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Evaluation to Possibility of Cellulose Fibers Isolated from Kapok Randu (Ceiba Pentandra) as A Precursor Growth of Carbon Micro Structures with The Assist of Metal Catalysts Abstract Cellulose fibers that contain hydrocarbon substances potentially could be used as precursor in synthesizing carbon micro-structure (CMTs). Due to the differences of fibers particularly in physical properties, **this study aims to** investigate the potential use of cellulosic material from kapok randu as a precursor in synthesizing carbon-based structures.

The isolation of cellulose was carried out via alkaline treatment, followed by mechanical disintegration. Meanwhile, the growth of CMTs was performed via heating treatment for 12 hours with various catalysts (i.e., Fe, Ni, and Cu). Cellulosic material obtained was assessed by using FTIR spectrum, while the morphological characteristic was done by using SEM images that depicted rectangular shapes. The thermal characteristic suggested that the decomposition was initiated at 300°C. The FTIR results **confirmed the presence of** functional groups in accordance to cellulose fiber, such as -OH (3418 cm<sup>-1</sup>), C-H aliphatic group (2900 cm<sup>-1</sup>), O-H (1635 cm<sup>-1</sup>), and C-H (1334 cm<sup>-1</sup>).

Whilst, the FTIR pattern also **confirmed the presence of** C=C stretching at 1600 cm<sup>-1</sup>, -CH in between 2800-3000 cm<sup>-1</sup>, indicating the activated carbon. Raman shift indicated the growth on G-band in the interval of 1500-1700 cm<sup>-1</sup>, suggesting the presence of C-C structures. Based on the morphological characteristics, the growth of CMTs were successfully obtained with different diameters and lengths due to the different catalysts used.

The iron (Fe) catalysts produced CMTs with a diameter about 200 nm and average length of 2-3 μm, whereas the Ni catalysts formed tubes with around 150 nm of length

and 50 nm of diameter. Meanwhile, the Cu catalysts formed amorphous particles with diameter below 10 nm. From these results, the evaluation of cellulose isolated from kapok randu as a precursor in the growth of carbon micro-tube with distinguished characteristics was demonstrated. Keywords: nanofibers cellulose, activated carbon, kapok randu, precursor, carbon based-structures Introduction The discovery of carbon-based nanostructures, such as carbon fibers, carbon nanotubes (CNTs), carbon microtubes (CMTs), and carbon nanodots, appears to be promising alternatives for practical applications.

In term of CNTs and CMTs, the structures have similarities such as hollow, tubular, and single- or multi- walled which only differs in the size, and since their discoveries (Ijima, 1991), they have been attracting scientists due to its characteristics such as high mechanical and strength properties as reinforcement material, excellent electricity and magnetism as storage-energy material. Ever since the discovery, another attempt to produce similar carbon-based structures, such as CMTs is carried out. Both CNTs and CMTs were fabricated by applying certain temperature of treatments which most of them were in high temperatures with high pressure condition.

Therefore, the synthesis of CMTs is influenced by three factors such as temperature, and to reduce the high temperature, the latter factors are catalysts and the carbon sources. Chemical vapor deposition (CVD), once was able to provide carbon onion(Kang et al., 2008) and multi-walled(Balogh et al., 2008) structures in CNTs, however, the precursors for synthesizing have become a challenge, which commonly is organic compounds such as methane, methanol and acetone(Janas, 2020).

Materials consisted of carbons were the most suitable precursors for synthesizing CNTs and CMTs, including hydrocarbon compounds such as methane(Kang et al., 2008) and ethanol(Kakehi et al., 2008). The drawbacks in using these two types of precursors require high temperatures of treatments even though its gaseous form reduces time reaction. Nonetheless, gaseous phases are not the only substances that could be used as precursors as a study had successfully synthesized CNTs from solid materials(Singh et al., 2002).

On the other hand, the needs of carbon microtubes (CMT) for several applications are in needs such as for oil adsorption(Zhao et al., 2019a), and batteries and energy storage(X. Huang et al., 2012; Salahdin et al., 2022), which are synthesized from solid compounds. Hence, finding strategies via availability of precursors and selective catalyst is promising advantages for future use. In term of availability, organic wastes from plantations provide advantages due to their availability of abundant material in carbon content, such as cellulose.

This organic material is commonly found in plants, for instance, angel's trumpet plant contains high variation of acidic compounds which also can be precursors for synthesizing carbon-based materials(Mokbli et al., 2021). Furthermore, In Indonesia, cellulose is organic waste that is annually produced by the plantations, including empty bunch of palm oil(Gea et al., 2020b), and even more a study utilized part of palm oil tree which is the kernel shell as precursor for bifunctional catalyst(Abdullah et al., 2020). Due to its structure with biomass elements, cellulose has been used as biofuel(Panneerselvam et al.,

2016), suggesting its high carbon content. Other reports have also successfully carbon nano- and micro-structures with mesoporous morphological features from paddy rice(Hao et al., 2019), poplar-catkin(F. Huang et al., 2021), and plant tissues(Zhao et al., 2019b). Another potential plants that can be a precursor is kapok randu, which is a native plant to Indonesia. The seed is covered by cellulosic fiber, which is similar to the cellulose from cotton plant. Cellulose fibers from kapok randu were reported to be used in the synthesis of activated carbon(Chung et al., 2013), subsequently; cellulose-fiber from this plan can be used as the precursors in synthesizing CMTs.

Attempts in synthesizing carbon-based microstructures seems to be high-cost due to the use of catalysts in order to overcome the non-steady phase of carbonization. Several reports have suggested the introduction of catalysts, such as silver nanoparticles (Gea et al., 2022) which is considered as high-cost. Whilst, the use of mono-(Co) combined with bimetallic (Fe-Co) catalysts during CVD synthesis is considered as complex strategies which also takes place in high energy(Balogh et al., 2008; Kakehi et al., 2008).

Although several reports have used transitional-metals (TiC, NiCl<sub>2</sub>, SnO<sub>2</sub>) and non-metal catalysts (Sulfur) with more controllable reaction, these show limited selection in finding affordable and available catalysts for synthesizing with low-cost aspects(Anil Kumar et al., 2022; Ariyanto et al., 2019; F. Huang et al., 2021). Hence, the purpose of this present work was to evaluate the possibility of cellulose fibers isolated from kapok randu to be used as the precursor for the synthesis carbon-based structures with affordable catalysts such as Fe, Ni, and Cu.

The heating treatments were applied along with the presence of metal catalysts supports. Materials and Methods Materials The Kapok Fibers (KFs) were collected from the fruit, in which the tree was located in the sub-district Tanjung Mulia, Deli Serdang Regency, Medan, Indonesia. The chemical reagents such as HCl, NaOH, H<sub>2</sub>O<sub>2</sub>, H<sub>2</sub>SO<sub>4</sub>, H<sub>3</sub>PO<sub>4</sub>, NaOCl, Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>, NaNO<sub>3</sub> and acetone were purchased from Sigma Aldrich Inc. Meanwhile, the metal catalysts, such as FeSO<sub>4</sub>.7H<sub>2</sub>O, NiSO<sub>4</sub>.H<sub>2</sub>O, CuSO<sub>4</sub>.5H<sub>2</sub>O were

supplied by Bratachem.

The gasses utilized to control the condition including nitrogen, methane, hydrogen and helium, were supplied by PT Aneka Gas. Preparation of Cellulose Kapok Fibers The amount KF that was from the fruit was separated manually via man-labor. Then, these amounts of fibers were dried directly under daylight, and from these amounts, 75 g of KFs were cut into 2-3 cm of sizes. The fibers were immersed in 1 liter of 3.5% HNO<sub>3</sub> and 10 mg NaNO<sub>3</sub> mixture for 2 hours at 90°C. Then, the mixture was filtered and the fibers were washed by using distilled water until the neutral pH was achieved.

Next, the fibers were soaked into 750 ml of 2% w/v NaOH and 2% Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution for 1 hour at 50°C, and followed by washing and filtrating processes. Afterward, these KF samples were bleached with 250 ml of 1.75% of NaOCl solution until its temperature boiled for 30 minutes. After being bleached, the samples were mixed with 500 mL of 17.5% NaOH for 30 minutes at 50°C to obtain cellulosic sample. Then, this cellulosic sample was washed by using distilled water, in which hereafter, 10% of H<sub>2</sub>O<sub>2</sub> were used to immerse the samples for 30 minutes at 60°C.

The cellulosic sample then was filtered and dried in an oven at 60°C to remove the residual water content, which was followed by storing it in a desiccator. Isolation of Nanofiber Cellulose Kapok Fibers The first step in isolating nanofiber cellulose (NFC) from KFs began by acid hydrolysis treatment, which was previously described in several studies (Gea, Panindia, et al., 2018b; Gea, Zulfahmi, et al., 2018; Zulham Efendi Sinaga et al., 2018). The cellulosic fibers were soaked in 45% H<sub>2</sub>SO<sub>4</sub> with ratio of 1:25 w/v% for 45 minutes at 45°C.

Then, into the mixture, some bi-distillation water with ratio 1:25 v/v% were added and the mixture was allowed to stand for 12 hours at room temperature to form suspension. The suspension was separated and washed to reach pH 7. Next, this mixture sample was placed in an ultrasonic bath for 3 hours, and then followed by homogenization step for 3 hours at 8000 rpm. Finally, these nano-cellulosic kapok fibers (NCKFs) were heated at 50°C in an oven to remove water content, and the dried samples of NCKFs were obtained which hereafter is stored in desiccator.

Thermal Gravimetric Analysis The characterization of TGA was performed to determine as basis of later heating treatments for the growth of CMT. The TGA was carried out via TGA DTG-60 in which the thermal rate was 10°C.min<sup>-1</sup> in nitrogen condition (flow rate 30 ml.min<sup>-1</sup>). The initial temperature was 27°C, and final temperature was 600°C, whereas the starting mass of the NCKFs was 3 mg. Synthesis of Activated Carbon from NCKFs Thermal Gravimetric Analysis (TGA) was used to obtain the decomposition

temperature.

The decomposition process started at 320°C based on the TGA result, so that the NCKFs were heated inside a furnace at 400°C for two hours to produce carbon. Afterward, the carbon samples were obtained, and the chemical activation were performed by immersing these carbon samples into 1M H<sub>3</sub>PO<sub>4</sub> for 90 minutes with ratio 1:10 w/v%. Then, this mixture was filtered, and the filtered carbon samples were dried at 150°C for 24 hours. Afterwards, 5N HCl was added to the dried carbon samples to perform reactivation.

Thus, the removal of excessive chloride ions was done by washing these dried carbon samples with distilled water to reach pH 7, and followed by additional washing with cool distilled water as well as filtering to remove the residual of phosphate anion. After being washed and filtered, the wet carbon samples were dried at 150°C for 24, and finally this sample was named after AC Synthesis of Carbon Microtubes from AC with Various Catalysts

#### The Preparation of Catalyst- Solutions

The preparation of 0.09 M Cu(NO<sub>3</sub>)<sub>2</sub>, 0.09 M Ni(NO<sub>3</sub>)<sub>2</sub>, and 0.09 M FeCl<sub>3</sub> catalyst solutions was carried out by respectively dissolving 1.08 g of Cu(NO<sub>3</sub>)<sub>2</sub>·3H<sub>2</sub>O, 1.3 g of Ni(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O, and 1.21 g of FeCl<sub>3</sub>·6H<sub>2</sub>O in acetone. Then, each solution was homogenized with constant stirring.

#### The Impregnation of Ni/Cu/Fe Catalysts

The preparation of catalysts for CMTs was performed by mixing 0.09 M Cu(NO<sub>3</sub>)<sub>2</sub>, 0.09 M Ni(NO<sub>3</sub>)<sub>2</sub>, and 0.09 M FeCl<sub>3</sub> with AC and ratio of 1:10 w/v%. Then, each of the mixture underwent ultrasonication for 2 hours at 70°C. The results of the impregnation samples were dried in oven for 12 hours at 70°C.

#### The Growth of Carbon Microtubes (CMTs)

An amount of impregnated AC samples was placed in a 25 ml porcelain dish into a gas furnace. On the surface of the dish, the end of furnace ceramic pipe was connected to gas source.

During the heating process, the dish was covered to prevent small carbon particles from escaping. The first step was calcination process, which was done by streaming the impregnated AC with heat at 500°C for two hours under inert conditions (nitrogen gas 100 ml.min<sup>-1</sup>). The second stage was a reduction process, where the temperature of 700°C and hydrogen gas (with flow of 60 ml.min<sup>-1</sup>) were applied for two hours. In this process, metal oxides would be removed and the metals were converted into metal nanoparticles.

In the third stage, the temperature in the reactor was increased to 950°C, and followed by the increase of nitrogen gas rate to 100 ml.min<sup>-1</sup>. When the reactor reached the set temperature, the nitrogen gas rate was increased to 200 ml.min<sup>-1</sup>. The next stage was the flowing of mixture methane and nitrogen gas which respectively 1:2 ratio (rate of

100 ml.min<sup>-1</sup>) for two hours at 950°C. Then, at the final process, Helium gas was flowed at 60 ml.min<sup>-1</sup> into the furnace, thus; the temperature within it would drop to room temperature. Inert condition during the final process was important in order to prevent the destruction of CMTs.

Characterization of Carbon Microtubes (CMTs) In this research, CMTs were analyzed by Transmission Electron Microscopy (TEM) instrument (JEM-1400) with acceleration voltage 120 kV. The photograph obtained from TEM was analyzed to observe the length and diameter of the tubes via Image-J application, as well as the structures of CMTs, which were different, depends on the catalyst used. Results and Discussion FTIR and Morphological Characteristics of NCKF The NCKF were analyzed by **Fourier Transform Infrared (FTIR)** instrument. Characterization by FTIR was done to confirm the functional groups in NCKF in Figure 1. Figure 1.

FTIR Pattern of Cellulose Fibers Isolated from Kapok Randu The FTIR pattern shows that the functional groups of the NCKF were the same as cellulose fibers. The -OH functional group was shown at peak 3418 cm<sup>-1</sup> with stretching vibration up to 2900 cm<sup>-1</sup>, whereas, the C-H aliphatic group was at 2900 cm<sup>-1</sup>. It could also be clearly seen that the O-H group related to carboxylate group was at 1635 cm<sup>-1</sup>. The bending vibrations of H-C-H, O-C-H, and C-H, and rocking vibration of -CH<sub>2</sub> were respectively at 1427 cm<sup>-1</sup>, 1373 cm<sup>-1</sup>, and 1334 cm<sup>-1</sup>, which these three vibrations were in C<sub>6</sub> glucose chain. The Figure 2 shows the morphological of NCKF. Figure 2.

SEM Photographic Image of NCKF (a) 250 times magnification, and (b) 100 times of magnification Figure 2 shows morphological images by SEM of NCKF obtained with 250 and 100 times of magnification. Unlike those obtained by previous studies, the NCKFs had different shapes from palm oil bunches and corncobs(Gea, Panindia, et al., 2018a; Zulham Efendi Sinaga et al., 2018). Although generally, kapok randu is one of tropical tress with fruits containing cellulosic fibers, its cellulosic material has been reported to be hydrophobic-oleophilic(J. R. Wang et al., 2018).

Hence, it is assumed that the thermal properties of NCKF were different due to its utilization as a precursor in the synthesis of CMTs. Thermal Gravimetric Analysis of NCKF As the NCKFs was used as the precursor to synthesize CMTs, TGA analysis was performed to determine the temperature breakdown. The Figure 3 depicts the comparison of TGA analysis of NCKFs Figure 3. TGA Analysis of NCKFs According to Figure 3, the initial temperature started from 27°C to 600°C, and the initial mass of NCKFs was 3 mg with 10°C.min<sup>-1</sup> of heating rate.

The significant observation can be seen at the temperature above 300°C as the NCKFs

started to decompose and reached 60% of mass change. Although the NCKFs differs to other biomass, this thermal analysis has confirmed similar data to what have been reported in several studies (Gea et al., 2018; Soykeabkaew et al., 2012). The region of initial and ending composition were above 300°C and 600°C respectively (Gea et al., 2020a). As it has been reported by several studies, around 32–47% cellulose was isolated from various raw materials (Gea et al.,

2018, 2020a; Gea, Panindia, et al., 2018a; Marpongahtun et al., 2018; SUN, 2004), where its physical and chemical structures may have been different from one to another due to different alkaline treatments. The use of sodium hydroxide in cellulose isolation has been concluded to provide distinguishing impacts on the morphological structures, including the stiffness and orientation of the fibrils (Chakraborty et al., 2011). FTIR Spectra of Activated Carbon (AC) from NCKF Figure 4. FTIR Spectra of Activated Carbon from NCKF In this study, the AC was obtained from NCKFs, and in Figure 2 and 3, the -OH, C-H aliphatic, and -CH<sub>2</sub> were confirmed.

However, the spectrum of AC in Figure 4 showed different results, particularly in the presence of new groups and the occurrence of reduction. The groups, such as C=C, C-O, and P=OOH was confirmed due to the treatments with H<sub>3</sub>PO<sub>4</sub>. This could occur as the surface of biomass was carbon-derived (Oginni et al., 2019), which were indicated the presence of C=C stretching band around 1600 cm<sup>-1</sup>, -CH stretching band in interval of 2800–3000 cm<sup>-1</sup>, and -P=OO<sub>4</sub> in 1100 cm<sup>-1</sup> (Xu et al., 2014). Figure 5. XRD pattern of activated carbon with SEM image of activated carbon (inset) Subsequently, the confirmation of activated carbon was also performed to ensure the success synthesis.

The following Figure 5 demonstrates the XRD result of sample activated carbon. In overall, two broad peaks were noticeable observed in 40–50° and 60–70° which is related to (002) [(Xu et al., 2014)]. It also can be observed that amorphous parts of activated carbons alongside, in which it could have attributed to the random stacking of layers, that were also noted from the SEM photographic images in Figure 5 (inset). TEM Analysis of CMTs from NCKFs Figure 6. TEM Photographic Images of Prepared Samples.

a) Commercial CNT; b) CNT activated carbon catalyst/Fe 6 hours; c, c\*) CNT activated carbon catalyst/Cu 11 hours; d) CNT commercial active catalyst/Ni 11 hours; e) CNT activated carbon catalyst/Ni 11 hours Figure 6 shows significant differences of TEM photographic images in each sample synthesized from different catalysts. The commercial AC samples were with diameter of 45–50 nm (Figure 6a). The image also showed metal nanoparticles attached to the cap of the CMTs with a mean tube length of 600 nm. Meanwhile, the sample with AC synthesized from NCKF with Cu catalyst for 11 hours had tube diameter and length of 50 nm and 100 nm respectively (Figure 6c).

In Figure 6c\*, with samples made of Cu catalyst, produced mostly spherical amorphous carbon particles with sizes of under 10 nm. Meanwhile, the AC from NCKF with Ni catalyst had tube diameter and length about 40-50 nm (Figure 6d). The first variation was sample of AC from NCKF with Fe catalyst treated for 6 hours to produce CMTs with diameters of 200 nm with the average tube length of 2-3 micrometer (Figure 6b). Then, the second variation was commercial AC with Ni catalyst treated for 11 hours, produced CMTs with diameters of 50 nm and a tube length of 150 nm (Figure 6e).

This happened due to **the solubility of carbon in metal** particles, which would form solid filaments alongside with the width of the diameters (Duc Vu Quyen et al., 2019). Another report has shown that the decreasing of diameter of CMTs from kapok randu for almost a half (from 20 $\mu$ m to ~12 $\mu$ m) after carbonization at the temperature of 500-1000°C (Zhao et al., 2019a). Figure 6a and 6b display tubular structure even though rough shapes were found. Therefore, high temperature used could reduce catalyst activities to bind the carbons in CMTs arrangement during the reaction, such as direct calcination at 500°C (J.

Wang et al., 2018). Due to the use of the metal catalysts which implied to the uncontrollable graphitization reaction (Ariyanto et al., 2019), the reaction caused the particles to agglomerate to each other, so that the reaction results tended to lead the formation of amorphous carbon particles with a size below 10 nm (A. Ahmad et al., 2018) as it is shown in Figure 6. In general, the synthesis reaction of CNTs via CVD was carried out over a time span of 30-60 minutes **for the growth of** CNTs by using precursors (Costa et al., 2008; Ramírez Rodríguez et al., 2018).

Several studies also mentioned that the process could take longer time (Fathy, 2017; Li et al., 2009). In this study, the results obtained based on TEM analysis showed a small amount of pile up (especially based on Figure 6a and b), which was assumed to occur due to thermal treatments. However, the tubes produced in this study were seen to be consistent compared to previous reports (S. Ahmad et al., 2019; Duc Vu Quyen et al., 2019).

The optimum growth of CMTs was 30-60 minutes, **more than 60 minutes** would not produce more CMTs as the surfaces of catalyst would have been overgrown with CMTs. In conclusion, the use of a longer time with the same temperature would reduce the yield of CMTs as previously reported (S. Ahmad et al., 2019). Raman Spectra of Prepared Samples from AC-NCKF The above Figure 7 is the Raman shifts of pyrolysis sample of AC from NCKF with different catalysts, i.e., the impregnation of Ni, Fe, and Cu.

The shifting could be clearly seen in the interval of 1500-1700  $\text{cm}^{-1}$ , indicating the first order of G band as well as implying the presence of C-C structures. Among three of them, sample 1 and 2 had the higher intensity, and also, in Figure 7B, the sample 2 which was treated with Cu as the catalyst, had higher intensity counts than that in sample 1 that was treated by Fe as the catalyst. The presence of this peak has confirmed the successful **growth of single-wall carbon nanotube** (SWCNT); as per what other studies have also reported the shifting 1500-1700  $\text{cm}^{-1}$  both in red and green laser (respectively 785 and 514 nm)(Costa et al.,

2008; Li et al., 2009). Figure 7. Raman Shifts of CMT Sample from kapok randu (a). The comparison of sample 1 (catalyst Ni), sample 2 (catalyst Fe), and sample 3 (catalyst Cu); (b). Specific Shifting for Sample 1 (Ni) and Sample 2 (Fe) Metal catalysts, mono-metals in particular, could affect the structural rearrangement structure of the carbonaceous materials(S. Ahmad et al., 2019). As this study only focused in the use of mono-metals, the selection of metals was based on the precursor, which is the cellulosic material(S. Ahmad et al., 2019; Duc Vu Quyen et al., 2019).

Both TEM and Raman results confirmed the differences in each growth samples in terms of the sample intensities as well as the length of the tubes. Copper (Cu) catalysts had amorphous structures, which were both proven by sample 3 in Raman shift results (Figure 7a). These results were in accordance to the studies which utilized Fe and Ni catalysts, producing long-shape tube growths via CVD method(Arjmand et al., 2016).

Meanwhile, another research reported Ni catalyst has successfully synthesized long-shape tubes with 10-40 nm diameters via microwave treatments(Burakova et al., 2018). Although this study investigated that sample Cu and Ni which are based on Figure 6a, 6b, and 6d, the growth of the tubes via AC from NCKF, nevertheless, have been successfully obtained. Conclusion The fibrous material from kapok randu that contains cellulose has the potential to be precursors for CMTs synthesis from activated carbon as the basis of growth.

By using various catalysts, such as iron, nickel and copper, the growth of CMTs has been successfully obtained via thermal treatments with the introduction metal catalysts of Fe, Ni, and Cu at moderate temperature (700-950°C) even though longer graphitization takes time. Based on the results, the size diameter of the CMTs were in between 50-200 nm with length diameter of less than 2  $\mu\text{m}$  in average, as the growth is observed in the micrograph results. The highest presence of CMTs' growth was observed in sample with nickel (Ni) catalyst, and the Raman shifts appears to show the growth with considerably peaks.

Thus, further investigation related to the physical parameters of catalysts and graphitization reaction in moderate temperature is required. Acknowledgement The authors would like to thank the rector of Universitas Sumatera Utara as its grants via TALENTA scheme program with given contract No. 2590/UN.5.1.R/PPM/2018.

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