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CHEMICAL COMPOSITION AND THERMAL ANALYSIS OF CELLULOSE EXTRACTED FROM SENDUDUK (*Melastoma malabathricum* L.)

IZATHUL SHAFINA SIDEK[1], SARIFAH FAUZIAH SYED DRAMAN[2*], SITI ROZAIMAH SHEIKH ABDULLAH[3] AND NORNIZAR ANUAR[4]

[1,2]School of Chemical Engineering, College of Engineering, Universiti Teknologi MARA, Bukit Besi, Terengganu, Malaysia. [3]Department of Chemical and Process Engineering, Faculty of Engineering and Built Environment, Universiti Kebangsaan Malaysia, Selangor, Malaysia. [4]School of Chemical Engineering, College of Engineering, Universiti Teknologi MARA, Shah Alam, Selangor, Malaysia.

**Corresponding author: sfauziah@uitm.edu.my*
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Abstract: *Melastoma malabathricum* L. (*M. malabathricum*), or known locally as "senduduk", is a dispersed shrub that can be found growing wildly and abundantly in nature. However, there have been limited studies on the chemical composition and utilisation of *M. malabathricum*, especially in terms of cellulose materials. Thus, this study aims to determine the chemical composition of *M. malabathricum* through its leaves and branches using the Technical Association of the Pulp and Paper Industry methods. Fourier transform infrared (FTIR) spectroscopy, thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) were used to characterise the extracted cellulose. The cellulose content extracted from the branches and leaves were 47.8% (w/w) and 36.5% (w/w), respectively. Results from the FTIR analysis confirmed that the peaks of extracted cellulose from leaves and branches have approximately the same pattern as commercial cellulose. TGA thermograms showed that the thermal degradation of cellulose obtained from the branches and leaves occurs around 350ºC and for DSC thermograms, the exothermic peak was observed at 330ºC. Both results are similar to the decomposition temperatures of commercial cellulose. The findings suggested that *M. malabathricum* has high potential in terms of being a source for plant-based cellulose for consumption and an alternative to conventional and/or synthetic cellulose in the market.

Keywords: Biodegradable, bioproducts, cellulose, *Melastoma malabathricum* (L.).

Introduction

Senduduk (*Melastoma malabathricum* L.) is a natural green foliage plant that belongs to the Melastomataceae family. Other native names for this plant include harendong, kenduduk, kluruk, kemunting, sekeduduk, sikadudok, senggani and Singapore rhododendron (Joffry *et al.,* 2012; Majid & Yu, 2007). *M. malabathricum* is a native plant that grows in the wild, found on the wayside, in wastelands, degraded land and secondary forests. *M. malabathricum* grows in China, Australia, the Indian Ocean islands, the South Pacific Ocean and Southeast Asia, including Malaysia (Wong, 2008). This plant has beautiful flowers in different sizes. It can be small, medium and big with white, dark purple-magenta or light pink-magenta petals (Figure 1). The leaves are physically simple, tapered, narrow with three great longitudinal veins and a bristly underside. The branches are coated with minor rough scales and are reddish and bristly. *M. malabathricum* is used traditionally to treat diarrhea, dysentery, wound healing, leucorrhoea, haemorrhoids and in post-partum treatments (Susanti *et al.,* 2008).

Figure 1: Senduduk (*M. malabathricum*)

The chemistry of *M. malabathricum* is limited, especially as an alternative renewable source of cellulose materials for bioproducts. Usually, bioproducts derived from cellulose materials can be used in the manufacturing of food packaging (Kumar & Thakur, 2017), electroacoustic devices (Ashter, 2016) and medical devices (Kalia *et al.,* 2011). Bioproducts that contain cellulose materials have good reinforcing capabilities (Alotabi *et al.,* 2020), good thermal properties (Khan *et al*., 2020a) and are eco-friendly (Shamsuddin, 2017). Besides, natural microorganisms, such as bacteria (Ali *et al.,* 2017), algae and fungi (Momani, 2009), can degrade bioproducts. Therefore, it is significantly interesting to develop bioproducts from cellulose materials that can be completely degraded and produced from natural fiber and abundant, renewable resources that is sustainable.

Cellulose is a semi-crystalline polysaccharide containing D-glucopyranosyl units joined by β-(1-4)-glucosidic linkages (Rosli & Ahmad, 2013). The end terminal of the cellulose polymer chain is stabilised with non-reducing and reducing sugar units (Nazir *et al.,* 2013). Cellulose is commonly found in different living species, such as plants, animals and a few microorganisms or bacteria. According to its nature and fibre form, cellulose can be amorphous or crystalline (Chandrahasa & Rajamane, 2014). It also exists in a significant constituent of plant cell walls, including lignocellulosic material (Saelee *et al.,* 2014). In this study, *M. malabathricum* is the lignocellulosic material that is focused on.

Several studies have revealed that cellulose is one of the most innovative materials due to its biodegradability (Flieger *et al.,* 2003), sustainability (Bohn, 2015) and renewability (Shanmugarajah *et al.,* 2015). Cellulose can be obtained from forest biomass, agriculture residue, municipal waste and herbaceous grass, such as from bacteria (Chen *et al.,* 2011), coir fibre (Abraham *et al.,* 2013), rice straw (Agustin *et al.,* 2014), empty oil palm fruit bunches (Lani *et al.,* 2014), agricultural and industrial waste (Hasan, 2014), sugarcane (Supranto *et al.,* 2015). Sugarcane-bagasse fiber waste, has a huge potential as raw material for production of the High Refined Cellulose (HRC and durian rinds (Fahma *et al.,* 2017). Cellulose has a few advantages, including its abundance (Joffry *et al.,* 2012; Khan *et al.,* 2020b; Khan *et al.,* 2021), lowcost (Yaqoob *et al.,* 2020), being environmentally friendly (Vijay *et al.,* 2020) and possessing biodegradable properties (Manzano, 2021; Alotabi *et al.,* 2020). These properties lead to extracted cellulose from *M. malabathricum* having the potential to be commercialised as bioproducts in a circular bio-economy. Most of the previous studies that investigated cellulose in the family Melastomataceae are for *Lavoisiera mucorifera* (Silva *et al.,* 2019), *Tessmannianthus carinatus* (Kriebel, 2014), *Miconia lucenae* (Goldenberg *et al.,* 2020) and *Conostegia* (Kriebel, 2016). However, three have not been many studies on cellulose from *M. malabathricum* compared with other species or genus from the same family (Silva *et al.,* 2019; Milanez & Machado, 2008). Samanaseh (2017) in his study, focuses on cellulose from leaves of *M. malabathricum.* For that reason, this study was conducted to determine the cellulose content and other constituents of *M. malabathricum* through its leaves and branches using the Technical Association of the Pulp and Paper Industry (TAPPI) method. Then, the characteristics of cellulose from *M. malabathricum* were investigated via Fourier transform infrared (FTIR) spectroscopy, thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) analysis.

Methodology

Materials

Raw senduduk *(Melastoma malabathricum)* branches and leaves were collected from wastelands in Bukit Besi, Dungun, Terengganu. All the reagent grade chemicals were used as received. Acetone (C_3H_6O), sulfuric acid (H_2SO4), acetic acid (CH_3COOH) were acquired from chemical manufacturing firm

Systerm. While sodium hydroxide (NaOH) and sodium chlorite ($NaClO_2$) were purchased from Friendemann Schmidt Chemicals and Sigma-Aldrich.

Methods

The raw materials were first cut into small pieces. The length ranges from 1 cm to 3 cm. The pieces are then rinsed in water before being dried overnight in a 60°C oven. The samples were ground in a grinder. The ground fibres were sieved using a 500 µm mesh screen. The ash content in the ground fibres (1 g) was determined by the weight loss after 6 hours of calcination at 525°C. The extractives content was determined by processsing the ground fibers (100 g) with Soxhlet extraction with acetone (C_3H_6O) for 16 hours and drying them overnight in a 60°C oven. This fibre is known as an extractives-free sample.

At 30°C for 1 hour, 0.3 g of extractives-free sample was hydrolysed with 3 mL of sulfuric acid (H_2SO_4, 72% (w/w)). The sulfuric acid was then diluted to 4%. The solution was autoclaved at 121°C for 1 hour. After cooling and filtering the leftover material, it was dried in oven at 105°C to a consistent weight. Insoluble lignin is the weight that was measured. The soluble lignin was determined by measuring the absorbance of the solution at 205 nm. The sum of the insoluble and soluble lignin contents was used to compute the total lignin content.

A total of 0.25 g of extractives-free sample was used to make the holocellulose. At 70°C for 1 hour, the extractives-free fibre was mixed with 5 mL deionised water, 1 mL of acetic acid (CH_3COOH) and 5 mL of sodium chlorite ($NaClO_2$, 0.7% (w/v)). The process was carried out four to five times more until the material turned white. The obtained material was filtered, rinsed in deionised water and dried to a constant weight at 105°C. The remaining weight was made up of quantifiable holocellulose.

After that, 0.1 g of the holocellulose was treated for 30 minutes at room temperature (RT) with 8 mL sodium hydroxide (NaOH, 17.5% (w/v)) and stirred every 10 minutes. Then after, 8 mL of distilled water was added to the solution. The reaction was kept for another 30 minutes. The material was filtered, rinsed with distilled water, then steeped for 5 minutes in 20 mL of acetic acid (CH_3COOH, 1.0 M). The material (residue) was washed with excess water and dried in the oven at 105°C until it reached a consistent weight. The cellulose content of these samples was determined. The lignin, holocellulose, cellulose and ash contents were determined in triplicate during the studies.

Chemical Composition

The chemical constituents of *M. malabathricum* were determined based on the TAPPI standards, which are TAPPI T9 wd-75 (2015) for cellulose, hemicellulose and holocellulose, TAPPI T211 om-12 (2015) for ash, TAPPI T280 wd-06 (2015) for extractives and TAPPI T222 om-11 (2015) for lignin (Pacheco *et al.,* 2018). The total amount of ash, extractives, lignin, holocellulose, hemicellulose and cellulose were calculated as follows (Tibolla *et al.,* 2014):

$$\text{Ash (\%)} = \frac{\text{weight of dried sample} - \text{dry ash residue}}{\text{weight of the initial fiber sample}} \times 100 \quad (1.1)$$

$$\text{Extractive (\%)} = \frac{\text{weight of dried sample} - \text{dry extractive residue}}{\text{weight of the intial fiber sample}} \times 100 \quad (1.2)$$

$$\text{Lignin (\%)} = \text{soluble lignin (\%)} + \text{insoluble lignin (\%)} \quad (1.3)$$

$$\text{Holocellulose (\%)} = \frac{\text{weight of dried sample} - \text{dry holocellulose residue}}{\text{weight of the intial fiber sample}} \quad (1.4)$$

$$\text{Cellulose (\%)} = \frac{\text{weight of dried sample} - \text{dry cellulose residue}}{\text{weight of the intial fiber sample}} \times 100 \quad (1.5)$$

$$\text{Hemicellulose (\%)} = \text{Holocellulose (\%)} - \text{Cellulose (\%)} \quad (1.6)$$

The cellulose functional group was identified using a FTIR Tensor 27 (Bruker, Germany) in the range of 400 cm^{-1} to 4000 cm^{-1} with three scans for each sample.

Thermal Analysis

The Netzsch model STA 449F3-Jupiter was used to conduct differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA). Under the dynamic nitrogen flow, 9-15 mg of the samples were placed in alumina pans and heated from 30°C to 800°C at 10 $Kmin^{-1}$(50 $mLmin^{-1}$).

Results and Discussion

The samples of the cellulose extracted from branches and leaves of *M. malabathricum* before and after treatment are shown in Figure 2. It can be seen that the cellulose extracted from the branches and leaves become pale yellowish in colour following the treatments.

Chemical Composition

The main constituents of senduduk *(M. malabathricum)* were investigated. The chemical composition determination of *M. malabathricum,* like extractives, ash, lignin, holocellulose, hemicellulose and cellulose, was performed based on the TAPPI standards. Table 1 shows the chemical composition of *M. malabathricum* branches and leaves.

This study found that the branches of *M. malabathricum* have a higher amount of ash than the leaves at 12.02% (w/w) and 8.97% (w/w), respectively. The lignin contents also

Figure 2: Extraction of cellulose: (a) *M. malabathricum* branches before treatment, (b) branch cellulose obtained after treatment, (c) *M. malabathricum* leaves before treatment and (d) leaf cellulose obtained after treatment

Table 1: The chemical composition of senduduk *(M. malabathricum)* branches and leaves

Constituents (%)	Senduduk *(M. malabathricum)*	
	Branches (%)	Leaves (%)
Extractives	8.60	7.75
Holocellulose	60.88	58.87
Hemicellulose	13.08	22.37
Cellulose	47.80	36.50
Lignin	13.20	9.68
Ash	12.02	8.97

demonstrated significant differences between branches (13.2% (w/w)) and leaves (9.68% (w/w)). The hemicellulose content was higher in the leaves (22.37%) compared with the branches (13.08% (w/w)). The percentage of cellulose found in branches and leaves is 47.80% (w/w) and 36.50% (w/w), respectively. The values of cellulose obtained in this study were significantly higher compared with other reported resources. According to Reddy and Rhim (2014), garlic skin has 42% (w/w) cellulose, rice husk 35% (w/w) cellulose (Johar *et al.,* 2012) and coffee silverskin 24% (w/w) cellulose (Sung *et al.,* 2017). Thus, the cellulose content is potentially useful for further application in bioproducts.

Fourier Transform Infrared (FTIR) Analysis

The FTIR spectra of cellulose derived from *M. malabathricum* were compared with the FTIR spectra of commercial cellulose as shown in Figure 3. The functional groups in each sample were identified using FTIR analysis.

The FTIR results demonstrated O-H stretching of the cellulose spectrum in the branches and leaves, with peaks ranging from 3500 cm^{-1} to 3200 cm^{-1} (Naduparambath *et al.,* 2018) and alkyl and aliphatic peaks ranging from 2980 cm^{-1} to 2570 cm^{-1} (Du *et al.,* 2016). Water absorption was attributed to the peaks from 1640 cm^{-1} to 1435 cm^{-1} (El Achaby *et al.,* 2018). Peaks between 1170 cm^{-1} and 1010 cm^{-1} indicated asymmetric C-O-C stretching (Moran *et al.,* 2008).

The cellulose spectrum peaks for commercial cellulose were shown to be 3334 cm^{-1}, 2900 cm^{-1}, 1639 cm^{-1} and 1029 cm^{-1}. The cellulose spectrum peaks for the leaf cellulose were at 3333 cm^{-1}, 2919 cm^{-1}, 1632 cm^{-1} and 1025 cm^{-1}, respectively. Furthermore, 3332 cm^{-1}, 2918 cm-1, 1610 cm^{-1} and 1022 cm^{-1} were recommended for the branches. The spectra of commercial cellulose and cellulose obtained from *M. malabathricum* followed the same pattern.

Thermal Analysis

Thermogravimetric analysis (TGA) is a thermal analysis method where the physical and chemical properties of materials are observed under temperature changes. TGA is valuable because of its high-throughput nature (at-line) and is ideal for assessing cellulose polymers. Therefore, TGA is used as a method to study the decomposition of the obtained cellulose. TGA

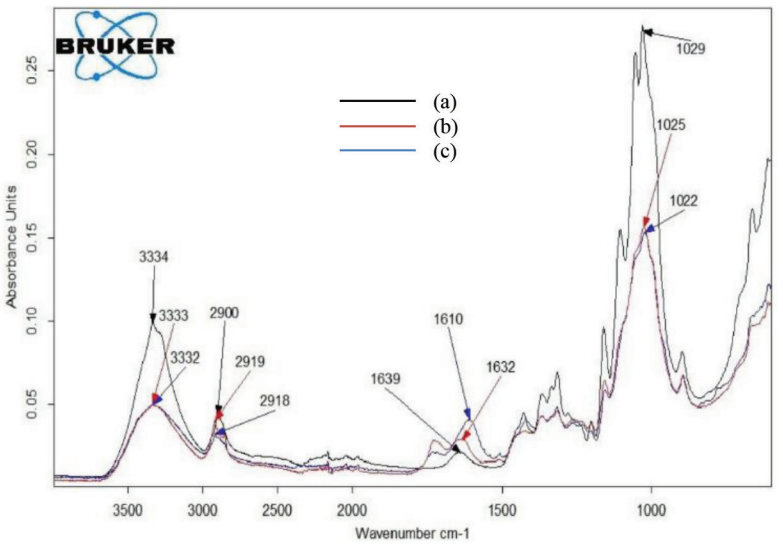

Figure 3: The FTIR spectra of (a) commercial cellulose, (b) cellulose from *M. malabathricum* leaves and (c) cellulose from *M. malabathricum* branches

has also been shown to be able to determine a compound's stability temperatures and the mass loss of compounds (Rasheed *et al.,* 2020). This is essential for the future application of cellulose in bioproduct materials.

The chemical structures of *M. malabathricum* leaves and branches decomposed at different temperatures. From a previous study, the decomposition of cellulose materials can be found in the thermal analysis. The decomposition of cellulose began at 315°C and proceeded until 400°C (Yang *et al.,* 2007; Khan *et al.,* 2020a; Alotabi *et al.,* 2020).

Figure 4 shows the TGA and curves of the derivative thermogravimetry (DTG) for commercial cellulose, leaf cellulose and branch cellulose. The first mass loss occurs below 100°C as a result of evaporation of the humidity of the materials. The decomposition of the hemicellulose is responsible for the second mass loss, which occurs at between 250°C and 300°C. The third mass loss began at about 350°C, which corresponds to the cellulose degradation.

The decomposisiton temperature for commercial cellulose is 351°C. The leaf cellulose began to decompose at 308°C and continued to 362°C. As shown in Figure 4, the thermal degradation of leaf cellulose occurs at 338°C. The branch cellulose, meanwhile, started to decompose at 318°C and continued to 365°C. The thermal degradation of the branch cellulose is 350°C, approximately similar to commercial cellulose. The maximum mass loss rate for commercial cellulose is at 34 wt.%, leaf cellulose at 28 wt.% and cellulose branches at 31 wt.%.

Differential Scanning Calorimetry (DSC) Analysis

The thermal behaviour of commercial cellulose and cellulose obtained from the leaves and branches of *M. malabathricum* were compared using the Differential Scanning Calorimetry (DSC) technique.

From Figure 5, it can be seen that commercial cellulose showed an exothermic peak at 343ºC. The exothermic peak for cellulose obtaimed from branches, meanwhile, is approximately 330°C. There was no clear fusion peak in cellulose derived from leaves. At temperatures ranging from 300°C to 350°C, all cellulose samples show an exothermic peak.

Conclusion

The amount of cellulose in senduduk (*M. malabathricum*) varies among the plant parts. The branches contain a higher amount of cellulose and has better thermal properties than the leaves. The plant could be considered as an alternative source of cellulose. Further studies on upscaling production are required for the development of an economical bioproduct.

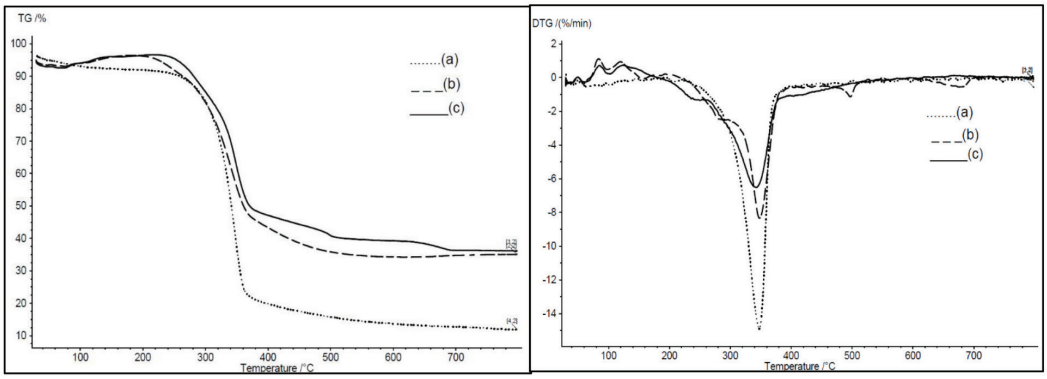

Figure 4: TGA thermograms and DTG curves of (a) commercial cellulose, (b) cellulose from *M. malabathricum* leaves (c) and cellulose from *M. malabathricum* branches

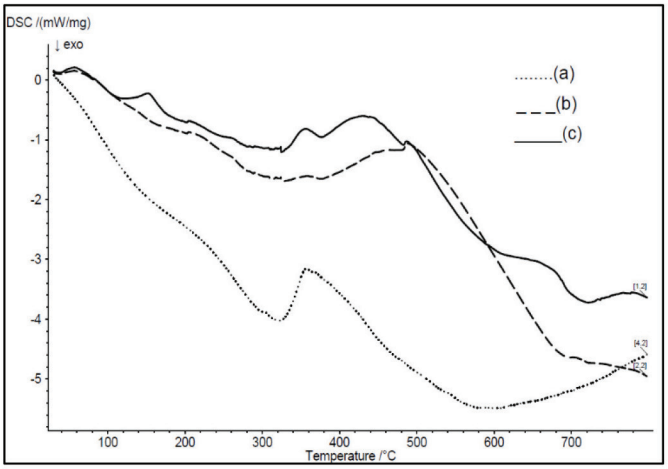

Figure 5: DSC thermograms of commercial cellulose (a), cellulose from *M. malabathricum* leaves (b), ellulose from *M. malabathricum* branches (c)

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