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Influence of Carbonization Conditions and Temperature Variations on the Characteristics of Coconut Shell Carbon

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ABSTRACT

This research aims to study the impact of carbonization atmospheres (ambient and nitrogen) and temperature on the properties of the coconut shell carbon (CSC) formed. To characterize the properties of CSC, the char yield percentage was calculated. Scanning Electron Microscopy (SEM) was used to study the surface morphology of CSC while X-ray Diffraction (XRD) analysis was done to identify the degree of graphitization. The carbon formed by carbonization under the nitrogen atmosphere yields lower char percentages compared to the ambient atmosphere. When the carbonization temperature elevated, both atmospheres produced a lower char yield percentage. This result is aligned with the SEM analysis where more and larger pores were observed from the carbon produced at higher temperatures and the result was further enhanced under a nitrogen atmosphere. It was found that the char yield of CSC decreased from 20.9% to 11.4% when the carbonization temperature increased from 400°C to 1000°C under the ambient atmosphere. More significant changes were formed through the carbonization process under the nitrogen atmosphere (from 18.3% to 6.03%). Pores formed when the volatile materials are released due to the elevated carbonization temperature, resulting in a reduction in total weight thus, the char yield percentage. From the XRD, all CSC produced from both atmospheres with varying temperatures poses an amorphous XRD pattern. However, the right shifted peak and the presence of an additional peak of ~40° suggest that under different temperatures and atmospheres, the crystallinity of the CSC produced was affected. This research provides insight for optimizing CSC production in the future to enhance the application of CSC.

Keywords: *Coconut shell carbon, Biochar, Carbonization, Heating temperature, Treatment atmosphere*

1. INTRODUCTION

As the natural energy sources continue to diminish, it then arises a clear concern in searching for sustainable and environmentally friendly energy solutions. This urgent issue has led to greater recognition of the importance of alternative energy sources that are both renewable and have a low impact on the environment. To reduce the negative effects of energy production on our fragile ecosystems, the main focus now by the researchers is on reducing the carbon footprint from human activities.

In the effort to reduce the carbon footprint, researchers are then focusing on utilizing the abundant biomass waste. By giving a second life to biomass waste, the goal of effectively managing it and generating valuable energy sources can be realized.

Carbon materials are one of the most popular materials that capture the attention of researchers as they can be produced from various kinds of biological products in human daily life. There is a range of carbon products that have been well utilized by researchers, particularly activated carbon, due to its notable adsorption properties and versatility, it has marked a rapid growth rate in industrial applications. Besides, the production of synthetic graphitic carbon due to its electrical properties also makes it one of the most popular research subjects.

Carbonization is a heat treatment process that helps to convert organic precursors into rich-carbon content materials [1]. Various materials have been employed as precursors for the carbonization process. Biomass waste materials have gained prominence among the precursors due to their cost-effectiveness, renewability, and sustainability. Through the process of carbonization, the carbon precursor produced is normally categorized as biochar. It is a good precursor for graphitic carbon growth because of its high carbon content [2] and potential for crystalline structure development. There are bunches of biomass raw materials from agricultural waste that can be used to produce graphitic carbon. For example, softwood, green tea waste, cornstalk, wood, bamboo and coconut shells [3]. Coconut shell waste, a biomass product, is abundant and widely used in numerous applications, due to its widespread availability, sustainability, unique

structures, and low cost [4]. However, the carbonization process especially when conducted in a nitrogen atmosphere will be accompanied by several challenges and environmental concerns.

The carbonization process which involves the degradation of organic materials [5] by elevating the temperature under a controlled atmosphere was one of the most applied processes in the production of carbonaceous materials [6]. Under high temperatures with the presence of oxygen, the impurities in the organic materials tend to form ash. Thus, nitrogen gas is generally applied in most industries during the carbonization process as a source of inert atmosphere. However, during the carbonization process, the nitrogen gas will remove the oxygen gas in the atmosphere by forming nitrogen oxides (NO_x) which are hazardous to both health and the environment [7].

By taking into consideration the safety and environmental concerns, it is crucial to investigate the effect of carbonization under different atmospheres and temperatures on the properties of carbon materials produced. In this study, to contribute to the utilization of biomass waste materials, the organic material used was coconut shell waste which can be abundantly found in agricultural countries. The effect on the properties of carbon materials produced with carbonization under both nitrogen gas and ambient atmospheres coupled with a range of temperatures will be mainly investigated. The outcomes of this research aim to provide insights as a guide to deciding the continued use of nitrogen gas during the carbonization process.

2. MATERIALS AND METHODS

2.1. Materials

The coconut shell waste (CSW) was collected from Pasar Besar Kangar, Perlis Malaysia.

2.2. Coconut Shell Carbon Preparation

The coconut husk was removed, followed by washing and rinsing the coconut shell with distilled water. It was then let dry in the oven (MEMMERT -UNB-200) at 110°C for 12 hours. Next, the coconut shell was crushed into small pieces and sieved into the range of 10-20mm size.

The coconut shell carbon was prepared by carbonization under two different atmospheres at various temperatures. Under ambient atmosphere, the carbon sample was produced by carbonizing at the temperatures of 400°C, 600°C, 800°C and 1000°C. For carbonization under a nitrogen atmosphere, temperatures of 400°C and 1000°C were used. The details about carbonization under ambient and nitrogen atmosphere are discussed in the next subsection.

2.2.1. Carbonization Under Nitrogen Atmosphere

An empty crucible boat was weighed, followed by the addition of 40 grams of coconut shell pieces. Then, it was placed into the tube furnace (CTF 12/75/700, Carbolite). The carbonization temperature was set at 400°C with a heating rate of 5°C/min, a holding time of 3 hours, and a cooling rate of 10°C/min, with nitrogen gas flow rate controlled at 50mL/min. After the carbonization process, the weight of the crucible boat was measured. The sample was denoted as CSC N-400. The process was repeated at 1000°C and the sample was denoted as CSC N-1000.

2.2.2. Carbonization Under Ambient Atmosphere

An empty metal crucible was weighed, followed by the addition of 350 grams of coconut shell pieces. The lid of the metal crucible was tightly closed to create a limited oxygen atmosphere for the carbonization of the sample. Then, it was placed into the muffle furnace (L27-1200, LT Furnace). The carbonization temperature was set at 400°C, with a heating rate of 5°C/min, a holding time of 3 hours, and a cooling rate of 10°C/min. After the carbonization process, the weight of the crucible boat was measured. The sample was denoted as CSC A-400. The process was repeated at 600°C, 800°C and 1000°C. The resulting samples were denoted as CSC A-600, CSC A-800 and CSC A-1000, respectively. After obtaining the coconut shell carbon pieces, they will be further ground into powder form for the characterization process to continue.

2.3. Char Yield Percentage

The percentage of char yield is an important parameter that might influence the efficiency of the carbon materials production process. In this research, the masses of coconut shell waste and coconut shell carbon were weighed before and after the carbonization process. The coconut shell carbon's yield percentage was then calculated using Eq. (1) [8].

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$$\text{Char yield}\% = \frac{\text{Mass of CSC}}{\text{Mass of raw coconut shell}} \times 100\% \quad (1)$$

2.3.2. Surface Morphology

For the surface morphology of the CSC sample, scanning electron microscopy (SEM), (JEOL JSM-6460 LA, Japan) was used. The coconut shell carbon powder was first coated with a platinum layer to prevent the charging effect. Subsequently, the coconut shell carbon surface was scanned at various magnifications to investigate the surface and porous structure.

2.3.3. Phase Analysis

Crystallinity and structural properties of carbon produced will be affected by high temperatures due to the formation

of various reactions during the carbonization process. The phase of the carbonized coconut shell was investigated by using X-ray diffraction (XRD), (D2 Phaser, Bruker, Germany) using Cu-Kα radiation (λ=1.5406 Å, 40 kV, 30 mA) in the range from (2Θ) 10° to 90° with scan rate 0.1 s/step. The analysis of XRD data was then carried out by using X'pert Highscore Plus software to determine the structural changes in the coconut shell carbon sample.

3. MATERIALS AND METHODS

3.1. Char Yield

The following data of the char yield percentage was calculated by using Eq. (1) and is tabulated in Table 1.

From the table, a similar decrement trend of the char yield percentage can be observed from the coconut shell carbon produced from different carbonization temperatures under both atmospheres. The percentage of char yield decreased significantly when the carbonization temperature increased.

Under ambient carbonization conditions, the char yield was 20.9% at 400°C, it then decreased to 11.4% when the temperature was increased to 1000°C. In the case of carbonization under a nitrogen atmosphere, the char yield decreased from 18.3% at 400°C to 6.1% at 1000°C. Biomass materials are generally high in volatile materials content which marked the percentage around 70-86% [9]. The decreased in char yield percentage as the temperature increased indicates that a greater proportion of volatile materials were released from the coconut shell at higher temperatures [10]. As the carbonization temperatures elevate, more heat energy is present, facilitating the breaking of chemical bonds for the impurities and volatile materials in the coconut shell waste. The disruption of chemical bonds causes further release of the volatile component, leading to a significant decrease in the overall weight [11]. Thus, this process contributed to the reduction in the char yield percentage of the coconut shell carbon produced.

From Figure 1, as compared to both atmospheres used, the decrement of char yield in the nitrogen atmosphere is more pronounced at 12.2% while the ambient atmosphere is 9.5% as the temperature increased from 400°C to 1000°C. A more substantial decrement in the char yield percentage can be noticed under the nitrogen atmosphere.

Table 1. Effect of carbonization temperatures and atmospheres on the char yield (%) produced from raw coconut shells

Sample Name	Carbonization Parameters		Char yield %
	Atmosphere	Temperature	
CSC A-400	Ambient Atmosphere	400	20.9
CSC A-600		600	18.8
CSC A-800		800	15.7
CSC A-1000		1000	11.4
CSC N-400	Nitrogen Atmosphere	400	18.3
CSC N-1000		1000	6.1

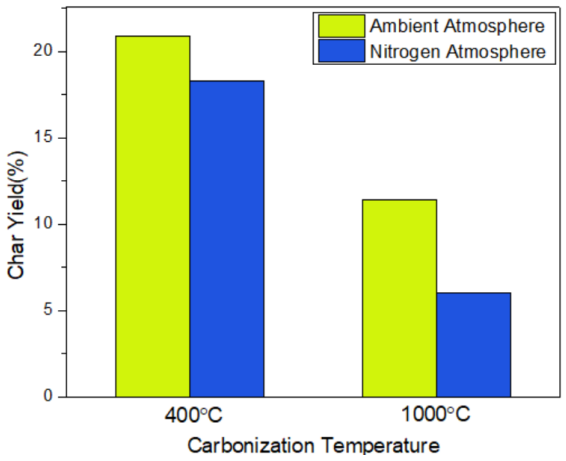

Figure 1. Char yield (%) produced from raw coconut shells under ambient atmosphere and nitrogen atmosphere at temperatures of 400°C and 1000°C

When it comes to the different carbonization atmospheres, the nitrogen atmosphere tends to promote a more complete decomposition of organic and volatile materials [12]. The presence of oxygen in the ambient atmosphere may promote an oxidation reaction that produces unwanted by-products that interrupt the formation of fixed-carbon content.

3.2. Surface Morphology of Coconut Shell Carbon

Figure 2 shows the SEM image of raw coconut shell powder, which was free from any further treatment. From the micrograph, it can be observed that there was no porous structure on the surface of the raw coconut shell powder. Therefore, to improve the value of coconut shell powder to be well-suited in various applications, further treatment is needed to create a porous surface or even change the structure of the coconut shell powder to obtain the desired properties.

Figure 3 shows the surface micrograph for the sample carbonized under ambient atmosphere where (a) and (b) is the sample heated at 400°C and (c) and (d) at 1000°C. Carbonaceous biomass materials consist of various components such as cellulose, hemicellulose, lignin and

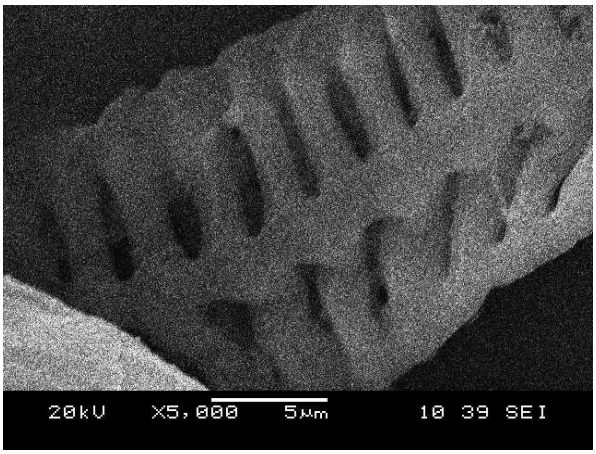

Figure 2. The SEM image of raw coconut shell powder

other impurities [13]. It also contains a large amount of volatile matter. During pyrolysis, these volatile matters will be released. As a result, the porous structure should be formed onto the surface of biomass waste due to the escapism of those matters [14]. Therefore, this explanation can be used for the SEM images in Figure 3 where the porous structures can be observed after carbonization in the ambient atmosphere at 400°C and 1000°C, respectively.

Based on Figures 3(a) to (d), it can be noticed that more pores were formed on the surface of coconut shell carbon (CSC) that was carbonized at 1000°C. Larger pore size also can be observed, which is 0.93 μm compared to CSC carbonized at 400°C (0.58 μm). The surface area of coconut shell carbon increases with the increase of the temperature [15]. As the temperature increased, the chemical bonding between the pore-blocking volatile substances was weakening. It causes the volatile materials on the coconut shell's carbon surface to expel. Also, due to high temperatures, the carbon tends to thermally crack, resulting in the formation of a porous structure and thus increasing the total surface area of the biochar [16].

The SEM image of the surface of coconut shell carbon produced under a nitrogen atmosphere is shown in Figure 4. Figures 4(a) and 4(b) are the carbon produced at 400°C while Figures 4(c) and 4(d) are at 1000°C. The same condition can be observed from the SEM images in Figures 4(a) to (d), where the raw coconut shell powder was carbonized under a nitrogen atmosphere. Both porosity size and porosity distribution are larger (2.77 μm) and higher for CSC powder that was carbonized at 1000°C, compared to the pore size of CSC carbonized at 400°C, which is 1.62 μm. Higher temperature promotes the release of volatile substances [17] and thus the formation of more and larger pores, resulting in an increased specific surface area [18].

The carbonization of coconut shell carbon under different atmospheres may significantly affect the pore distribution and the complexity of the porous structure [19]. As seen in Figures 3 and 4, both samples CSC A-400 and CSC A-1000 depicted lower porosity distribution and smaller porous structure than CSC N-400 and CSC N-1000. When compared, the porous structure and its distribution are found to be more idealized in CSC carbonized under the nitrogen atmosphere.

The reason is that, without the application of nitrogen or air current, more tarry matter deposits and decomposes in the microporous network, partially filling or blocking most of

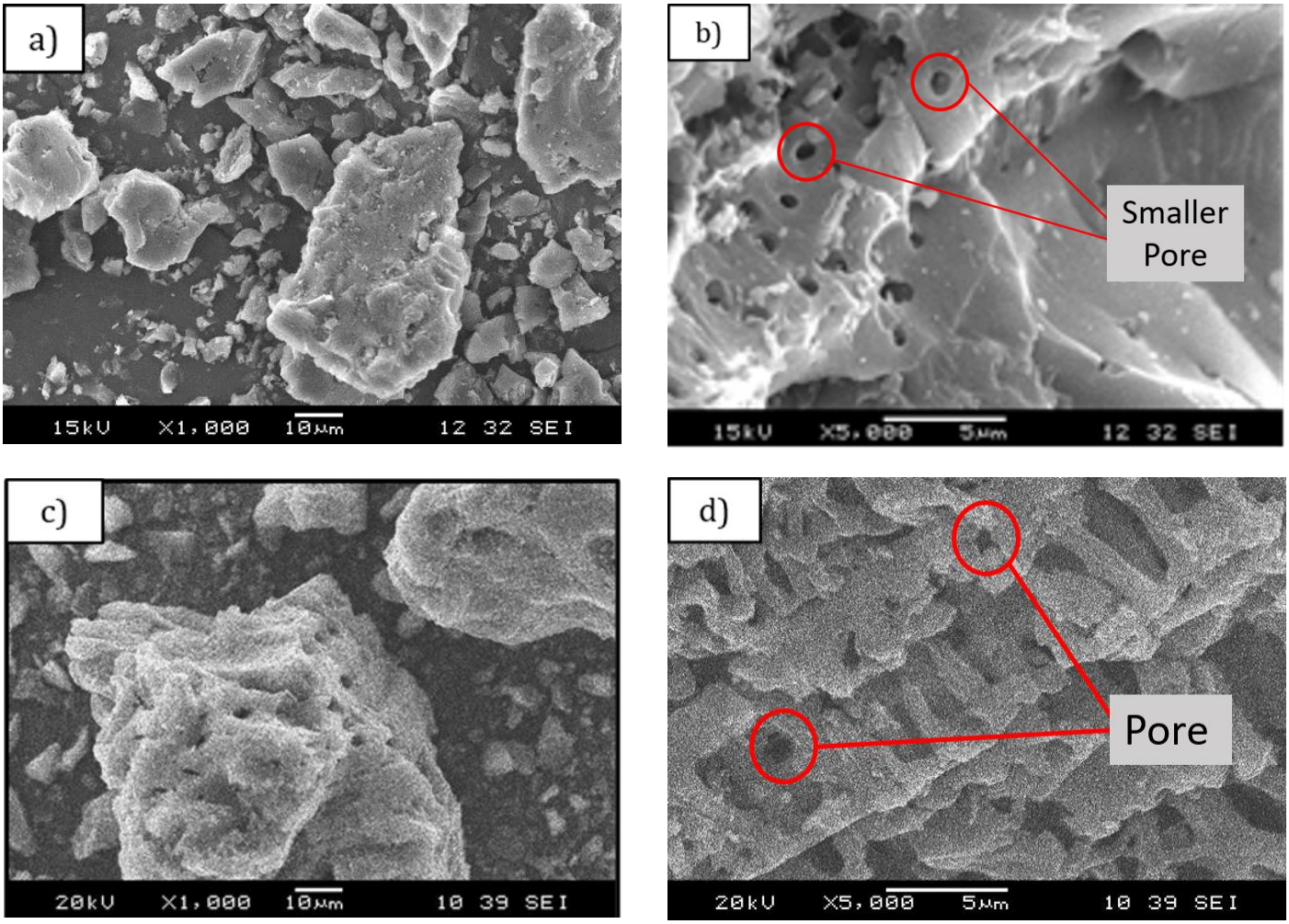

Figure 3. The SEM images of coconut shell carbon powder carbonized in ambient atmosphere at 400°C, CSC A-400 (a & b) and at 1000°C, CSC A-1000 (c & d) under 1000X and 5000X magnification

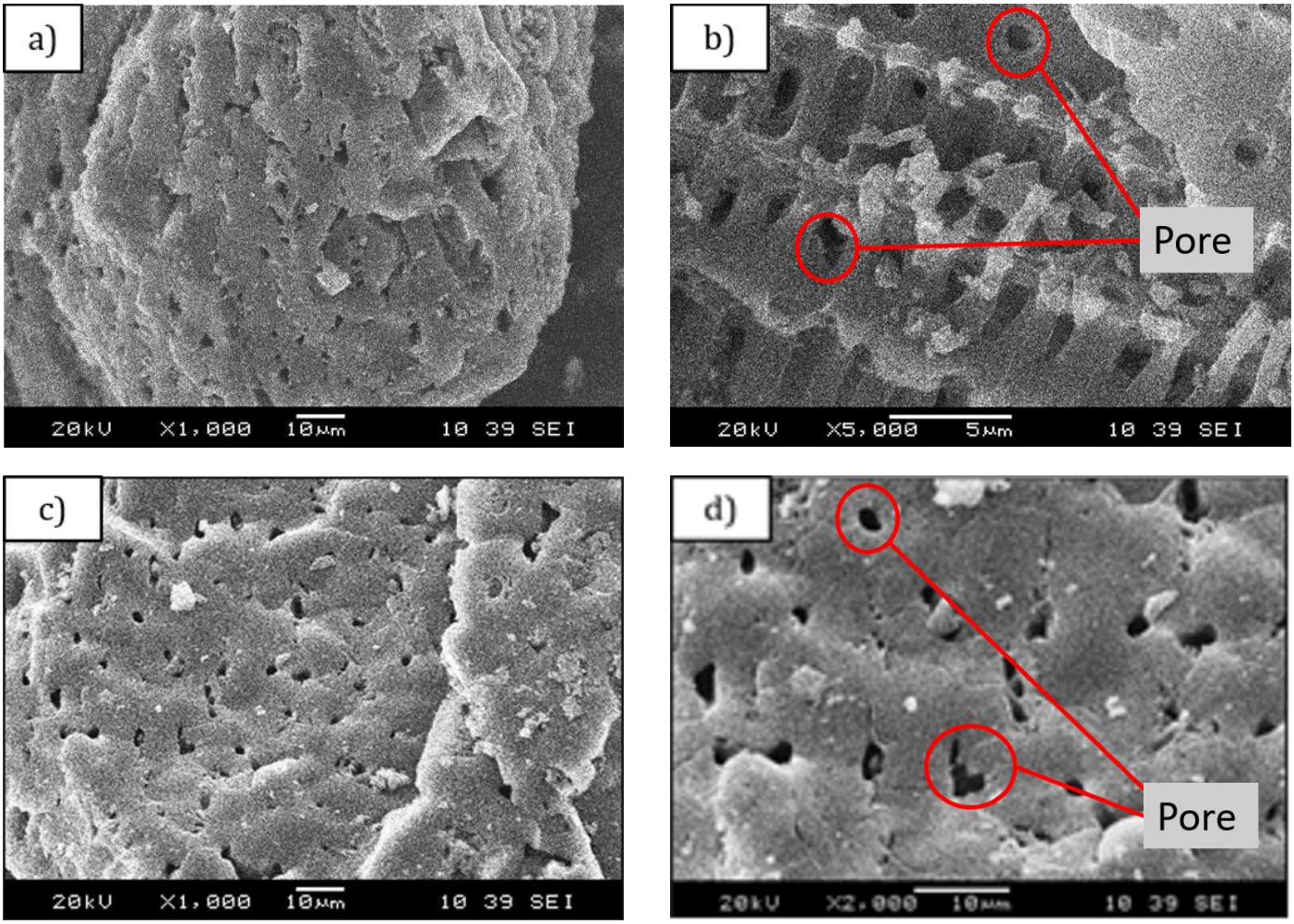

Figure 4. The SEM images of coconut shell carbon powder carbonized in nitrogen atmosphere at 400°C, CSC N-400 (a & b) and at 1000°C, CSC N-1000 (c & d) under 1000X and 5000X magnification

the micropores and thus generating more constrictions for the formation of the porous structure [12].

In the presence of nitrogen gas during the carbonization, more complete decomposition of the organic and volatile material can occur, resulting larger porous structure and higher porosity distribution. The presence of oxygen during carbonization will affect the carbon produced [6]. Carbonization without oxygen avoids the possibility of oxidation reaction occurring on the coconut shell carbon, which will create unwanted by-products that affect the production of porous structure on the surface.

3.3. Phase Analysis of Coconut Shell Carbon

Figure 5 shows the XRD pattern for the raw coconut shell before undergoing any reaction. Raw coconut shells exhibit a higher crystallinity sharp peak compared to the carbonized coconut shell carbon. This can be explained by the composition of the coconut hard shell, which consists of various components, with the three main ones being cellulose, hemicellulose, and lignin [13]. The cellulose in the coconut shell was one of the main contributors to its sharp crystalline peak, as cellulose has a crystalline structure in nature. A typical natural cellulose fibre exhibits sharp crystalline diffraction peaks around 19°, 22.25° and 34.67° [20]. Therefore, the presence of sharp crystalline peaks in the raw coconut shell sample is attributed to the presence of cellulose fibers in coconut shells.

The XRD pattern of the coconut shell carbon is shown in Figure 6. Based on the figure, all the samples show a broad peak between 10°-30° of 2θ degrees. A broad peak around

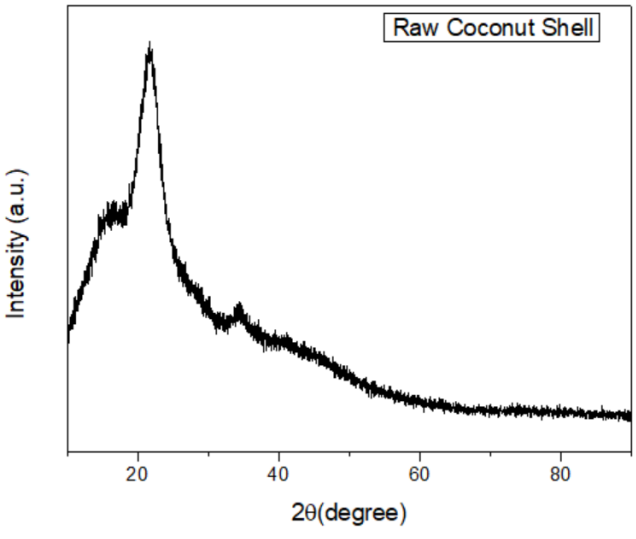

Figure 5. The XRD pattern of raw coconut shell waste

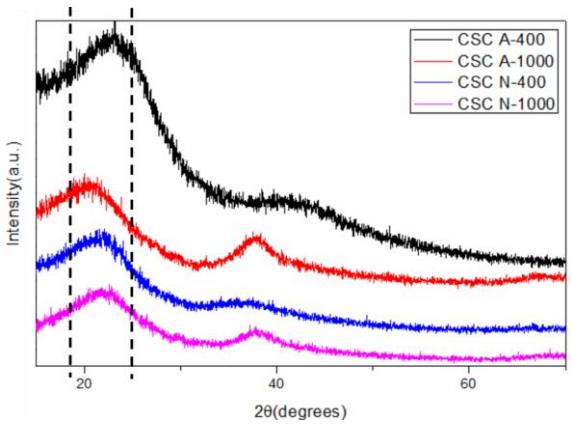

Figure 6. The XRD pattern of carbonized coconut shell waste

23° indicates the presence of amorphous carbon in the sample [21]. Those humps suggest that the carbonization temperatures used might be insufficient to induce the crystallization of amorphous carbon or to produce a graphitic carbon structure [22].

From Figure 6, compared to the carbonization atmosphere, both samples carbonized under ambient atmosphere show a broader peak. This suggested that a lower degree of crystallinity formed with the absence of nitrogen gas during carbonization.

Nevertheless, at 1000°C for both atmospheres used, an additional hump can be observed between 35° and 40° of 2θ degrees. This situation suggested that there might be a change in the structure of the amorphous carbon as a function of temperature. Weak peaks at ~40° can be referred to as the characteristic reflections originating from the (100) planes within disordered carbon materials [23]. Under higher temperatures, carbon materials tend to achieve a higher degree of crystallization [24]. The increase of heat energy leads to the promotion of further reaction as more energy is available to support the breaking and forming of new chemical bonds. For this reason, structural change in the coconut carbon shell may occur, resulting in the amorphous coconut shell carbon approaching a higher degree of crystallization.

When comparing the XRD patterns of raw coconut shell and coconut shell carbon that underwent the carbonization process, obvious changes from the sharp crystalline peak to the broad amorphous peak can be noticed. As suggested by Chen et. al, the hemicellulose, cellulose and lignin components in carbonaceous material thermally decompose at different temperature ranges [25]. Therefore, even after the carbonization, part of the component was yet to be decomposed into carbon, affecting the crystallinity of the carbon produced. The presence of lignin in the sample may contribute to the amorphous nature of the peaks observed [26]. Therefore, the XRD peak of the carbon formed retains the characteristic broad amorphous peak.

4. CONCLUSION

Carbonization temperature and atmosphere are among the most decisive factors that affect the biomass char yield percentage. The char yield percentage of coconut shell carbon decreased significantly with the increase in carbonization temperature. At 400°C it was 20.9% and decreased to 11.4% at 1000°C. A more obvious decrease trend of the char yield can be observed through the carbonization of coconut shells under a nitrogen atmosphere. The char yield decrement is about 12%, from 18.25% at 400°C to 6.04% at 1000°C. It can be concluded that the presence of nitrogen gas and high temperature during the carbonization process led to a significant decrease in the char yield percentage.

The SEM micrograph suggested that through the carbonization process, porous structures and voids can form on the coconut shell carbon. This outcome further supports that when the carbonization temperature increased, more pores formed, resulting in the reduction of total weight, and consequently causing the char yield percentage to decrease. The presence of a nitrogen atmosphere promotes a more complete carbonization process, contributing to the formation of larger and more pores and therefore, lower char yield percentage.

XRD analysis demonstrated that the carbonization process under a nitrogen atmosphere enhanced the crystallinity of coconut shell carbon formed for both temperatures. However, under both atmospheres, increasing carbonization temperature will also promote the crystallinity of the coconut shell carbon. Therefore, it can be summarized that the best condition for high crystallinity of the coconut shell carbon form was a carbonization temperature of 1000°C with the presence of a nitrogen atmosphere.

From this research, it is demonstrated that the use of nitrogen gas and carbonization at high temperatures may bring various harmful substances to both the environment and human health. However, the benefits of the application of these two conditions are significant. Therefore, it is suggested that further research needs to be done to determine the replacement of carbonization conditions that produce similar properties of coconut shell carbon without the usage of nitrogen gas. It is highly recommended to involve the usage of green catalysts which can help to lower the carbonization temperature while producing high carbon content carbonaceous materials. Research can also be done to investigate methods for reducing the oxidation reactions during the carbonization process in the absence of nitrogen gas.

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