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Properties of Cellulose Extract from Different Types of Oil Palm Biomass

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Abstract. This study focuses on the characteristics of cellulose extracts from different oil palm biomass namely oil palm mesocarp fiber (OPMF), oil palm empty fruit bunch (OPEFB) and oil palm frond (OPF). Cellulose was obtained from these biomass through soda pulping process followed by a totally chlorine free (TCF) bleaching step using peracetic acid solution. It was found that the cellulose extract for OPMF, OPEFB and OPF had 88.8%, 93.0% and 94.6% purity. Further analyses on the properties of cellulose in terms of morphology, thermal stability and crystallinity were conducted. The OPF cellulose obtained after pretreatment exhibited the highest crystallinity at 84.5%, followed by OPEFB (82.4%) and OPMF (69.2%). This directly affected the thermal stability of the celluloses, in which OPF cellulose was more thermally stable compared to celluloses from OPEFB and OPMF, as being recorded by a thermogravimetry analyzer (TGA). Results from this study provide new insight not only for fundamental understanding, but also to forecast the potential applications of cellulose extracts from different type of oil palm biomass, based on the performance of the celluloses reported herein.

1. Introduction

Oil palm or namely as *Elaeis guineensis* was early introduced in central of West Africa and its production was expanded worldwide in many countries due to the acceptance in global market. Malaysia and Indonesia have the largest area for oil palm plantation and are the biggest producer of palm oil in the world, mainly due to the favorable agro ecological zone which contributes to the high yield of oil palm [1-2]. Currently, the uses of oil palm can be seen in food and non-food applications. The large amount of palm oil being produced is in parallel with the high production of biomass, either from the palm oil mill or plantation. About 80 million tons of oil palm biomass is being generated in Malaysia annually [3].
Various types of biomass can be obtained from the oil palm plantation and palm oil mill, such as oil palm mesocarp fiber (OPMF), oil palm empty fruit bunches (OPEFB), oil palm fronds (OPF), oil palm trunks (OPT), leaves and roots. For example, the OPF will be cut during pruning seasons for harvesting oil palm fresh fruit bunches. It has been reported that the OPF tend to increase its production due to frond pruning before the age of 13 years [4]. OPF represents the major oil palm biomass from plantation area [5]. At present, oil palm biomass are mainly used for fuel generation, biocomposting and mulching.

It has been reported that cellulose represents about 1.5 x 10^{12} tons of the annual biomass production [6]. In biomass, cellulose is accompanied by other components such as hemicellulose, lignin and small amount of extractives. This structure is known as lignocellulosic materials which exhibit by its architectural of biological origin. Complex carbohydrate in cellulose composed by chain of polymer with unbranched β (1, 4) linked ᴅ glucopyranosyl units. There are intramolecular hydrogen bonds which are responsible for rigidity and stiffness of cellulose polymer, and bring the strong interaction between cellulose chains. This layer with anhydroglucopyranose unit causes the cellulose chain to held together by weak van der Waals forces [7]. The cellulose can form in both crystalline and amorphous structure by it placement at plant cell wall. For hemicellulose, it tends to have amorphous structure due to its long branches of polymer chain. Various monomeric sugars are found in hemicellulose such as glucose, galactose, mannose, xylose, arabinose and rhamnose. Hemicellulose represents about 25-30% in plant cell walls and comprises a mixture of a polysaccharide chain with low molecular weight as compared to cellulose. Whereas, lignin is a complex polymer in aromatic ring or refer as phenolic monomers. Phenol is very stable organic molecule benzene with an alcohol functional group. The main building alcohol of lignin are p-coumaryl alcohol, coniferyl alcohol and sinapyl alcohol [8].

Cellulose has become of interest in product development for commercialization purpose such as in paper making, composite production, textiles, pharmaceutical products, cosmetics and other advanced materials. Nevertheless, cellulose from different bioresources may have different characteristics, and the potential applications of cellulose from various bioresources could actually be expanded if the characteristics are well studied. To the best of our knowledge, there is lack of detailed study on the characteristics of cellulose obtained from oil palm biomass. Since there is abundant of oil palm biomass in Malaysia and they are readily available, it is important to have a set of data on the characteristics of cellulose obtained from different oil palm biomass in order to forecast the actual potential applications of each biomass. In this study, cellulose obtained from pre-treated OPMF, OPEFB and OPF were characterized for their chemical, morphological, thermal stability and crystallinity properties. Results obtained showed variation, suggesting the potential applications of these materials could be varied.

2. Experimental

2.1 Materials
Three types of biomass were used namely oil palm empty fruit bunch (OPEFB), oil palm mesocarp fiber (OPMF) and oil palm frond (OPF). OPEFB and OPMF were collected from Seri Ulu Langat Palm Oil Mill, Dengkil Selangor, Malaysia. OPF was obtained from Taman Pertanian Universiti (TPU), Universiti Putra Malaysia. OPF was shredded and pressed in order to remove its juice. Afterwards, all samples were thoroughly washed with tap water and sun dried to lower its moisture content in order to avoid fungal growth in samples. The dry samples were kept in sealed plastic bag and stored at room temperature prior to further treatment.

2.2 Chemical pulping
Prior to pulping process, the size of the biomass samples was standardized by cutting it into 1-2 cm in length. Pulping was conducted in a 15L batch reactor of lab-scale twin digester (model GTD-15L, GIST Co Ltd., Korea). Sodium hydroxide solution (14% w/v) was used. Fiber samples were mixed with the
alkaline solution at ratio of 1:4 and the pulping process took place at 160°C with pressure at about 0.5 MPa. The condition for soda pulping was modified from [9]. Pulping process was conducted for 1 – 1.5 hour. After cooling, the obtained cellulose pulp was washed several times using tap water until the pH became neutral. The pulp was spun to remove water and dried at 60°C overnight.

2.3 Pulp bleaching

Dried cellulose pulp was subjected to bleaching process using a totally chlorine free (TCF) method. Peracetic acid (PA) solution was prepared by mixing 50% acetic acid and 30% hydrogen peroxide at equal volume ratio (1:1 v/v) with addition of catalyst, i.e. 98% sulphuric acid (H₂SO₄) at concentration of 1.1% (w/v) (modified from [10]). The pure PA solution was allowed to stand for 3 days prior to bleaching process. For bleaching, dried cellulose pulp samples were mixed with distilled water and PA solution at ratio 1:5:5 and vigorously stirred for 24 hours at 70°C. Afterwards, the solid residues were collected and washed several times with tap water until the pH became neutral. The obtained cellulose samples were dried at 60 °C overnight. After the samples were cooled, they were kept in an airtight container until further use.

2.4 Chemical composition analysis

Samples were analyzed for their chemical composition using a Standard Test Method of TAPPI (Technical Association of Pulp & Paper Industry). The analysis was conducted to characterize the composition of untreated and treated fibers based on following TAPPI standard of cellulose (T-203cm-99), holocellulose (Tappi useful Method 249-75), lignin (T-222om-88) and ethanol-toluene extractives (T-204cm-97). The chemical analysis was done in replicates to obtain the mean and standard deviation values.

2.5 Morphology

Surface morphology of cellulose was observed using a scanning electron microscope (SEM), JCM-6000PLUS NeoScope Benchtop SEM (JEOL Ltd., Japan). Firstly, the samples were coated with Pt using Hitachi Ion Sputter E-1030 and the magnifying view was at 500x -1000x magnification with 15kV of beam voltage. Diameter size of cellulose was analyzed using a polarized microscope.

2.6 Thermal stability

Thermal stability of samples was analyzed using a thermogravimetry analyzer (TGA) model EXSTAR6000 (Hitachi High-Tech Science Corporation, Tokyo, Japan). TG and DTG thermograms were obtained by conducting the TG analysis at temperature range in between 50 °C to 550 °C with heating rate of 10 °C/min. The analysis required pure nitrogen as the inert gas which was purged at flow rate of 100ml/min.

2.7 Crystallinity

Samples crystallinity was determined using a benchtop X-ray Diffractometer (XRD) model Rigaku MiniFlex. Operating conditions set were 40kV and 15mA. The peak measurement was conducted by taking 2θ value between 5 to 50° at a scan speed at 2.00°/min. The crystallinity index (CrI) was calculated based on calculation by Segal method [11] as following:

\[
CrI = \left( \frac{1002-\text{lam}}{1002} \right) \times 100
\]
Where, $I_{002}$ 2θ was from highest intensity at 22° and $I_{am}$ 2θ was from minimum intensity at 18° which known as amorphous scattering.

3. Results and Discussion

Three different types of oil palm biomass were used to investigate the effect of pretreatment process on various anatomical parts of oil palm. The success of pretreatment process in extracting cellulose from oil palm biomass can be determined physically by observing the color changes after the pretreatment. The original color of the fibers was brown. After pretreatment, the color changed to white indicates the major composition of the fiber which is cellulose. The change in color could be contributed by solubilization of lignin at high temperature in alkaline charge solution [12]. The cellulose is known as represent white in color. In this study, all samples turned to white color after pulping and bleaching. The composition of the samples before and after pretreatments is shown in Table 1. OPF had the highest composition of cellulose after the pretreatment with cellulose composition of 94.6%, followed by OPEFB and OPMF with 93.0% and 88.8%.

It is seen that the chemical composition varied greatly depending on the part of the plant. For instance, OPMF exhibited the highest composition of lignin at 32.4%, as compared to OPEFB and OPF at 18.6% and 16.9%, respectively. Cellulose on the other hand is the least in OPMF, followed by OPEFB and OPF. This observation is similar to that reported by [13] in which different chemical composition was reported for oil palm fiber from different anatomical part. This could be explained by the function of each fiber. For instance in palm fruits, oil is stored in mesocarp. Since oil is hydrophobic, lignin content in OPMF is hence high, because lignin is the hydrophobic part which could function to allow the fruit to store the oil and protect it from wet condition, for example during raining.

After pretreatment, all samples exhibited non-detectable amount of lignin which indicates that the lignin component was being removed almost completely after the pretreatment. Despite of lignin removal, some hemicellulose retained after pretreatment as shown by the % of hemicellulose remained in the samples. OPMF showed the highest hemicellulose content as compared to OPEFB and OPF. This is expected to be contributed by the content of recalcitrant hemicellulose in the sample. It has been shown that some portion of hemicellulose is trapped within cellulosic microfibrils. Also, covalent linkages between arabinose and lignin have been proposed [14]. These two conditions lead to a condition which is called recalcitrant hemicellulose. Since OPMF contained the largest amount of lignin initially, it is expected that more hemicellulose fraction is bound covalently to lignin in OPMF compared to the OPEFB and OPF. It is hence the highest amount of residual hemicellulose was found in OPMF. This is in agreement with earlier report which exhibited that hemicellulose hydrolysis is limited by high lignin content [14].
**Table 1.** Chemical composition (%) of different oil palm biomass pretreated with pulping and TCF bleaching.

| Oil palm biomass | Cellulose (%) | Hemicellulose (%) | Lignin (%) | Extractives (%) |
|------------------|---------------|-------------------|------------|-----------------|
| OPMF             |               |                   |            |                 |
| Untreated OPMF   | 28.2 ± 0.8    | 32.7 ± 4.8        | 32.4 ± 4.0 | 6.5 ± 0.1       |
| OPMF cellulose extract | 88.8 ± 1.7  | 11.2 ± 1.7        | ND         | ND              |
| OPEFB            |               |                   |            |                 |
| Untreated OPEFB  | 37.1 ± 4.4    | 39.9 ± 0.75       | 18.6 ± 1.3 | 3.1 ± 3.4       |
| OPEFB cellulose extract | 93.0 ± 0.5  | 7.0 ± 0.5         | ND         | ND              |
| OPF              |               |                   |            |                 |
| Untreated OPF    | 45.0 ± 0.6    | 32.0 ± 1.4        | 16.9 ± 0.4 | 2.3 ± 1.0       |
| OPF cellulose extract | 94.6 ± 3.2  | 5.4 ± 3.2         | ND         | ND              |

*Note: Chemical composition (%) expressed on oven dry basis (OD). Data represent in replicates (mean values). ND indicates not detectable.

Higher amount of extractives was found in OPMF sample, which was contributed by the residual oil content in the OPMF. OPMF stores oil, and some oil is still present after pressing of the oil palm fruits [15].

![SEM images of untreated OPMF, OPEFB and OPF (a1-c1) and cellulose extracts from OPMF, OPEFB and OPF (a2-c2)](image)

**Figure 1.** SEM images of untreated OPMF, OPEFB and OPF (a1-c1) and cellulose extracts from OPMF, OPEFB and OPF (a2-c2).

SEM images of cellulose obtained from pretreated fibers are shown in Figure 1 (a2-c2). Several microfibrils are seen from all images, indicating disruption of lignocellulosic structure as compared to the untreated oil palm biomass in Figure 1 (a1-c1). This observation is mainly due to the removal of lignin
and hemicellulose [16]. OPMF shows incomplete microfibrillation as compared to OPEFB and OPF, as seen by the presence of non-fibrillated structure. This may indicate the presence of other components, which support the chemical composition results shown in Table 1. The diameter size of fibers was less than 10μm as estimated from SEM images.

**Figure 2.** Polarized images showing cellulose fibers from (L-R) OPMF, OPEFB and OPF

Polarized microscope images (Figure 2) further exhibit the diameter size of the microfibrils cellulose. All the biomass had almost similar diameter size, in the range of 9 - 11μm. However there was no image shown for untreated biomass due to large micro bundles fibrils to be seen by polarized microscope.
Figure 3. TG and DTG chromatograms of (a) untreated and (b) cellulose extracts from OPMF, OPEFB and OPF.
Table 2. Data evaluated from TG and DTG curve

| Oil palm biomass | \( T_{d10\%} \) (°C) | \( T_{d50\%} \) (°C) | \( T_{\text{max}} \) (°C) |
|-----------------|--------------------------|--------------------------|--------------------------|
|                 | Untreated                | Extract cellulose         | Untreated                | Extract cellulose         | Untreated                | Extract cellulose         |
| OPMF            | 280                      | 255                      | 338                      | 340                      | 335                      | 351                      |
| OPEFB           | 247                      | 280                      | 332                      | 349                      | 345                      | 361                      |
| OPF             | 253                      | 284                      | 340                      | 347                      | 359                      | 360                      |

*\( T_{d10\%} \) and \( T_{d50\%} \) represent the degradation temperature at 10% and 50% material degradation from TG data.
*\( T_{\text{max}} \) (°C) is the maximum thermal decomposition of cellulose from DTG chromatogram.

TG analysis for lignocellulosic materials can be used to determine thermal stability of each component in the material, as well as to qualitatively determine the presence of components in the material. From observation in Figure 3a, a different thermal degradation of the untreated fibers due to the presence of non-cellulosic components in the raw fibers associated by DTG curve which represent two peaks in the range temperature of hemicellulose and cellulose at around 290°C and 370°C. It has been reported that the degradation temperature range for lignin, hemicellulose, and cellulose are within temperature ranges of 160–900, 220–315 and 315–400 °C [16]. From Figure 3b, it can be observed that all oil palm biomass cellulose samples exhibited single step degradation in the temperature range of 230 – 380 ºC, which indicates the degradation was mainly due to one main component. This is supported by the DTG chromatograms which showed single peak for all samples. Weight loss was observed at temperature around 100 ºC, due to moisture in the samples. Meanwhile, \( T_{d10\%} \) was observed in the range of 255-284 ºC for the three samples, suggesting that early degradation of all the samples could be contributed by hemicellulose content. This is in agreement with the results shown in Table 1, which exhibited that some amount of hemicellulose was left after the pretreatments. \( T_{d50\%} \) for all untreated samples were almost similar, show increase thermal stability after pretreatment to the range of 340 – 349 ºC (Table 2). Based on previous report, this range is attributed to the degradation of cellulose. \( T_{\text{max}} \) values also showed increase the degradation temperature of cellulose after the pretreatment because the removal of non-cellulosic material provided highly dense of cellulose fibrils [17].

In term of thermal stability, it is observed that OPMF is the least stable while OPEFB and OPF showed almost similar thermal stability. Thermal stability could be contributed by several factors, for example the content of thermally unstable component, crystallinity and degree of polymerization. Based on chemical composition showed in Table 1, OPMF contained the highest amount of hemicellulose, which is the most thermally unstable component in lignocellulose materials. This explains why OPMF has the lowest thermal stability. Apart from that, crystallinity also is an important factor which affects the thermal stability. Increased crystalline region in cellulose could increase its thermal stability [10].

In corresponding to the crystallinity, XRD data was analyzed and the results are shown in Table 3. It is apparent that the highest crystallinity was recorded for OPF cellulose extract (84.5%) followed by OPEFB (82.4%). Distinct observation can be seen for OPMF cellulose extract, in which the crystallinity of the sample was only 69.2%. This observation could be contributed by hemicellulose content in OPMF cellulose extract. It has been widely known that hemicellulose is amorphous [18]. The higher content of hemicellulose in OPMF cellulose extract was hence influencing the overall crystallinity of the sample. In comparison to other reports, the crystallinity of oil palm biomass before pretreatment was almost similar.
for all biomass. There were variations in crystallinity of samples after pretreatment, which is expected due to the pretreatment methods used.

**Table 3.** Crystallinity of cellulose from untreated and treated oil palm biomass fibers in comparison with other studies.

| Oil palm biomass | Crystallinity (%) | Reference |
|------------------|-------------------|-----------|
|                  | Before pretreatment | After pretreatment (Cellulose extract) | |
| OPMF             | 34.3              | 69.2      | This study |
|                  | 15 - 18           | 51 - 55   | [19]     |
|                  | 38.5              | 56.8      | [20]     |
| OPEFB            | 45.0              | 82.4      | This study |
|                  | 43                | -         | [21]     |
|                  | 50                | 62        | [22]     |
| OPF              | 54.5              | 84.5      | This study |
|                  | 35                | -         | [23]     |
|                  | -                 | 57        | [24]     |

4. Conclusions
Characteristics of cellulose from different bioresources may vary despite of its similar chemical structure. The characteristics could be vary due to the nature of the bioresources which may influence the physical characteristics of the cellulose and hence, its performance for product development. In this study, we demonstrated the characteristics of different oil palm biomass: OPMF, OPEFB and OPF. OPF and OPEFB exhibited superior performance in terms of cellulose purity, thermal stability and crystallinity compared to OPMF. Variation in properties of cellulose from oil palm biomass may suggest its suitability in different applications. For instance, pretreated cellulose from OPF and OPEFB which showed better crystallinity and thermal properties may be suitable as reinforcement material for biocomposites. Cellulose from OPMF on the other hand can be used as starting material for cellulose nanofiber production since it has been reported that some amount of hemicellulose in cellulose sample may assist in nanofibrillation. The results from this paper are important to provide fundamental understanding on oil palm biomass cellulose as well as to estimate its actual potential for product development. The variation in the results obtained suggesting that different applications of material could be proposed and developed.

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