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## Hydrothermal Treatment to Produce Honeycomb-like Structure of Hierarchical Carbon from *Intsia, spp* Waste Wood: Preparation and Characterisation

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# Hydrothermal Treatment to Produce Honeycomb-like Structure of Hierarchical Carbon from *Intsia*, spp Waste Wood: Preparation and Characterisation

Darma Santi<sup>a\*</sup>, Triyono<sup>b</sup>, Wega Trisunaryanti<sup>b</sup>, Iip Izul Falah<sup>b</sup>  
and Jacson Victor Morin<sup>a</sup>

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## ABSTRACT

Hydrothermal treatment of *Intsia*, spp wood waste has succeeded in producing hierarchical carbon structures such as honeycomb-like structures. This study aims to examine the difference between hydrothermal and non-hydrothermal activation treatments in the hierarchical synthesis of carbon. Sequentially, the hydrothermal treatment was created with  $ZnCl_2$  as an activating agent and  $H_2O_2$  as an oxidising agent. The hydrothermal process is followed by carbonisation at  $700\text{ }^\circ\text{C}$  under  $N_2$  gas (HC-ZH) and oxidation at  $350\text{ }^\circ\text{C}$  in an oxygen stream (HC-ZH-Ox). All carbonised products were analysed using XRD, FT-IR, the Boehm method to determine the quantity of oxygenated functional groups (OFGs), the  $NH_3$  base gas adsorption method to determine the total acid amount, and the Surface Area Analyser (SAA) and SEM. Hierarchical carbon with the amorphous structure shown by XRD analysis. Hydrothermal treatment increased the OFGs content, 50.25% for carboxylic groups, 354.62% for phenolic groups, and 3.9% for lactone groups. The presence of OFGs was proven through identification using FTIR. Hydrothermal treatment increased acidity by 387.83%, the highest surface area produced by hierarchical carbon non-hydrothermal (HC-Ox) of  $443.5\text{ (m}^2\text{/g)}$ . The HC-ZH-Ox has a surface area of  $232.2\text{ (m}^2\text{/g)}$ . SEM image shows a structure of stacked honeycomb-like, with different thicknesses in the samples HC-ZH-Ox and HC-Ox.

*Keywords: Hierarchical carbon; hydrothermal;  $H_2O_2$ - $ZnCl_2$  activation; Intsia; spp; oxidising agent; honeycomb-like structures.*

## 1. INTRODUCTION

Currently, there is a need for more research into the production and study of carbon-based materials. It is related to the advancement of new scientific and engineering sectors that necessitates the creation of improved carbon materials, which includes the synthesis of allotropic carbon forms and the production of varied porous materials in various diverse carbon forms [1]. Physical activation is one of the most common ways to make activated carbon. It does, however, yield microporous activated carbon in most cases [2]. Furthermore, the synthesis of mesoporous materials frequently necessitates high-temperature moulding [3-5]. Chemical activation methods can also be used to create porous carbon compounds and this method [6-7].

Chemical activation has been shown to increase the presence of oxygenated functional groups (OFGs), including carbocyclic, lactones and phenolics in their precursors. The use of  $H_2O_2$  as an oxidising agent to increase the formation of OFGs on precursors during pre-treatment of coconut shell is expected to increase the affinity of the activating agent ( $ZnCl_2$ ) on the surface of the precursor in

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the activation process [8]. OFGs increase chemical activation by  $\text{ZnCl}_2$ , thereby promoting a larger area and volume of the mesoporous region. The use of  $\text{ZnCl}_2$  as an activating agent should also be considered because it can be [9–14] and its use does not have certain limitations for environmental issues.

Chemical activation can be done on a commercial scale with zinc chloride [1], which can dehydrate and promote the breaking of glycosidic bonds.  $\text{ZnCl}_2$  removes hydrogen and oxygen atoms from the water in carbon materials but not from organic oxygen molecules, allowing pores to form and the carbon content to rise [15], as well as the production of OFGs. The use of hydrogen peroxide followed by hydrothermal activation of zinc chloride can yield mesoporous activated carbon with a large surface area [8]. Due to the increased available content of OFGs on the surface due to  $\text{H}_2\text{O}_2$  activation, zinc chloride activation is possible. In this study, using  $\text{H}_2\text{O}_2$  as an oxidising agent is expected to increase the affinity of  $\text{ZnCl}_2$  as an activating agent on the surface of the precursor, resulting in increased volume and mesoporous area.

Merbau wood (*Intsia, spp*) has previously been shown to have potential as a carbon precursor [16], [17] due to the presence of lignin, hemicellulose, and lignin in the sawdust [18]. The surface properties of the carbon are related to the relationship between OFG in the precursor as a functional attribute of the original material and the mesoporous area on the carbon. This study examines the hydrothermal preparation of carbonaceous materials with zinc chloride as an activating agent and hydrogen peroxide as an oxidative agent, followed by physicochemical activation in a furnace with an oxygen gas stream.

## 2. EXPERIMENTAL

### 2.1 Material

Carbon materials from waste wood samples of *Intsia, spp* were collected in Manokwari, Indonesia. *Intsia, spp* wood chips were dried at  $110^\circ\text{C}$ . Hydrothermal treatment is carried out before the carbonisation and oxidation process to promote the chemical activation of the sample. Merck's analytical grades include  $\text{NaOH}$ ,  $\text{NaHCO}_3$ ,  $\text{Na}_2\text{CO}_3$ ,  $\text{ZnCl}_2$ ,  $\text{H}_2\text{O}_2$  (30%), and  $\text{HCl}$ . A vertical stainless steel reactor is used for carbonisation and oxidation.

General procedure:

**Hydrothermal treatment preparation:** The wood chips were dried at  $105^\circ\text{C}$  for 24 hours before mashed into 3-5 mm samples with an industrial blender. A hydrothermal treatment with a mixture of wood chips and  $\text{H}_2\text{O}_2$  (10% by weight) in an autoclave at  $150^\circ\text{C}$  for 20 minutes. The product was dried at  $105^\circ\text{C}$  for 12 hours after the reactor temperature reached room temperature. In an autoclave, this product was combined with 37.5 mL of water and 11.25 g of  $\text{ZnCl}_2$ , and the reaction was performed at  $200^\circ\text{C}$  for 20 minutes.

**Carbonisation and oxidation:** The previous stage dry sample is fed into a stainless steel tube and placed in the furnace tube reactor. For 2 hours, at a rate of  $10^\circ\text{C}/\text{min}$ , a carbonisation temperature of  $700^\circ\text{C}$  was maintained under  $\text{N}_2$  gas at a flow rate of 20 mL/min, then was cooled to room temperature under nitrogen gas with a constant flow rate. After that, the oxidation process was continued at  $350^\circ\text{C}$  with oxygen flow and flow rate of 15 mL/min. Before the oxidation process, the hydrothermal treatment's product hierarchical carbon (HC) is recorded as HC-ZH, and the product of the non-hydrothermal/dry raw material following oxidation is recorded as HC. The sample HC created after oxidation is labelled HC-Ox, and the HC-ZH formed after oxidation is marked HC-ZH-Ox.

**Characterisation of carbonised samples:** The thermogravimetric method was used to determine the decomposition rate and temperature range of Merbau wood as a raw material. It was accomplished utilising a DTG60, which was used to heat 2.889 mg of sample from  $30^\circ\text{C}$  to  $600^\circ\text{C}$  at a rate of  $10^\circ\text{C}/\text{min}$ . 4.100 mg of material was processed in a DSC60 Plus with a 30 mL/min nitrogen flow rate.

The synthesised materials' X-ray diffractogram (XRD) was recorded using a Rigaku Miniflex600 (= 1,54, 40 kV, 15 mA). the carbonised materials' adsorption isotherm, BET surface area (SBET), and pore volume were determined by a gas sorption analyser, (SAA, Quantachrome NovaWin Series). The samples were degassed at 300°C for 3 hours before the nitrogen adsorption-desorption experiments.

An acidic site on the surface of porous carbons was explored by Boehm titration was used. The samples were combined with 0.05 M NaHCO<sub>3</sub>, Na<sub>2</sub>CO<sub>3</sub>, and NaOH reaction bases in the modified method [19]. The suspensions were stirred for 24 hours at room temperature using a magnetic stirrer, filtered, and 10 ml of the filtrate pipetted and titrated with 0.05 M HCl to determine the amount of acidic site [20-22]. The qualitative examination of test acidity using Boehm titration of (OFGs) and FT-IR spectroscopy to determine adsorption vapour base of ammonia.

Scanning electron microscopy was used to examine the texture of the carbonised samples (SEM, Jeoul JSM-6510). At a 15 kV accelerating voltage, carbonised Merbau woods were attached to carbon tape with a sputter-coated platinum layer on the sample surface.

### 3. RESULTS AND DISCUSSION

#### 3.1 TGA/DTA Curves of Merbau Woods

Lignin, cellulose, and hemicellulose are commonly found in lignocellulosic materials like Merbau wood. Under slow pyrolysis, the breakdown of hemicellulose and cellulose begins at 250°C and reaches a maximum mass loss at 400°C [23]. Others have found that lignin, cellulose, and hemicellulose decompose at 300-400°C, 180-240°C, and 230-310°C, respectively [24]. Lignin begins to degrade at low temperatures (160-170°C) and continues to do so slowly until it reaches 900°C. After that, they were followed by cellulose in a narrow temperature interval from about 200 to 400°C. The TGA and DTA curves of Merbau wood as a raw material are shown in Fig. 1. The carbonisation process was revealed to have three steps. At temperatures ranging from 30 to 170°C, the first stage of dehydration occurred. Water (such as free water and bound water) and light volatile components were eliminated, resulting in a weight reduction of only 9.21% [25]. The second stage occurs at 170-380°C, associated with hemicellulose breakdown and a weight loss of 46.63 %. At 380-521°C, 44.65% of the weight was lost due to cellulose and lignin pyrolysis. The breakdown of lignin at higher temperatures causes TGA/DTA curves to flatten. The findings were consistent with those of prior studies [26,27] however, the long flat tail found at high temperatures is thought to be due to lignin breakdown. It is commonly known that when the carbonisation temperature rises, the unstable volatiles on the carbon samples decreases. As a result, the weight losses of Merbau wood between 500 and 600 °C were greater than those at temperatures over 600°C. These findings support the theory that lignin is superior to cellulose and hemicellulose in the production of carbons [26].

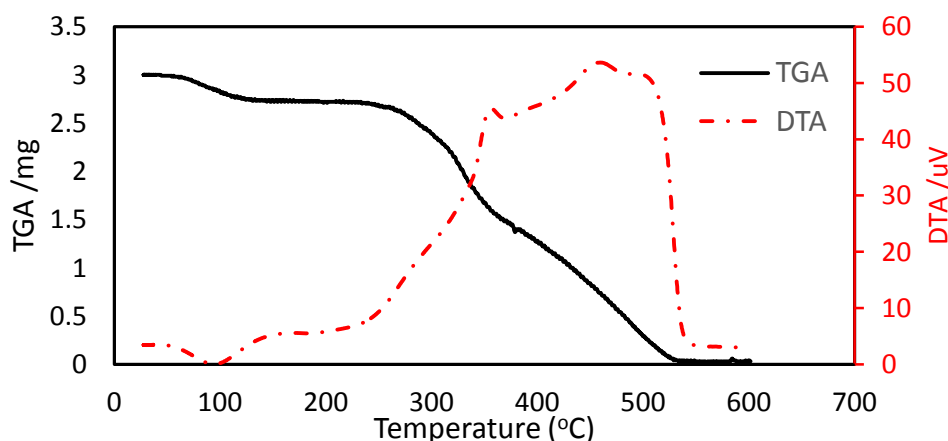
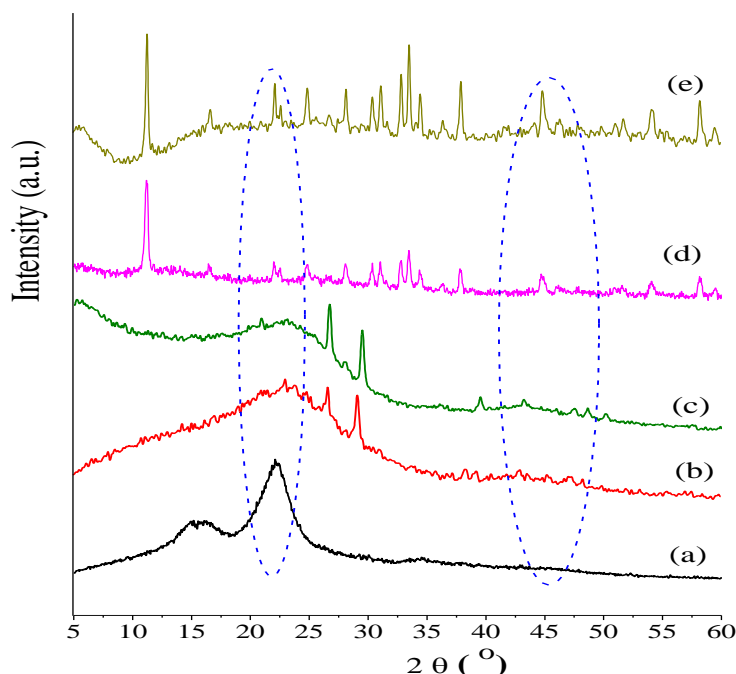


Fig. 1. TGA/DTA curves of Merbau woods



### 3.2 XRD Analysis

As seen by the XRD patterns, the carbonisation and hydrothermal processes modified the structure (Fig. 2). Three peaks were visible in the raw diffractogram, comparable to those described by Jain et al. [28]. This pattern matched the XRD pattern of microcrystalline cellulose with amorphous carbon. The designs of all carbonised and oxidised revealed two large peaks centred at  $2\theta = 23^\circ$  and  $43^\circ$ . These peaks, which correspond to the (002) and (101) reflection planes in turbostratic carbon structure [29], [30] suggested randomly arranged graphitic layers in the material associated with amorphous carbon.

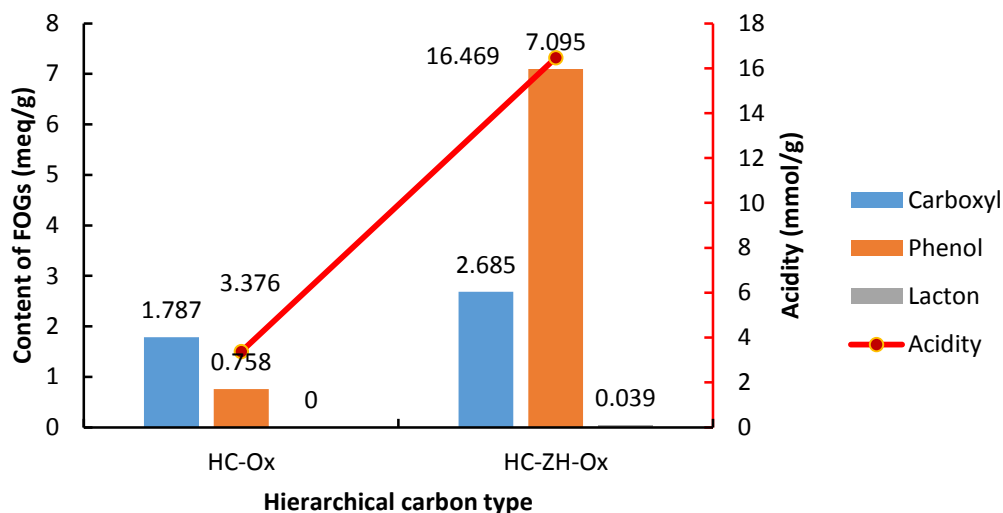


**Fig. 2. XRD patterns of raw of (a) Merbau woods, (b) HC, (c) HC-Ox, (d) HC-ZH, and (e) HC-ZH-Ox**

The structure of carbonised samples was changed by hydrothermal treatment of HC samples, as illustrated in Fig. 2. The two broad peaks detected in the HC and HC-Ox sample patterns did not appear in the HC-ZH and HC-ZH-Ox sample patterns. It could be explained by the influence of  $ZnCl_2$  and  $H_2O_2$  on cellulose structure defects, as seen by the decreased intensity of microcrystalline cellulose peaks.  $ZnCl_2$  easily disrupted the glycosidic connections, whereas  $H_2O_2$  as an oxidising agent converted the cellulose to oxy-cellulose, possibly due to the production of carboxylic groups[11].

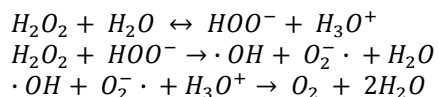
### 3.3 Analysis of FOGs by Boehm Titration Methods

Fig. 3 depicts the number of FOGs on carbonised materials after non-hydrothermal (HC-Ox) and hydrothermal (HC-ZH-Ox) treatments. The action of  $ZnCl_2$  as an activating agent and  $H_2O_2$  as an oxidising agent on the precursor surface increased the concentration of OFGs. In solution, coordinated water molecules operate as nucleophiles and  $Zn^{2+}$  as hydration shells [31]. These nucleophiles were attracted to the free electron pairs of the oxygen atoms in the OFGs, allowing more  $Zn^{2+}$  to reach the surface of the precursor, resulting in improved lignocellulosic biomass dehydration [8]. The presence of  $H_2O_2$  to trigger the production of OFGs on the precursor aided this advantage. The carbonised surface samples contained carboxylic, lactonic, and phenolic groups.



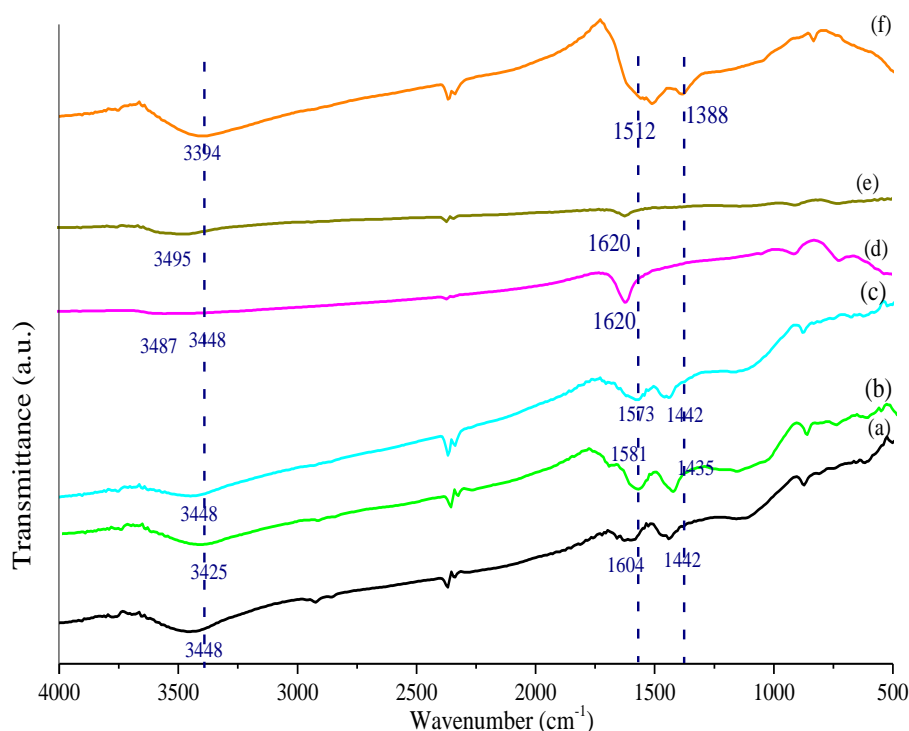
**Fig. 3. Acidic groups (FOGs) (meq/g) of HC-Ox and HC-ZH-Ox samples based on Boehm Titration method and the acidity (mmol/g) of the catalyst was determined gravimetrically using adsorption  $\text{NH}_3$  basic of gas**

Hydrogen peroxide generally dissociates into hydroperoxy anions ( $\text{HOO}^-$ ), then hydroperoxy anions can react again with undissociated  $\text{H}_2\text{O}_2$  to form highly reactive hydroxyl radicals ( $\cdot\text{OH}$ ) and superoxide ( $\text{O}_2^- \cdot$ ) [32]. The presence of hydroxyl radicals ( $\cdot\text{OH}$ ) and superoxide ( $\text{O}_2^- \cdot$ ) will cause degradation of lignocellulosic raw materials. Superoxide and hydroxyl radicals under acidic conditions can react and form oxygen and water.



### 3.4 Acidity Test Based on Ammonia Vapour Adsorption and FT-IR Analysis

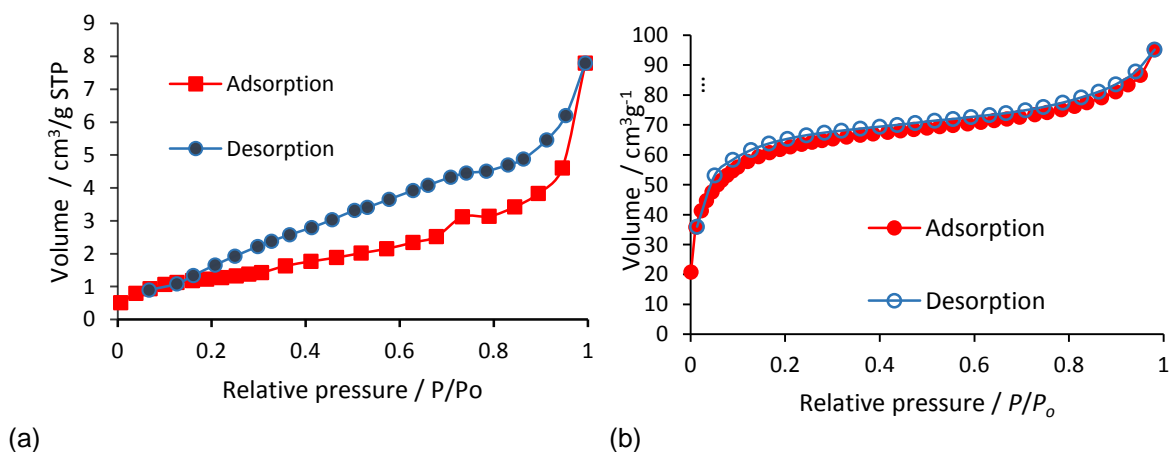
Ammonia vapour adsorption and FT-IR analysis are used to determine acidity. Hydroxyl and carboxylic groups were indicated as the primary surface OFGs. All of these had the following bands in their FT-IR spectra: 3495, 3487, 3448, 3425  $\text{cm}^{-1}$ , which corresponded to OH bond stretch. CH stretching was linked to vibrations between 3000 and 2810  $\text{cm}^{-1}$ . In this region, the spectrum of oxygenated carbonised materials reveals a low-intensity band. All the prepared AC samples showed peaks at the wavelength of 1525 1581, 1573, 1512, 1604 and 1620  $\text{cm}^{-1}$  matched to aromatic C=C stretching and  $\text{CH}_3$  C-H deformation [33]. Both hydrothermal and non-hydrothermal treatment of samples demonstrated a rising intensity band of  $\text{NH}_3$  oxidation and adsorption. Peaks observed at 1442 and 1435  $\text{cm}^{-1}$  in non-hydrothermal materials were associated with OH-groups of carboxylic acids and phenols [34]. Following the adsorption of  $\text{NH}_3$ , hydrothermal treatment samples showed a new band at 1388  $\text{cm}^{-1}$  and a broad absorption band at 3394  $\text{cm}^{-1}$ . The overtones of the N-H and O-H stretching vibrations are thought to be responsible for the band at 3394  $\text{cm}^{-1}$  [33]. The vibration frequency of  $\text{NH}_4^+$  chemically adsorbed on Brönsted acid sites is close to the peak at 1388  $\text{cm}^{-1}$  [35]. The first was the  $\text{NH}_3$  adsorption mechanism on carbonised samples, in which ammonia molecules diffused into the pore of the carbonised materials and were physically adsorbed at active locations on the surface. The molecules of adsorbed  $\text{NH}_3$  accept the proton to form ammonia complexions ( $\text{NH}_4^+$ ), so that act as the Brönsted acid from acidic neighbour groups [21].



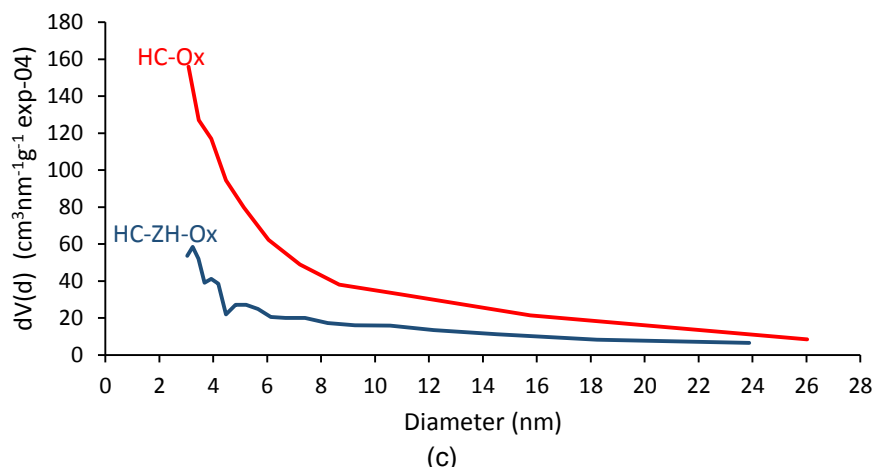
**Fig. 4. FT-IR spectra of (a) HC, (b) HC-Ox, (c) HC-Ox-Ammonia (after adsorbing NH<sub>3</sub>), (d) HC-ZH, (e) HC-ZH-Ox, and (f) HC-ZH-Ox-Ammonia(after adsorbing NH<sub>3</sub>)**

### 3.5 Surface Characteristics Analysis

Fig. 5(a) and (b) show carbon materials surface character analysis using the nitrogen adsorption-desorption isotherm for hydrothermal and non-hydrothermal treatments. Type IV isotherms with hysteresis III (H3) and hysteresis IV (H4) revealed the presence of mesoporous areas in HC-Ox and HC-ZH-Ox, respectively. Typical properties of a blend of micro and mesoporous materials are shown by the H3 and H4-type hysteresis loops in the relative pressure range of 0.15 to 1.0. The H3 type is distinguished by its large pores and gaps between parallel sheets [36]. The occurrence of sheet micropores is linked to Type H4 [37]. These two types of hysteresis are found in materials containing slit-shaped pores (in the case of H3) or plate-like particles (in the case of H3) [38].







**Fig. 5. Nitrogen adsorption-desorption isotherm of (a) HC-Ox, (b) HC-ZH-Ox, and (c) pore size distribution curves of HC-Ox and HC-ZH-Ox**

Table 1 presents that HC-Ox has a greater BET surface area of 443.5 m<sup>2</sup>/g than HC-ZH-Ox, which has a surface area of 232.2 m<sup>2</sup>/g. It is conceivable because hydrothermal activation promotes the creation of pores and the formation of additional micropores, resulting in a larger total volume and smaller pore diameter. Furthermore, high molecular weight volatile molecules may sinter with ZnCl<sub>2</sub> salt residues during the carbonisation process, resulting in depolymerisation of the molten carbonised sample. Some of the pores in the carbon structure may be closed by the intermediate melt. It corresponds to the decrease in pore diameter and surface area after hydrothermal treatment, shown in Table 1. The pore distribution curves of HC-Ox and HC-ZH-Ox also prove this phenomenon, as shown in Fig. 5. (c). Activated carbon derived from other natural precursors and different activators by previous researchers were tabulated in Table 1, showing varying porosity characters. IR10W and ML10W were synthesised from the Indian Rubber leaves, and Mint leaves using H<sub>3</sub>PO<sub>4</sub> solution as activator agent via impregnation method and temperature of pyrolysis at 500 °C. Based on these data, the surface area of all samples shows a similar trend. The difference seen is in the mean pore diameter. The IR10W and ML10W samples show material with micropores. In contrast, the HC-Ox and HC-ZH-Ox samples are mesoporous materials with a distribution indicating the presence of a mixture of mesoporous and slightly microporous areas, which follows the data in Fig. 5.

**Table 1. Characteristics of porosity**

Sample	D <sub>average</sub> (nm)	V <sub>tot.</sub> (cm <sup>3</sup> /g)	S <sub>BET</sub> (m <sup>2</sup> /g)	Reference
HC-Ox	5.437	0.010	443.50	Present study
HC-ZH-Ox	2.536	0.147	232.20	Present study
IR10W	1.177	0.082	278.49	[7]
ML10W	1.010	0.111	402.92	[7]

### 3.6 Morphology Analysis using SEM

SEM images of non-hydrothermal carbonised (HC) and hydrothermal (HC-ZH) samples (Fig. 6 c-d) before and after the oxidation process are shown in Fig. 6 (a-b). All of them have overlapping tunnels with stacked honeycomb-like structures, demonstrating that the raw material's morphology significantly impacts the carbonaceous material's morphology. The difference in a smoother surface and the development of a cleaner tunnel than without oxidation indicates the oxidation impact. The non-hydrothermal and hydrothermal samples show differences in the shape and thickness of the walls. It could be due to high molecular weight volatiles sintering with ZnCl<sub>2</sub> salt residues, causing depolymerisation during the carbonisation process.

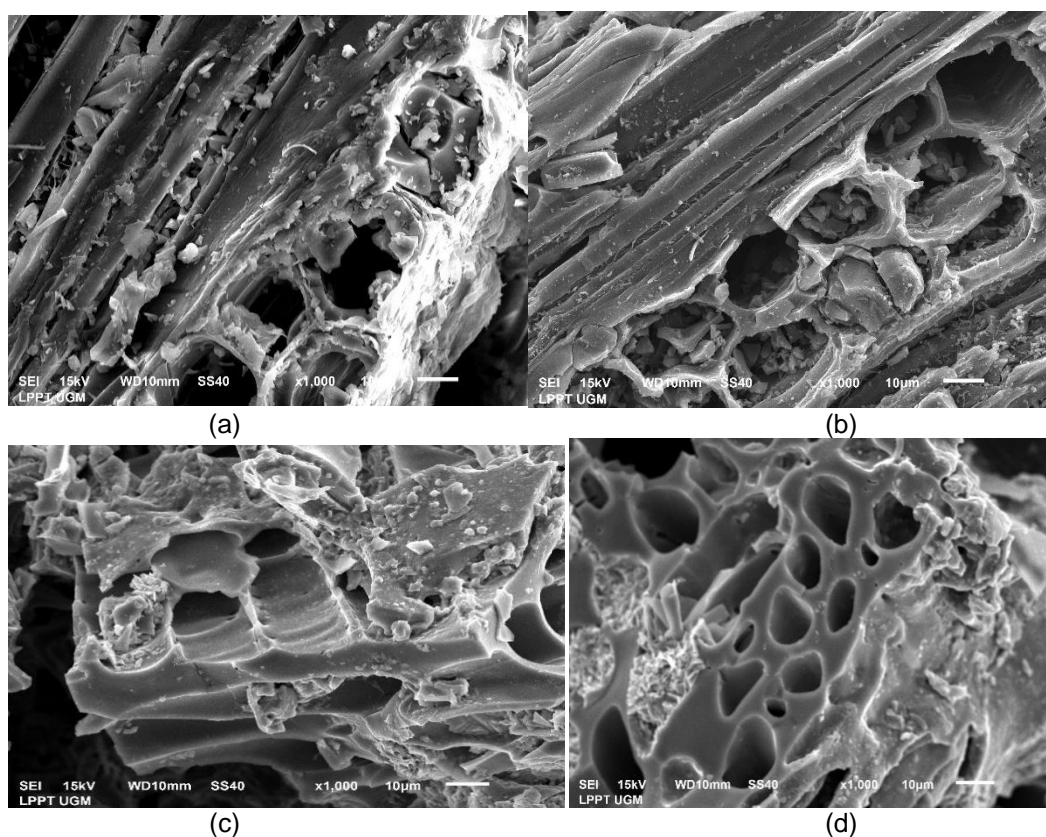


Fig. 6 SEM images of the (a) HC, (b) HC-Ox, (c) HC-ZH, and (d) HC-ZH-Ox

#### 4. CONCLUSIONS

Waste wood of *Intsia, spp* is treated with non-hydrothermal and hydrothermal methods to produce hierarchical carbon material. The stacked honeycomb-like structure of the hierarchical carbon produced has a large surface area with mixed mesoporous micropores. It demonstrates that the composition of the raw material largely determines the carbon substance's form. The surface area and diameter of the sample were reduced as a result of the hydrothermal treatment. The intermediate melt formed by the depolymerisation of  $ZnCl_2$  can block some of the pores in the carbon structure, and the high molecular weight volatiles can do the same. The thickness and shape of the hierarchical carbon wall structure alter as a result of hydrothermal treatment.

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#### COMPETING INTERESTS

Authors have declared that no competing interests exist.

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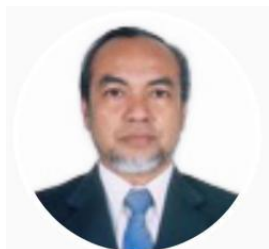
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