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Characterization of sugar palm (*Arenga pinnata*) fibres

Tensile and thermal properties

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Abstract The aim of this study was to characterize tensile and thermal properties of sugar palm (*Arenga pinnata*) fibres obtained from different heights (1, 3, 5, 7, 9, 11, 13, and 15 m) of sugar palm tree. This study has confirmed that in a mature sugar palm tree, degradation was occurred and altered the properties of its fibre. Fibres obtained at the area of live (green) palm frond were found to have a better tensile properties as a result of its optimum chemical composition especially cellulose, hemicelluloses and lignin. For the fibre obtained from the upper part of sugar palm tree, it shows slightly decreasing trend in tensile properties compared to mature fibres. It is due to the fibres are juvenile where their cell walls are progressively built up thus give slightly lower properties than matured fibres. For the fibre obtained from the area of dead palm frond, the fibres are considered to be degraded biologically. It is believed that polymeric chains in microfibrils were broken and their cellulose content was decreased which demonstrated inferior properties (tensile strength, modulus, elongation at break and toughness). The use of such fibre for application as reinforcing fibre in composite is not recommended since the strength of the fibre and composite will be reduced. There were four phases of decomposition of the fibres where the sequence of decomposition started with decomposition of moisture, followed by hemicelluloses, then cellulose and next is lignin while the ash was the last component left. The thermal degradation of these components were found in ranges of 45–123, 210–300, 300–400, 160–900 and 1723 °C, respectively. Thermogravimetric analysis and derivative thermogravimetric analysis curves showed that the fibre of 1 m showed higher thermal stability than the fibres of 3–15 m. The different thermal stability for each fibre was due to different chemical compositions especially when the fibre containing high ash content which result in higher thermal stability.

Keywords Sugar palm fibre · *Arenga pinnata* · Thermal degradation · Thermal properties

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Introduction

Sugar palm (*Arenga pinnata*) trees have been around for making variety of by-products for hundreds of years [1]. In Malaysia, especially in state of Negeri Sembilan, it is a popular with activity of tapping palm sap as the material for making traditional sugar blocks locally known as *gula enau* or *kabung* [2, 3]. Until now, there are a number of local palm sap tappers who are still getting income by making traditional sugar blocks while some locals use the

fibre for making traditional brooms. Traditionally, it is processed for making traditional sugar blocks and can also be processed to be crystal and brown sugar as an alternative to the commercialized sugarcane granular sugar. It can also be fermented to produce bioethanol for production of varieties of products (chemical products, solvents, pharmaceutical, medicines, beverages, etc.) and would also be used for production of biofuel. The next important part after palm sap is its fruit. It can be processed for making pickles, juices, desserts, for canned foods, and also being cooked for making traditional sugary syrup. There are other commercial purposes of sugar palm such as production of sago, its hard wood, etc.

However, the most important part after its palm sugar and its fruit is its fibre. Its black fibre or locally known as *ijuk* fibre is used for making ropes, brooms, brushes, paint brush, septic tank base filter, roofing, fishing tools and for handicrafts [4–7]. There are three main advantages of sugar palm fibre; it has high tensile strength and can withstand a longer life to degrade; is not effected by heat and moisture compared to coir fibre, it is durable and good resistance to sea water. Traditionally, sugar palm fibres were used for making ropes for ship cordages and proven to have good properties in sea water [8–10]. Not like other natural fibres (kenaf, banana pseudo-stem, bagasse, pineapple leaf, oil palm fibres), it does not require secondary processes such as water retting or mechanical decorticating process to yield the fibres [10]. It is due to the fibres were originally wrapped along the base of the palm leaf ribs [2, 11, 12]. Thus, the fibres can directly be used where their fibres are readily in the form of fibre. In addition, the increasing of interest in the use of natural fibre as composite reinforcement is one of the factors that encourage the use of sugar palm fibre. There are a number of studies have been carried out on the properties of sugar palm fibre and its composites in order to reveal its performance and to promote its use to the users and industries [13–24]. Continuous research is being carried out and has found that the sugar palm fibres have great potential to be used as reinforcement in polymer composites as alternative to glass fibre [2, 25, 26]. Since natural fibres are natural materials, they exhibit great variability in their properties. This has caused difficulties in the use of sugar palm fibre for making high quality and performance of composite products. Also, studies of biocomposites have been usually focused on their mechanical properties but less attention is given on the thermal stability [27]. A number of studies were carried out and have proven that thermal properties give significant contribution in understanding the behaviour of their fibres and composites [28–36].

This paper studies the tensile and thermal properties of sugar palm fibres obtained from different heights (1, 3, 5, 7, 9, 11, 13 and 15 m) of sugar palm tree. It is aimed to provide background information which will hopefully be useful in selection the optimum fibre properties of sugar palm fibre.

Materials and method

Preparation of fibres

Sugar palm fibres were obtained from Kampung Kuala Jempol, Negeri Sembilan, Malaysia. In this study, a matured sugar palm tree (after undergoing flowering stage with age more than 6 years) with the height of 20 m was selected. The fibres were taken at the height of 1, 3, 5, 7, 9, 11, 13 and 15 m of the sugar palm trunk. The fibres were washed and air dried for 24 h before being dried in the oven at 85 °C for another 24 h.

Determination of fibre tensile properties

In order to get results with statistical meaning and considering that the natural fibre is a anisotropic material which has diverse variations, the specimen of fibres was carefully selected randomly after being gently separated from the strand bundle sugar palm fibre. The selected fibres were carefully observed using optical microscope in order to confirm that there was no damaged on the fibre. The fibre was attached and glued to the tab shape which was designed with the gauge length of 20 mm and tested according to the standard test method for single fibre tensile test ASTM D 3379. The tab shape was cut at mid-gauge length after being gripped in test machine before the fibre is being tested. The specimen was tested with a crosshead speed of 1 mm/min with replication of 25 specimens using universal testing machine model Instron 3366 with the load capacity of 5 kN.

Individual fibre breaking loads were recorded and their diameters were measured using optical microscope type Lieca MS 5. The average cross-sectional area of fibres at area of failure was calculated as follows:

$$A = \frac{\sum a_{\mathrm{f}} \times 10^{-6}}{N(M_{\mathrm{f}})^2} \quad (1)$$

where A average fibre area in m^2, a_{f} area of one fibre in mm^2, N number of fibres measured, M_{f} photomicrograph magnification factor

Tensile strength of fibres was calculated as follows:

$$T = \frac{F}{A} \quad (2)$$

where T tensile strength in Pa, F force to failure in N, A average fibre area in m^2

Determination of thermal properties

Thermogravimetric analysis (TG) was carried out using a Perkin Elmer Thermal Analyzer model TGA7/DTA7, at temperature range of 35–800 °C with heating rate of 10 °C/min with holding time of 1.0 min at 35 °C under nitrogen atmosphere.

Results and discussion

Tensile properties

The sugar palm fibres (1, 3, 5, 7, 9, 11, 13 and 15 m) were tested for single fibre test and their stress strain behaviour is shown in Fig. 1. The trend shows the stress increases linearly with the increase of strain values, while when it reaches at certain stress values, nonlinear behaviour is observed for all curves where strain increases at low stress value before undergoing failure. This irregularity curves show that the properties of fibre are differ to each other especially at the fibre of 1 and 3 m where significant lower in tensile strain was found.

The low stress–strain values of the fibres of 1 and 3 m may attribute to poor strength of fibre when the fibre is pulled under the action of force. It can be seen in Fig. 2 that tensile strength of fibre obtained at the lowest part (1 m) was much lower and start to increase with the increases of the height of tree before consistently in ranges of 266–292 MPa (for the fibre of 9–15 m).

Since the fibres obtained from the matured sugar palm tree, the fibre had undergone several kinds of degradations especially at the bottom part of the tree. It can be seen during harvesting of the fibre, the degradation took place especially at the lowest part of sugar palm tree where brown or broken dead palm frond was observed at this region. This is a normal growing process of palm species like coconut, beetle nut, oil palm and sago trees where new palm frond grows above (at higher location) the dead palm

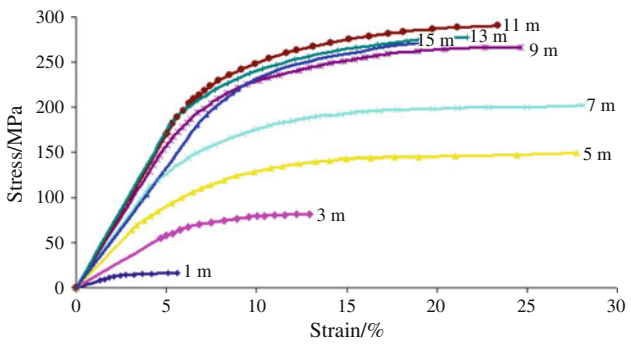

Fig. 1 Stress–strain behaviour of sugar palm fibre obtained from different heights of sugar palm tree

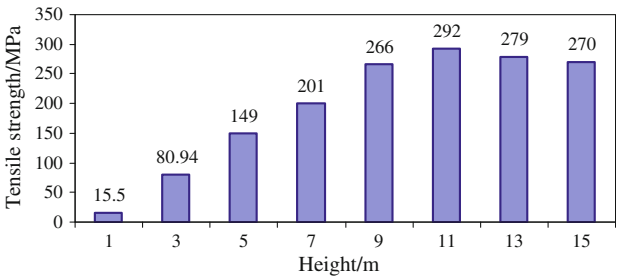

Fig. 2 Tensile strength of sugar palm fibre obtained from different heights of sugar palm tree

frond. This is due to the fact that palms are monocots which mean they do not have a continued outward growth in their trunks as do other typical trees. However, in reality, it is formed with expansion of their sugar palm trunk since the first tissue is formed. When the growing reaches to their ultimate height, the growing of tree will decline and die. As a new frond emerges, it is followed by an old frond dying. As the new frond will take place the dying frond to produce a continuous supply of food to grow. This is a normal growing process of palm species while only the number of frond is different to each palm species. According to Harpini [26], a normal sugar palm tree produces about 30–50 palm fronds during its life time depending on the size of the tree.

As the fibres produced by dead palm frond were experiencing degradation, they were attacked by various sources of degradations that can weaken the fibres. Figures 3 and 4 show the cross-sectional views of sugar palm fibres (normal and degraded fibres, respectively). Figure 3 [13] shows the macrofibrils were well arranged to form a sugar palm fibre while Fig. 4 shows the damaged macrofibrils due to the degradation of cell wall structures. It depicts that the fibre has undergone degradation where defects were found in cellular structures and this led the poor tensile strength of fibre. This is due to the fact that the strength of

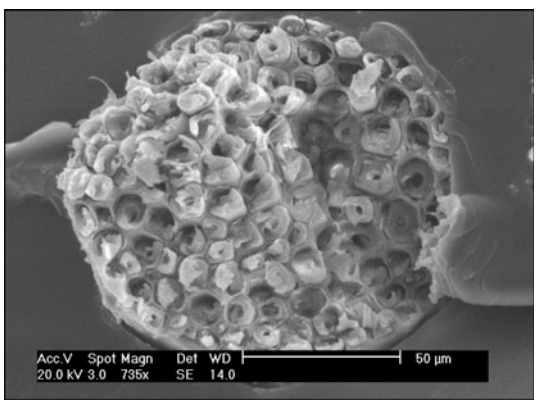

Fig. 3 Cross-section of normal sugar palm fibre. ×735

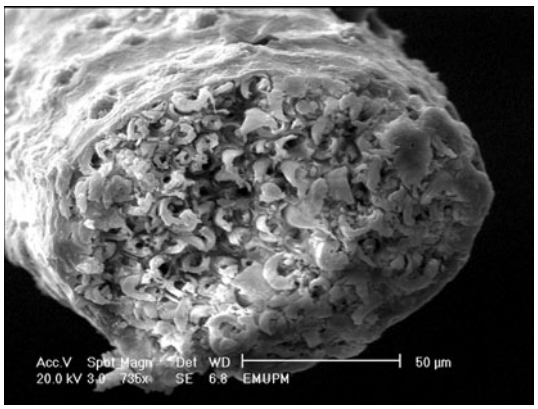

Fig. 4 Cross-section of degraded sugar palm fibre. ×735

fibre is provided by macrofibrils (individual macro fibres) which were held together to form a fibre.

From Fig. 4, it is believed that polymeric chains in microfibrils were broken and their cellulose content was decreased. It is a known fact that the reduction of cellulose content will decrease the tensile strength of fibres. This is due to the fact that cellulose is the main structural component which provides mechanical strength to the fibres [37]. The reduction of cellulose content after undergoing degradation has been proven in our previous study [38] as shown in Table 1. The results of this current study supported by findings of other researchers have shown that degradation altered the chemical composition of fibres.

The cellulose content of fibres was found to be lower at lower height especially for the fibres obtained at 1 and 3 m. This process is found to be normal for a tree where at the end of the life of a tree, all of the chemical compositions are broken down and the constituent atoms are returned to the environment. They are available as the building blocks for new organisms [39].

Similar to cellulose, hemicelluloses contents for fibres at 1 and 3 m were found to be lower than those of higher height. Hemicelluloses does not give significant contribution to the strength of fibre as cellulose does but it acts to bind the microfibrils which provides as structural reinforcement to microfibrils of the fibre. This is because, among all important variables that influence the overall properties of natural fibres, which include structure, microfibril angle, cell dimension, defect and chemical composition, chemical composition is found to be one of the main factor. Details for the chemical composition of sugar palm fibres will be presented in a future work. The next important factor that influences the overall properties (particularly tensile strength) is the structure must have minimum defect. In tensile strength test if defect presents in a structure, the failure is most likely to start at the point where the defect occurred.

Tensile modulus represents the ability of the fibre to resist deformation when stress is applied, and it was decreased due to the degradation. With similar trend to tensile strength, tensile modulus of the fibre obtained at the bottom part was significantly lower and increases exponentially as shown in Fig. 5. The results were in the range of 2.68–3.37 GPa for the fibre of 7–15 m. Thus, it means that the stiffness of fibre decreases due to poor properties of fibre. It was observed during the fibre harvesting process that there was no green palm frond was found at this range (1–3 m).

It shows a good agreement with the increase of lignin content as shown in Table 1. It was found that tensile modulus increases with the increasing of lignin. It is due to the fact that [40], lignin is the compound that gives rigidity to the plants. It plays an important role to bind the fibre cell wall which is similar to the function of adhesive or resin to bind the reinforcement fibres or particles in composite materials. Thus, the lower lignin resulting to lower tensile modulus as can be seen at the bottom part of fibres.

Similar to elongation at break, this region has shown the lowest elongation at break. Due to the poor strength of rotten fibres, when stress is applied on the fibre, the fibre does not elongate and tends to break immediately, as depicted in Fig. 6.

The poor properties of this region can be translated with their poor toughness. Based on stress–strain behaviour, it gives a mean of poor toughness of fibre 1 and 3 m as it were measured with the area underneath the stress–strain

Table 1 Chemical compositions of sugar palm fibre obtained from different heights of sugar palm tree

Composition/%	Height/m							
	1	3	5	7	9	11	13	15
Cellulose	37.3	49.36	55.28	56.55	56.8	55.75	54.42	53.41
Hemicelluloses	4.71	6.11	7.36	7.68	7.93	7.92	7.89	7.45
Lignin	17.93	18.941	20.89	20.45	23.6	22.96	24.27	24.92
Ash	30.92	14.04	5.8	4.23	2.06	4.09	3.98	4.27
Extractive	2.49	2.019	1.71	1.41	1.35	1.48	1.21	0.85
Moisture content	5.36	8.64	7.92	8.37	8.19	7.72	8.12	8.7

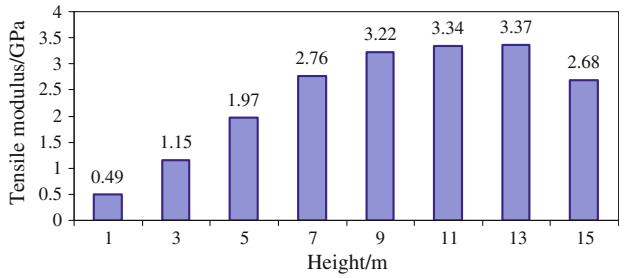

Fig. 5 Tensile modulus of sugar palm fibre obtained from different heights of sugar palm tree

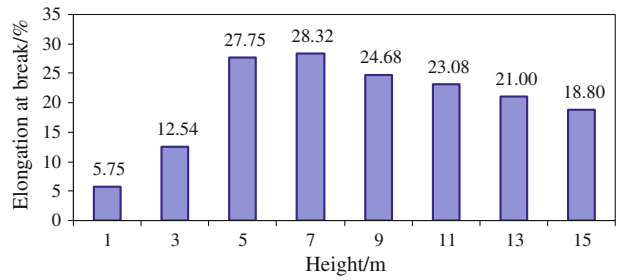

Fig. 6 Elongation at break of sugar palm fibre obtained from different heights of sugar palm tree

curve (Fig. 1). This shows the lower energy that the fibre of 1 and 3 m (0.58 and 7.36 J/m^3, respectively) can absorb before undergoing failure compared to others as shown in Fig. 7.

It is also important to discuss in this study, the fibre obtained at the highest part (15 m) was experiencing slightly decreasing trend as seen in tensile strength, tensile modulus, elongation at break and toughness (Figs. 2, 5, 6 and 7, respectively). This may be attributed to the problem of juvenile fibre where it has poor properties. Similar to the problem dealing with juvenile wood, juvenile fibre has larger microfibril angle in its cell wall than matured fibres. It has large longitudinal shrinking and swelling when it absorbs or desorbs moisture which means it has poor dimensional stability. As a result of juvenile fibre (15 m), it also has lower cellulose content as shown in Table 1, thus,

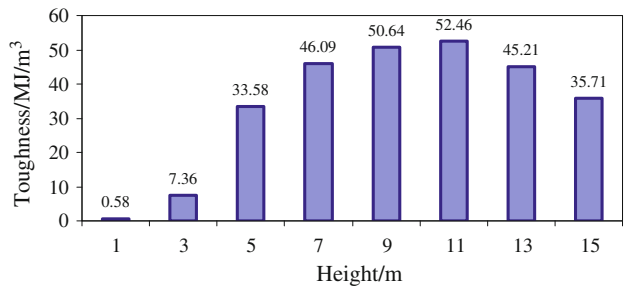

Fig. 7 Toughness of sugar palm fibre obtained from different heights of sugar palm tree

the lower cellulose content and the larger microfibril angle resulting in lower strength and durability than matured fibre. This is because a high cellulose content and low microfibril angle are the desirable properties of a natural fibre to be used as reinforcement in polymer composites [41].

In this study, it is worth noting that the properties of sugar palm fibre are not dependent on the position of fibres obtained, but it depends on the condition of the fibre. For the fibre obtained from the area of green palm frond, the fibre is considered to be in matured stage where it has the optimum cellulose content which gives maximum fibre strength. For the fibre obtained at the area of upper part of tree, the fibres are assumed to be in juvenile stage where it is in growing process. For the fibre obtained from a matured tree (where the tree is in flowering stage) a portion of dead palm fronds is often seen, thus the fibres obtained in this area were assumed to be weak where they were degraded biologically. Thus, the fibre taken at this region (dead palm frond) is not recommended for the application which requires fibre strength such as for application as reinforcing fibre in composite. The use of such fibre should be avoided since the strength of the fibre and composite will be reduced.

Thermal properties

Thermogravimetric analysis

This section studies on thermal degradation of sugar palm fibre. The results of the effect of increasing temperature to mass and derivative mass loss rate of sugar palm fibre are shown in Figs. 8 and 9, respectively. In short, there are two great differences found for both thermogravimetric analysis (TG) and derivative thermogravimetric analysis (DTG) behaviours of the fibres; that is 1 and 3–15 m. In general, the thermal decomposition of these fibres consists of four phases. The first phase was moisture evaporation, then decomposition of lignocellulosic components of

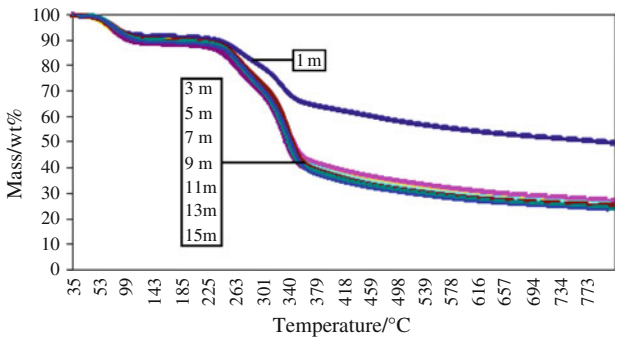

Fig. 8 TG of sugar palm fibres obtained from different heights of sugar palm tree

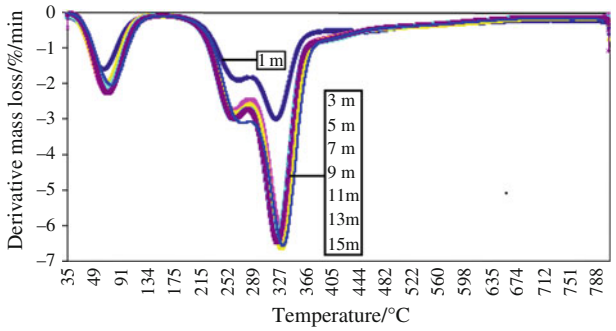

Fig. 9 DTG of sugar palm fibres obtained from different heights of sugar palm tree

hemicelluloses, followed by cellulose, lignin and lastly their ash. The decreasing trend follows the result obtained by Yang et al. [42].

The first phase was the evaporation of the moisture absorbed in fibres ranging from 45 to ~123 °C. As fibre is heated, there is a decrease in weight of the material initially, due to the loss of bound water and volatile extractives, with less volatile extractives tending to migrate to the surface of the fibre. The migration of volatile extractives was due to the movement of water from interior of fibre to the surface of fibre (due to migration from lower to the higher potential of water at surface of fibre) since the water on fibre surface was evaporated. Thus, the volatile extractives were brought together by water and left on the fibre surface. It can be observed that the evaporation of moisture of fibre (height of 1 m) was completely evaporated at ~110 °C compared to fibres of 3–15 m height (~123 °C) as shown in Fig. 8. This is due to the lower moisture content (MC) of 1 m fibre (5.36%) resulted in lower mass loss compared to the others; 3–15 m (where in average of 8.24%) as shown in Table 1. It can be seen in Fig. 9 that the average maximum mass loss rates for the fibres of 3–15 m were 1.99 wt%/min at 72.44 °C while for 1 m fibre was 1.59 wt%/min found at 68.26 °C. Heating at lower temperature results in low mass loss, which mainly associated with only loss of free (in cell lumen) and bound water (in cell wall) and volatiles in fibre. While significance mass loss (up to 70%) can be found from its basic constituents; cellulose, hemicelluloses and lignin where they can occur starting from temperature of 100–140 °C and above. The sequence of decomposition among these three components are generally supposed to decompose with hemicelluloses, followed by cellulose and lastly with lignin [42].

The second phase was decomposition of hemicelluloses. As the temperature is increased, there are chemical changes in their hemicelluloses components due to the cellular breakdown. Study by Yang et al. [42] reported that hemicelluloses decompos at 220 °C and substantially completed at 315 °C. However, in this study hemicelluloses decomposes starting from temperature of 210 °C and had completely decomposed at 300 °C as shown in Fig. 8. The results also show that there was no difference in term of temperature required to decompose the hemicelluloses for all fibres (1–15 m) which were in the range of 228–312 °C. However, in term of mass loss rate, at same temperature of 270.83 °C, 1 m fibre was lower (1.91%/min) than the fibres of 3–15 m (~2.95 wt%/min). This is due to lower hemicelluloses content (4.71%) compared to the fibre of 3–15 m (7.47%) as shown in Table 1.

There are several reasons why hemicelluloses degrade first compared to other compositions. According to Yang et al. [42], hemicelluloses consists of various saccharides (xylose, mannose, glucose, galactose, etc.) and they appear as random, amorphous structure, rich of branches, which are very easy to remove from main stem and to degrade to volatiles evolving out (CO, CO_2 and some hydrocarbon, etc.) at low temperatures. For these reasons, hemicelluloses are less thermally stable than cellulose. Cellulose has a number of chains which are closely associated via extensive H-bonding networks to form the microfibril which it is well known as the primary reinforcing agent in the cell wall. Hemicelluloses are degraded to a greater extent than the other macromolecular components (cellulose and lignin) [43].

As soon as hemicelluloses had completely decomposed, the decomposition of cellulose will take place as the third phase of decomposition. Because of its highly crystalline nature of their cellulose chain than amorphous, cellulose has relatively thermally stable. It does not start to decompose until hemicelluloses had completely decomposed which normally starts at higher temperature at about 315 °C [42]. This refers to the degradation of crystalline cellulose. The degradation (amorphous cellulose) actually occurs earlier where similar thermal properties of hexose components of hemicelluloses. This is due amorphous regions were known more susceptible to thermal degradation than crystalline structure. Thus, a few amount of cellulose content (amorphous cellulose) was degraded earlier while the degradation of this temperature (315 °C) refers to the degradation of crystalline cellulose. In this study, it was observed in DTG of fibres (Fig. 9) where crystalline cellulose degrades in the temperature range of 300–370 °C with the highest peak of decomposition occurred at average temperature of 330 °C. This is supported by Kim et al. [44] where they reported that the critical temperature of decomposition of crystalline cellulose is 320 °C. They also reported that the crystalline cellulose did not decompose at 300 °C, but completely decomposed at 340 °C. However, different to Yang et al. [42] where they reported that cellulose decomposes at higher temperature where it is converted to non-condensable gas organic vapours and aerosols once 400 °C is attained. Thus, wide range of

thermal degradation of crystalline cellulose can be concluded where it occurs in the range of temperature of 300–400 °C. Although the decomposition of crystalline cellulose requires higher temperature to decomposed, once the required temperature is achieved, the decomposition occurs at a very high mass loss rate. It is observed in Fig. 9 that the portion of crystalline cellulose shows its highest peak during their decomposition process. This is similar to the finding of Yang et al. [42] as shown in DTG curve (Fig. 10) where the highest peak was cellulose compared to hemicelluloses. Similar to the case found in cellulose, due to lower cellulose content of fibre of 1 m (37.3%) compared to the fibres of 3–15 m (54.51%) (as shown in Table 1), mass loss rate of cellulose of 1 m was found to be lower (3.01 wt%/min at 327.54 °C) than the fibre of 3–15 m (6.59 wt%/min at 332.39 °C).

The fourth phase was the decomposition of lignin. It is the most difficult to decompose compared to hemicelluloses and cellulose. Although the decomposition of lignin had started as early as 160 °C, it decomposes slowly and extends its temperature as high as 900 °C to complete its decomposition [42]. This is contributed by lignin which is very tough component and known as the compound that gives rigidity to the plant materials. It is responsible for providing stiffness to the cell wall and also serves to bond individual cells together in the middle lamella region [39]. Thus, higher lignin content resulted in increase in stiffness of the plant materials. Based on Figs. 8 and 9, there was no significance difference in terms of TG and DTG curves of all fibres. The mass loss rate for both curves steadily show in reaching the equilibrium state and there was no significance mass loss since almost lignocelluloses composition had been decomposed. Its decomposition happened slowly under the whole temperature range from ambient to 900 °C, but at a very low mass loss rate (<0.14 wt%/°C) [42]. Due to its wide range of decomposition and very low mass loss rate, it is hard to see the peak of lignin by observing its DTG curve in Fig. 9 as well as in Fig. 10.

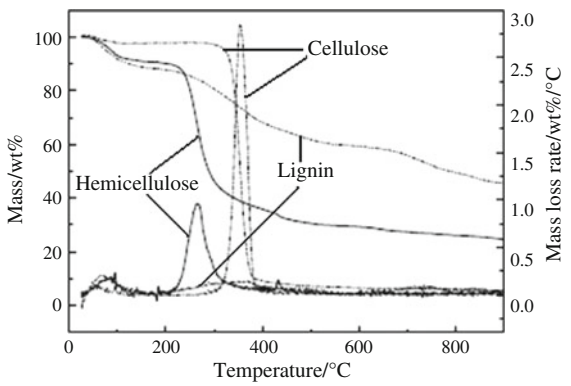

Fig. 10 Pyrolysis curves of hemicelluloses, cellulose and lignin in TG/DTG [42]

Finally, when the lignin had completely decomposed, the component that is left is inorganic material in the fibres which can be assumed as ash content. This is due to the presence of inorganic materials such as silica (silicon dioxide, SiO_2) in the fibre which can only be decomposed at a very high temperature of 1723 °C [45]. Figure 8 clearly indicates that the ash content of 1 m fibre showed consistently higher content than others. The presence of silica in the fibre is higher due to the relative position of the fibres to the ground (1 m). Thus, as the fibre has higher ash content which is about 31% (as shown in Table 1) the ash had blocked reactive chemical groups and suppressed the percentage weight loss of the fibre. Hence, this is the main reason of higher thermal stability of the fibre of 1 m compared to others. This is also the main reason of TG and DTG curves of 1 m fibre had a great difference and consistently higher than the others (3–15 m).

Conclusions

Tensile and thermal properties of sugar palm fibre obtained from different heights (1, 3, 5, 7, 9, 11, 13 and 15 m) were investigated. This study has confirmed that in a matured sugar palm tree, degradation had occurred and altered the properties of its fibre. Fibres obtained from the area of live (green) palm frond were found to have a better tensile properties as a result of their optimum chemical compositions especially cellulose, hemicelluloses and lignin. For the fibre obtained from the upper part of sugar palm tree, it shows slightly decreasing trend in tensile properties compared to matured fibres. It is because the fibres are juvenile where their cell walls are progressively built up in the fibres thus they give slightly lower properties than matured fibres. For the fibre obtained from the area of dead palm frond, the fibres are considered to be degraded biologically. It is believed that polymeric chains in microfibrils were broken and their cellulose content was decreased which demonstrated inferior properties (tensile strength, modulus, elongation at break and toughness). This study also qualitatively proved the existence of main chemical components (cellulose, hemicelluloses and lignin) in fibres. Thermal degradation of these compositions was found to be present in four phases where the sequence of decompositions is: moisture $\gg$ hemicelluloses $\gg$ cellulose $\gg$ lignin $\gg$ ash. The thermal degradations of these components were found to be in the range of 45–123, 210–300, 300–400, 160–900 and 1723 °C, respectively. In general, there were only two different curves observed in TG and DTG where the fibre of 1 m shows consistently higher thermal stability than the fibre of 3–15 m. This was due to the fact that much higher ash content was believed to be containing higher silica content for fibre of 1 m. It resulted in significantly higher

thermal stability. In conclusions, the properties of sugar palm fibres depend on the condition of fibre, the use of fibres obtained at region of dead palm frond for application as reinforcing fibre in composite should be avoided since the strength of the fibre and composite will be reduced.

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