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Green simple microwave-assisted extraction (MAE) of cellulose from *Theobroma cacao* L. (TCL) husk

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Abstract. This work had successfully produced Theobroma cacao L. (TCL) husk-based cellulose used efficiently and friendly environmentally simple microwave-assisted extraction (MAE) method. The microwave-assisted heating time optimization of alkaline-treatment was carried out for 10, 20, 30 and 40 minutes. Microwave-assisted bleaching process was also conducted using green bleaching agent H_2O_2, and also utilizing microwave heating for 60 minutes. The followed treatment was filtered, neutralized, washed and overnight freeze-dried. Fourier transform infrared (FTIR) analysis confirmed common cellulose functional groups of TLC, are β-(1,4)-glycosidic bonds at ~897 cm^{-1}, O-H (hydrogen bond) at 3412 cm^{-1}, C-H vibration at 2902 cm^{-1}, C-O-C asymmetric at 1161 cm^{-1}, and C-OH out-of-plane bending at 665 cm^{-1}. FTIR analysis, in addition, also studied crystallinity ratio (*CrR*), hydrogen bond energy (E_H) and hydrogen bond distances (R_H), while particle size analyzer (PSA), X-ray diffraction (XRD) and thermogravimetry analysis (TGA) were carried out to confirm particles sizes, crystallinities and thermal stability properties of TCL husk, cellulose of TCL husk and commercial-microcrystalline cellulose (MCC), respectively.

1. Introduction

Searching and utilization of cellulose in industrial and research activities are the answer to issue in reducing environmentally unfriendly fossil fuels. Cellulose is appealing ones due to easily and costly obtained, renewable, biodegradable, biocompatible, good mechanical strength, and good thermal stability properties [1].

Cellulose is the most abundant and renewable biopolymer in nature. Cellulose is a main component of plants, strengthening the cell wall [2], forming one-third to one-half of plants tissue and photosynthesis reproduced [3]. Biosynthetically, cellulose is as much as 10^{11} tons produced every year [4]. In its chemical structure, cellulose is a homopolymer composed of units of D-glucopyranose bound by β (1 → 4) glycoside bonds [5].

Cellulose is widely used in industries such as papermaking, food as stabilizer, anti-caking agents, suspending agents and packaging, pharmaceutical applications as a binders, absorbents, flowability and innovative composite applications [6]. Natural cellulose fibers are used as a reinforcing material in thermoset and thermoplastic cellulose matrix especially glass fibers due to unique characteristics, such as renewability, low density and high specific strength [3].

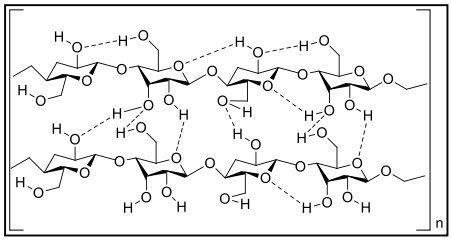

Figure 1. Supramolecular hydrogen bond of cellulose [7].

A reason to be widely applied in some applications such as an alternative to synthetic materials of electrochemical devices caused cellulose has excellent characteristics, are strength and modulus, large surface area, high aspect ratio, high strength, and stiffness, inexpensive, lightweight, recyclable and biodegradable [6]. Cellulose material has excellent properties and meets green friendly environmentally and sustainable requirements of lithium-ion batteries (LIBs) separator membranes compare with others [8,9], such as polyethylene (PE), polypropylene (PP), polyethylene oxide (PEO), polyvinyl alcohol (PVA) [10], and polyvinylidene fluoride (PVDF) [11]. Several reports have been published as a source of cellulose such as sugarcane bagasse [12,13], seaweed [6], durian rind [3], pineapple peel [14], cassava peel [15], and others [16].

Indonesia is the third highest *Theobroma cacao* L. (TCL)-producing country in the world, after Ghana and Republic of Cote d'Ivoire. In Indonesia TCL is often popular called as *kakao*, *cocoa* and *buah* (*pohon*) *cokelat* [17]. Every year Indonesia produces 800,000 tons of TCL, and contribute 16% of world TCL production. TCL has 73-75% husk, 2-3% placenta and 22-24% seed. TCL husk contains 32-45% crude fiber in the form of lignin and cellulose. Unfortunately, TCL husk waste was only used as animal feed [18,19]. However, there were not many reports to explore lignocellulose of TCL husk for last two decades.

Cellulose extraction consists of two important stages, commonly, are an alkaline treatment (NaOH/KOH) and bleaching process (bleaching agent: NaClO/ $NaOCl_2$). Conventional alkaline treatment aims to break bonds between lignin and holocellulose, to obtain lignin, hemicellulose, and cellulose. The alkaline treatment using strong bases such as NaOH and KOH requires neutralizing process the pH of the extracted product. The filtration process is then followed by a process of washing with excess distilled water, then using acetic acid in neutralizing the extraction product. This product is usually called holocellulose. Holocellulose consists of hemicellulose and cellulose. Bleaching process aims to separate the cellulose-bonded hemicellulose, and also to make the product brighter (white). The bleaching agent which usually used is acidified sodium chlorite ($NaOCl_2$) after alkaline treatment in breaking lignin completely. The conventional cellulose extraction long takes 4-6 hours interval for alkaline treatment and then the bleaching process needs 1-2 hours [3,12,13,15].

This study utilized a new method *green simple microwave-assisted extrac*tion (MAE), applied of electromagnetic radiation to a material [20], which can make more efficient so that cellulose extraction process can be done in the short duration (between 10-60 minutes). MAE is an interesting technique due to able it can accelerate biomaterials isolation method [21]. Microwave irradiation can reduce the dewaxing process in cellulose extraction. Microwave irradiation increases heating perfectly to alkaline, bleaching, and hydrolysis treatment. And also, it needs environmentally friendly hydrogen peroxide (H_2O_2) bleaching agent to replace $NaClO_2$ [6].

A microwave is a form of electromagnetic energy which exists in the electromagnetic spectrum (300-300,000 MHz). There are two basic mechanisms of energy transfer of microwaves in the matter, through dipole rotation and ionic conduction. Dipole rotation is the interaction between polar molecules as a result of the rapidly changing electric field of microwaves. The ion conduction mechanism includes a momentary superheating of ionic substances due to the ion motion generated by an electric field. As temperatures increase, energy transfer becomes more efficient [22].

Previous studies stated that microwave irradiation convert electromagnet heating into energy, a familiar method to organic synthesis with many advantages such short duration time, need small amount of solvent and produce high yields [23]. Santos [24] and Smiderle [23] described the effectiveness of microwave-assisted extraction of cellulose. Likewise, in synthesizing cellulose derivatives, the use of

microwave heating is practically highly preferred. Biswas [25] reported that alkyl cellulose (methyl cellulose and ethyl cellulose) have been successfully synthesized using microwave-assisted heating.

Based on reasons above, so this work studied and developed a green simple microwave-assisted extraction of cellulose from TCL husk. We investigated effect of alkaline treatment with various time using this method, continued bleaching process. Bleaching product, as cellulose product, was characterized with Fourier transform infrared (FTIR), particle size analyzer (PSA), x-ray diffraction (XRD) and thermogravimetry analysis (TGA), and this result was compared with TCL husk and commercial microcrystalline cellulose (MCC).

2. Experiment

2.1. Material

TCL (*Theobroma cacao L.*) husk was obtained from Hilisimaetano Village (Lahusa Subdistrict, South Nias Regency, Province of North Sumatera, Indonesia), hydrogen peroxide (H_2O_2), sodium hydroxide (NaOH), distilled water and ethanol were commercially obtained from Merck.

2.2. Characterization Instrument

Functional groups of samples were carried out with fourier transform infra-red, FTIR (type: Prestige 21 Shimadzu), crystallinities of samples were carried out with polymer electrolytes membranes were carried out with diffractometer (type XRD Rigaku Smartlab), particle size of sample was measured with PSA Delsa™ Nano C (Merk Beckman Coulter), morphology images of samples was carried out with SEM instrument, Type JEOL-JSM-6510LV, and thermal analysis of samples were carried out with thermal gravimetry analysis (TGA, merk Linseis, type STA PT 1600).

3. Methods

3.1. Klason lignin content

Klason lignin was investigated using Viera *et al*.-developed method [26], with some modifications. A 1 gram of TCL husk was subjected into round bottomed flask 100 mL, and then added 7.5 mL sulfuric acid 72% under stirring in the room temperature for 1 hour. After previous treatment, the solution was diluted with 280 mL distilled water. In addition, the system was stand for residue precipitation, next it was filtered dan washed with 250 mL distilled water. The product was dried in the vacuum freeze-dry, weighed and analyzed by FTIR.

3.2. Holocellulose content

Holocellulose was also investigated using Viera *et al*.-developed method [26], with some modification. 2.5 g of TCL husk was added to 125 mL beaker, containing 0.75 g of sodium chlorate ($NaClO_3$), 0. 5 mL glacial acetic acid, 50 mL of distilled water was added and the collision was agitated until chlorate homogenized. The mixture was than heated and stirred at 75°C for 1 h. Each 1 hour for a total duration time of 3 h, the reagents added into beaker containing mixture. The mixture was subjected into ice water and next filtered. The bright product was washed with ethanol and dried in the vacuum freeze dry, weighed and FTIR analyzed.

3.3. Extraction of cellulose

The method of cellulose extraction in this study refers to a method developed by Santos *et al*. [24] and Singh *et al*. [6] with some modification. The ratio of TCL husk and 2% NaOH solution is 1: 20 (m/v). Microwave CEM reactor settings were power: 100 watts, temperature: 50°C, stirring: medium, time variations: 10, 20, 30 and 40 minutes. The product of the NaOH treatment, separated from the mixture with 2% NaOH, was neutralized with aquadest, followed by ethanol. The separated product was dried in vacuum freeze dry and then weighed.

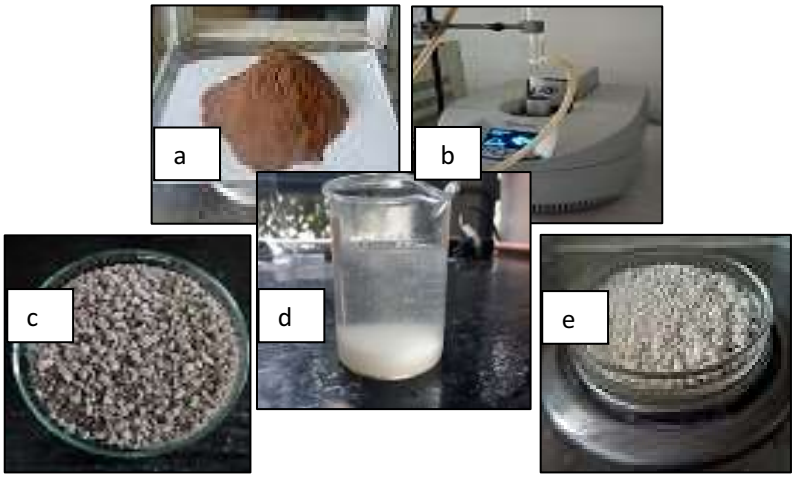

Figure 2. (a) ground TCL husk; (b) microwave-assisted alkaline treatment; (c) product of alkaline treatment; (d) after bleaching process; (e) cellulose product.

Alkaline treated products were then bleached by green bleaching agent H_2O_2 using heating with microwave. Microwave CEM reactor settings were power: 100 watts, temperature: 50°C, stirring: medium, time: 1 hour. The bleaching treatment product, separated from the mixture using a glass filter, was then neutralized with distilled water until the odor of the bleaching agent removed, then again washed with ethanol, so that the bleaching product can be terminated by water disturbance. The filtered product was then dried in freeze dry and after drying it was milled again and weighed.

3.4. Yields calculation

Weight of both alkaline-treatment and bleaching process product were calculated using formulation below.

$$\%\ yields = \frac{weight\ of\ product}{weight\ of\ sample} x\ 100\ \% \quad (1)$$

Weight of product = weight dried product (alkaline treatment/bleaching process)
Weight of samples = weight of TCL husk initially.

3.5. Crystallinity Index (C.I) Calculation

C.I calculation used a Segal *et al.*-proposed formulation [27,28].

$$C.I. = \frac{I_{200} - I_{am}}{I_{200}} x\ 100\% \quad (2)$$

where I_{200} = maximum intensity of lattice diffraction; I_{am} = amorph region of diffraction intensity.

3.6. Crystallinity rasio (CrR) calculation

$$CrR = \frac{A_{1372}}{I_{2900}} \quad (3)$$

where A_{1372} is absorption peak around 1372 cm^{-1} and A_{2900} is absorption peak around 2900 cm^{-1}.

3.7. Hydrogen bond energy (E_H)

$$E_H = \frac{1}{k}\left[\ \frac{(v_0 - v)}{v_0}\ \right] x\ 100 \quad (4)$$

where v_0 is standard wave number relate to free OH group (3650), v is wave number of bonded and observed -OH group to cellulose samples FTIR spectra, dan k is constant (1/k = 2.625 x 10^2 kJ).

3.8. Hydogen bond distances (R_H)

$$\Delta v(cm^{-1}) = 4430\ x\ (2.84 - R_H) \quad (5)$$

$\Delta v = v_0 - v$, v_0 is OH^- stretching wave number at 3600 cm^{-1} and v is observed-OH stretching wave number to cellulose samples FTIR spectra.

4. Results and Discussion

The microwave-assisted extraction method of cellulose from TCL husk meets green chemistry principle. Designing energy efficiency is mainly principle approved in this method [29]]. Short time duration of cellulose isolation is promising for many applications. A hydrogen peroxide (H_2O_2) was bleaching agent also fulfill one of green chemistry principle, that is designing safer chemicals [29], if we compare to common bleaching agent of sodium (hypo)chlorite ($NaOCl/NaOCl_2$) which not friendly to environment.

4.1. FTIR analysis

Theobroma cacao L. (TCL) husk is one of most lignocellulose biomass, mainly comprised of lignin and holocellulose. TCL husk component analysis is very important employed quantitively. In this work, klason lignin and holocellulose extraction were also carried out. Fig 4 shows the differences of the main functional groups FTIR absorption of lignin and holocellulose, whereas Table 1 performs their main functional groups FTIR absorption differences compare to isolated cellulose product. The wavenumber of 1521.81 and 1253.73 cm^{-1} are lignin-typical peaks, C=C stretching of aromatic ring and C-O stretching ether linkage. Those bands are disappeared on holocellulose and cellulose (see figure 3).

Differences of TCL husk to cellulose by FTIR analysis, are mainly shown to wave number at 898 (commercial MCC) and 896.89 (~897) cm^{-1} (bleaching product of NaOH-treatment). The wave number is a C-O-C bond between the anhydroglucose unit (AGU), known as β- (1.4) -glycosidic bonds. We concluded that the bleaching product of NaOH-treatment is isolated cellulose product.

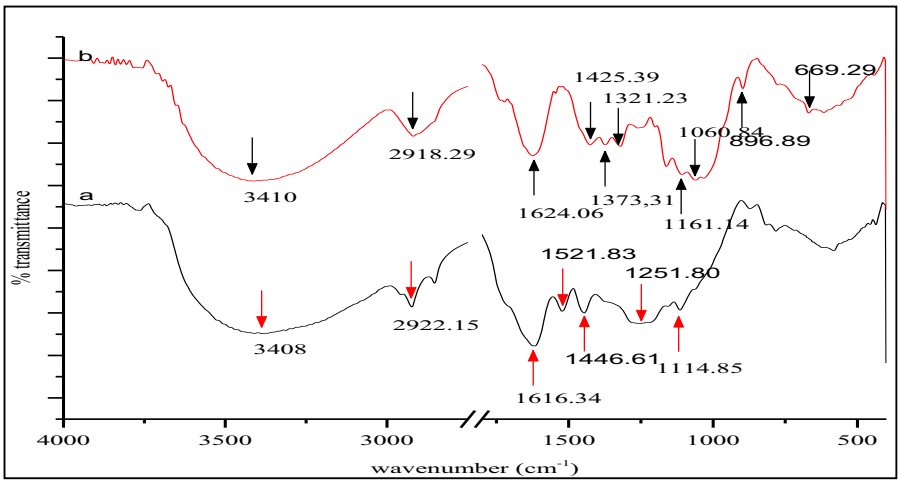

Figure 3. FTIR spectra: (a) klason lignin; (b) holocellulose.

Holocellulose and cellulose have significantly similar of FTIR absorption, but they are some different on wavenumber shift. Holocellulose and cellulose product expose the β- (1.4) -glycosidic bonds absorption at 896.89 (~897) cm^{-1}.

The complete assignment of main functional groups absorption of lignin, holocellulose and cellulose are depicted in Table 1 below.

Table 1. Assignment of main functional groups absorption of lignin, holocellulose and cellulose.

Functional group absorption	Klason lignin	Holocellulose	Cellulose product of TCL husk
OH stretching	3408	3410	3412
CH stretching	2922.15	2918.29	2902
H-O-H bending of absorbed water	1616.34	1624.06	1641
C=C stretching of aromatic ring (lignin)	1521.83	-	-
CH_2 bending	1446.61	1425.39	1431
C-H deformation	-	1373.31	1373
C-O stretching of ether linkage	1251.80	-	-
C-O-C antisymmetric bridge stretching	-	1161.14	1161-1163
C-O symmetric stretching of primary alcohol	-	1060.84	1080.91
β- (1.4) -glycosidic bonds	-	896.89	896.89 (~897)

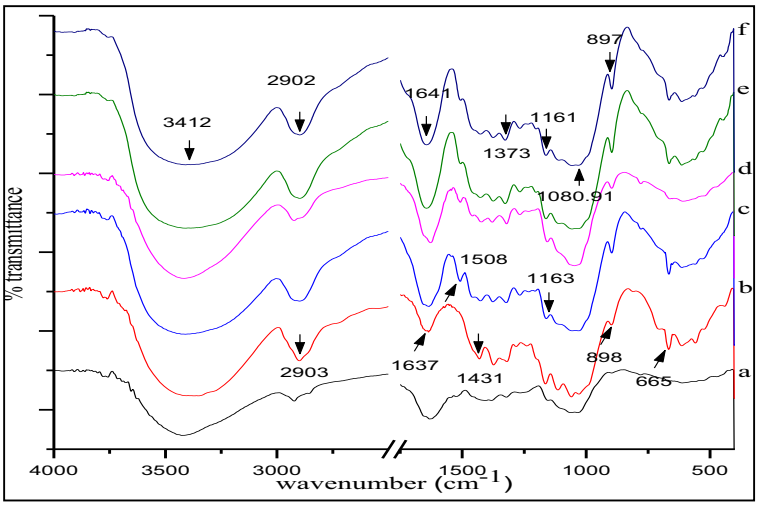

Figure 4. FTIR spectra: (a) TCL husk; (b) commercial MCC; (c) bleaching product of NaOH-treatment at 10 minutes; (d) bleaching product of NaOH-treatment at 20 minutes; (e) bleaching product of NaOH-treatment at 30 minutes; (f) bleaching product of NaOH-treatment at 40 minutes.

The time optimization of the NaOH-treatment produces different yields. Table 2 shows the yields of the isolated cellulose product with time optimization. The longer the NaOH-treatment, the lower the product yields. This is because of the amount of lignin and hemicellulose attached to cellulose decreases. In figure 3, it can be explained that existence of lignin is appeared at 1508 cm^{-1} on the duration of NaOH-treatment to the TCL husk at 10 minutes and 20 minutes. However, the typical peak of the lignin is decreased, even disappeared in the NaOH-treatment time of 30 minutes and 40 minutes. With considering its purity and yields, so bleaching product of NaOH-treatment time of 30 minutes that was next followed to analysis.

Table 2. NaOH 2%-treatment product and TCL husk bleaching yields (setting Microwave reactor: Power = 100-watt, T = 50°C, stir = average).

Samples	Long time of NaOH-treatment	% weight of NaOH-treatment product	% weight of bleaching process product	Typical lignin group (~1500 cm^{-1})
Sample 1	10 minutes	79.450	35.044	appeared
Sample 2	20 minutes	73.150	31.180	appeared
Sample 3	30 minutes	68.080	25.220	disappeared
Sample 4	40 minutes	65.240	11.810	disappeared

Based on the obtained results, the use of cellulose extraction methods from TCL husk with microwave heating compared to conventional extraction methods (reflux with heating using hotplate) is more efficient especially in the use of time and temperature of heating. A very short duration of time in the treatment of NaOH with the best condition of 30 minutes, greatly accelerates the process of propagation, further work, and other analyzes. While based on several previous reports, the process of treatment of NaOH needed time duration between 4 and 6 hours.

Table 3. Crystallinity ratio (CrR), hydrogen bond energy (E_H) and hydrogen bond ranges (R_H).

Samples	CrR	E_H (kJ)	R_H (A°)
commercial MCC	1.082	17.25	2.797
Sample 1	1.010	17.25	2.797
Sample 2	1.031	16.97	2.798
Sample 3	1.048	17.25	2.797
Sample 4	1.048	17.25	2.797

Some absorption peaks can be used to study the polymorphic types of cellulose samples. The absorption peaks around 1420, 1160, 1110 and 890 cm^{-1} can be used to study cellulosic polymorphic types. From the analysis of these wavelength numbers, cellulose of TCL husk is identified as cellulose I. It is exposed that wavenumber shifts around 1420 to 1431 for all conditions, confirmed to trend of cellulose I, whereas cellulose II content is further decreased [2,30]. Similarly, the absorption peak around 890 will shift to about 896 cm^{-1} confirming the presence of cellulose I, which is not possessed by TCL husk.

Calculation of CrR depict that commercial MCC crystallinity as shown in table 3 is higher that all samples. While among of cellulose products of TCL, the bleaching products of alkaline-treatment for 30-40 minutes have higher CrR than the bleaching products of alkaline-treatment for 10-20 minutes. E_H and R_H calculation confirm that similarity of all samples, indicating that cellulose properties are displayed.

4.2. *Particle size analyzer* (PSA) *and Scanning Electromotive Microscopy* (SEM*)*

Based on particle size analysis data, we obtained mean diameter of isolated cellulose product of TCL is 2,439.7 nm (~2.4397 μm). The particles were expected as micrometer-sized, and not as nanometer-sized.

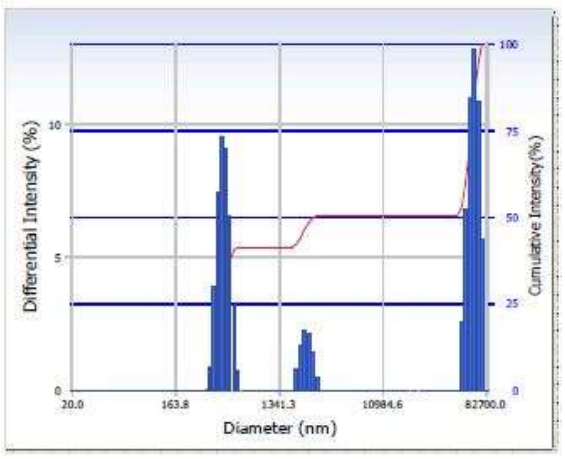

Figure 5. Intensity distribution of isolated cellulose product particle size.

From the histogram above, depicted that there are three peaks of distribution results at 431.2, 2306.9 and 69147.1 nm.

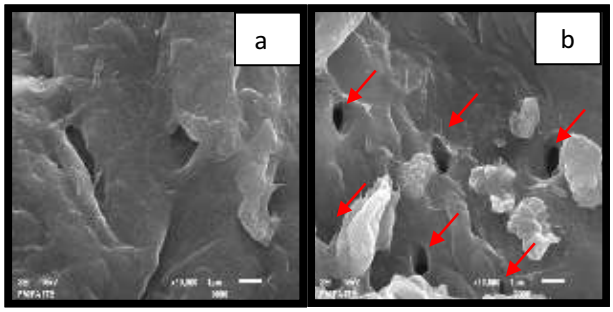

Figure 6. SEM analysis of (a) Commercial MCC and (b) Isolated cellulose of TCL husk.

Scanning electromotive microscopy (SEM) analysis was performed to both commercial MCC and isolated cellulose of TCL husk for 10,000x. Figure 6 explains the different of morphology appearance of the samples. Commercial MCC shows dense morphology than isolated cellulose of TCL husk. Some pores of isolated cellulose of TCL husk are depicted by arrow in figure 6b. Pores presence confirm crystallinity index of isolated cellulose of TCL husk decrease, it means increase amorph region as effect of molecular weight decreasing, if we compare to commercial MCC. Molecular weight decreasing weakens hydrogen bond interaction intra- and interchain of cellulose polymer chain.

4.3. XRD analysis

Figure 7 below shows X-ray diffraction patterns for each sample (a) TCL husk, (b) isolated cellulose and (c) commercial MCC cellulose respectively. The commercial MCC X-ray diffraction pattern shows three major peaks located at 2θ: 22.48; 14.29 and 34.36. The X-ray diffraction pattern for cellulose of TCL husk shows a similar pattern a commercial MCC. The cellulose of TCL husk shows the main peaks at shift 2θ: 22.40; 15.20 and 34.7.

The X-ray diffraction pattern of the commercial MCC is appeared having a strong intensity with three major peaks, indicating the crystal structure. While the cellulose of TCL husk is appeared weaker intensity and with the wide area at the shift of the main peaks, indicating lower crystallinity than commercial MCC. XRD pattern of TCL husk shows amorphous structure, there is no strong peak appeared. Crystallinity Index (*C.I.*) calculation of TCL husk, isolated cellulose, and microcrystalline cellulose are shown in Table 4 below. The calculation refers to *Equation* 2.

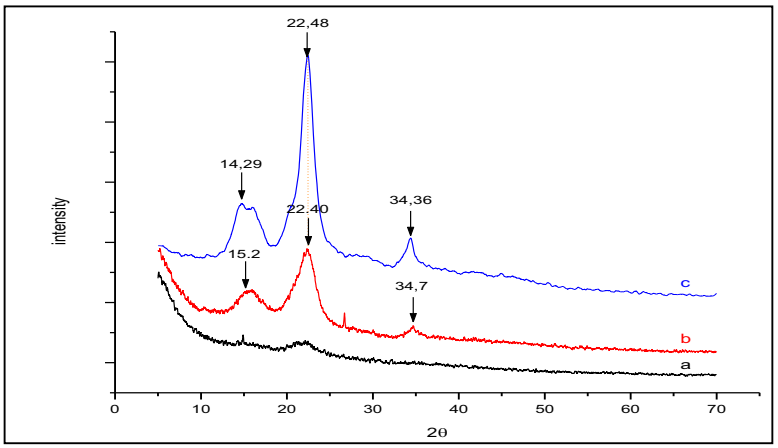

Figure 7. X-ray Diffraction Pattern: (a) TCL husk; (b) cellulose product dan (c) commercial MCC.

Table 4. Crystallinity Index of Samples.

Sample	I_{200}	I_{am}	*C.I. (%)*
TCL Husk	-	-	-
Cellulose product of TCL husk	335	199.36	40.48
Commercial MCC	1029	320	68.90

We didn't calculate *C.I. (%)* of TCL husk, because it's totally amorphous structure, no crystalline peak appeared. TCL husk's amorphous fraction is contribution of lignin content. While, the low crystallinity index, *C.I. (%)* of cellulose of TCL husk compared with commercial MCC, is predicted caused of lower molecular weight. However, this prediction needs some studies forward. The low molecular weight cellulose from biopolymer provides advantages, fortunately, such as cellulose is more easily modified. This statement is similar to previous report (Hutomo, et al. In press) and also confirmed by preceding SEM analysis. The low molecular weight indicates hydrogen bonding inter/intra-chain weak that relate to crystallinity index, *C.I. (%)* of cellulose of TCL husk reducing.

The XRD pattern provides information of crystalline allomorphs structure of cellulose. There are four crystalline allomorph are celluloses I, II, III and IV [28]. As we know, the cellulose XRD pattern of the cellulose of TCL husk is significant similar to commercial MCC, refers to cellulose-I allomorph structure. It approves that the product is native cellulose, an abundant biopolymer in nature.

4.4. TGA analysis

The thermal stability of cellulose products from TCL husk was analyzed by Thermogravimetry analysis (TGA). The following thermogram shows the differences in thermal stability of the sample. Based on Moran's report [31], hemicellulose and cellulose degradation began at temperature intervals of 220 and 250 °C respectively, whereas lignin was degraded at lower temperatures, at 200 °C. At higher temperatures, heat-resistant lignin is higher than hemicellulose and cellulose, due to the slow rate of degradation. Weight loss between 200 and 300 °C is primarily due to decomposition of hemicellulose and slow parallel decomposition of lignin, whereas weight loss between 250 and 500°C is caused by cellulose decomposition (250-350°C) and lignin (200-500°C). At temperatures higher than 400 °C, oxidation and breakdown of residues take place into gas products with lower molecular weight [24].

According to Trache *et al*. [16], degradation processes of cellulose, commonly, consist of both releasing of water (60-140°C) and dehydration, decarboxylation, depolymerization and decomposition of glycosyl unit of cellulose (250-450°C). Water releasing of polysaccharide is detected with a little weight loss appeared for samples TGA thermogram [32]. From TGA thermogram above showed that TCL husk exhibits higher weight than commercial MCC and cellulose product of TCL husk indicating TCL husk absorbed higher water than another both samples.

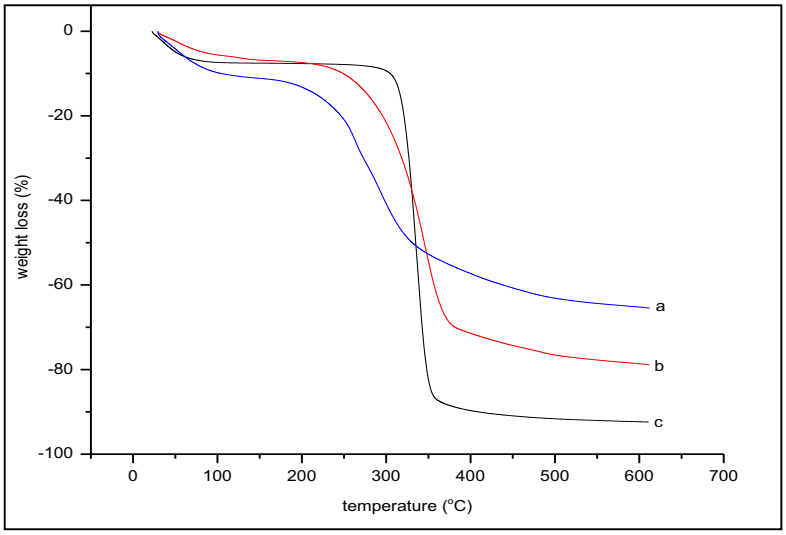

Figure 8. Thermogram of: (a) TCL husk; (b) Cellulose product of TCL husk and (c) commercial MCC.

Table 5. Initial and final decomposition temperatures of samples

Samples	Initial decomposition temperature (°C)	Weight loss of initial (%)	Final decomposition temperature (°C)	Weight loss at final (%)	Weight remains (%)
TCL HUSK	203.5	13.35	332.9	50.72	49.28
Commercial MCC	308.0	9.7	352.6	86.60	13.40
Cellulose product of TCL husk	248.7	9.8	375.5	69.60	30.40

By comparing the obtained data from the table 5, can be depicted that isolated cellulose products from TCL husk have good thermal stability compared to their original TCL husk. The weight loss of isolated cellulose product occurs at higher temperature temperatures (248.7 °C) while TCL husk first undergoes weight loss at lower temperatures (203.5°C). The mass that is lost too rapidly on TCL husk is the component of lignin and hemicellulose, whereas in the first weight loss of isolated cellulosic unit is first estimated from the cellulose itself.

When compared with MCC, the thermal stability is better compared with the isolated cellulose. This is due to the molecular weight of MCC is much greater than that of isolated cellulose from TCL husk. From the above data has been obtained information that the mass remaining to temperatures above 600°C. As mentioned before, after temperatures above 400 °C, there is a decrease in the decomposition rate of lignin. Consequences of the rate, then the largest residual gain is on TCL husk is 49.28% whereas on the cellulose of TCL husk and the commercial MCC are lower at 30.40% and 13.40% respectively. The high char residue of TCL husk showing no significant lignin component remains else degrade at the final decomposition and the next. Cellulose of TCL husk and commercial MCC, however, are stable at the high temperature, just remain lower residue of char.

5. Conclusion

Green simple microwave-assisted extraction (MAE) of cellulose from TCL husk had succeeded to employ short duration of alkaline-treatment, with best condition of 30 minutes. Applying of low concentration of alkaline solution (NaOH 2%) and H_2O_2 as a bleaching agent indicated that the extraction method was greener compared to conventionally extraction methods. Designing energy efficiency and chemical safer are mainly approved of green chemistry principle from the MAE method. Delignification of TCL husk had produced lignin and hemicellulose-free cellulose product, which depicted to FTIR peaks absorption differences. The cellulose products of TCL husk were confirmed having pure cellulose-typical important groups by FTIR analysis, they are –OH group at 3000, CH_2/CH_3 group at 2902, C-O-C group at 1161-1163 and β-(1,4)-glycosidic bond at ~897 cm^{-1}. PSA analysis shows that isolated cellulose of TCL husk has average sizes 2,439.7 nm (~2.4397 μm). Morphology study depicts some pores of isolated cellulose of TCL husk, while commercial MCC have dense morphology and tends has no pores. XRD analysis indicated that cellulose product has cellulose-I crystalline structure with 2θ: at 21.89; 14.20 dan 34.64 and has crystallinity index ~40.48%. Cellulose product of TCL husk has good thermal stability, with decomposition temperature between 248.7 and 375.5°C, which was analyzed using TGA.

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