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Investigation of Structural, Thermal, Magnetic and Functional Properties of Sustainable Carbon Materials from Calcined Coconut Shell Charcoal

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ABSTRACT: The effect of calcination of the coconut-shell charcoal on thermal, structural, chemical, morphological, textural, vibrational and magnetic properties has been investigated. The TG/DTA results indicated that moisture and volatile contents were removed up to about 200 °C, unstable ones were decomposed between about 200 and 450 °C, and a thermally stable carbon framework was formed above approximately 600 °C. The XRD patterns showed broad reflections near 28° and 45° in 2θ, which confirmed the amorphous-to-turbostratic nature of the carbon. The crystallites average size was 4 nm before calcination and 6 nm after calcination. FTIR analysis showed the presence of hydroxyl, aliphatic C–H, carbonyl, aromatic C=C and C–O bands, and the decrease in their intensities following calcination, suggested deoxygenation and dehydration. The SEM study indicated that the surface was irregular, rough, fractured and porous with many interconnected cavities, cracks, grooves and natural channels. The BET analysis was employed to study the surface area, pore volume, pore diameter and the pore size distribution, and the hysteresis behavior observed reveals the presence of mesoporous nature along with the microporous regions. Raman spectra exhibited the typical D and G bands at 1350 and 1580 cm^{-1}, respectively, and the changes in the intensity ratios of these bands after calcination indicated carbonization, aromatization, defect formation and partial graphitic ordering. VSM analysis results indicated that the magnetic reactivity of the charcoal changed because of the calcination, which could have been due to the reorganization of the organic carbon matrix and/or the possible concentration or modification of the inorganic mineral phases. In summary, the process of calcination transformed coconut-shell biomass into a carbon material with thermal stability along with chemical reorganization and porous structure that may have some degree of order and functionality for use in adsorption, catalysis, electrochemistry and energy-related applications.

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1. INTRODUCTION

With the rapidly depleting fossil fuels, the global rise of ecological pollution and the increased demand for the development of new functional materials with environmental protection, low cost and long-term sustainability, the development of eco-friendly, low-cost, long-lasting materials has become a high priority. The conversion of agricultural and agro-industrial residues into gas or vapor has emerged as one of the interesting aspects of realizing the conversion of renewable biomass into valuable products carbon-based materials. Coconut Shell is one of the available biomass resources around the world which is a remaining substance of coconut despite being abundantly available, it is a renewable byproduct, a low-cost material and an underused material. Coconut processing produces significant amounts of shells, which are often disposed of via open incineration, unchecked disposal or low value uses. These practices could cause environmental damage and the release of heat-trapping particulate matter and gases into the atmosphere [1-2]. Therefore, the process of repurposing the waste of coconut shells into valuable carbon materials for technology is an eco-friendly method to reduce waste, reclaim resources and produce functional materials of enhanced value. Coconut shell has a complicated structure. The fibers consist of mainly cellulose, hemicellulose, lignin and mineral components, which make it particularly suitable as a raw material for the synthesis of carbon-based materials. The strong cell shape and high natural carbon content of the coconut shell helps to provide a stable carbon-based fundamental structure during thermal processing. The volatile organic constituents can be released slowly by controlled carbonization and subsequent calcination since the carbon-rich material undergoes structural rearrangement, aromatization and condensation. As a result, coconut that has been calcined Shell charcoal has the potential to act as an effective precursor for environmentally friendly carbon-based materials with possible uses in energy storage, adsorption, catalysis and environmental cleanup, sensing and electrochemical technologies [3-5]. Thermal degradation properties of the lignocellulosic components play a very significant role in the conversion of biomass to carbon materials. During the thermal treatment process, Hemicellulose usually degrades at relatively lower temperatures, followed by the degradation of cellulose and slow degradation of lignin at a broader range of temperatures. Calcination also results in a sudden increase in the ability to remove the remaining contents and unstable oxygenated compounds, which leads to an increase in the saturation of carbon and structural integrity [9-11]. The manufactured material may exhibit a higher level of basic carbon than non-treated or biomass that is only partially carbonized. Grasping these thermal changes is thus crucial for enhancing processing conditions and determining relationships between calcination factors and the outcome structural and functional characteristics. Another important criterion of suitability of coconut shell charcoals as a sustainable carbon source is the thermal properties of the charcoals. Thermogravimetric analysis (TGA) and differential thermal analysis (DTA) can be utilized to examine mass-loss characteristics, thermal breakdown phases, remaining moisture, gaseous substances and the thermal stability of the charred material. Compared with unprocessed coconut shell, the carbonized and calcined materials are expected to have lower volatiles content and increased resistance to further thermal decomposition. The thermal process might consequently change to a chemically intricate biomass

Converts a precursor to a relatively stable substance that contains carbon. Turns a precursor into a relatively stable carbon-rich product. In particular, the Assessment of thermal stability is key for applications that require high temperatures. Temperatures, repeated heat and cooling cycles, catalytic reactions or the use of high-temperature energy storage. Further, thermal analysis is an important tool for identifying suitable processing temperatures and the relationship between the thermal history of the material and final material properties [9-10]. Structural characteristics of carbon materials derived from biomass play important role in the technological capabilities of these materials. The X-ray diffraction (XRD) technique provides valuable information on the structural organization, crystallinity and growth of turbostratic carbon regions. Carbon from a lignocellulosic source usually exhibits broad

diffraction peaks associated with perhaps less regular graphitic structures. Heat treatment can produce changes in strength, location and organization of the aromatic region, range of these unique reflectabilities and signposting changes in interlayer distances. Studies of the more oxygen-containing functional groups such as carbonyl, carboxyl and hydroxyl groups, using supplementary spectroscopic techniques such as Fourier-transform infrared spectroscopy (FTIR), can identify the progression of these groups. These structural changes are important as they lead to changes in surface chemistry which play an important role in determining adsorption behavior and chemical reactivity, wettability and interactions with neighboring species [11-12]. The surface morphology and textural properties of coconut shell charcoal are of great scientific interest besides the structural properties. Morphological changes such as channels, cavities, cracks and heterogeneous surface features that can occur due to thermal degradation can be detected by scanning electron microscopy (SEM). Naturally vascular and cellular architecture of coconut shell could be used as an intrinsic template to create porous carbon structure. Carbonization and calcination may occur that can create or expand internal voids in the carbon matrix. Such changes can enhance the surface area for the basic carbon and aid in the mass transport of material. The formation of micro and mesoporous structures is very desirable for adsorption and electrochemical applications as it increases the amount of active sites and shortens the diffusion paths [13-14]. There is significant structural change in coconut-shell carbon derived after calcination observed from the Raman spectroscopy. Uncalcined sample shows the characteristic D bands (~1350cm-^{1}) and G bands (~1580cm-^{1}) which are related to disordered carbon domains and graphitic sp^{2}-bonded domains, respectively. The D and G bands are enhanced after calcination, suggesting that the carbon-network has rearranged and aromatized with increasing severity. The level of structural disorder/density of defect is represented by I_D/I_G ratio. The shift in peak position, intensity and bandwidth indicates improved graphitic ordering, and the residual defects indicate that biomass-derived carbon has a heterogeneous character. The results again validate the effectiveness of the thermal transformation and improved integrity of the carbon structure. For the coconut-shell carbon, a noticeable change is observed in the magnetic response after its calcination, as examined by the vibrating sample magnetometry (VSM). The uncalcined sample, overall, displays low magnetic properties, particularly low values of saturation magnetization (M_s), remanent magnetization (M_r) and coercivity (Hc) due to its chief organic carbonaceous matrix. Following the process of calcination, thermal decomposition and structural transformation may be directed toward certain inorganic mineral phases and cause changes in the magnetic centres associated with defects which may result in a changed hysteresis reaction. The observed changes in M_s, M_r and Hc indicate alteration in magnetic interactions and the activity of domains, which is a confirmation of the change in magnetic properties of the material due to calcination [15-16]. Their eco-friendly source, plentiful resources, affordable manufacturing expenses and adjustable physical and chemical properties offer notable benefits compared to various traditional carbon sources obtained from non-sustainable resources.

Although significant strides have been made in biomass-based carbon research, it is still important to define a clear link between the change in the structure due to calcination and functional efficiency. Thus, thermo-chemical treatment (thermo-chemical calcination) is mandatory to achieve an adequate balance between carbonization, structural integrity, surface properties and porosity. This study is not merely for making a single kind of carbon material from biomass; its significance lies in its potential to yield a variety of other valuable carbon materials. It demonstrates the potential of coconut shell waste as an eco-friendly source of carbon. Precursor and provides a scientifically proven pathway towards converting the abundant agricultural waste product into a value-added functional product [17-18]. So, the progress of such materials simultaneously aids in sustainable waste management and carbon-resource utilization,

along with the development of next-generation functional materials, aiding the wider shift towards a circular, low-emission, and resource-efficient economy.

2. Experimental

2.1 Materials

Coconut shells were chosen as a renewable source of carbon rich precursor of lignocellulosic matrix. They used to wash the shells thoroughly in deionized water, dry them and crumple them by scrunching and then carbonized them under optimum conditions, followed by calcination to get the carbon-rich charcoal powder. An inert atmosphere was used if necessary, using high purity nitrogen. The basic technique was used for all the materials and deionized water was used throughout the preparation of prepared solution to minimize the impurities in the prepared solution.

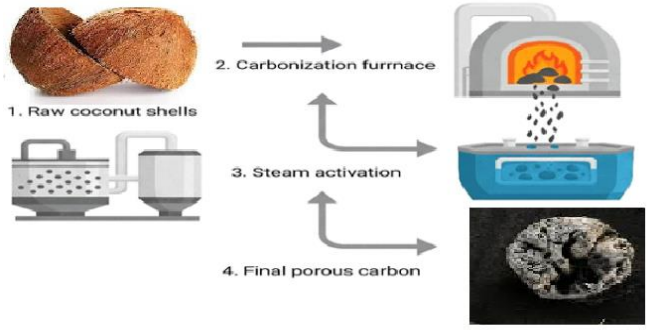


Figure 1 Preparation of Coconut Shell Charcoal.

2.2. Synthesis

Coconut shells were carefully cleaned with deionized water to remove the impurities and dried at 105 °C for 24 hours as shown in Figure 1. Pyrolysis of dried shells was carried out in an oxygen deficient atmosphere at 500 °C for 2 hours, and the obtained coconut shell charcoal was chopped into small pieces. The charcoal was further structurally modified and carbonised by calcination for 2 hrs at 750 °C in an inert (N_2) atmosphere, resulting in a uniform particle size of the charcoal. Natural cooling to room temperature, then the product was recovered and kept in a closed system for further structural, thermal, morphological, spectroscopic, surface-area and magnetic characterization.

2.3. Measurements

Synthetic coconut shell charcoal was systematically characterized to know its structural, thermal, morphological properties and the functions. The crystallinity, phase evolution and structural ordering were investigated by X-ray diffraction (XRD). Thermal stability and decomposition behaviour were determined by thermogravimetric analysis (TGA) and differential thermal analysis (DTA). To identify the functional groups on the surface and chemical-bond transformation of this process, the FTIR technique was used. Scanning electron microscopy (SEM) was used to study surface morphology, particle structure, and porosity. Specific surface area, pore volume and pore-size distribution were measured by the Brunauer–Emmett–Teller (BET) analysis. The laser power was limited to less than 5 mW to avoid thermal degradation of the sample, and Raman spectra were obtained in the spectral range 100–3200 cm^{-1} with a spectral resolution of 2 cm^{-1}. After baseline correction, the spectra were fitted to obtain the D/G-Band intensity ratio, crystallinity and structural disorder. Lastly, vibrating sample magnetometer (VSM) was used to record magnetic hysteresis loops at room temperature for calculating the saturation magnetization, remnant magnetization, coercivity, and magnetic susceptibility. The powdered sample was placed in a nonmagnetic sample holder in the amount of about 10 to 25 mg and the instrument was checked for accuracy with a standard reference material before use. To gain an understanding of the magnetic characteristics and structural changes of the carbon formed from the coconut shells before and after calcination, magnetic susceptibility and hysteresis were measured. The measurements enabled a full correlation between processing conditions, material structure and functional performance.

3. RESULTS AND DISCUSSION

3.1. TG / DTA Analysis

The thermogravimetric (TG) and differential thermal analysis (DTA) features were used to assess the thermal decomposition and stability of the coconut-shell charcoal before and after calcination as presented in Figure 2. The TG curves show a small initial weight loss in the temperature region below about 200 °C that may be due to desorption of moisture and low-boiling volatile compounds. The mass loss is more distinct from 200 to 450 °C, which is associated with the loss of any remaining oxygen-containing functional groups and other thermally unstable organic components. When the temperature exceeds about 450 °C, the mass loss is slow, with progressive degradation, rearrangement and carbon enrichment of carbonaceous structure. The mass stability of both samples is relatively good above around 600 °C, indicating the formation of a thermo-resistant carbon framework. The resultant sample after calcination has a reduced total mass loss and is more stable than the uncalcined sample, thus indicating that a significant amount of the volatile and unstable constituents has been removed by the calcination process. Above about 800 °C, the contribution of the carbon matrix, mainly due to its stability, and inorganic ash to the remaining mass is predominant [21–22].

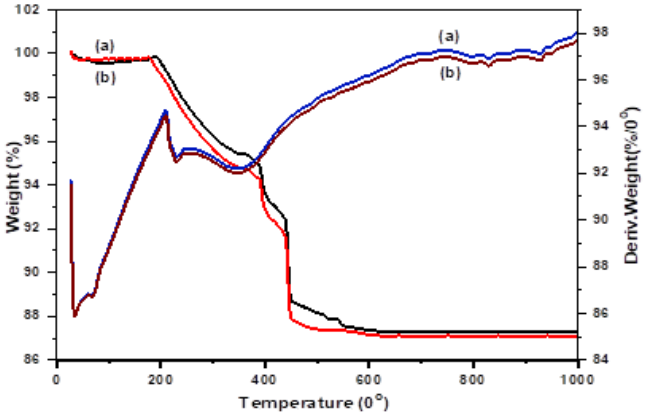


Figure 2 TG / DTA analysis of the Coconut Shell Charcoal samples (a) Before calcined (b) After calcined.

3.2. X-Ray Diffraction Analysis

The crystalline structure, phase evolution, and structural order of the coconut shell charcoal were analyzed by using X-ray diffraction (XRD) technique, as illustrated in Figure 3. A diffraction pattern was recorded at a set 2θ range for Cu Kα radiation (λ = 1.5406 Å) with a constant scanning rate. The average crystallite size (D) of the nanoparticles was calculated from the Debye-Scherrer equation,

$$D = \frac{K\lambda}{\beta \cos\theta}$$

Where the grain shape factor (K) = 0.94 for spherical grains, λ is the X-ray wavelength (0.1504 nm) and θ is the diffraction angle at FWHM (β), defined in radians [23-24].

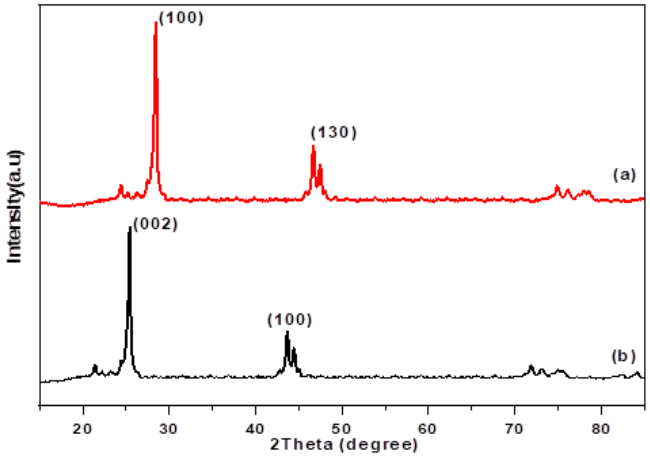


Figure 3 XRD pattern of the Coconut Shell Charcoal powder samples: (a) Before calcined (b) After calcined.

The average crystallite size was 4 nm prior to calcination and 6 nm following calcination. The diffractogram obtained showed broad features typical of mostly disordered or turbostratic carbon. A broad reflection at 2θ ≈ 23-26° is generally associated with the (002) plane composed carbon structure, showing the presence of partially ordered graphitic domains, and a weaker feature around 2θ ≈ 43-45° may be due to the in-plane ordering of carbon atoms, (100) reflection. Calcination helps with dehydration, deoxygenation, volatilization and aromatization. The increase in peak width can be attributed to the reduced crystallite growth and maintenance of the carbon basic structure as amorphous-to-semi

crystalline. The results show efficient conversion of lignocellulosic bioresource into structurally stable carbonaceous material.

3.3. FTIR Analysis

The surface functional groups and changes of chemical bonds in calcined coconut shell charcoal were characterized by Fourier-transform infrared (FTIR) spectroscopy (Figure 4). The wide band of absorption in the range 3150-3550 cm^{-1} is attributed to the stretching of the O-H groups associated with phenolic structures or moisture. Observed bands are in the range of 2850–2950 cm^{-1} correspond to aliphatic C–H stretching vibrations [27-28].

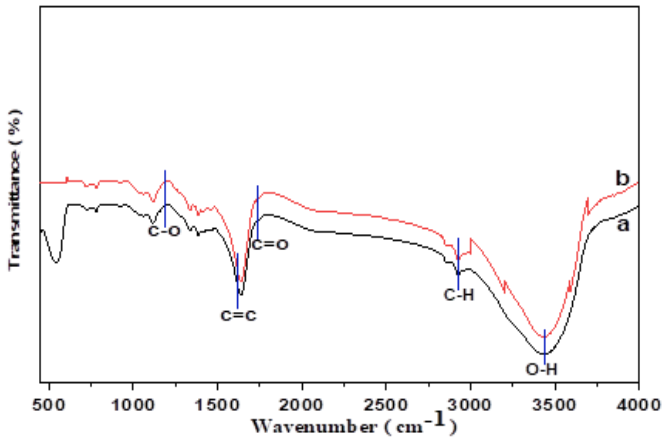
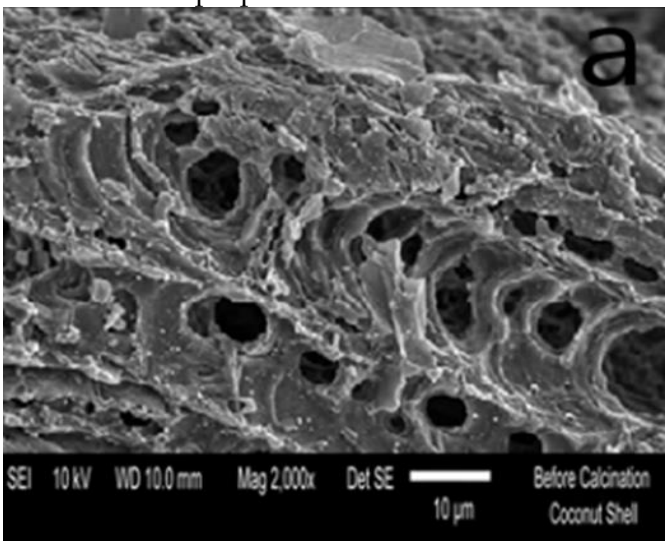


Figure 4 FTIR pattern of the Coconut Shell Charcoal powder samples: (a) Before calcined (b) After calcined.

The absorption between 1645 and 1760 cm-1 is one of the characteristics of the C=O stretching in the carbonyl, carboxyl or ester functional groups. Lastly, the band around 1600 cm-1 is linked to aromatic C=C skeletal vibrations and is an indication of the presence of condensed aromatic carbon domains. The absorptions occurring between 980 and 1290 cm-1 are generally indicative of the C–O stretching vibrations of alcohol, ether and phenol [29-30]. The loss of intensity of this oxygen containing functional group bands after the calcination suggests the gradual loss of water from them and the conversion of the solid material to gaseous form. In general, FTIR analysis results prove that there is a considerable change in chemical structure and that the base of the carbon structure can form a stable structure.

3.4. SEM Analysis

The surface morphology and microstructural changes of the coconut shell charcoal after calcination were investigated by scanning electron microscope (SEM) as exhibited in Figure 5. The irregular and heterogeneous nature of the carbonaceous surface, with interconnected cavities, cracks, grooves and naturally occurring porous channels, were revealed in micrographs. These morphological characteristics are obtained by means of the decomposition and conversion of the lignocellulosic components in the carbonization process and the calcination process. If there are abrupt changes in the components, it encourages the opening and remodeling of the pores, that can result in an increase of the surface area and active components. The observed rough and fractured biomass structure [31-32] indicated that the original biomass structure was greatly changed after heat treatment. The presence of connected pores can benefit application for adsorption and electrochemical applications because of the enhancement of surface accessibility and mass movement. The investigation proves that coconut shell biomass can be successfully transformed into structurally developed porous carbon material that has functional properties.



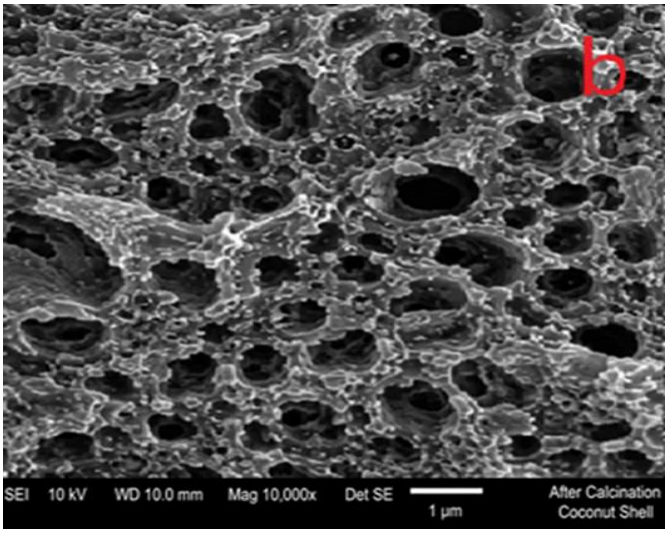


Figure 5 SEM image of the Coconut Shell Charcoal powder samples: (a) Before calcined (b) After calcined.

3.5. BET Analysis

The textural characteristics of calcined coconut shell charcoal, such as specific surface area, total pore volume, average pore diameter and pore-size distribution, were thoroughly assessed using Brunauer–Emmett–Teller (BET) surface-area analysis as shown in Figure 6,7. Nitrogen (N_2) adsorption and desorption measurements have been made at 75 K and isotherms were analyzed using IUPAC classification. A hysteresis loop and the shape of the isotherm can be used for classification of the adsorption behavior [33-34].

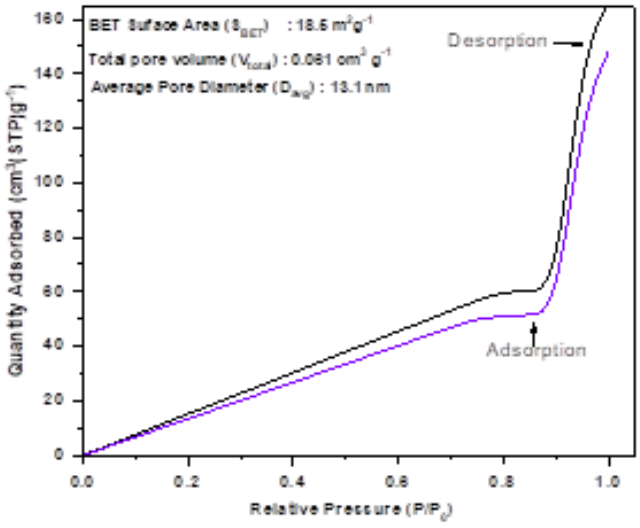


Figure 6 BET Isotherm- Coconut shell (Before Calcinations).

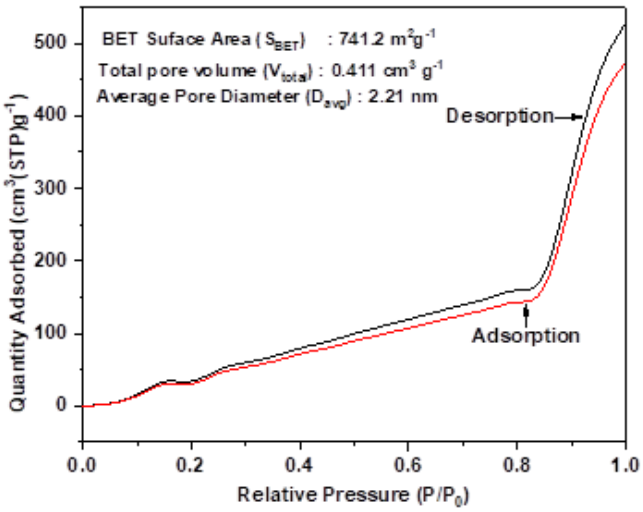


Figure 7 BET Isotherm- Coconut shell (After Calcinations).

Type IV behavior with hysteresis loop is indicative of the presence of mesopores and capillary condensation, while Type I is usually associated with microporous materials. Mesopores (2–50 nm) allow molecules to pass through and provide surface area for the interior of the material, while the micropore area (<2 nm) contributes greatly to adsorption. Macropores > 50 nm in size that could act as transport channels. The appropriate range of relative pressure for the adsorption branch was selected to calculate the BET surface area. However, suitable adsorption models were employed to calculate the pore-volume and pore-size distributions. Volatile components removed by the calcination process may expand the accessible surface area, because of opening of pores and structural reorganization [35–36]. These features demonstrate the usefulness of the carbon produced for electro-chemical, catalytic, and adsorption processes.

3.5. Raman Analysis

Raman spectroscopy is also an important tool to provide information about the defect features, carbon ordering and the history of the carbon extracted from coconut shell before and after the calcination process as seen in Figure 8. The

uncalcined coconut shell's Raman spectra typically shows strong D and G bands about ~1350 and ~1580 cm^{-1}, respectively. The G band corresponds to the inplane vibration of laying-graphitic carbon atoms, while the D band is associated with the breathing mode arising from defects in sp^2 domains of carbon atoms. Significant structural disorder and diverse carbon domains are indicated by the comparatively high ID/IG ratio and functional groups that contain oxygen that come from lignocellulosic components. The volatile decompositions and carbonaceous structure reorganizations are incomplete at high temperature calcinations and cause significant change in the Raman characteristics. The relative sharpness and distinctness of the D and G bands indicate increased growth of sp2-bonded carbon networks. The competition of local graphitic ordering, aromatization and defect formation during thermal treatment is reflected in the ID/IG changes. Calcination can create edge and vacancy defects through the process of volatile elimination, as well as carbonization and the formation of the smaller connected graphitic domains [39-40]. The 2D band at ~2700 cm^{-1} provides more evidence for the structural structure and graphitic stacking when examined. Raman study confirms that calcination significantly changes the carbon structure, leaving the carbon more aromatized, thermally stable and energy storing, which is suitable for adsorption.

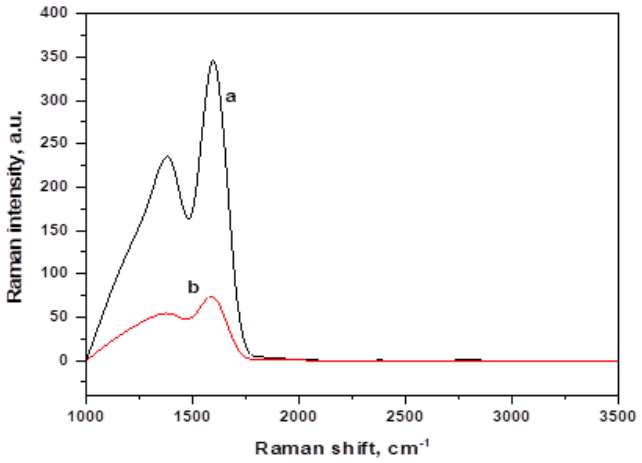


Figure 8 Raman spectrum of the Coconut Shell Charcoal powder samples: (a) Before calcined (b) After calcined.

3.6. VSM Analysis

The magnetism study of carbon compounds from the coconut shells was done before and after the calcination process by vibrated sample magnetometer (VSM) analysis as presented in Figure 9. By measuring the magnetization (M) as a function of the applied magnetic field (H), which is often depicted as a M–H hysteresis loop, the magnetic response was assessed. The determination of saturation magnetization (Ms), remanent magnetization (Mr) and coercive field (Hc) can provide useful information about the magnetic nature and magnetic ordering of the material and is a particularly beneficial application of VSM characterization [41-42]. As the majority of the structure of the coconut shell charcoal is composed of carbon phases and oxygen containing functional groups, it is expected that it will have very weak magnetic properties before it is calcined and the presence of naturally occurring inorganic minerals may be in trace amounts. After the calcination, considerable structural and compositional modifications occur. Thermal treatment can remove the sudden organic constituents of the material and bring about carbonization, oxidation, breakdown or concentration of inorganic constituents, depending on the treatment conditions and temperature. (Fe, Mn, Ni, Co)-oxide minerals may form crystalline oxide phases. This means that the magnetic response of the calcined sample can be different than the untreated sample. While a decrease in magnetization (Table 1) may happen when magnetic species are eliminated during thermal treatment or oxidized into weakly magnetic phases, a rise in magnetization may suggest the production or enrichment of magnetic/paramagnetic inorganic phases [43–44]. Important details regarding the magnetic properties of the calcined material can be found in the hysteresis loop acquired by VSM. The presence of a discernible hysteresis loop signalizes stronger magnetic interactions, and the formation of magnetic domains, whereas a small coercivity and a narrow loop may signal soft-magnetic or weakly magnetic behavior. Thus, the comparison of

the magnetic properties of Ms, Mr and Hc before and after calcination will help to understand the effect of heat treatment.

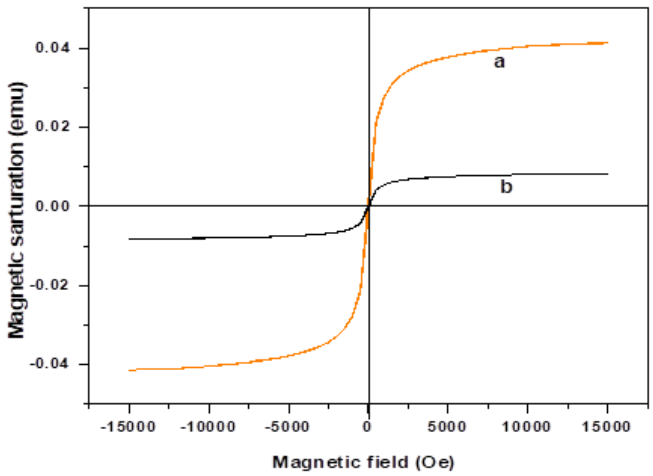


Figure 9 VSM graph of the Coconut Shell Charcoal powder samples: (a) Before calcined (b) After calcined.

Table 1 Summary of before and after calcined coconut.

Shell / 750◦ C	Ms (Saturation magnetization) (emu/g)	Mr (Remnant magnetization) (emu/g)	Squareness ratio(SQR)= (Mr/Ms)	Hc (Coercivity) (Oe)
Before Calcined	0.0411	0.0079	0.1975	82
After Calcined	0.0081	0.00150	0.185	101

5. CONCLUSION

This study reveals that coconut shell charcoal can be used as a source of biomass-derived carbon with added value to produce sustainable carbon materials. The characterization results were all combined, which showed that the properties of coconut-shell charcoal were significantly changed by the calcination process. The progressive loss of moisture and volatile, thermally unstable groups, and the stabilization of a carbon framework, was observed by TG/DTA analysis at high temperatures. The X-ray diffraction pattern showed broad reflections near 2θ = 28 and 45, characteristic of the amorphous/turbostratic carbon structure; and the crystallite size before and after calcination was 4 and 6 nm, respectively. FTIR identified hydroxyl, aliphatic C–H, carbonyl, aromatic C = C and C–O groups, with the lower intensity for oxygen-containing bands after calcination indicating that the hydroxyls, oxygen atoms and chemical structures of the polymer were removed and/or altered. SEM imaging showed that the surface was rough, irregular, fractured and heterogeneous, featuring interconnected pores, cavities, grooves and channels which could enhance surface accessibility and mass transfer. The BET analysis showed that calcined charcoal had micro and mesoporous properties, which could be used for adsorption, catalytic and electrochemical applications. The presence of D and G bands was confirmed by Raman spectroscopy, which also showed that the levels of carbon defects, aromatization and ordering of sp^2 carbon changed after calcination. The analysis also revealed that, based on the type of transformation, removal or concentration of inorganic mineral phases, the magnetic behavior of the material is modified by heat treatment using VSM. Overall, the calcination process successfully yielded a coconut shell-based carbonaceous material that is thermally stable, chemically reorganized, porous, partially ordered, and multifunctional that has potential applications for environmental remediation, energy storage, catalysis and adsorption.

Conflicts of interest

There are no conflicts to declare.

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