has been processed to enhance its surface area and porosity. This processing enables it to function as an efficient adsorbent, attracting and retaining other molecules on its surface. In our environment, many types of biomass waste are inexpensive and renewable. Recently, there has been a growing interest in producing products from renewable resources. A prime example is the use of biomass to produce activated carbon. This includes various plant materials such as leaves, bark, fruit peels, roots, seeds, and grasses. This study focuses on the process of producing activated carbon from the leaves and stems of the Cryptolepis buchananii plant. Biomass-derived activated carbon (AC) has extensive applications in energy conversion, energy storage devices, adsorption processes, water purification by removing organic pollutants, and air filtration. The research primarily emphasizes the synthesis of activated carbon from the Cryptolepis buchananii plant. Carbon is a naturally occurring component of plant biomass. In this study, we activate carbon produced through the pyrolysis of this biomass using a physical method involving nitrogen gas. The process involves three main steps: first, synthesizing the activated carbon; second, activating the prepared carbon material; and third, characterizing the activated carbon using a particle size analyzer, UV analysis, and BET analysis.

Keywords: Activated Carbon, Biomass, Renewable resources, Cryptolepis buchananii, Surface area, Porosity

INTRODUCTION

One of the primary sources of human energy is plants. Preparing activated carbon from plant biomass is both efficient and environmentally friendly. There are several advantages to using plant biomass for activated carbon production, including environmental clean-up, sustainable resource utilization, and potential business opportunities through agricultural waste [1]. Activated carbon is typically produced from charcoal or coal; however, the sustainable nature of deriving activated carbon from renewable resources makes this method particularly appealing. Various industrial applications exist for carbonizing naturally occurring activated carbon from different parts of plants and biomass, including: - Grass, Rice husk, Date seeds, Sunflower seeds, Melaleuca cajuputi leaves, Banana with bagasse stems, Wood apple fruit shells, Orange peels and Pomegranate peels [2 – 10]. By utilizing these plant materials, we can promote sustainability while creating valuable products.

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Cryptolepis buchananii, commonly known as the wax-leaved climber, is a climbing shrub indigenous to the Indian subcontinent. It thrives in semi-evergreen and deciduous forests in India and is valued in traditional Ayurveda medicine for its therapeutic properties. Traditionally, various parts of the plant, including the leaves, root, and stem, have been used to treat a wide range of ailments. These include inflammatory conditions, tumours, blood purification, cough relief, bacterial infections, and arthritis, among others [11].

Charcoal offers several advantageous properties, including a larger surface area, significant adsorption capability, microporous structure, and enhanced surface reactivity [12]. Carbon materials derived from biomass present a flexible and sustainable alternative for various applications in energy generation and storage due to their substantial surface area, excellent electrochemical characteristics, and adjustable porosity. Activated carbon obtained from plant biomass has gained popularity as a suitable material for preparing electrodes in supercapacitor cells [13]. Activated carbons (AC) are among the materials utilized in electric double-layer capacitors (EDLCs) [14]. As the demand for renewable energy technology increases, these materials are also used in energy storage devices [15], fuel cells [16], rechargeable batteries [17], and catalytic cells [18]. Since they are produced from abundant and sustainable biomass sources, such as agricultural waste [19], activated carbons provide an affordable and eco-friendly alternative to traditional fossil fuel-based carbon materials.

Any lignocellulosic material that has a high carbon content and low levels of organic matter and volatile substances can generally be used to produce activated carbon. Specific lignocellulosic materials can yield highly porous activated carbons with large surface areas. The production of activated carbon typically involves carbonizing or pyrolyzing these lignocellulosic materials, known as biomass, followed by either chemical or physical activation. The choice of organic matter (biomass) precursor significantly affects the properties and effectiveness of the resulting activated carbon (AC). [20, 21].

This study is based on naturally occurring component of plant biomass - activate carbon synthesized using a physical method - Pyrolysis involving nitrogen gas. Our objective is to create activated carbon from the stems and leaves of the *Cryptolyptis buchanaii* plant. The resulting carbon is characterized using a BET surface area analyzer, while its particle size is determined with a particle size analyzer. Additionally, activated carbon is identified through ultraviolet-visible spectroscopy.

THE MATERIALS AND METHODS

Activated Carbon Preparation

Leaves of the *Cryptolypies buchananii* plant were collected from the forest area of Wai District in Satara. After the collection, the plant material was promptly submitted to the Agarkar Institute for authentication and to obtain a voucher number. Following this, the collected material was shade-dried for 24 hours. The stems were then cut into small pieces and heated in a muffle furnace for 36 hours at a temperature of 100°C. After heating, the sample was crushed and passed through sieves [22.] Next, the prepared powder was activated using a physical activation process. This sample was subsequently heated in a nitrogen environment in a microwave at 400°C for 4 hours [23].

Experimental Analysis Techniques

The sample was analyzed to determine its characteristics and surface area.

ANALYSIS OF BET

The nitrogen adsorption isotherm at 77.35 K was used to determine the textural properties of all the activated carbon manufactured using automated adsorption equipment. The nitrogen adsorption isotherm is the most common method for evaluating the porous nature of activated carbon [24]. Adsorption-desorption isotherms for nitrogen were utilized to identify the specific surface area and porous structure of the produced activated carbon.

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Figure 1 illustrates the results of the BET (Brunauer-Emmett-Teller) and BJH (Barrett–Joyner–Halenda) methods used to determine the surface area of activated carbon. These methods reveal that the broad hysteresis loop observed at high relative pressures may result from the increasing volume adsorbed as relative pressure rises, which is related to the micropore structures of the produced activated carbons. The nitrogen adsorption isotherms show distinct shapes among the activated carbons. All materials exhibit typical type I isotherms, as classified by the IUPAC, which are also referred to as Langmuir isotherms [25]. This type I isotherm is associated with microporous structures [26]. The maximum surface area (SBET) of 11.5022 m²/g was found for activated carbon derived from *Cryptolypies buchananii*, with an average pore diameter of 2.6637 nm. According to Ettish et al., cinnamon-based activated carbon also has a significant surface area along with a considerable pore volume [27].

Figure 2 highlights the Pore Size Distribution (PSD) of activated carbon, which is crucial for understanding variations in its structure. The PSD provides fundamental data regarding the porous nature of the material, offering a statistical analysis of pore sizes that represent each distinct pore configuration. This representation of activated carbon can be envisioned as non-interacting, meticulously designed model pores. Given that most industrial applications utilize porous carbon materials, it is evident that their kinetic and equilibrium properties are of significant interest [28, 29].

Scanning Electron Microscope (SEM) Analysis
SPPU University in Pune does SEM analysis on activated carbon samples.

Scanning electron microscopy (SEM) was used to examine the surface morphology of the synthesized activated carbon. The SEM images (Figure 3) showed a highly porous structure with an irregular and rough surface, characteristic of well-developed pore networks; many cavities and channels were observed, indicating effective activation and carbonization processes; the presence of interconnected pores is likely to enhance the surface area and adsorption potential of the material; the activated carbon showed a significant increase in surface roughness and pore formation when compared to the raw precursor, indicating a successful removal of volatile components during activation. These morphological features confirm the suitability of the synthesized activated carbon for adsorption and electrochemical applications.

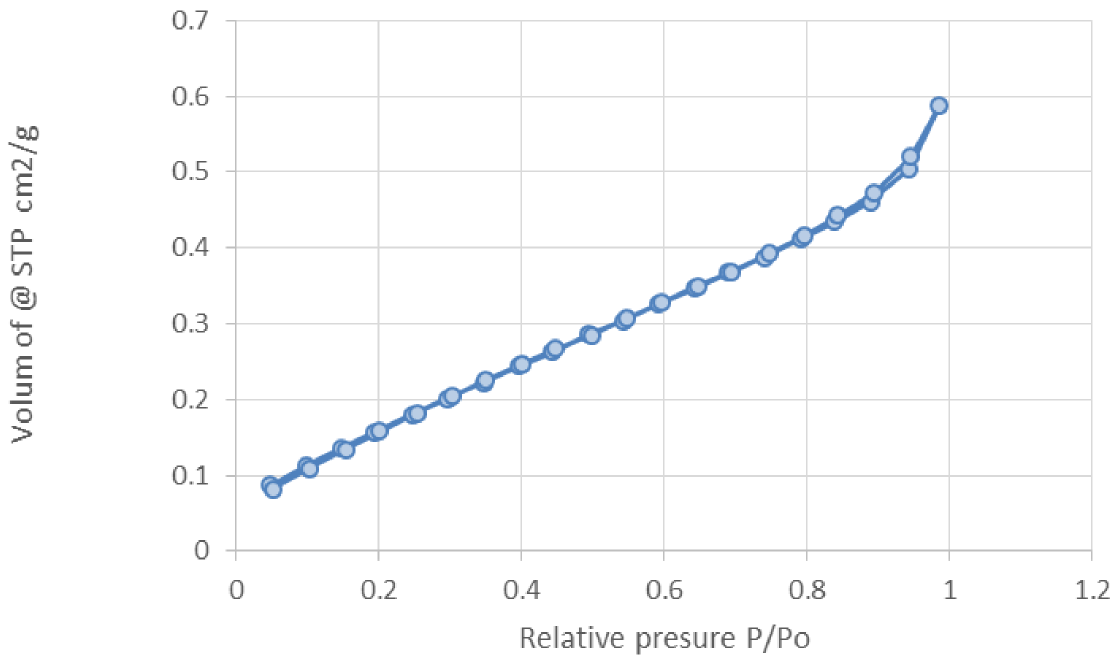

Figure 1. N_2 Adsorption isotherm at 77.35 of Activated Carbon.

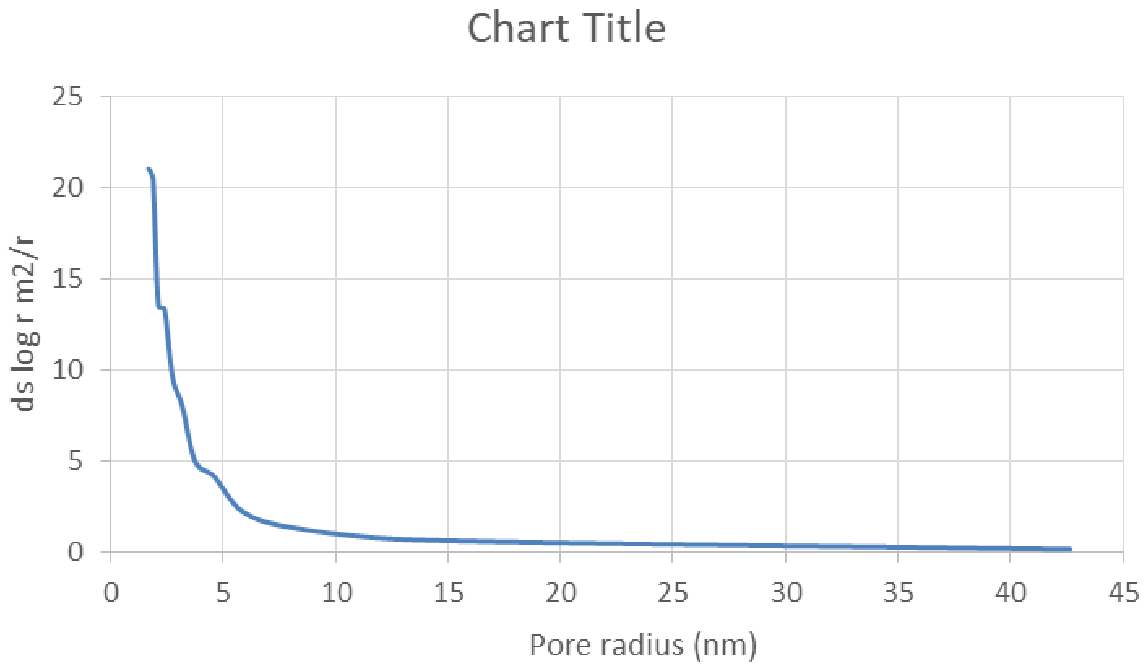

Figure 2. Activated carbon pore size dispersion.

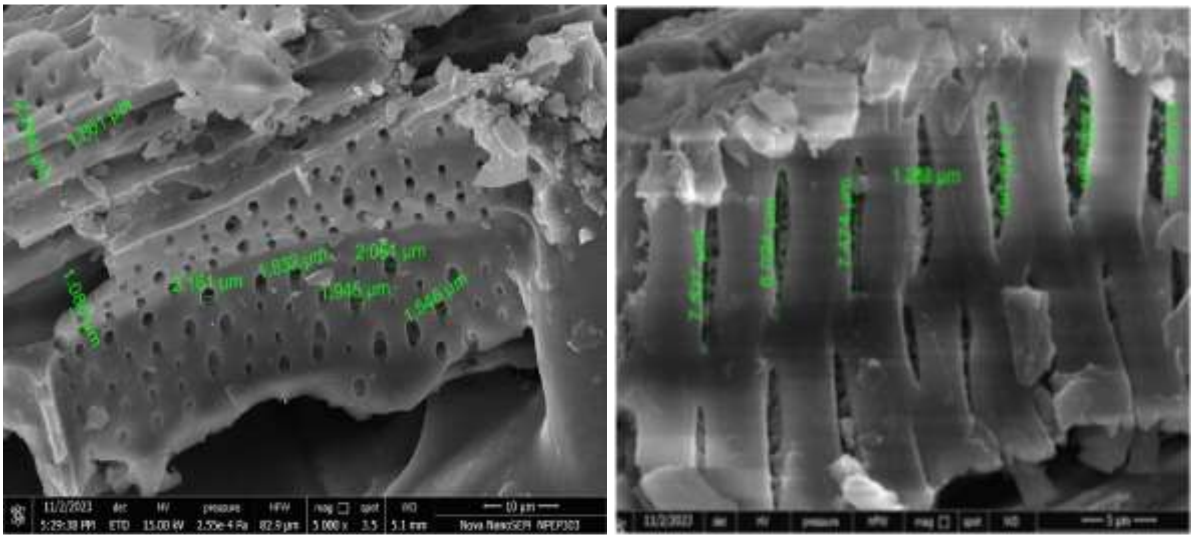

Figure 3. Four SEM micrographs taken at various magnifications (a) 10,000x (b) 5,000x

SEM, or scanning electron microscopy, is an efficient and widely used technique for examining the surface morphology, topography, and porosity of activated carbon. Through direct observation and characterization of aspects that are essential for comprehending activated carbon's performance in many applications, it offers researchers important insights into the structure of activated carbon at the micro and sub-micro level [30].

We make activated carbon from n leaves. By using scanning electron microscopy (SEM) to analyse the surface morphology and porosity of an activated carbon sample made from the stem of the Cryptolepis buchanani plant, it is confirmed that the sample has sizable pores on it. According to morphological observations made using a scanning electron microscope, new granular activated carbon has a wide variety of pores. The average pore size measured in Figure 4 for 5000x and 10000X was 1.546 to 2.204 nm and 1.268 to 8.024 nm, respectively [31].

PARTICLE SIZE ANALYZER AND ZETA POTENTIAL

Measuring the zeta potential of particles is important for assessing their stability in solution and their tendency to aggregate. Zeta potential analysis effectively illustrates the interactions between the adsorbent and contaminant through electrostatic repulsion. The Malvern Particle Size and Zeta Potential Analyzer uses dynamic light scattering (DLS) and electrophoretic light scattering to measure particle and molecule sizes ranging from less than a nanometre to several microns. Particle stability can be inferred from the magnitude of the zeta potential. To determine the zeta potential, a solution is added to a cell containing two gold electrodes. The larger the magnitude of the potential, the greater the electrostatic repulsion and, consequently, the stability of the particles. When a voltage is applied to the electrodes, the particles move toward the electrode with the opposite charge. The Doppler method is used to measure particle velocity as a function of voltage. Zeta potential analysis can also be performed using Microtrac instruments based on Dynamic Light Scattering (DLS). The analysers in this product line provide data on molecular weight, concentration, zeta potential, and particle size—all within a single instrument. The Zetasizer instrument primarily operates by measuring particle size in a liquid medium using the dynamic light scattering (DLS) technique or photon correlation spectroscopy (PCS).

The particle size distribution and zeta potential of the activated carbon derived from *Cryptolepis buchananii* are presented in Figures 4 and 5, respectively. As shown in Figure 4, the activated carbon exhibited a narrow particle size distribution, while Figure 5 illustrates a highly negative zeta potential, indicating good colloidal stability.

Observation Table

Their zeta potential measures the amount of charge on nanoparticles. The more stable the colloid dispersion, the greater the value (whether positive or negative). Good stability is typically indicated by a value greater than thirty. Typically, if the zeta potential of a particle is more positive than +30 mV or more negative than -30 mV, it is considered stable. Zeta potential levels greater than ± 30 mV are often associated with strong physical stability in Nano dispersions [32]. In this zeta potential analysis of bioactivated carbon sample derived from *Cryptolypies buchananii* we get -52.4 (millivolts (mV). The zeta potential value, which indicates the bioactive carbon sample has good physical stability shown in Table 1,2 and 3.

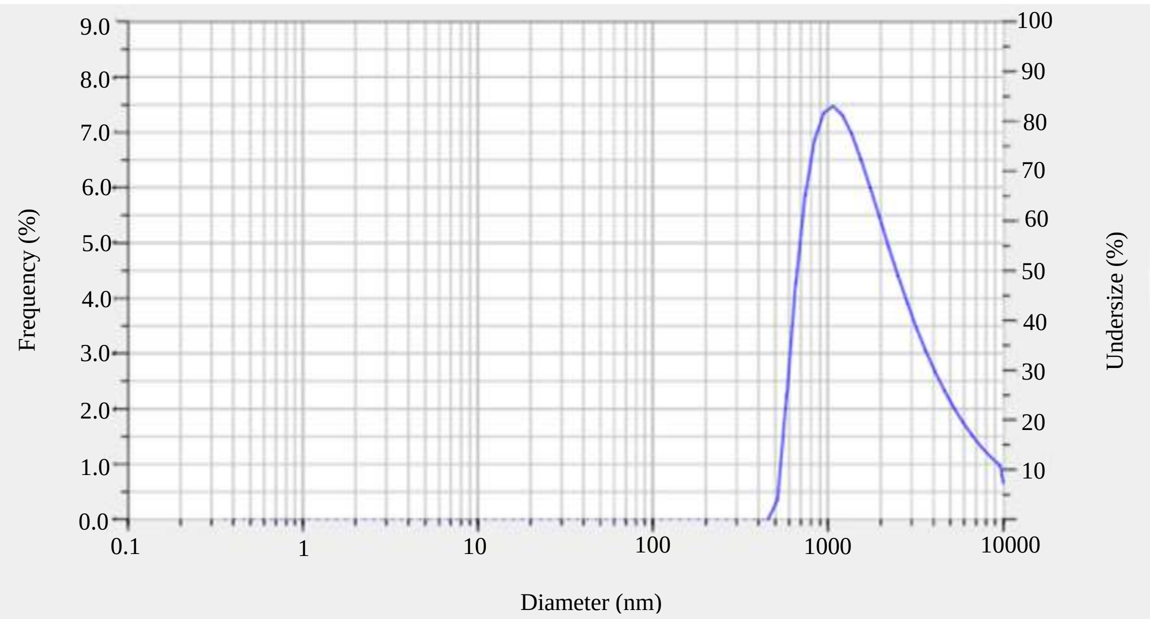

Figure 4. Particle size distribution of activated carbon prepared from Cryptolepis buchananii plant measured using a Particle Size Analyzer.

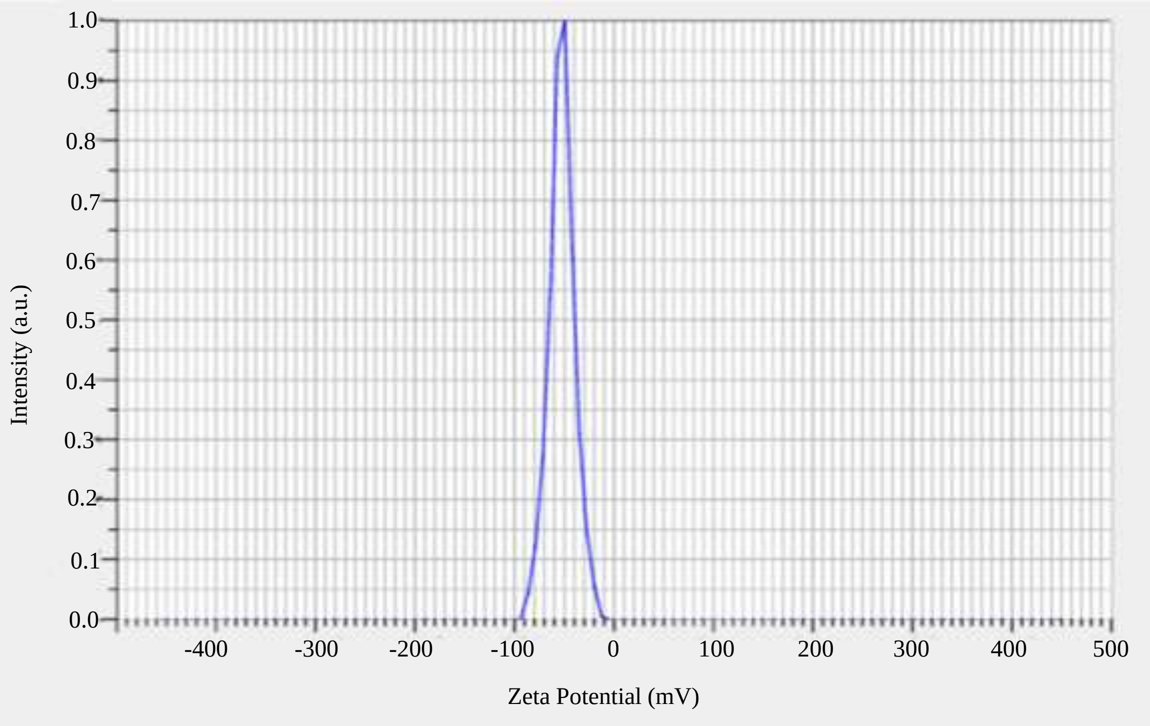

Figure 5. Zeta potential of activated carbon prepared from Cryptolepis buchananii plant, measured using a Zeta Potential Analyzer.

Table 1. Statistical parameters of particle size distribution from the particle size analyser.

Peak No.	S.P.Area Ratio	Mean	S. D	Mode
1	1.00	2023.4 nm	1686.8 nm	1004.9 nm

Table 2. Measured zeta potential and electrophoretic mobility values for the activated carbon sample.

Peak No	Zeta Potential	Electrophoretic Mobility
1	-52.4Mv	-0.000406cm^2/Vs

Table 3. Zeta potential-based stability classification of activated carbon dispersion.

Zeta potential (mV)	Stability degree
0	Slight to no stability
15	Less stability with light settling
30	Moderate stability
45	Decent stability with possible settling

UV ANALYSIS

Method for Preparing Samples: 50 mL of distilled water was used to distribute about 10 mg of the activated carbon nanopowder for UV-Visoptical investigations. In order to ensure even particle dispersion, the suspension was ultrasonically agitated. After that, a 1 mL aliquot ofthe suspension was put into a sterile quartz cuvette so it could be measured.

RESULTS AND DISCUSSIONS

Optical Band Gap and Optical Absorbance UV-Vis absorption experiments were conducted using aqueous solutions of the nanopowders for optical measurements. We mixed 50 mL of distilled water with approximately 10 mg of the nanopowder sample. To determine the absorbance of the activated carbon after preparation, 1 mL of the suspension was placed in a cuvette and subjected to ultrasonic treatment. The results are illustrated in Figure 2. Below a wavelength of 217 nm, all nanopowders exhibited a significant UV absorption band. This band results from the π–π* transitions occurring between 180 nm and 260 nm in all carbon compounds. The UV-Vis absorption spectra of various carbon compounds in the 200 nm region are shown in Figure 6, where the activated carbon sample displays notable absorption [33].

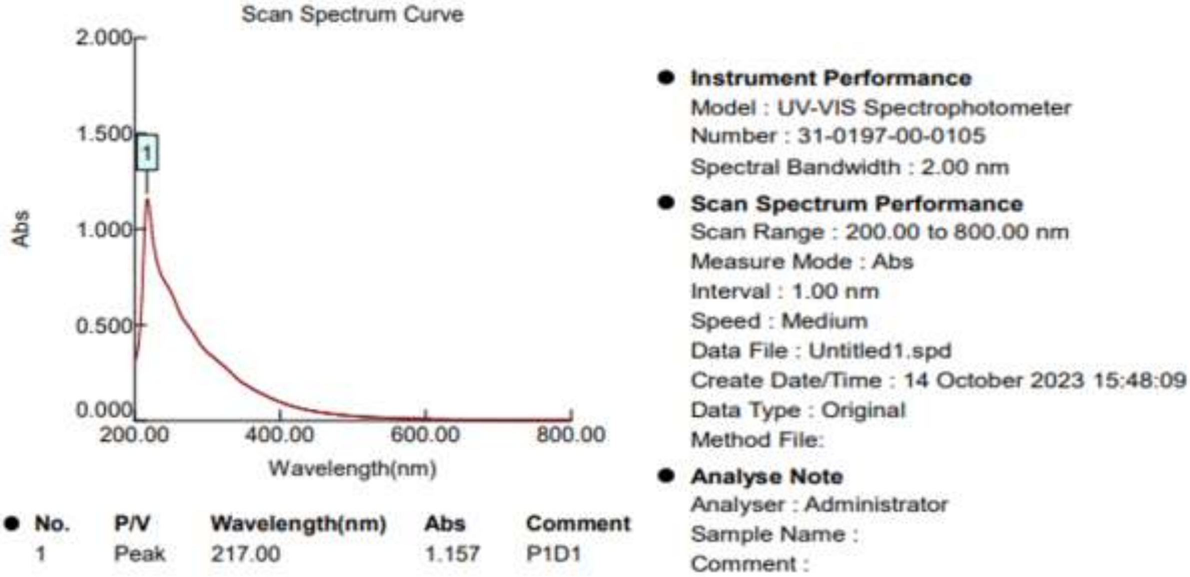

Figure 6. UV Analysis of Activated.

APPLICATIONS OF BIOACTIVE CARBON

Energy Storage Device

Activated carbons derived from biomass are intriguing resources for long-term energy storage devices, such as aqueous supercapacitors. Their abundance, affordability, customizable porosity, and rich heteroatom architectures contribute to their appeal. Activated carbons (ACs) are widely used as electrode materials due to their affordability, non-toxicity, extensive surface area, well-developed porosity, tunable surface chemistry, and excellent electrical conductivity and physicochemical stability. However, conventional activated carbon is often produced from non-renewable sources like petroleum coke and coal, which can have negative environmental impacts. In contrast, using biomass—a green and renewable carbon precursor—offers a sustainable and environmentally friendly method for producing activated carbons [34, 35]. Biomass-derived activated carbons have been extensively utilized in the manufacturing of supercapacitors. Additionally, due to their cost-effectiveness, activated carbon electrodes made from biomass also represent a promising avenue for research in energy storage using battery technology [36-39].

Water Treatment

The primary applications of activated carbon globally are in water treatment and purification. This includes the removal of impurities from drinking water, groundwater, and wastewater. High-quality activated carbon is effective in eliminating phenols and other harmful organic and inorganic substances from both drinking water and wastewater. Additionally, activated carbon can remove a variety of pollutants and carcinogenic compounds and serves as an efficient adsorbent for heavy metals such as zinc, mercury, lead, chromium, and cadmium [40, 41].

CO_2 Adsorption

Activated carbons can be produced from readily available agricultural waste and other types of biomass, making them suitable for various industrial applications, including CO2 adsorption. Several methods can be employed to convert biomass into activated carbons, resulting in a variety of textural characteristics. Additionally, these carbons can be made more effective for CO2 extraction by incorporating nitrogen-based groups into their composition [42]. Helena et al. reported on the synthesis and CO2 absorption capacities of activated carbons derived from biomass sources such as Camellia japonica and Jujun grass. The raw materials, known as hydrochars, were produced through hydrothermal carbonization and were then activated using KOH to yield the final carbon products. These are among the earliest examples of porous materials made from biomass specifically known for their exceptional CO2 absorption capabilities. The study indicates that the efficiency of these carbons is notable because, even in samples with large surface areas, the pore size distribution tends to favor small micropores. Consequently, these carbons are well-suited for CO2 uptake at both high and low pressures, which enhances their overall performance [43].

Gas Purification

In this study, Elsayad et al. evaluate the effectiveness of various commercially available coconut-based activated carbons in removing hydrogen sulfide (H_2S) at both low and high concentrations. The primary objective of the study is to investigate how different experimental conditions affect H_2S adsorption. This includes examining the influence of humidity, water content, and impregnation on the activated carbon's ability to facilitate H_2S oxidation. To gain a deeper understanding of the mechanism of reactive adsorption, the samples were analyzed both before and after H_2S adsorption. This analysis allowed for the identification of the types of products deposited on the exhausted materials [44]. Additionally, activated carbon has traditionally been used to extract and recover Volatile Organic Compounds (VOCs) from gases, with extensive literature detailing these adsorption processes. Given the wide variety of potential VOCs present in gaseous ambient samples, a summary of the key concepts is provided [45].

Catalysis

Activated carbons have been extensively studied as catalysts and catalytic supports in recent decades. These materials possess exceptional qualities, including a high specific surface area and excellent chemical and thermal stability. Moreover, they can be derived from various types of lignocellulosic waste. Bifunctional catalytic beds based on activated carbon have been utilized to investigate the direct synthesis of dimethyl ether (DME) from syngas. For catalyst preparation, two types of activated carbons were produced: one through chemical activation of olive stones with phosphoric acid and the other through physical activation via CO2 partial gasification. [46, 47]. In a study by Mendez et al., the potential of physically activated carbon derived from biomass was examined as a catalyst for extracting copper from a copper sulfide concentrate rich in chalcopyrite. The leaching experiments were conducted under two distinct reaction times [48].

CONCLUSION

The most commonly used method for evaluating the porous characteristics of activated carbon is nitrogen adsorption isotherm analysis. Based on the BET surface area characterization of activated carbon derived from the leaves of the *Cryptolyes buchananii* plant, we concluded that activated carbon with a microporous structure has a larger BET surface area. The microporous nature of these carbon materials makes them advantageous for industrial applications. However, during the chemical activation process, the micropore structure may expand or develop further. The size of the zeta potential indicates the stability of the colloidal system. Since each particle in suspension carries a high negative zeta potential, they tend to repel one another, preventing aggregation. Using activated carbon derived from sustainable resources offers two significant benefits: low cost and environmental friendliness. This is especially important given that the prices of high-end activated carbon products have increased over time.

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