SPECTROSCOPIC, THERMAL, AND ANATOMICAL CHARACTERIZATION OF CULTIVATED BAMBOO (*GIGANTOCHLOA* SPP.)

Irshad-ul-Haq Bhat,[a] Mohd. Tamizi B. Mustafa,[b] Abd. Latif Mohmod,[b] and H. P. S. Abdul Khalil [a,*]

This paper presents spectroscopic, thermal, and morphological properties of two bamboo species viz. *Gigantochloa brang* and *Gigantochloa wrayi*. The nature of cell wall structure and distribution of vascular bundles in *G. brang* and *G. wrayi* were studied by scanning electron microscopy and transmission electron microscopy techniques. *Gigantochloa* spp. at various positions and locations showed identical thermal stability and are stable up to 200 °C. The decomposition of cellulose and hemicelluloses component of the culm occurred between 220 °C and 390 °C, while the degradation of lignin takes place above 400 °C.

Keywords: Bamboo; FTIR; Thermogravimetric analysis (TGA); Scanning electron microscopy (SEM); Transmission electron microscopy (SEM)

Contact information: a: School of Industrial Technology, Universiti Sains Malaysia, 11800, Penang, Malaysia; b: Forest Research Institute Malaysia, Kepong, 52109 Kuala Lumpur, Malaysia.
** Corresponding author: akhalilhps@gmail.com*

INTRODUCTION

Bamboo, which grows abundantly in most of the tropical countries, is extensively used in construction, agriculture, and the chemical industry. Total production of bamboo pulp is about 100 million T/a, most of which is used for papermaking, textiles, Lyocell, etc. (Ma et al. 2004). Bamboo is strong, lightweight, flexible, economical, and handy to use in building. Bamboo is a vital resource from the perspective of environmental problems because of its renewable and sustainable properties (Nakajima et al. 2010). Bamboo utilization has been increased in many Asian countries (Uchimura 2007), and reports on advanced utilization have been reported recently (Yamashita 2005). On the one hand, bamboo has been viewed as an agricultural waste material with a low value product for pulping and paper industry. On the other hand, bamboo is known as a material source for furniture, building, pulp, particleboard, bioenergy, food, forage, and medicine. It plays a vital role in bio-diversity preservation, soil and water conservation, and has waste purification potential (Kelecha 1980; Ayre-Smith 1963; Embaye 2000).

The structural organization of the bamboo cell wall and the vascular bundles at the inner, middle, and outer positions of the internode and the node of the culms of *Gigantochloa brang* and *Gigantochloa wrayi* have been investigated using SEM and TEM. Thorough understanding of thermal stability and influence of the processing temperatures in relation to the processing duration is essential. Therefore, the spectroscopic, thermal, and morphological characterization of two bamboo species

(*Gigantachloa brang and Gigantachloa wrayi*) at different locations (internode and node) and position (outer, middle and inner) of the culm was carried out.

EXPERIMENTAL

Materials and Procedures

Three years old bamboo (*Gigantachloa brang and Gigantachloa wrayi*) culms were collected from the experimental fields of the Forest Research Institute Malaysia (FRIM), Selangor, Malaysia (Latitute 3° 14′ N, Longitude 101° 38′ E, Altitude 80-90 m). The selected bamboo culms were cut at 15 cm above ground level with diameters ranging from 11 to 17 cm. Ten representative bamboo culms (10 poles/species) for each species were further cut into internodes and nodes. The epidermis of each strip was first slightly scraped off with a fine blade. The remaining material was divided evenly based on volume into inner, middle, and outer layers along the radial direction. The 15-cm-long fresh bamboo sections cut from each culm locations were washed with water and then dried in an oven at 50 °C for 72 h. Some portion of the bamboo samples were reduced into chips using a commercial chipper, then screening to the size between 40 mesh (425 μm) and 60 mesh (250 μm).

FT-IR Characterization of *Gigantachloa* spp.

FT-IR spectra were obtained with dried bamboo powdered recorded as KBr disks on a Nicolet AVATAR 360 Fourier Transform Infrared Spectrophotometer. Approximately 1 mg samples were dispersed in a matrix of KBr (100 mg) and pressed to form pellets. The spectra were measured at a spectral resolution of 4 cm^{-1}, and 64 scans were recorded per sample.

Morphological studies of *Gigantachloa* spp.

The microscopic appearance of bamboo samples was examined with a scanning electron microscope (Leo Supra, 50 VP, Carl Ziess, SMT, Germany, SEM) using small samples of 1 to 2 mm thickness. Specimens were sputter-coated with gold to a thickness of ca. 10 nm in order to prevent charging during the examination. An accelerating voltage of 15 kV was used to collect the SEM images. The transverse sections of thickness 1 μm were cut using a Sorvall Ultra microtome (MT 500) with a glass and diamond knife. For anatomical characterization and lignin distribution determination, embedded samples were stained with 1% toluidine blue and were viewed under a polarized microscope (Olympus BX50) for transverse and longitudinal section. For the ultra-thin sections (0.1 μm), samples were stained with 2% uranyl acetate and lead citrate. The transverse sections were examined with a Phillips CM12 transmission electron microscope (TEM).

Thermogravimetric Measurements

Thermogravimetric and differential thermogravimetric analysis of bamboo samples were carried out using a Shimadzu (Model 30) thermal analyzer at heating rates 20 $^{o}C\ min^{-1}$, and the temperature ranged from 30 to 900 ^{o}C under nitrogen.

RESULTS AND DISCUSSION

FTIR Spectroscopy

FTIR spectra of *Gigantochloa brang* and *Gigantochloa wrayi* extracted from three different layers of the node and internodes were found to be identical in all respects (Figs. 1 and 2). Three different portions (inner, middle, and outer layer) of the internodes or nodes showed the same absorption bands with no significant differences in intensity. This revealed that irrespective of the location, the chemical compositions of the bamboo fiber were all essentially the same.

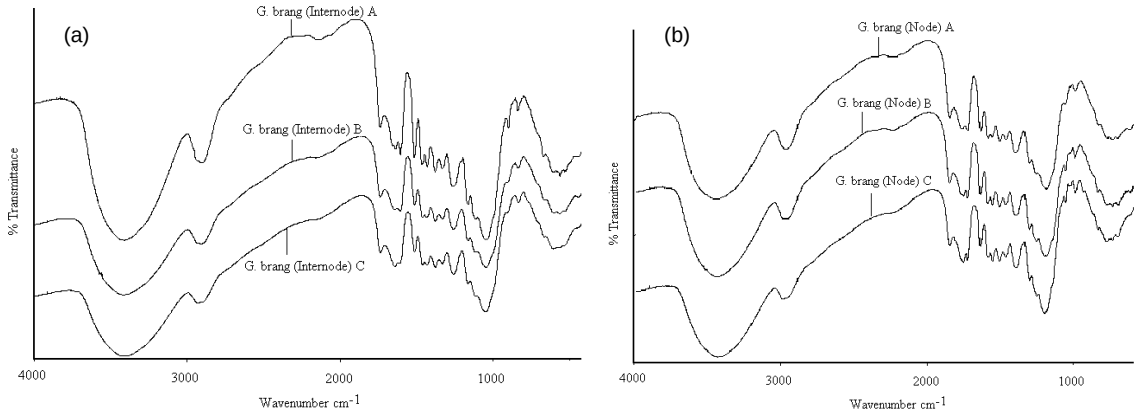

Figure 1. FTIR spectra of *Gigantochloa brang* internode (a), and node (b)

All FTIR spectra were dominated by the bands at 3411 cm^{-1} and 1048 cm^{-1}, indicating the stretching vibrations of ν(OH) and ν(CO), respectively, present in the bamboo constituents.

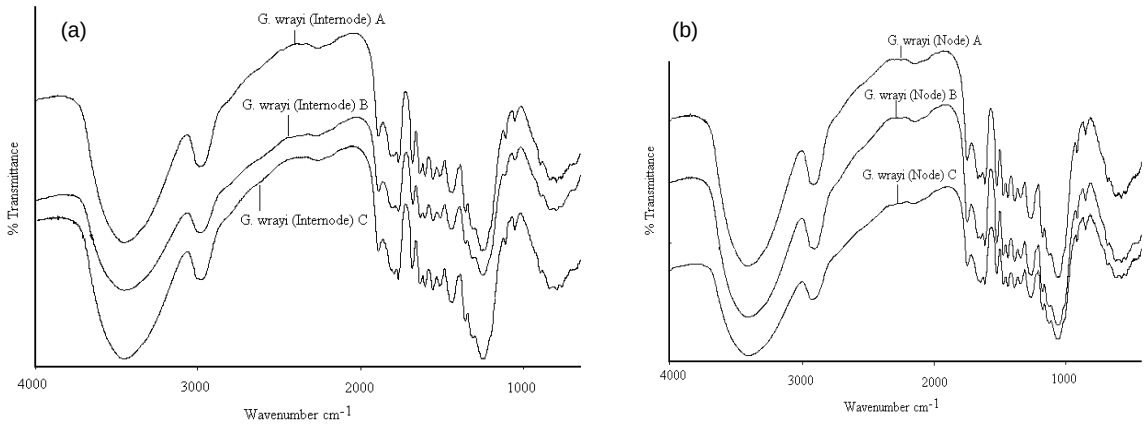

Figure 2. FTIR spectra of *Gigantochloa wrayi* internode (a), and node (b)

The chemical composition of bamboo comprises mainly cellulose, hemicellulose, and lignin, in common with other lignocellulosic materials such as wood, empty fruit bunch fiber, coir fiber, etc. (Scurlock et al. 2000; Li et al. 2007). The absorption bands at 3411 cm^{-1} and 2906 cm^{-1} were ascribed to stretching frequencies of ν(OH) and ν(CH_2), respectively. The absorption band at 1733 cm^{-1} is ascribed to the stretching vibration of

the carbonyl group ν(C=O) of the cellulose and hemicelluloses component in the bamboo (Herrera-Franco and Valadez-Gonzalez 2005; Silverstein et al. 2005). The absorption at 1510 cm^{-1} is associated with the ν(CH) bending vibration of the aromatic ring bond present in lignin component (Tserki et al. 2005; El Oudiani et al. 2009). Furthermore, the small absorption bands at 1603 cm^{-1}, 1510 cm^{-1}, 1459 cm^{-1}, and 1335 cm^{-1} correspond to the aromatic skeletal vibrations and ring breathing with ν(C−O) stretching in aromatic ring of the lignin (Silverstein et al. 2005). The absorption at 1510 cm^{-1} is associated with the ν(CH) bending vibration of the aromatic ring bond present in the lignin component (El Oudiani et al. 2009). The slight variations in the intensity of bands in the region around 1603 cm^{-1}, 1459 cm^{-1}, and 1428 cm^{-1} were observed. These observations could be due to the variations of lignin content of the culms (Ganan et al. 2008).

Thermal Stability of Various Bamboo Species

The thermal studies of *G. brang* and *G.wrayi* were carried out in the presence of nitrogen and are given in Figs. 3 and 4. Also, the DTG curves of bamboo are shown in Figs. 5 and 6, while the summary of the major peak temperature obtained from these TGA curves are given in Table 1. Figure 3 shows the TGA curves of the inner, middle, and outer layer of *G. brang* internodes, while Fig. 5 shows the DTG curves. The TGA curves showed a slight weight loss before 100 °C, which can be attributed to the evaporation of water.

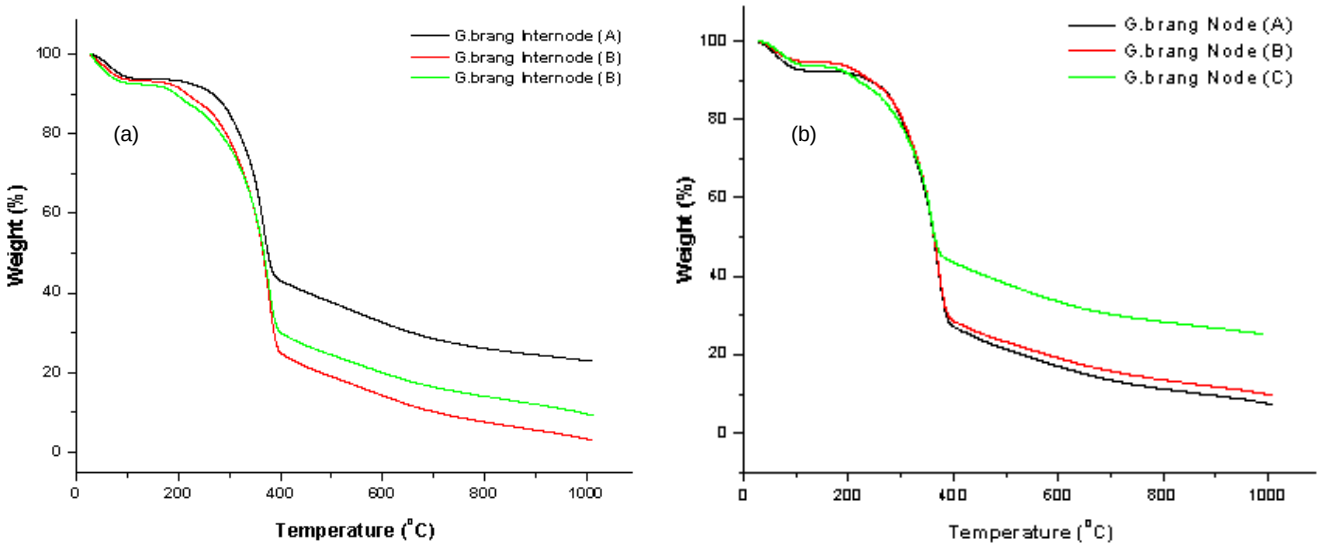

Figure 3. Thermogravimetric analysis of *Gigantochloa brang* internode (a), and node (b)

The slight weight loss ranged between 6.4 to 7.5%, in which the middle position (*G. brang* Internode B and *G. wrayi* Internode B) exhibited the lowest value. The lower values of moisture content recorded (Table 1) might be due to reduction in hydrophilic tendency associated with the reduction of the free hydroxyl of the phenolic groups present in the cellulosic and lignin components.

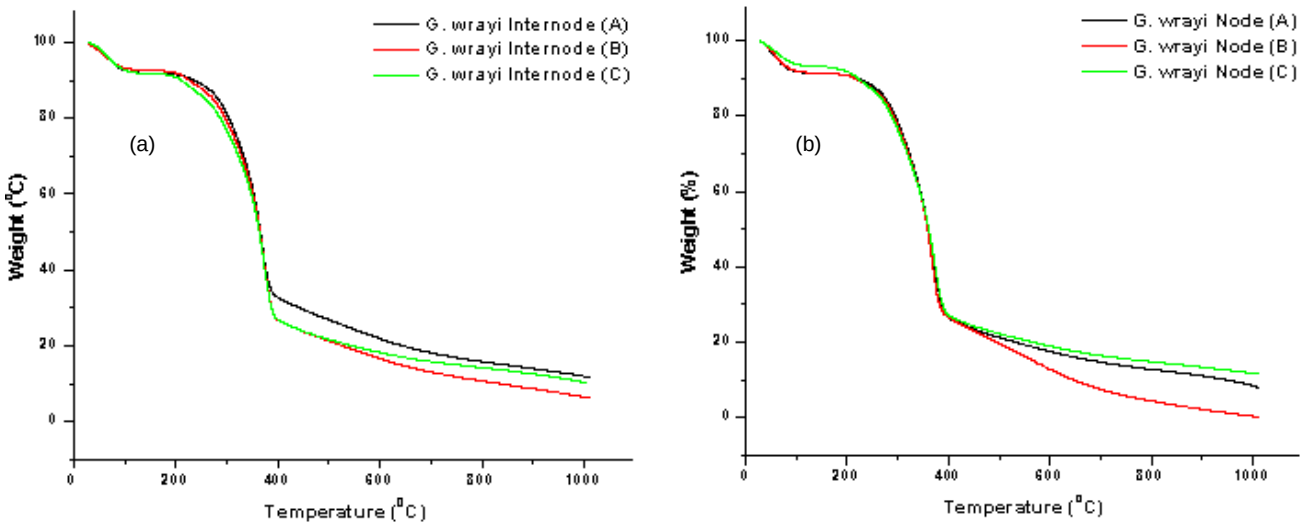

Figure 4. Thermogravimetric analysis of *Gigantochloa wrayi* internode (a), and node (b)

The slight weight loss at the node ranged between 4.9 to 7.1%, in which the middle position (B) had the lowest value. The *G. brang* and *G. wrayi* internodes samples were stable up to 210 °C, and thereafter they started to decompose.

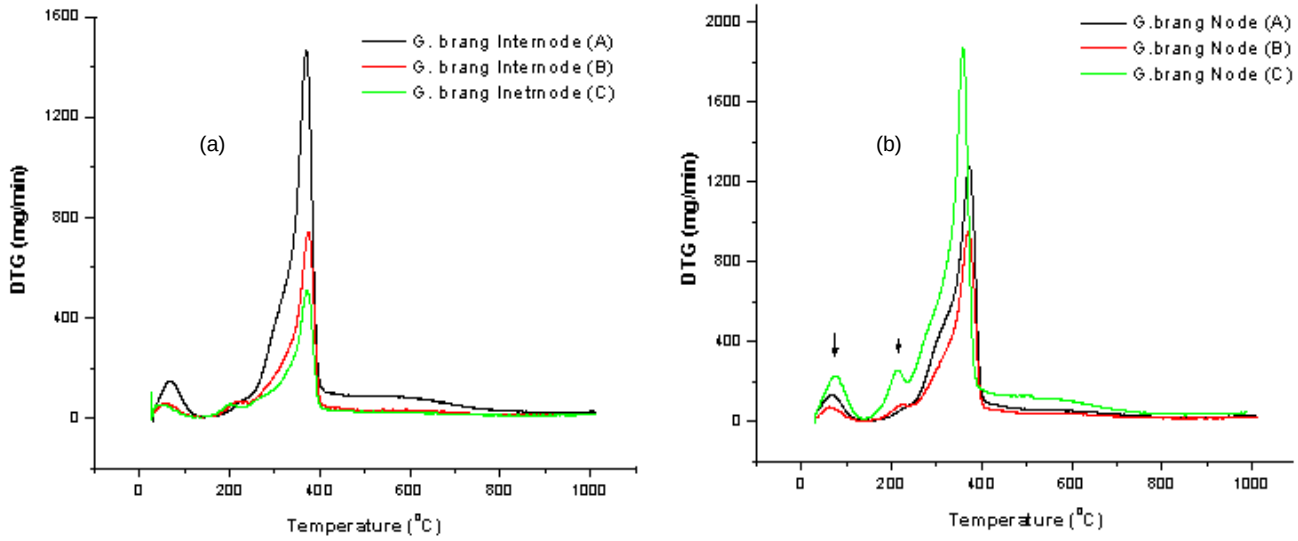

Figure 5. DTG analysis of *Gigantochloa brang* internode (a), and node (b)

Similar observations were made with respect to the node (Table 1). In all cases, the outer, middle, and inner locations of the *G. brang* and *G. wrayi* fiber at the node and internode decomposed in two stages. The first region, between 210 and 390 °C, was associated with of the bamboo constituents, which are mainly cellulose, lignin, and hemicellulose (Nabi Saheb and Jog 1999; Gomez et al. 2007). The second stage of degradation between 390 °C and 800 °C in the entire sample might involve decomposition of lignin. The thermal degradation of lignin is a complex process because

the materials have many components with different decomposition pathways, including competitive and/or consecutive reactions. Lignin thermally decomposes over a broad temperature range, because various oxygen functional groups from its structure have different thermal stabilities, their scission occurring at different temperatures (Brebu and Vasile 2010). Thermal studies on different bamboo fibers are well documented in literature (Das M and Chakrabarty 2008; Rajulu et al 2002). Table 1 shows the comparison of the weight losses, the maximum temperature, and the residual weight loss at 800 °C.

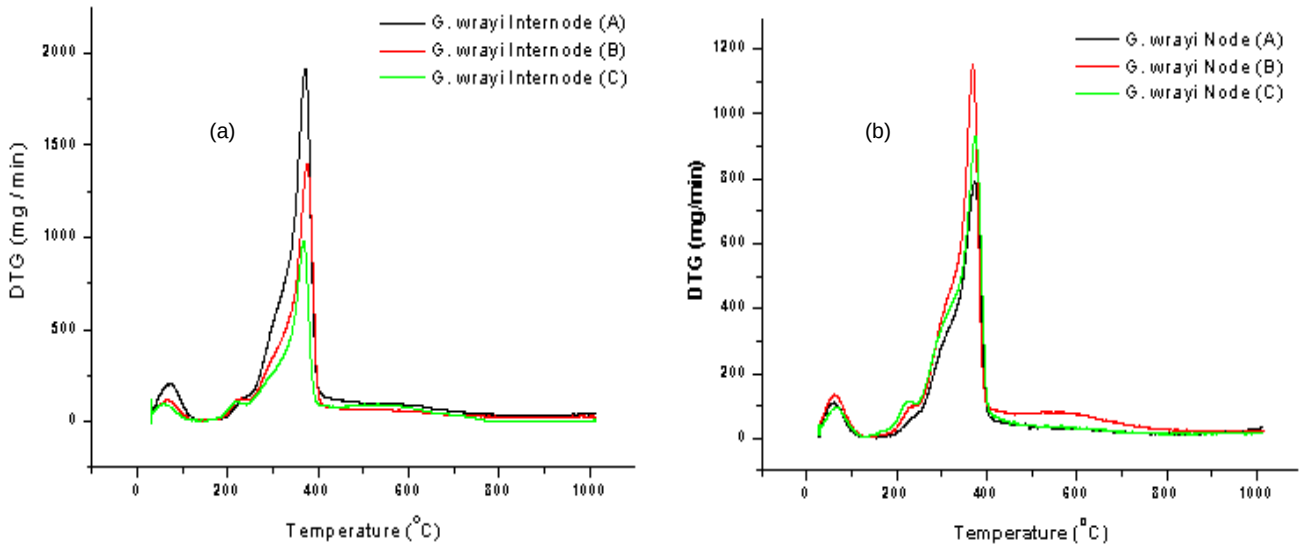

Figure 6. DTG analysis of *Gigantochloa wrayi* internode (a), and node (b)

Table 1. Thermal Degradation Temperatures and Residue Weight of *Gigantochloa brang*

Position (800 °C)	Moisture	T_{10} (°C)	T_{30}(°C)	T_{50} (°C)	T_{70} (°C)	Residual weight %
G. brang Internode (A)	7.5	268	347	375	659	26.1
G. brang Internode (B)	6.4	216	327	365	387	7.6
G. brang Internode (C)	7.4	195	325	367	401	14.1
G. brang Node (A)	7.1	238	327	362	387	11.2
G. brang Node (B)	4.9	241	330	363	390	13.5
G. brang Node (C)	6.0	216	329	364	711	28.3

The degradation temperatures for the inner layers at different time intervals were higher than those for the middle and outer layer at the internode and node.

Table 2. Thermal Degradation Temperatures and Residue Weight of *Gigantochloa waryi*

Position (800 °C)	Moisture	T_{10} (°C)	T_{30}(°C)	T_{50} (°C)	T_{70} (°C)	Residual weight %
G. wrayi Internode (A)	7.8	240	332	366	446	15.8
G. wrayi Internode (B)	7.1	229	328	364	386	10.7
G. wrayi Internode (C)	7.8	214	322	363	387	14.2
G. wrayi Node (A)	8.4	221	322	360	385	12.8
G. wrayi Node (B)	8.2	214	320	358	381	4.4
G. wrayi Node (C)	6.5	220	318	360	587	14.8

The thermal depolymerization of the hemicellulose and the cleavage of the glycosidic bonds of the cellulose are responsible for the degradation at about 230 to 300 °C. However, the higher degradation above 390 °C is due to decomposition of lignin (Manfredi et al. 2006). Further heating to 800 °C led to an average residual weight of 4 to 28 % for all the samples.

Table 3. Differential Thermogravimetry (DTG) Temperatures of *Gigantochloa brang*

	(DTG temperatures peaks)		
Sample	Moisture	Minor peak (T1)	Major Peak (T2)
G. brang Internode (A)	68	-	369
G. brang Internode (B)	59	-	373
G. brang Internode (C)	49	-	370
G. brang Node (A)	67	223	371
G. brang Node (B)	64	221	370
G. brang Node (C)	74	211	357

Table 4. Differential Thermogravimetry (DTG) Temperatures of *Gigantochloa wrayi*

	(DTG temperatures peaks)		
Sample	Moisture	Minor peak (T1)	Major Peak (T2)
G. wrayi Internode (A)	70	233	371
G. wrayi Internode (B)	66	228	375
G. wrayi Internode (C)	58	220	367
G. wrayi Node (A)	61	235	372
G. wrayi Node (B)	61	234	369
G. wrayi Node (C)	64	228	374

The middle position recorded the lowest residual weight in almost all cases (Table 1). This implies that the middle positions are least stable. This observation must have been due to lower lignin content of middle position compared with inner and outer position at both the internodes and nodes. It is concluded that the thermal stability of bamboo samples are similar irrespective of location and position in both species. Remarkable differences were observed in thermal stability above 380 ^{o}C. The thermal behavior of the bamboo species is comparable to other lignocellulosic fiber reported in the literature. This result is important if the bamboo fibers are to be applied as reinforcement or filler in thermoplastic polymers with high processing temperature. In addition, an overall summary of TGA and DTG data are given in Tables 1 to 4.

ANATOMICAL STUDY

The bamboo culms are made of three basic tissues namely epidermal, vascular, and ground. The vascular bundles consist of the xylem (which are mainly protoxylem and two metaxylem vessels) and the phloem, with thin walls, as shown in scanning electron micrograph in Fig. 7. The vascular bundle is oval in shape and varies in size depending on the location/position as well as species. Sclerenchyma sheaths surround the xylem and phloem, and are known to be principal supporting tissues within the vascular bundles (Grosser and Liese 1971). The microstructures are similar in both species and are identical at both the internode and node. The basic patterns of sclerenchyma bundle arrangements within the vascular bundles fall within Type III as proposed by Grosser and Liese (1971). The central vascular strand is separated from one distinct fiber strand outside, and the second fiber strand is located inside the central vascular strand. The width of the vascular bundle of G brang and G wrayi were 357 to 628 μm and 386 to 849μm, while the length also ranged from 729 to 1138 μm and 683 to 1122 μm, respectively.

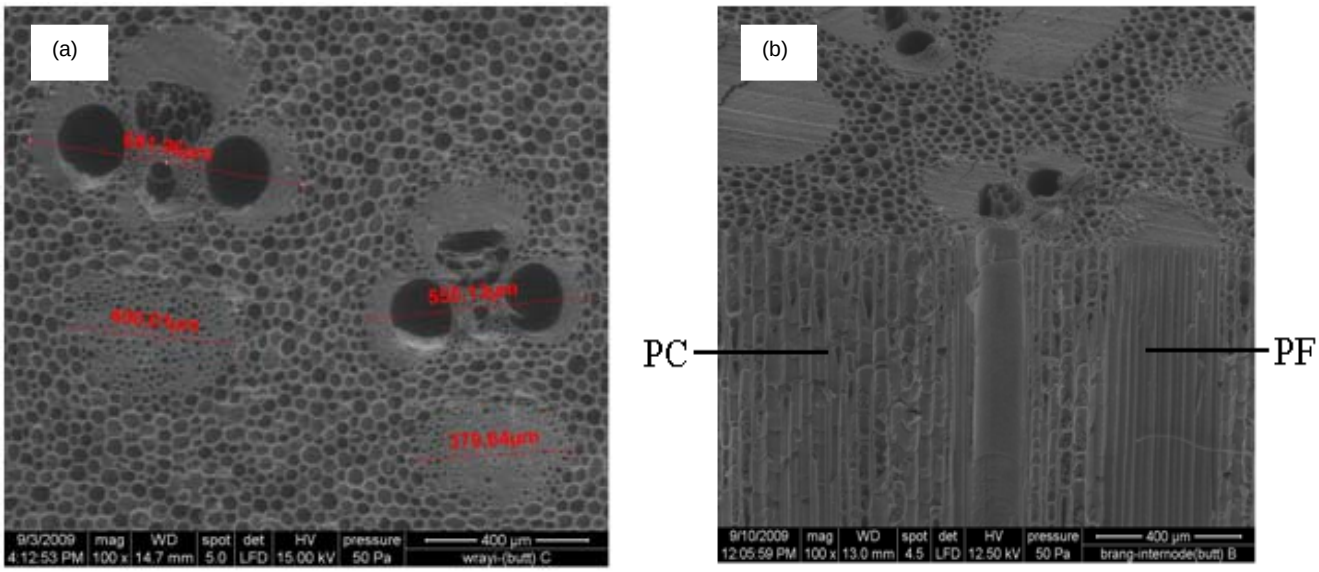

Figure 7 (a). TS of a typical vascular bundle from internode of *G. wrayi* showing fiber strand (F), metaxylem (MX), protoxylem (PX), phloem (P) of the vascular elements. (b) LS showing elongated fibres (F) and parenchyma (PC)

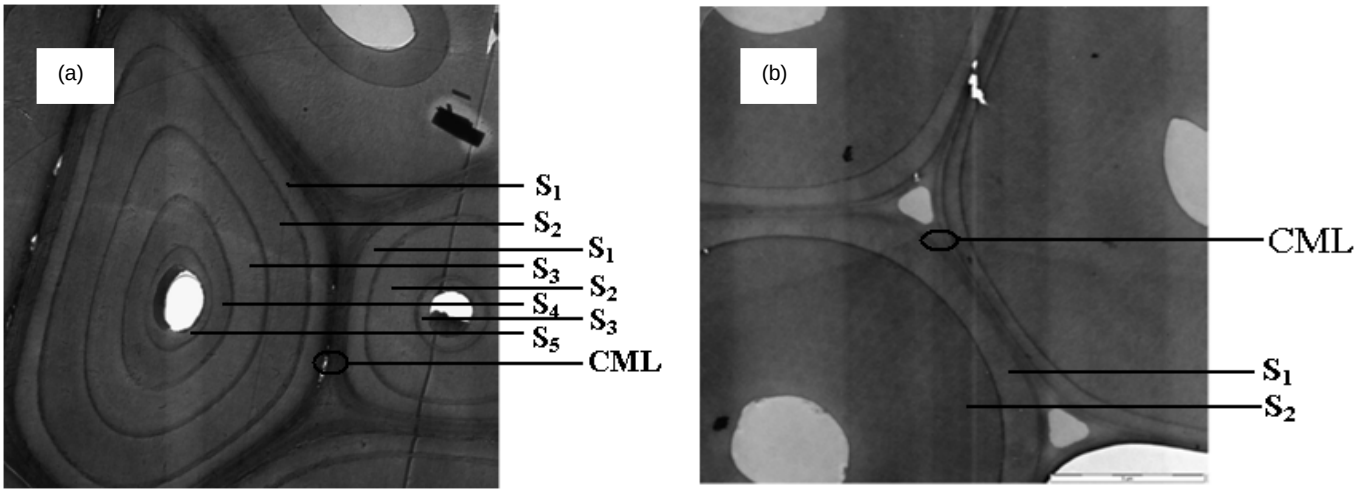

Figure 8. (a).TEM micrographs of cell walls of: A) four fibers from the node showing the middle lamella (CML) and varying secondary wall layers can be distinguished (S1–S5); (b) three fibers from the internode showing the middle lamella (CML), a thin dark transition layer, and two layers of secondary wall (S1 and S2).

The size of the vascular bundles in both species increased from outer through the middle to the inner position and this type of anatomical property is well reported (Wahab et al 2010). The magnitude of the length and width are higher at the node compared with the internodes except the width on the inner. The average numbers of vascular bundle per 4 mm^2 at the internode are higher compared with the node. The average number per surface area of the vascular bundle is of the order of outer> middle> inner. Thus, the concentration at the outer position is distinctly higher and well over 200% higher than in the inner.

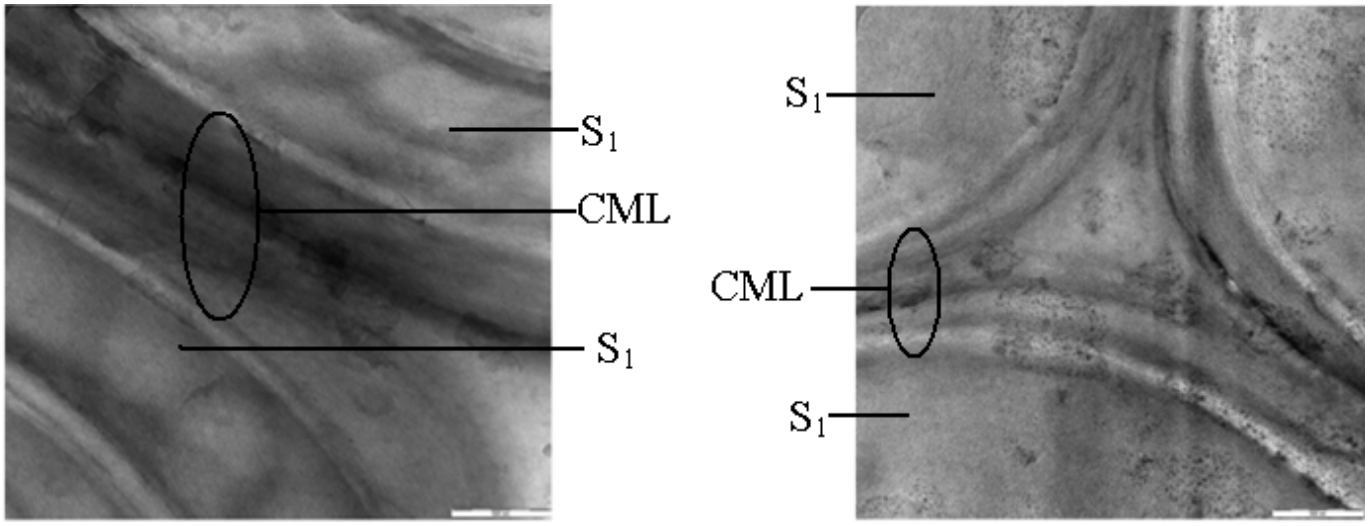

Figure 9. Cell walls of fibres showing secondary wall layer (S1, S2), Variation in coloration is due to changes in the concentration of lignin. The CML is darker indication more lignin concentration.

The increase in number of cells in the vascular bundles with secondary thickenings may be responsible for the observed increase in specific gravity (Li et al. 2007). The transmission electron micrographs revealed the variability in size, shape, and structure of the cell wall fibers, as shown in Fig. 8. The cell wall fibers were round to polygonal in shape. The fiber cell wall of various bamboo fiber exhibited a poly-lamellate structure with alternating lamellae. The middle lamella separates two cells and appears as the outer-most layer, followed by the primary wall and the secondary wall, as shown in the Fig. 9. The middle lamella binds the cells together and may sometimes not show any clear transition to the adjacent wall layers. In such instance, the middle lamella and both adjacent primary walls are termed the compound middle lamella (CML). The secondary wall *Gigantochloa* is made up of two or more layers, represented as S1, S2, S3, etc. The various cell wall layers could be identified clearly in the TEM micrographs. For instance, two distinct secondary walls at the internodes while more than two secondary walls were visible at the nodes (Fig 8). The observed darkening due to toluidine blue staining in the middle lamella indicated that it was highly lignified (Abdul Khalil et al. 2006, 2010). Toluidine blue is a polychromatic stain that has been widely used to differentiate lignified tissues, which are generally stained bluish, from non-lignified ones (Abdul Khalil et al. 2010). The transverse section of bamboo wall demonstrated that CML were well stained very dark, whereas the primary walls, the secondary walls were less stained are lighter (Fig 9). Changes in the concentration gradient within the secondary walls due to differences in the staining intensity were observed. The fiber walls of many monocotyledons were characterized by successive lignifications, as cell walls of living fibers thicken with increase in age (Li et al. 2007). The high lignin content of the cell wall of bamboo fibers contributes to its high heating value and its structural rigidity, with the latter making it a valuable building material (Li et al. 2007). The lignin particles occur in high concentrations throughout the CML and intermingle with the microfibrils of the S1, which appears brighter indicating a low lignin concentration. The transition between S1 and S2 is characterized by a change in microfibril direction and also by an increase in lignin concentration. Similar behavior of lignin distribution in bamboo has been reported by other workers (Gritsch and Murphy 2005; Li et al. 2007).

CONCLUSIONS

A spectroscopic, thermal, and morphological investigation of *Gigantochloa brang* and *Gigantochloa wrayi* was carried out in order to evaluate the basic fundamental properties of these bamboo species. FTIR spectra of culms did not exhibit any major differentiation in spectra. However, thermal studies of different portions showed variation in weight loss from different portions of bamboo spp. as observed with different residual weight percentage. Different degrees of distribution of vascular bundles, xylem, and phloem were observed. The results revealed that study on fundamental properties of different bamboo section can be an alternative measure to be used in conjunction with the strength analysis of bamboo for construction use.

REFERENCES CITED

Abdul Khalil, H. P. S., Alwani, M. S., and Mohd Omar, A. K. (2006). "Chemical composition, anatomy, lignin distribution, and cell wall structure of Malaysian plant waste fibers," *BioResources* 1, 220-232.

Abdul Khalil, H. P. S., Yusra, A. F. I., Bhat, A. H., and Jawaid, M. (2010). "Cell wall ultrastructure, anatomy, lignin distribution, and chemical composition of Malaysian cultivated kenaf fiber," *Ind. Crop. Prod*. 31, 113-121.

Beckers, E. P. J., Meijer, De M., Miltiz, H., and Stevens, M. (1998). "Performance of finishes on wood that is chemically modified by acetylation," *J. Coat. Tech*. 70, 59-67.

Brebu, M., and Vasile, C. (2010). "Thermal degradation of lignin – A review," *Cellulose Chem. Technol*. 44, 353-363.

El Oudiani, A., Chaabouni, Y., Msahli, S., and Sakli, F., (2009). "Physico-chemical characterisation and tensile mechanical properties of *Agave americana* L. fibres," *J. Text. Inst.* 100, 430-439.

Embaye, K., Christersson, L., Ledin, S., and Weih, M. (2003). "Bamboo as bioresource in Ethiopia: Management strategy to improve seedling performance (*Oxytenanthera abyssinica*)," *Biores. Tech*. 88, 33-39.

Embaye, K. (2000). "The indigenous bamboo forests of Ethiopia: An overview," *Ambio* 29, 518-521.

Ganan, P., Zuluaga, R., Restrepo, A., Labidi, J., and Mondragon, I. (2008). "Plantain fibre bundles isolated from Colombian agro-industrial residues," *Bioresource Technol*. 99, 486-491.

Gomez, C. J., Meszaros, E., Jakab, E., Velo, E., and Puigjaner, L. (2007). "Thermogravimetry/mass spectrometry study of woody residues and an herbaceous biomass crop using PCA techniques," *J. Anal. Appl. Pyrol*. 80, 416-426.

Gritsch, C. S., and Murphy, R. J. (2005). "Ultrastructure of fibre and parenchyma cell walls during early stages of culm development in *Dendrocalamus asper*," *Annals of Botany* 95, 619-629.

Grosser, D., and Liese, W. (1971). "On the anatomy of Asian bamboos, with special reference to their vascular bundles," *Wood Sci. Technol*. 5, 290-312.

Li, X. B., Shupe, T. F., Peter, G. F., Hse, C. Y., and Eberhardt, T. L. (2007). "Chemical changes with maturation of the bamboo species *Phyllostachys pubescens*," *J. Trop. For. Sci*. 19, 6-12.

Ma, N. X., Zhang, W. Y., and Chen, G. C. (2004). "Viewpoints on increasing the national bamboo pulp production for paper making," *China Forestry Sci. Tech.* 18, 8-11.

Manfredi, B. L., Rodriguez, E. S., Wladyka-Przybylak, M., and Vazquez, A. (2006). "Thermal degradation and fire resistance of unsaturated polyester modified acrylic resins and their composites with natural fibers," *Polym. Degrad. Stab*. 91, 255-261.

Nabi Saheb, D., and Jog, J. P. (1999). "Natural fiber polymer composites: A review," *Adv. Polym. Tech*. 18, 351-363.

Nakajima, M., Kojiro, K., Sugimoto, H., Miki, T., and Kanayama, K. (2010). "Studies on bamboo for sustainable and advanced utilization," In Press., *Energy,* xxx 1-6.

Rajulu, A., Reddy G. R., Chary, K.N., Rao, G. B., and Devi, L. G. (2002). "Thermogravimeteric analysis of *Dendrocalamus strictus* bamboo fibers," *J. Bamboo and Rattan*. 1, 247-250.

Scurlock, J. M. O., Dayton, D. C., and Hames, B. (2000). "Bamboo: An overlooked biomass resource?" *Biomass Bioenerg*. 19, 229-244.

Silverstein, R. M., Webster, F. X., and Kiemle, D. J. (2005). *Spectrometric Identification of Organic Compounds*, John Wiley & Sons, Inc.

Tserki, V., Zafeiropoulos, N. E., Simon, F., and Panayiotou, C. (2005). "A study of the effect of acetylation and propionylation surface treatments on natural fibres," *Compos. Part A: Appl. Sci. Manuf*. 36, 1110-1118.

Uchimura, E. (2007). *Takenomiryokutokatsuyou*, Soshinsha, Tokyo [in Japanese].

Wahab, R., Tamizi, M. M., Sulaiman, O., Aminuddin, M., Affendy, H., and Izyan, K. (2010). "Anatomical and physical properties of cultivated two- and four-year-old *Bambusa vulgaris*," *Sains Malaysiana* 39, 571-579.

Yamashita, O., Yokochi, H., Imanishi, H., and Kanayama, K. (2005). "Biodegradable plastic substitute from bamboo," Proceedings of the 3rd Dubrovnik Conference on Sustainable Development of Energy, Water and Environmental Systems, Dubrovnik, Croatia, 133-140.

Article submitted: January 24, 2011; Peer review completed: March 11, 2011; Revised version received and accepted: April 1, 2011; Published: April 3, 2011.