ROLE OF PYROLYSIS TEMPERATURE IN COCONUT FIBER BIOMASS CONVERSION

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This study examined the influence of pyrolysis temperature on the properties of biochar produced from coconut fiber, a waste product from mattress manufacturing. Coconut fiber was pyrolyzed in a muffle furnace at 300–700 °C to evaluate its suitability for biochar production and to analyze how temperature affects yield and physicochemical characteristics. The yield decreased from 67.1% at 300 °C to 15.8% at 700 °C, showing a strong negative correlation with temperature. In contrast, fixed carbon, ash content, and pH increased, while volatile matter and the volatile matter/fixed carbon ratio declined. Electrical conductivity exhibited variable behaviour. SEM and FTIR analyses revealed that higher temperatures produced more porous structures and enhanced aromatic carbon groups due to transformations of chemical functional groups. The study confirms that pyrolysis temperature is a key factor in optimizing coconut fiber biochar properties for environmental and energy applications aligned with green analytical chemistry principles.

KEYWORDS

coconut fiber, biomass, pyrolysis temperature, biochar, sustainable materials

INTRODUCTION

Biomass accounts for approximately 12.83% of the world's renewable energy stock, with its utilization projected to continue for decades. Significant quantities of biomass are generated from agricultural cultivation, harvesting, processing, and consumption [Adewole 2020]. Traditionally, these residues are treated as waste, often ending up in landfills. However, plant residues from various sources, including banana, plantain, fruit peels, and especially coconut, are suitable feedstocks for thermochemical processes [Olatunji 2023].

Coconut waste is an abundant biomass resource, with a global production of 62.5 million tons per year across more than 90 countries [Siengchum 2012, Azeta 2021]. With increasing cultivation and production, the volume of coconut waste has dramatically risen, as only about 40% of the plant is utilized. Despite its year-round availability, especially from shells and husks, a significant portion of coconut waste is often discarded improperly. Coconut waste biomass is an attractive option due to its low cost, low carbon emissions, and rigid polymer structures, including cellulose, lignin, and hemicellulose [Abrahim 2019, Lomeli-Ramirez 2018]. Despite its potential, the vast amount of waste from coconut cultivation and processing poses significant environmental challenges. In 2019, an estimated 62 million tons of coconuts produced worldwide generated about 20 million tons of husks, yet only a fraction is utilized [Stelte 2022]. The slow biodegradation of these highly recalcitrant husks leads to unpleasant odours, methane emissions, storage issues, and watercourse clogging [Dumasari 2020, Nunes 2020]. Thus, sustainable management of coconut residues is urgently needed.

Pyrolysis, a thermochemical process occurring in the absence of oxygen, produces gases, solid char, and liquid hydrocarbons [Zhou 2016]. Compared with coal pyrolysis, biomass pyrolysis offers a more sustainable pathway with fewer harmful volatiles [Wang 2019, Adewole 2020]. The pyrolysis process, as a thermochemical conversion, requires precise control of temperature and atmosphere, directly dependent on the quality and design of the experimental apparatus [Zhu 2023]. In the broader context of high-temperature engineering research, optimizing technical design and understanding heat transfer in complex environments are crucial for process efficiency [Pivarciova 2019]. Coconut biomass, containing roughly 45% cellulose, 35% hemicellulose, and 15% lignin, decomposes at varying rates during pyrolysis, yielding valuable products with industrial and economic potential [Azeta 2021]. Converting coconut residues into biochar also brings environmental benefits, including greenhouse gas reduction, soil enhancement, and promotion of a circular economy [Ighalo 2023]. The efficiency of an adsorbent is primarily determined by its physicochemical properties, such as polarity, surface area, pore size distribution, and total pore volume. Highly porous materials can reach specific surface areas exceeding 1500 $m^2.g^{-1}$, significantly improving adsorption performance. Micropores and submicropores dominate the adsorption of water and trace compounds, while larger pores mainly facilitate transport [Ciahotny 2016].

Carbonaceous adsorbents have long been used for sampling and preconcentration of volatile and semivolatile organic contaminants [Matisova 1995]. Despite decades of research, the development of new sorbents with tailored properties remains relevant, driven by the demand for greener and cost-effective materials [Merlo 2023; Stratulat 2023]. Adsorption is considered one of the most efficient techniques for removing persistent organic pollutants from water. Although activated carbon remains the benchmark due to its high surface area and porosity, its cost and regeneration requirements limit large-scale use [Kobeticova 2017]. Hence, attention increasingly turns toward sustainable, low-cost sorbents derived from waste biomass [Michalek 2021], among which coconut-derived biochar stands out as a promising, eco-friendly alternative that supports waste valorization and circular economy principles.

Coconut residues biochar generally contains a substantial amount of carbon and relatively low levels of ash, which makes it an ideal material for diverse applications such as soil amendment, carbon sequestration, and importantly, water treatment. Previous studies also indicate that a higher lignin content contributes to a greater biochar yield, and a rich organic carbon content promotes the formation of aromatic structures in the biochar, which in turn enhances its adsorption properties [Wang 2022]. This study will therefore focus on optimizing biochar quality through the systematic investigation of pyrolysis temperature in the conversion of coconut fiber biomass, aiming to leverage its unique chemical composition for enhanced biochar characteristics specifically for sorption applications.

1 MATERIALS AND METHODS

The sample (biomass) of coconuts fibers (Fig. 1a) were obtained from a local company specializing in the production of

mattresses in Bratislava, Slovakia (SEGUM, Bratislava, Slovakia). Their products contain a layer of coconut board, 2 to 3 cm thick, which is produced by pressing coconut fibers and had been bonded with latex. The waste material produced during the cutting of these coconut boards was used for the preparation of the biochar.

1.1 Preparation of Biochar and Characterization

The collected coconut fibers were cut and shredded in a knife mill to a size of approximately 2 mm. After cutting, the fibers were dried in a dryer at a temperature of 100 °C for 48 hours, to remove excess moisture.

The slow pyrolysis reaction of the coconut fibers was conducted in a modified muffle furnace under controlled conditions. For each experiment, 5 grams sample of oven-dried coconut fibers was loaded into the furnace. The furnace was then heated at a constant rate of 10 °C per minute until it reached the desired pyrolysis temperature. The residence time for the sample in the furnace was set at 120 minutes for every experiment. In the slow pyrolysis process, an oxygen-deficient atmosphere was maintained. This was achieved by continuously flowing nitrogen gas at a rate of 20 mL per minute, ensuring inert conditions for the pyrolysis reaction. After the pyrolysis process was complete, the furnace was cooled to room temperature, and the biochar samples were collected and stored in desiccators. The slow pyrolysis process was repeated at five different temperatures: 300 °C, 400 °C, 500 °C, 600 °C, and 700 °C. For each of these temperatures, the entire procedure was replicated three times to ensure the reliability of the results. The obtained biochar yield data was then averaged to get a representative value for yield experiments performed at each temperature. For illustration, Fig. 1b shows the biochar sample.

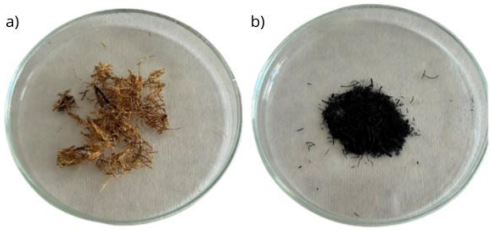

Figure 1. Samples of coconut fibers before (a) and after pyrolysis process (b)

The percentage of biochar yield was determined based on experiments conducted on a dry basis. The biochar yield was calculated using the following equation:

$$biochar\ yield\ (\%\ feedstock) = \frac{mass\ of\ biochar\ (g)}{mass\ of\ feedstock\ (g)} \times 100 \quad (1)$$

Proximate analysis was conducted to determine key characteristics of both the initial biomass and the resulting pyrolyzed biochars: specifically, their volatile matter (VM), ash content, and fixed carbon (FC). All these values were calculated on a dry basis. The ash content in the biomass samples was determined following the standard STN EN ISO 18122: Solid biofuels – Determination of ash content (ISO 18122:2015). The percentage of ash was calculated as:

$$Ash\ content\ (\%) = \frac{weight\ of\ residue\ (g)}{weight\ of\ dried\ sample\ (g)} \times 100 \quad (2)$$

VM determination was performed according to the ISO 562:2010 standard on Hard coal and coke − determination of the volatile matter. The percentage of VM was calculated as:

$$VM\ (\%) = \frac{weight\ of\ dried\ sample\ (g) - weight\ of\ sample\ after\ heating\ (g)}{weight\ of\ dried\ sample\ (g)} \times 100 \quad (3)$$

Fixed carbon was a calculated value. FC was calculated on a dry-basis and determined as follows:

$$FC(\%) = 100 - Ash(\%) - VM\ (\%) \quad (4)$$

Since the samples were dried for 48 hours before analysis to eliminate excess moisture and all components were subsequently determined on a dry basis, moisture content was excluded from the calculations.

Thermogravimetric analysis (TGA) was performed using a STA 449 F5 Jupiter (Netszch, Germany). The measurements were done under inert atmosphere by a high purity nitrogen with a gas flow of 100 mL.min^{-1}. The heating of the samples proceeded uniformly at a rate of 10 °C.min^{-1}. The temperature range of the measurement was set from 100 °C to 900 °C, and the sample weight was 10±0.5 mg.

Scanning electron microscopy (SEM) (JSM-7600F, FEG-SEM, JEOL, Japan) was used to analyze the surface morphology of the raw material and pyrolyzed samples. Images were primarily taken at magnifications of 500× and 2000×. An additional image at 140× magnification was obtained to provide a broader structural overview (as illustrated in Fig. 1a).

A suspension of 1:20 (biochar to deionized water, w:v) was prepared for determining the **pH** and **EC (Electrical Conductivity**). The suspension was agitated for 1.5 hour for equilibration before pH and EC analysis. The pH and EC were measured using the same suspension rather than making different replicates of the suspension [Rajkovich 2011]. A pH meter pH510 CyberScan (Eutech Instruments, Singapore) and an EC meter MD 800 L (VWR International, USA) with Multi Meter IDP 761-C (VWR International, USA) were used for the pH and EC measurement respectively.

Fourier Transform Infrared Spectroscopy (FTIR), Varian FT-IR Spectrometer 660 (Agilent Technologies, Inc., USA) was used for spectroscopic analysis of the raw materials and biochar, which were directly applied to diamond crystal of ATR GladATR (Pike TechnoLogy Inc., USA). The spectrometer utilized an attenuated total reflectance (ATR) accessory in the wavenumber range of 3400–400 cm^{-1}, each spectrum was measured 256 times.

2 RESULTS AND DISCUSSION

2.1 Effect of pyrolysis temperature on biochar yield

The yields of biochars produced from coconut fiber are presented in Fig. 2 as a function of pyrolysis temperatures ranging from 300 °C to 700 °C (with increment of 100 °C). It was observed that the biochar yields gradually decreased with increasing pyrolysis temperatures. This observed gradual decline in biochar yield with increasing pyrolysis temperatures is a well-established characteristic of biomass pyrolysis, directly attributable to the progressive decomposition of lignocellulosic components and the extensive devolatilization of organic compounds at elevated temperatures [Mohammed 2015]. This trend is further corroborated by studies on similar feedstocks; for instance, Noor et al. [Noor 2019] investigated how varying the pyrolysis temperature impacted the characteristics of coconut flesh-produced biochar. The results showed that raising the pyrolysis temperatures increased the fixed carbon and BET (Brunauer-Emmett-Teller) surface area of biochar while decreasing the yield percentage.

The highest biochar yield was recorded performing the pyrolysis at the temperature of 300 °C and was reaching 67.1±1.4%. This yield sharply declined to 31.0±2.9% at pyrolysis temperature of 400 °C. Applying the temperature 500 °C and higher, the yield showed only a minor decrement up to experiments with pyrolysis temperature up to 700 °C, indicating a slower rate of weight loss at higher pyrolysis temperatures. At

pyrolysis temperature of 700 °C, the biochar yield was the lowest, reaching value of 15.8±1.1%. These findings are consistent with the general understanding of slow pyrolysis, which typically yields 30–40 wt% biochar within a pyrolysis temperature range of 300–800 °C [Dhyani 2019, Liu 2015]. Furthermore, slow pyrolysis is widely recognized as the preferred method for maximizing the yield of solid char (biochar), as opposed to liquid bio-oil or gaseous syngas [Daful 2020]. Additionally, the biochar characteristics, such as increased porosity and increased surface area, can influence the yield, as more extensive devolatilization and gasification during pyrolysis may lead to a lower solid product yield. However, coconut residues, as a lignocellulosic biomass rich in lignin, are known to consistently produce substantial biochar yields, typically ranging from 23.8% up to 92% [Ighalo 2023].

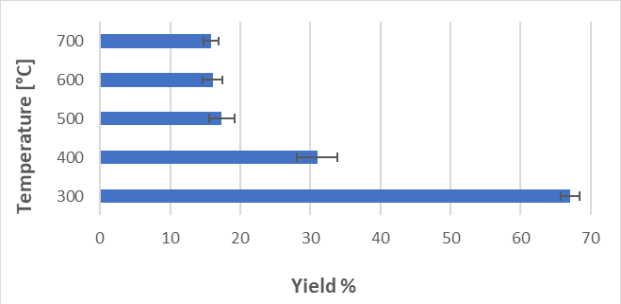

Figure 2. The percentage of biochar yield at five different pyrolysis temperatures

2.2 Effect of pyrolysis temperature on proximate analysis

The proximate analysis of coconut fiber and biochars, which quantifies volatile matter, fixed carbon content, and relative ash content, revealed substantial differences across varying pyrolysis temperatures (Tab. 1). The results indicate a negative correlation between volatile matter and pyrolysis temperature, while fixed carbon and relative ash content exhibited a positive correlation with increasing temperature. This analysis was performed on a dry basis to ensure accuracy and avoid moisture-related bias.

Specifically, the volatile matter (VM) content drastically decreased from 80.3±0.6% in the coconut fiber feedstock to 58.7±0.4% at pyrolysis temperature of 300 °C, further reducing to 14.6±0.5% at temperature of 700 °C. VM typically decreases as the pyrolysis temperature rises because the volatile particles will be expelled at higher temperatures [Noor 2019]. This reduction is primarily attributed to the devolatilization of feedstocks, the breakage of lower molecular weight hydrocarbons, and increased aromatization occurring at higher pyrolysis temperatures [Domingues 2017]. Correspondingly, the fixed carbon (FC) content showed a substantial increase from 14.5±0.7% in the coconut fiber (CF) to 34.5±0.6% at pyrolysis temperature of 300 °C, reaching yield 75.4±0.7% at temperature of 700 °C. This observed increment in FC aligns with established research, which demonstrates that the transformation of coconut shell and husk biomass into biochar through pyrolysis or gasification inherently leads to an increase in FC and elemental carbon [Ajien 2022]. Biochars produced at lower pyrolysis temperatures retained a higher amount of volatile matter, likely due to the initial high lignin content of coconut fibers, which is subsequently lost as devolatilization intensifies at higher temperatures.

Concurrently, the ash content significantly increased with rising pyrolysis temperature, from 5.6±0.2% in the coconut fiber to 10.3±0.4% at temperature of 700 °C. This finding is consistent with literature reporting a considerable amount of ash in coconut shell and husk biochar produced via pyrolysis and gasification [Ajien 2022]. The increase in ash content is often attributed to the concentration of inorganic materials as organic matter volatilizes. During the conversion of coconut biomass to biochar, alkali-earth elements such as potassium (K) and sodium (Na) likely nucleate, condense, and coagulate, contributing to the formation of ash on the biochar surface [Jia 2012]. The resulting carbon-rich material, along with its intrinsic surface functional groups (such as carboxyl, ester, hydroxyl, and aromatic groups) and mineral content, are crucial features that enhance the adsorption capacity of the biochar [Ajien 2022].

The Volatile Matter to Fixed Carbon (VM/FC) ratio, as presented in Tab. 1, serves as a crucial indicator of biochar stability and its resistance to further thermal and biological decomposition. Our findings reveal a sharp decrease in the VM/FC ratio of biochars from 1.70 obtained at pyrolysis temperature of 300 °C to the value of 0.52 at pyrolysis temperature of 400 °C. This trend aligns with general principles of biomass pyrolysis, where increasing temperatures lead to greater devolatilization and carbon enrichment, thereby reducing the volatile matter relative to fixed carbon [Zhang 2022]. The VM/FC ratios became stable when the pyrolysis temperature was set at 600 °C and 700 °C, respectively, obtaining values of VM/FC approximately 0.22 and 0.19, respectively. This stabilization at higher temperatures signifies a high degree of carbon stability, indicating that the biochar products become increasingly resistant to further thermal degradation and biological breakdown [Crombie 2012]. Our observations are strongly supported by Nartey and Zhao (2014), who reported that biochar produced via slow pyrolysis achieves significant thermal and biological stability when its VM/FC ratio falls within the range of 0.21 to 0.28, preventing further thermal decomposition.

Furthermore, analysis by Leng (2018) and other studies confirm that higher pyrolysis temperatures generally lead to lower H/C and O/C ratios, which are also indicators of increased aromaticity and carbon stability [Wang 2021]. The reduced VM/FC ratio at elevated temperatures reflects the formation of more condensed and recalcitrant carbon structures, characteristic of highly stable biochar [Crombie 2012, Wang 2021]. This enhanced stability makes biochar produced at higher temperatures particularly suitable for applications requiring long-term carbon sequestration and resistance to environmental degradation.

Table 1. Proximate analysis, pH, EC, and yield of coconut fiber (CF) biochars (BC) derived at different pyrolysis temperatures

	Pyrolysis temperature [°C]					
	RAW	**300**	**400**	**500**	**600**	**700**
Name of samples	CF	CF-BC300	CF-BC300	CF-BC300	CF-BC300	CF-BC300
Yield (dry basis) [wt%]	-	67.1 ±1.4	31.0 ±2.9	17.4 ±1.8	16.1 ±1.4	15.8 ±1.1
Proximate analysis [wt%]						
VM	80.3 ±0.6	58.7 ±0.4	31.2 ±0.1	18.4 ±0.2	15.9 ±0.8	14.6 ±0.5
FC	14.5 ±0.7	34.5 ±0.6	59.8 ±0.1	71.7 ±0.2	73.7 ±0.9	75.4 ±0.7
Ash content	5.6 ±0.2	6.6 ±0.5	9.0 ±0.1	9.8 ±0.1	10.7 ±0.1	10.3 ±0.4
VM/FC	5.54	1.70	0.52	0.26	0.22	0.19
Other properties						
pH [-]	7.15	6.55	6.12	6.02	6.41	9.29
EC [dSm^{-1}]	2.58	0.15	1.41	4.52	4.18	3.50

2.3 Effect of pyrolysis temperature on pH and EC

The pH and EC (Electrical conductivity) are crucial characteristics of biochar derived from coconut fibers, influencing its potential applications. These properties depend on the specific production method, the characteristics of the source material, and the process variables [Hossain 2020].

The pH and EC values for the raw coconut fiber and the produced biochars across different pyrolysis temperatures are summarized in Tab. 1.

The raw coconut fiber, obtained as a waste product from mattress manufacturing and containing residual latex adhesive, exhibited a near-neutral pH of 7.15 and an EC of 2.58 dSm^{-1}. The presence of this latex might have influenced these initial values, and potentially affected the subsequent pyrolytic transformations and the final pH and EC characteristics of the biochars. Upon pyrolysis, the pH values of the biochars showed initial variations. Biochar produced at pyrolysis temperature of 300 °C had a pH of 6.55, which slightly decreased to 6.12 at pyrolysis temperature 400 °C, and further to 6.02 at temperature 500 °C, indicating slight acidification at lower pyrolysis temperatures. However, a notable shift towards alkalinity was observed at higher temperatures: CF-BC600 showed a pH of 6.41, and CF-BC700 demonstrated a significant increase to a highly alkaline pH of 9.29. While biochar typically exhibits a slightly alkaline pH between 7 and 9 [Fidel 2016], this general trend of initial slight acidification followed by an increase in pH to alkaline values at higher pyrolysis temperatures is commonly observed in biochar production from lignocellulosic biomass. Higher pyrolysis temperatures also play a vital role in pH control, often resulting in increased alkalinity due to the oxidation of acidic functional groups and the concentration of alkali and alkaline earth metals in the biochar matrix [Khan 2024]. Although, studies on the pyrolysis of latex sediment alone obtained at pyrolysis temperature of 500 °C report the resulting liquid product to be highly alkaline with a pH of 9.45 [Wongdaeng 2024], the biochar reported in this work (the solid product) from the latex-bonded coconut fiber only exhibited a significant alkaline pH 9.29 at pyrolysis temperature 700 °C, remaining around pH 6 for material obtained at pyrolysis temperature 500 °C. This difference highlights that the final pH of the biochar is a complex interplay between the decomposition of both the lignocellulosic coconut fiber and the latex, where the solid residue's alkalinity might only become dominant at higher temperatures after the complete devolatilization of acidic compounds from both components.

The EC values for the raw coconut fiber and biochars, as summarized in Tab. 1, show distinct trends with increasing pyrolysis temperature. Upon pyrolysis, CF-BC300 exhibited a significantly lower EC of 0.15 dSm^{-1}. Subsequently, the EC notably increased with rising pyrolysis temperature: CF-BC400 had an EC of 1.41 dSm^{-1}, CF-BC500 reached a peak of 4.52 dSm^{-1}, and CF-BC600 showed a slightly reduced value of 4.18 dSm^{-1}. At the highest temperature, CF-BC700 had an EC of 3.50 dSm^{-1}. The general increase in EC with pyrolysis temperature is a widely reported phenomenon. This is primarily attributed to the concentration of inorganic elements (such as K, Na, Ca, Mg) in the biochar's ash content as volatile organic matter is released during carbonization. As the mineral content of the feedstock is largely retained and becomes concentrated in the resulting biochar due to the gradual loss of carbon, hydrogen, and oxygen during processing, the EC increases significantly [Rehrah 2014]. The slight decrease in EC observed from 600 °C to 700 °C might be due to the potential volatilization of some alkali salts at very high temperatures or the formation of less soluble mineral forms [Khan 2024].

2.4 Effect of pyrolysis temperature on functional groups of coconut fiber and biochar (FTIR analysis)

FTIR analysis presented in Fig. 3 was employed to identify the functional groups present in the CF (not treated by pyrolysis) and the biochars produced at varying pyrolysis temperatures. The changes in these functional groups provide crucial insights into the chemical transformations occurring during the pyrolysis process, reflecting the progressive decomposition of biomass components and the formation of new structures. Due to its cellulose, hemicellulose, and lignin content, raw coconut fiber typically contains several functional groups such as hydroxyl (O-H), carboxyl (C=OOH), carbonyl (C=O), and methylene (CH_2) [Adeniyi 2019]. Any modification of the fibers can cause a change in the functional groups, with modifications often reducing the lignin content of the fibers [Siakeng 2018].

In the 3400–2500 cm^{-1} range, both the broad O-H stretching band (~3300–3200 cm^{-1}) and the C-H aliphatic stretching peaks (~2920 cm^{-1} and ~2850 cm^{-1}) were prominent in the coconut fiber but significantly diminished with increasing pyrolysis temperatures [Yang 2007]. These aliphatic C-H stretching bands are characteristic for raw coconut fiber and also for natural rubber latex [Cesar 2019]. It indicates extensive dehydration and the thermal degradation of aliphatic structures (cellulose, hemicellulose, and latex components) as carbonization progresses.

The 1800–1500cm^{-1} region revealed a transition from oxygenated to more aromatic structures. A band around ~1730–1710 cm^{-1}, assigned to C=O stretching (from esters, carboxylic acids, or aldehydes, possibly from hemicellulose or latex), largely disappeared at higher temperatures. This disappearance, particularly for conjugated aromatic carbonyl/carboxyl C=O bonds at 1719 cm^{-1} at temperatures above 700 °C, is explained by the instability of these groups due to dehydration [Zhao 2015]. Conversely, the band at ~1620–1580 cm^{-1}, characteristic of aromatic C=C stretching (lignin), persisted and often intensified at higher temperatures, signifying the stability and progressive condensation of aromatic structures in the biochar [Dhar 2020]. Further evidence of biomass decomposition was observed in the 1400–1000 cm^{-1} region. Bands corresponding to C-H bending (~1375–1380 cm^{-1}) and various C-O stretching vibrations (~1250–1030 cm^{-1}), associated with alcohols, ethers, and esters in polysaccharides (cellulose, hemicellulose) and potentially latex, decreased significantly or vanished at 600–700 °C [Singh 2012, Dhar 2020]. This points to the thorough breakdown of these complex structures, leading to a more carbonized material.

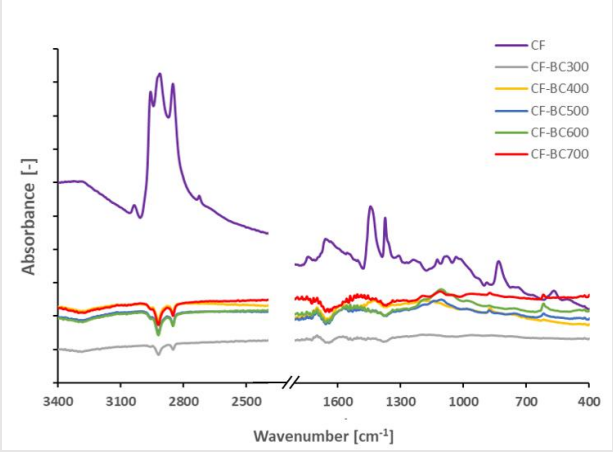

Figure 3. FTIR spectra of coconut fiber (CF) and derived biochar from coconut fiber (CF-BC) at five different pyrolysis temperatures (Note: the number in legend of color coding is the pyrolysis temperature)

Finally, in the <900 cm^{-1} region, the appearance or increased intensity of bands (e.g., around 800–600 cm^{-1}) indicative of C-H out-of-plane bending in aromatic rings confirmed the enhanced aromaticity of biochars produced at higher temperatures [Dhar 2020].

2.5 Effect of pyrolysis temperature on surface morphology

Biochar derived from coconut fibers is particularly recognized for its high surface area and porosity, making it an effective material for both soil amendment and water treatment. The efficacy of biochar in pollutant removal and nutrient exchange is largely dictated by its porosity and surface area [Eletta 2021]. Elevated pyrolysis temperatures are known to enhance these characteristics by expelling volatile organic compounds, leading to an expansion of micropores and a corresponding increase in the biochar's surface properties and carbon content [Wang 2019]. To visually investigate these morphological features and their development, SEM micrographs of the coconut fiber biochars, presented in Fig. 4 (and specifically detailed in a, b, c, d, e and f), illustrate significant surface morphological variations and structural changes, particularly concerning porosity, as a function of pyrolysis temperature.

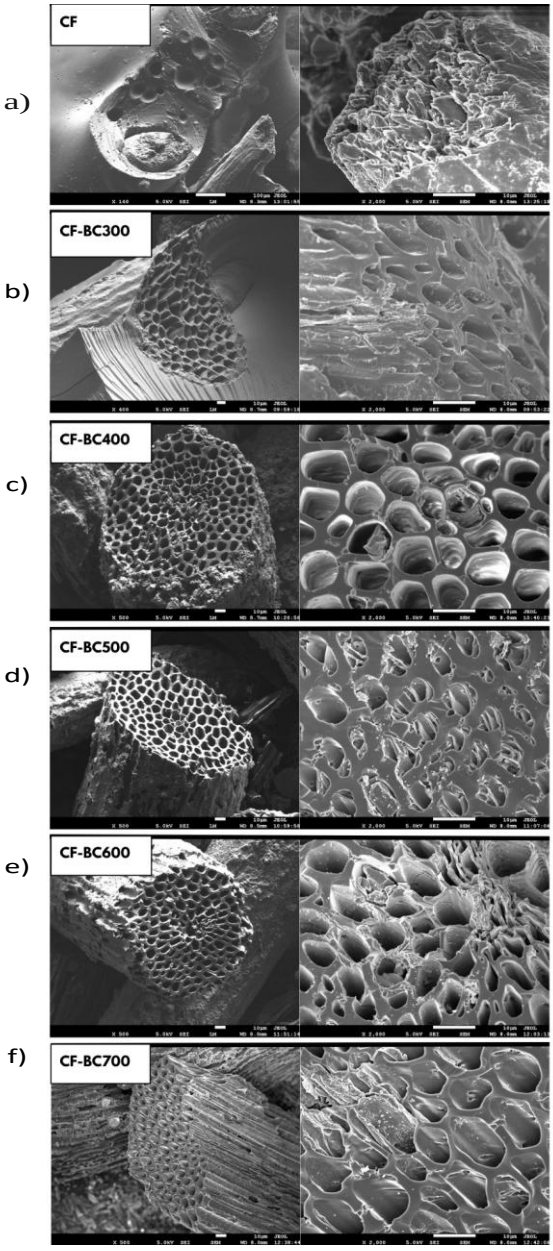

Figure 4. SEM micrographs of coconut fiber (a) and derived biochars from CF at five different temperatures (b–f)

The varying conditions of different pyrolysis temperatures clearly played a significant role in altering the surface morphology of the solid products.

Overall, the images revealed dense surfaces interspersed with intricate networks of heterogeneous porous structures. The formation of these structures and associated shrinkages is attributed to the flow of volatile matter during the pyrolytic process, leading to an uneven surface structure. For comparison, previous studies on raw coconut fibers noted rough surfaces with regularly spaced pits [Yan 2015]. These pits, referred to as spherical voids by A. Khan et al. [Khan 2014] and observed to reduce with chemical treatment, can be seen in Fig. 4a CF (referring to the raw feedstock CF on the left side). Different forms and scales of macropores, micropores, and mesopores were evident throughout the images. The SEM micrographs (Figures 4b – 4f) revealed that the coconut fiber biochars formed non-uniform elongated shapes after pyrolysis. Figures 4b and 4c (representing lower pyrolysis temperatures of 300 and 400 °C) show that the amount of porosity was relatively less. At these temperatures, the biochar obtained at pyrolysis temperature 300 °C and 400 °C had a rigid, uneven surface with the formation of different size and shapes of pores. This corresponds to the degradation of less stable hemicellulose (220–315 °C) and the initial stages of cellulose degradation (315–400 °C), releasing some volatiles that begin to form porosity through mass loss [Kalina 2022]. At pyrolysis temperature 400 °C, the micrographs reveal more open pores and slightly thinned walls, reflecting early stages of structural breakdown as aliphatic cellulose residues are removed and aromatization processes commence [Zhang 2021]. The less significant effect of temperature on lignin-based woodcut feedstock compared to cellulose/hemicellulose-rich materials (like oat bran and corn biomass) observed by Kalina et al. [Kalina 2022] further supports the idea that initial changes in coconut fiber biochar at lower temperatures are driven by hemicellulose and cellulose decomposition.

The presence of porosity consistently increased with rising pyrolysis temperatures, with pores appearing irregular and sturdy in shape (Figures 4d and 4e). Noticeably, higher pyrolysis temperatures (500–600 °C) yielded much greater surface porosity. At 500 °C, the images reflect the full degradation of hemicellulose and most cellulose, leading to significant volatile release and the formation of initial porosity [Kalina 2022]. By 600 °C, the biochar (Fig. 4e) obtained showed only some cracks developing on the surface when compared to the biochar from 500 °C (Fig. 4d), however, at this temperature, cellulose-derived char transitions towards more graphite-like microstructures with increased aromatization and a substantial increase in overall porosity and surface area. SEM imagery reveals heterogeneous pore networks with irregular cell-wall fragments, indicative of extensive devolatilization and collapse of cellular structures [Zhang 2021]. The biochar acquired at 700 °C showed a miscellaneous range of shapes in the pores (Fig. 4f) and exhibited a highly porous surface area. At this elevated temperature, the continuous breakdown of lignin, which has a broad degradation range extending up to 900 °C, primarily contributes to the enlargement of existing pores rather than the formation of new ones [Mukherjee 2022, Kalina 2022].

2.6 Effect of pyrolysis temperature on thermal stability of coconut fiber and derived biochars

The results of the thermogravimetric analysis (Fig. 5 and Tab. 2) indicate an increase in thermal resistance with rising treatment temperature. The often-described phase of moisture reduction in the literature [Mothe 2009, Ezekiel 2011, Mittal 2019] is not visible in the graphical representation of the individual sample results, as the initial measurement temperature was 100 °C.

The mass of the original material decreases in several stages due to its composition. During the TG curve of coconut fiber, the main decomposition stage occurs between 250 °C and 370 °C. This indicates the decomposition hemicelluloses, cellulose, and lignin [Mothe 2009]. Hemicelluloses are the least thermally stable, with their decomposition primarily occurring at 220–315 °C. This is followed by cellulose (315–400 °C), and lignin is the most resistant. Its decomposition happened slowly under the whole temperature range from ambient to 900 °C, but at a very low mass loss rate [Yang 2007]. In addition to these lignocellulosic components, the latex adhesive used for bonding the fibers also contributes to the mass loss during thermal decomposition. According to Haque (2020), the thermal decomposition of waste latex is reported to begin as early as 169 °C to 200 °C with an initial 3.6% weight loss for volatile compounds. Significant mass loss, corresponding to the structural decomposition of the latex polymer, typically occurs in a broader range, with a maximum weight loss (67.8%) observed between 289–330 °C and a second phase decomposition (14.4%) occurring from 423–488 °C. Minor mass loss (4.9 wt%) can continue up to 1000 °C. This multi-stage decomposition of latex overlaps with the degradation temperatures of hemicellulose and cellulose, thereby influencing the overall TG curve of the coconut fiber.

As can be seen, even in the case of biochar produced at 300 °C, there is an increase in thermal stability and also an increase in the carbonaceous residue. Given the decomposition temperatures of the individual pseudo-components mentioned above, this shift is primarily caused by the removal of most hemicelluloses during the production process. At a treatment temperature of 400 °C, cellulose decomposition also occurs, which causes a significant difference between the results of samples CF-BC300 and CF-BC400. Higher biochar preparation temperatures continue to shift the resistance of the resulting product to higher temperatures; however, this shift is less pronounced and can be attributed to the transformation of lignin into fixed carbon. Finally, samples CF-BC700 show only a very slight mass loss, with more than 80% of their mass remaining even at 900 °C.

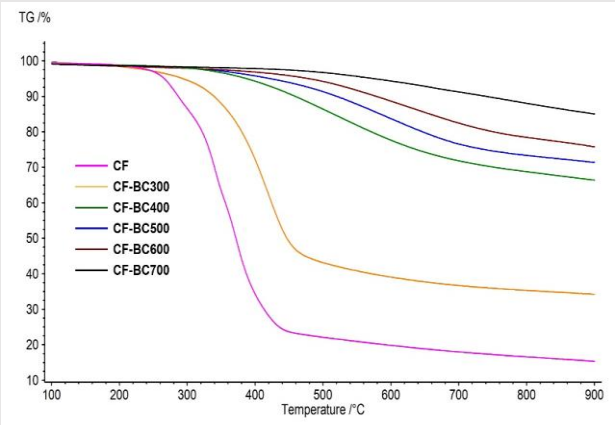

Figure 5. Thermogravimetric curves of raw material (CF) and prepared biochar at five different temperatures

Table 2. Results of thermogravimetric analysis of raw material (CF) and prepared biochar at five different pyrolysis

Sample	$T_{95\%}$ [°C]	T_{max} [°C]	Residue at 900 °C [%]
CF	269	372	15.3
CF-BC300	300	421	34.4
CF-BC400	401	497	66.9
CF-BC500	430	594	71.3
CF-BC600	495	650	76.1
CF-BC700	607	777	85.8

2.7 Future application of waste coconut fiber pyrolysis products

For further research, the biochar produced from waste coconut fiber is currently undergoing testing for its application in dispersive solid-phase microextraction (DSPME). This ongoing work aims to fine-tune the biochar preparation method specifically to optimize its performance for microextraction purposes, exploring its potential as an efficient and sustainable sorbent in isolation and enrichment of different volatile and semivolatile organic components for the purpose of green sample preparation in environmental analysis.

3 CONCLUSIONS

In this study, we investigated the feasibility of coconut fibers (a waste product from mattress manufacturing, bonded with latex) as a source for biochar production using the slow pyrolysis process. The effects of different pyrolysis temperatures on the yield and physicochemical properties of the resulting biochars was thoroughly analysed.

The experimental findings revealed that biochars produced across the pyrolysis temperature range from 300 to 700 °C exhibited significant variations in their structures and physicochemical characteristics. Specifically, the yield of biochar consistently decreased with increasing pyrolysis temperature, dropping from 67.06% at 300 °C to 15.83% at 700 °C. Conversely, fixed carbon, ash content, degree of aromaticity, and porosity significantly increased with rising temperatures.

Volatile matter content and the volatile matter/fixed carbon ratio were inversely correlated with pyrolysis temperature. EC displayed a variable trend and pH of the biochars, starting near neutral, exhibited slight acidification at lower temperatures before shifting to highly alkaline values (pH 9.29 at 700 °C) at the highest temperature, a phenomenon potentially influenced by the latex content in feedstock. FTIR analysis confirmed the progressive loss of oxygenated and aliphatic functional groups and the concurrent development of more aromatic structures. SEM images further revealed the formation of elongated and highly porous structures at elevated temperatures. These disparities in structure and properties highlight the critical role of pyrolysis temperature in tailoring biochar for specific applications in the development of an analytical procedures for environmental analysis in accordance with requirements of green analytical chemistry.

In future research, we can focus on the possibilities of using digital image processing [Frankovsky 2022] and comparing it with other materials [Majko 2022].

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