CONTROLLING NANOSTRUCTURE AND POROSITY: THE INFLUENCE OF HIGH-TEMPERATURE INERT-GAS PYROLYSIS AND TIME-DEPENDENT BALL MILLING ON COCONUT SHELL CHAR

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ABSTRACT

Pyrolyzed coconut shell char has potential applications in various fields, including energy storage and water purification. This study aims to investigate the effects of pyrolysis in inert atmosphere and size reduction of the char on the properties of coconut shell char. Coconut shell samples was pyrolyzed at 800 °C for 3 hours in a nitrogen atmosphere. The char obtained divided into three portions. Ball milling treatment at 250 rpm for 24 and 48 hours was applied to two separate portions of chars. The resulting chars were characterized for the morphological, textural and structural properties using scanning electron microscopy (SEM), Brunauer-Emmett-Teller (BET) nitrogen adsorption, X-ray diffractometry (XRD), Fourier Transform Infrared spectroscopy (FTIR) and Raman spectroscopy method. The morphology revealed the particles to be agglomerated, irregularly shaped with rough surfaces and varying pore sizes. While possessing increased surface area of 196.255 m^2/g for the untreated char to 361.244 m^2/g for the 48 hours ball milled char. XRD patterns showed the presence of broad peaks centred between 2θ angles of 24º - 26º which are characteristic of a turbostratic carbon structure, while the ball milling effect shown progression in char's crystalline order. The FTIR and Raman analyses indicated the existence of various functional groups (OH, C=O, C-O) and aromatic compounds (C=C). The graphitic character accompanied by the high surface area and mesoporous nature of the chars suggest that the pyrolyzed char may be a suitable precursor for graphene oxide synthesis. The results of this study provide valuable insights into the effects of thermal treatment and ball milling on coconut shell char properties.

Keywords: Ball milling, Coconut shell char, nanostructure, Porosity, Pyrolysis, Surface morphology, Turbostratic Carbon.

INTRODUCTION

The escalating worldwide need for sustainable and renewable carbon sources has fuelled a rapid increase in biomass utilization research [1]. Given their global abundance, biomass are increasingly recognized as a sustainable and renewable carbon resource which offers momentous potential across diverse sectors such as environmental purification, pharmaceutical manufacturing, catalysis substrates and source of carbon for advanced energy storage technologies [2]. Pyrolysis of biomass offers a sustainable way to produce carbon with well-defined features for industrial applications. The flexibility of feedstock sources for biochar production makes it an attractive option [3].

A significant challenge persists in effectively synthesizing high-performance biochar materials with systematically tuneable structural and functional characteristics. As a

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ubiquitous agricultural waste, coconut shell offers a sustainable source of biomass feedstock. The material's inherent characteristics—specifically its high percentage of volatile compounds, excellent carbon yield, and low inorganic ash content—make it highly suitable for various carbon-based applications [4]. While considerable research has focused on the generation of biochar from diverse biomass sources, a significant knowledge gap remains regarding the specific processing required for coconut shell char. Despite its inherent promise, there is a lack of comprehensive investigation into how both inert-atmosphere thermal treatment parameters and subsequent mechanical post-treatment methods (such as ball milling) specifically influence and enable the tailored properties of the resulting coconut shell char.

Therefore, this study aims to investigate the effects of carbonization atmosphere such as a nitrogen and ball milling on the properties of coconut shell char. The novelty of this research lies in its comprehensive analysis of the combined effects of thermal treatment and mechanical processing on the structural, morphological, and adsorption properties of coconut shell char. The findings of this study will contribute to the development of optimized production methods for high-quality coconut shell char, paving the way for its applications in various fields, including environmental remediation, energy storage, and catalysis.

MATERIALS AND METHODS

Sample Collection and Pretreatment

Coconut shells were procured from the Gwagwalada market and underwent to a series of pretreatment steps. Initially shells were washed to remove dirt's with water, sun dried for 48 hours. Then, pulverized to smaller particles size using a Trapp Animal ration Shredder hammer mill foliage TRF 80. The crushed particles were subsequently sieved using a Haver EML digital plus vibrational shaker with 75 µm to 2 mm.

Preparation of Coconut Shell Char

Approximaetly 200 g of the prepared coconut shell materials, with particle size in the range of <600 µm to >150 µm was loaded into the stainless-steel combustion tube (50 mm diameter by 7500 mm length). The sample was subjected to pyrolysis at 800 °C for 3 hours under a continuous stream of nitrogen gas (inert atmosphere). The resulting char was cooled, weighed and then two-third portion was divided into two parts. These portions were milled separately at 250 rpm for 24 and 48 hours respectively. The three final coconut shell char samples were systematically labelled as CC N_2 Atm (Unprocessed char), CC N_2 Atm BM24 (ball milled for 24 hours) and CC N_2 Atm BM48 (ball milled for 48 hours).

Characterization of Precursor and Char

The CC N2 Atm, CC N2 Atm BM24, and CC N2 Atm BM48 samples were analysed for surface morphology, textural surface area, crystalline properties, functional groups and molecular structure The equipments used

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included: Thermo Fisher Scientific Scanning Electron Microscopy, equipped with secondary electron and backscattered electron detectors, Quantachrome Nova 4200e gas adsorption/desorption, Rigaku Miniflex G6 X-ray diffraction, Agilent Cary 630 Fourier transform infrared (FTIR) spectroscopy with diamond attenuated total reflectance and ProRaman-L-785-B1S series 196166 spectroscopy.

RESULTS AND DISCUSSION

Surface Morphological Investigation of Coconut Shell Char

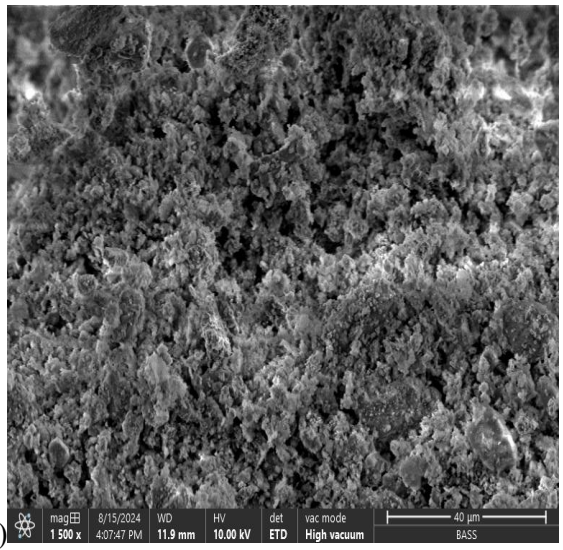
The surface morphology the coconut shell char obtained under nitrogen atmosphere (CC N_2 Atm) at magnification of 1500x (Figure 1a) shows an irregular shaped and highly heterogeneous multi-particle agglomerates; retained on the original raw coconut shell's skeletal backbone. The observed agglomeration and irregularly fractured particles suggest a low bulk packing efficiency and potentially low mechanical strength of individual fragments. This will influence handling, attrition and pressure-drop when used as packed beds. In addition, some micro-flaky particles are also visible over the highly rough surface as evident by the presence numerous fine asperities, micro-protrusions and cavities. The strong scanning electron contrast (bright tips and darker pits) are consistent with a rough and porous external topology. The surface of the biochar possesses multi-scale waviness overlaid with finer roughness (sub-µm to ~µm protrusions). These hierarchical macro-undulations plus micro-roughness is visible across the field of view. The sharp edges and broken fragments, evident in the field of view suggest mechanical fragmentation or brittle behaviour of the precursor initial preparation.

The morphological appearance shows inter-particle macropores and cavities at the µm scale as well as intra-particle micropores. Many pores are irregular and tortuous, interconnected in a random network; typical for chars produced by pyrolysis without deliberate activation. The visibly connected macropores and tortuous channels indicate reasonable fluid access at the particle-scale suitable for mass transfer to accessible surfaces which is essential for adsorption kinetics. The 24 hours ball milled coconut shell char sample (CC N_2 Atm BM24) in Figure 1b, shows increase in morphological fragmentation across the field of view, with an angular or plate-like sized fragments acting as evident of brittle fracture of the char particles due to the ball milling treatment. The char displays a high degree of surface roughness with pronounced fractured faces and acute edges, indicative of brittle failure. While being defined by irregular surface relief, characterized by micro-fractures that delineate stepped fragments and complex, jagged topography relative to the pre-milled image of CC N_2 Atm sample. The milling treatment also revealed particles adherence on the surface as seen by the re-deposition of smaller particles debris on semi and large particles surface. This increased opening of fissures, generate fresh

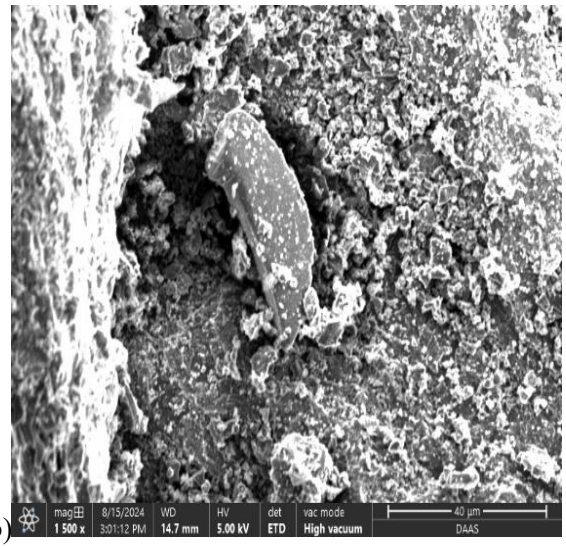
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surfaces likely to improve adsorption/desorption kinetics suitable for rapid uptake matters. The 48-hours ball-milled samples (CC N_2 Atm BM48) exhibit modified surface morphologies, with tiny micro-sized particles adhering to the surface of large fragmented particles (Figures 1c). The increased ball milling time of 48 hours resulted in enhanced rougher surface topology with additional micropore development, indicating improved adsorption potential. In addition, the surface exhibits an increased number of high-irregularity surface relief features, as characterised by extensive microfractures with stepped fragments and jagged topography. The expanded hours of the mechanical treatment caused particle adherence, as evidenced by the re-deposition of smaller particles, debris, semi- and large-sized particles onto the char surface. This suggests agglomeration and the integration of fine fragments generated during milling. Overall, the scanning electron microgram images suggest that the thermal treatment and ball milling processes significantly enhanced the surface area, pore structure and adsorption potential of the coconut shell biochar.

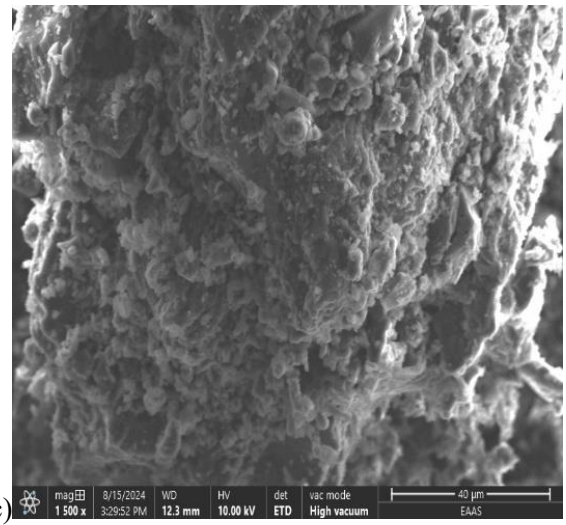
(a)



(b)



(c)



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Figure 1: Surface morphology image of coconut shell char

Surface Area, Pore Size and Pore Volume of Coconut Shell Chars

The nitrogen adsorption isotherms for the coconut shell char obtained in a nitrogen atmosphere (CC N_2 Atm) and ball milled coconut shell char are depicted in Figure 2. The BET plot for the coconut shell char sample (CC N_2 Atm) lead to a lower surface area value of 196.255 m^2/g, which is suspected to be due to partial blocking of the initial pores formed by the pyrolysis process as the residual tars or condensed carbon, limits accessibility of nitrogen gas during the adsorption isothermal investigation.

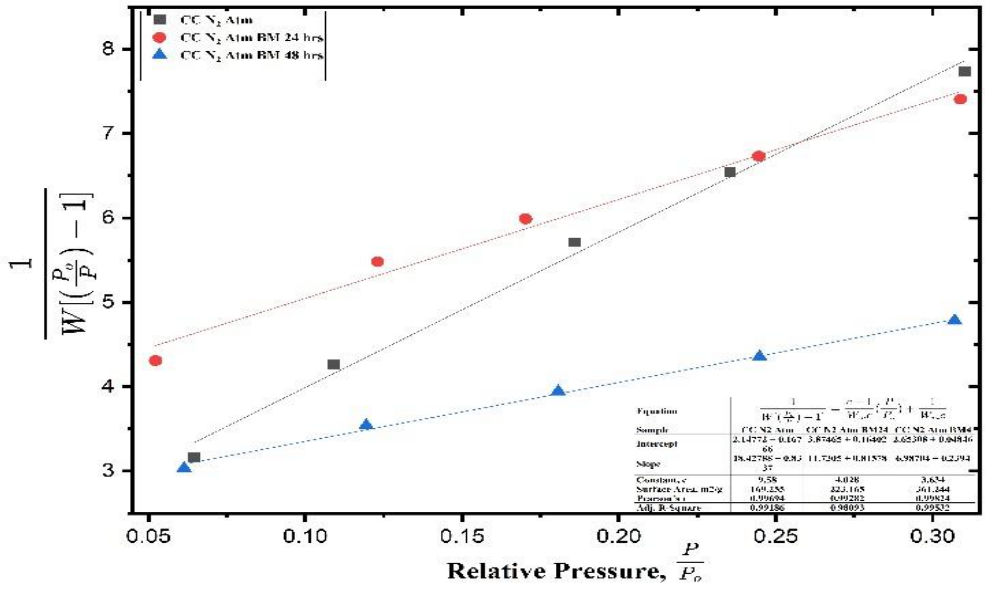


Figure 2: BET plot for Surface Area of coconut shell char

When compared to those of the ball milling treated chars with the surface area value of 223.165 m^2/g obtained for 24 hours (CC N_2 Atm BM24) and 361.244 m^2/g for 48 hours (CC N_2 Atm BM48) samples. Ball milling progressively increased the accessible surface area of coconut-shell char, as demonstrated by SEM images, which shows that unprocessed char consists of irregular, aggregated clumps with multi-scale surface waviness and limited exposed fissures. Whereas 24 hours of ball milling treatment produced a moderate increase from 196 to 223 m^2/g occasioned by the initial fragmentation as evident by the fines, angular fracture faces and opened channels in the SEM image Figure 1b. The substantial increase to 361 m^2/g after 48 hours, reflects extensive comminution, fissure formation and exposure of previously inaccessible microporous and mesoporous domains. The BM48 surface is dominated by fine particulates, ripped-open channels, and fresh fracture surfaces, all of which enhance nitrogen accessibility during adsorption, accounting for the markedly higher surface area. The observed BET increase can be attributed to morphological changes, including increased external surface due to comminution and improved connectivity between exterior

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networks. Typical chars produced at 800 ºC and above in an inert atmosphere, have been reported to exhibits an isotherm that is a combination of Type I and Type IV [5] which is characteristic of the co-existences of microporous and mesoporous materials [6]. The rapid initial rise in adsorbed volume at low relative pressures also suggest micropore filling, where the strong adsorption forces within narrow pores cause gas molecules to condense quickly [7]. The quantitative impact of different ball milling durations on the porosity of coconut shell char was examined using the Density Functional Theory (DFT) model; by focusing on the narrow range of 1.7 nm to 3.5 nm, as this is the range which represents the highest-energy adsorption sites and the most active pore structure in the char. Essentially, the non-local DFT (NLDFT) equilibration approach, simulates N_2 adsorption at 77 K within a carbon slit pore size, to evaluate the structural modifications. The pore size distribution by the DFT shown in Figure 3, reveals a clear structural pattern of the unprocessed char (CC N_2 Atm), 24-hour milled char (CC N_2 Atm BM24), and 48-hour milled char (CC N_2 Atm BM48).

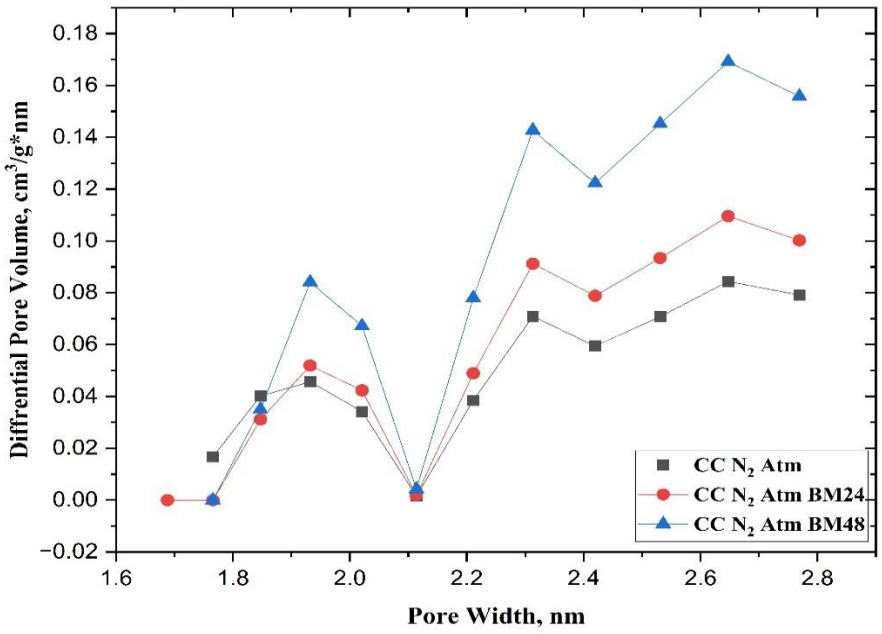


Figure 3: pore size distribution of the raw coconut shell and coconut shell char samples

All the chars maintained a stable pore size of 2.65 nm which is mesoporous according to IUPAC guidelines [8]. The DFT model result reveals that the initial effect of the 800 ºC pyrolysis treatment in the inert atmosphere on the porous char led to a baseline volume of 0.0662 cm^3/g and cumulative surface area of 64.68 m^2/g for the unprocessed char (CC N_2 Atm). For the 24-hour ball milled treated char, a marginal volume increase of 0.0687 cm^3/g and decrease in the cumulative surface area of 57.16 m^2/g was observed which suggest, that the ball milling treatment phase is dominated by particle fracture and pore unblocking, though not sufficient to break particle agglomerates and unblock sealed pores as evident from SEM

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observation of particle adherence and re-deposition by smaller debris. Thus, the high-energy collisions during milling likely cause some of the finest, highest-surface-area micropore entrances to be shielded or filled by re-deposited carbon fragments and debris, effectively blocking access to the internal surface area and resulting in a quantitative loss in the DFT surface area measurement. Whereas, the 48-hours ball mill treatment resulted in a massive increase in pore volume of 0.1064 cm^3/g representing a 55 % surge over the 24-hour treated char volume of 0.0687 cm^3/g. This substantial increase is the clearest evidence that the prolonged high-energy milling successfully fractured the carbon walls and dramatically expanded the accessible volume within the micro/small-mesopore range. In addition to the surface area recovery and enhancement noted from the increase in the total cumulative surface area from 57.16 m^2/g to 88.33 m^2/g. This recovery and subsequent enhancement demonstrate that the aggressive mechanical stress eventually overcame the initial surface shielding/re-deposition effects, leading to the generation of the fresh surfaces as observed in the SEM result of CC N_2 Atm BM48.

X-ray diffraction of coconut shell Char

X-ray diffraction patterns for the coconut shell char obtained after pyrolysis at 800 °C for 3 hours in a nitrogen atmosphere (CC N_2 Atm) and ball milling of coconut shell char (CC N_2 Atm BM24 and CC N_2 Atm BM48) is depicted as stacked plot with reference graphitic phase in Figure 4. Pyrolysis, especially at high temperatures of 600 ºC and above, is known to drives off volatile matter and promotes the initial formation of turbostratic carbon structures [9]. The chars exhibit broad, weak peaks around 2θ angles of 23º for the (002) plane and 43º for the (100) plane commonly associated with amorphous graphitic carbon [10,11]. The result, suggest that the graphitization of porous carbon from the coconut shell is quite weak as can be observed by the increasing peak broadening due to the reduction in crystallite size (nano-crystallinity) and the introduction of mechanical strain occasioned by the increase in ball milling time.

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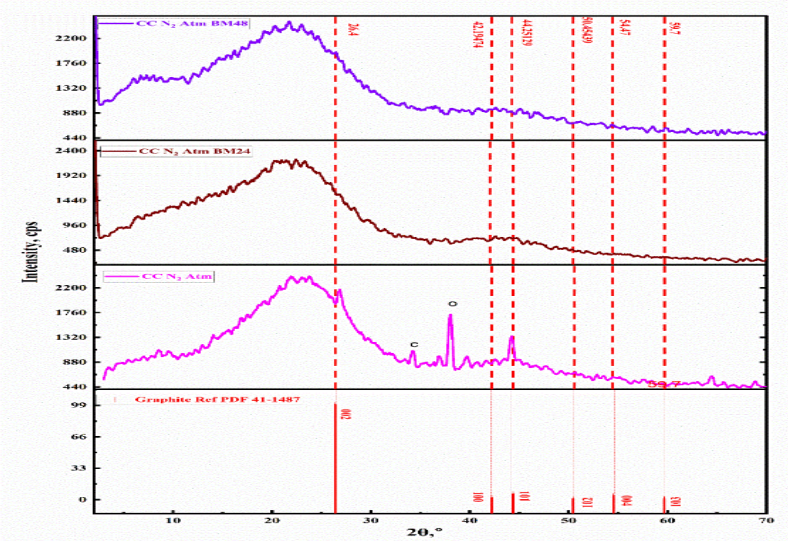


Figure 4: XRD of the coconut shell powder compared with graphite and stacked chars

The CC N_2 Atm char sample exhibits a relatively sharp peaks between the 2θ angle of 23º to 26º which suggest a 002 reflection as it corresponds to the 002 graphitic reference phase. While the 101-reflection occurred between 42º to 45º 2θ angle. The appearance of the 002 and 101 graphitic phases serves as baseline appearance of the turbostratic carbon structure. The peak being the sharpest among the three char samples, indicates highest degree of structural ordering achieved by the post pyrolysis treatment. In addition, the pattern shows two distinct, sharp, narrow peaks at 2θ angle of 36º and 38º. These are characteristic of residual mineral matter or ash that survived the 800 ºC pyrolysis treatment in inert atmosphere. These sharp peaks confirm the presence of crystalline inorganic phases alongside the amorphous carbon structure. The influence of the 24-hours ball milling treatment on the coconut shell char (CC N_2 Atm BM24) was observed as the peaks show clear broadening and reduced intensity, resulting to an initial crystallite breakdown and introduction of structural defects. For the 48-hours ball milled sample (CC N_2 Atm BM48), the peaks appeared as the broadest, weakest and slightly shifted towards lower 2θ. It exhibits the maximum structural disorder. The structural information relating to crystallite stack height (Lc) and diameter (La) calculated from the Scherrer equation is shown in Table 1.

Table 1: Structural information of samples determined from XRD patterns

Sample	$2\theta_{002}$ (º)	β_{002} (°)	d_{002}(nm)	Lc(nm)	$2\theta_{100}$ (º)	β_{100} (º)	d_{100}(nm)	La(nm)
CC N_2 Atm	23.48	6.92	0.3789	1.1735	44.22	6.02	0.2048	2.9144
CC N_2 Atm BM24	23.18	7.48	0.3837	1.085	42.50	3.32	0.2127	5.2530
CC N_2 Atm BM48	21.76	7.04	0.4084	1.15	46.76	3.82	0.1943	4.6357

The maximum separation d_{002} value was observed to increased monotonically from 0.3789 nm for the unprocessed char to a maximum of 0.4084 nm at 48 hours for the CC

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N_2 Atm BM48 char sample. This increase is demonstrated by the significant 2θ angle shift from 23.48º to lower angles of 21.76º. This extreme expansion of the d_{002} spacing proves that the prolonged, high-energy ball milling effectively destroyed the weak stacking order of the turbostratic carbon layers. The layers are forced farther apart, creating maximum structural disorder. This disorder is the physical mechanism that generates the new porosity; the separation of these carbon sheets directly forms the slit pores that the DFT model identified and quantified. The calculated crystallite sizes confirm the material is fundamentally amorphous, since all the stacking height values were extremely small, clustering around 1.1 nm, indicating that the carbon consists of only a few stacked layers across all treatment times. Additionally, the Full Width at Half Maximum (FWHM) for the (002) reflection is large (6.9º to 7.5º), confirming the highly amorphous nature of the char inherited from the 800 ºC pyrolysis. The lateral size (La) values show inconsistency; likely due to the low intensity and high noise of the (100 and 101) peaks in amorphous carbon produced by the ball milling treatment. The large lateral size calculated for the milled samples is an artifact of peak fitting but does not contradict the overall picture of fragmentation and disorder.

Raman Spectroscopy of Raw coconut shell powder and char

The Raman spectrum of the raw coconut shell powder revealed was previously described [12]. The Raman spectrum of the coconut shell char carbonized at 800 °C for 3 hours in a nitrogen atmosphere (Figure 5) exhibited prominent peaks at 696, 1406, 1424, 1432 and 2202 cm^{-1}, assigned to aliphatic C-H vibrations, aromatic C-C vibrations and C≡N stretching vibration, respectively. However, the high intensity of the peaks at 1424 cm^{-1}, also have obscured the small peak at 1396 cm^{-1} which is suspected to be the D-band usually found at 1350 cm^{-1} position, while the expected peak at 1580 cm^{-1}, typically associated with the G band, was clearly overshadowed with the noisy background signal. This makes it difficult to associate any maximum peak intensity to that of the expect G-band. The Raman spectrum of the char ball-milled for 24 hours showed two prominent peaks associated with aromatic C-H vibrations (588 and 3008 cm^{-1}). The disappearance of the peaks observed in the CC N_2 Atm sample, maybe associated to the ball milling effect, which have introduced defects in the carbon structure, altering the functional group composition, as such the shift from initially observed position to a newer position.

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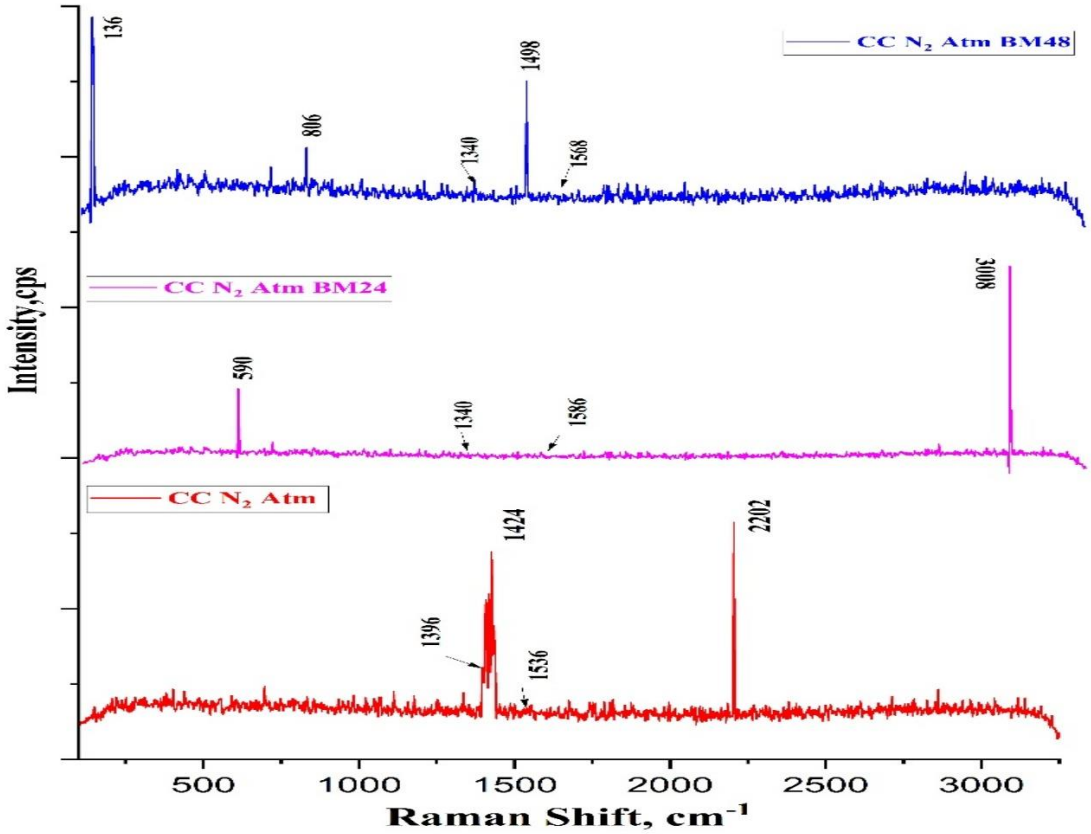


Figure 5: Stacked Raman spectra of raw coconut shell, Char and ball milled chars

The char ball-milled for 48 hours, exhibits two prominent peaks at different position which differs from those observed in the CC N_2 Atm and CC N_2 Atm BM24 samples. The peak observed at 136 cm^{-1} was assigned to aliphatic C-H vibration, while those observed at 806 and 1498 cm^{-1} were equally assigned to aromatic C-H vibrations. This suggests that both aliphatic and aromatic skeleton fragments presence are affected by the ball milling treatments, which may have introduced defects in the carbon structure, altering the functional group composition positions. Particularly, the disappearance of the initially observed peak at 1396 cm^{-1} in the CC N_2 Atm sample, from both the CC N_2 Atm BM24 and CC N_2 Atm BM48 samples. The 1396 cm^{-1} positioned peak is considered the closest peak resemblance of the D-band and its disappearance with ball mill treatment reinforce the previously observed anomaly from the XRD pattern where the graphitic phase of 002 disappeared completely in the same samples.

FTIR of Raw Coconut Shell and Char

The functional group investigation the CC N_2 Atm, CC N_2 Atm BM24, and CC N_2 Atm BM48 samples are depicted in Figure 6. The three distinct peaks between 3854-3540 cm^{-1} are ascribed to O-H stretching vibrations of residual phenolic, carboxylic, and alkanol groups [12,13]. The 2970-2870 cm^{-1} region commonly associated to C-H stretching vibrations of aliphatic chains or aromatic fragments was observed to exhibit reduced intensity in the charred samples suggesting loss of aliphatic components due to the pyrolysis

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condition leading to the decomposition and possible reordering of carbon to form aromatic rings. Notably, the peak at 2870 cm^{-1} matches the symmetrical stretching vibration of the C-H bond in aromatic rings, as reported by Venkatraman *et al.* [14]. The FTIR spectra revealed additional functional groups present in the charred coconut shell samples. The peak at 1722.02 cm^{-1} was ascribed to the carbonyl group (C=O) of carboxylic functional groups, consistent with the expected range of 1725-1700 cm^{-1} [15]. The peak at 1595 cm^{-1} was assigned to the C=C-C stretching vibration of aromatic rings [16], falling within the expected range of 1615-1580 cm^{-1} according to Nandiyanto *et al.* [15]. In addition, all the samples (CC N_2 Atm, CC N_2 Atm BM24 and CC N_2 Atm BM48 exhibited distinct peaks at 1632 cm^{-1} position which is attributed to the C=O group from esters or carboxyl families. These notable peaks at 1632 cm^{-1} were associated with both C=C and C=O functional groups. Rampe *et al*. [17] proposed that the peaks between 1681.93-1427.32 cm^{-1} indicate C=C functional group vibrations of aromatic compounds, while Sujiono *et al.* [18] ascribed this position to the carbonyl groups C=O stretching vibration of oxidized graphitic domains in chars.

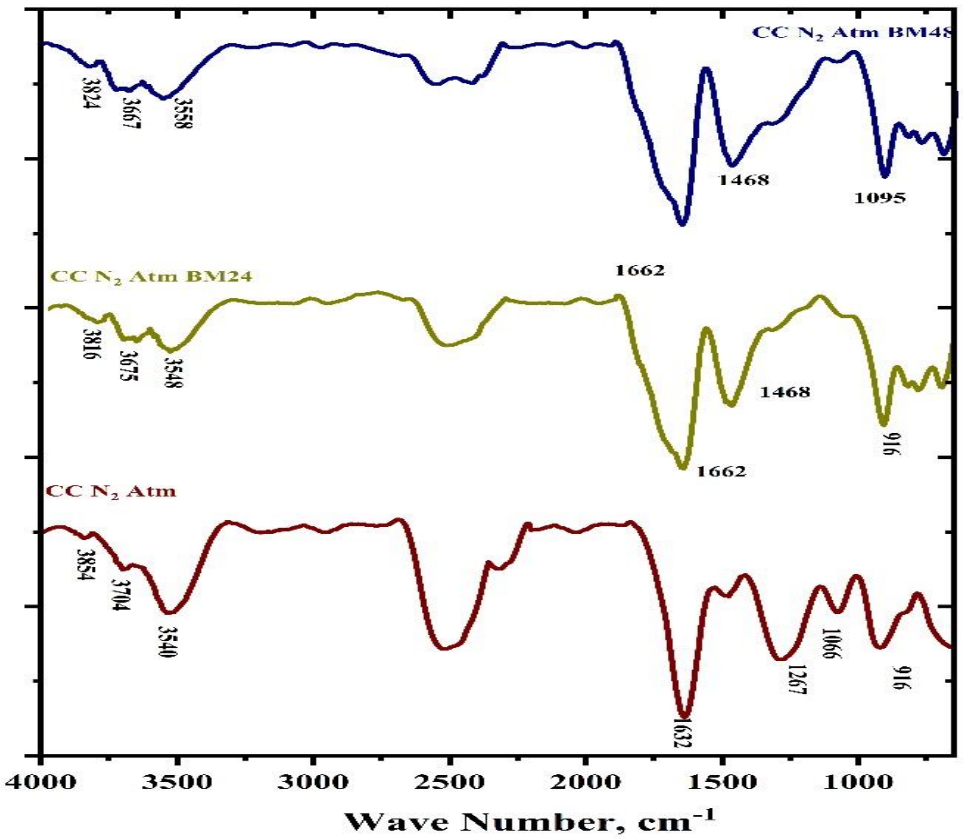


Figure 1: FTIR spectra of coconut shell, char and ball milled char

The FTIR spectra showed noteworthy changes in the functional groups of the coconut shell samples upon pyrolysis and ball milling. The region between 1468-1095 cm^{-1}, associated with C-O stretching and C-H bending vibrations, showed a variety of peaks in the raw

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coconut shell sample [12], indicating the presence of oxygen-containing functional groups. In contrast, the charred samples exhibited changes in this region, signifying the loss of oxygen-containing functionalities and the development of the carbon structure. The peaks at 1468 and 1095 cm^{-1} in the ball-milled samples may indicate the formation of new structural features or changes in the existing ones. Specifically, the medium peak at 1468 cm^{-1} was assigned to the C-H bending vibration of methyl groups or benzene ring vibrations, as reported by Jnawali *et al.* [19]. The presence of peaks at 1237 and 1028 cm^{-1} indicated the presence of C-O functional groups, specifically esters, supported by Adeniyi *et al.* [20]. The region between 916-767 cm^{-1}, characteristic of C-H out-of-plane bending, suggested the presence of aromatic rings in the raw coconut shell sample [12]. Charring and ball milling introduced significant changes in this region, indicating alterations in the aromatic structure. FTIR spectra demonstrate that ball milling causes a significant chemical degradation and stripping of oxygen-containing functional groups through reduced intensity of peaks observed for O-H and C-O. This chemical cleaning action, coupled with the profound structural destruction confirmed by XRD disappearance of 002 phase and Raman disappearance of 1396 cm^{-1} D-band, results in the optimal treatment at 48-hours milled char. This final product possesses a clean, highly fractured surface with maximized porosity, making it superior for adsorption applications.

Conclusion: The study demonstrated that the prolonged ball milling time of 48 hours was critical for the physical activation in producing high-performance char. It does not create new pore types, but rather optimizes the existing microporous network by maximizing the separation of graphitic layers (d_{002}), introducing a maximum density of structural defects, fragmenting the particle morphology and increased surface area. These changes collectively lead to the char having the largest accessible internal surface area, optimizing the microporous network by expanding the slit-pore entrances and pore volume, establishing the 48-hour ball-milled char (CC N_2 Atm BM48) as the optimal product for applications requiring high adsorption capacity such as catalysis and water purification due to their mesoporous characteristics and strong adsorption potentials.

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