Thermal Degradation Analysis on 4-year-old Culms of Cultivated Tropical Bamboo *Bambusa Vulgaris*

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Abstract

The thermal degradation of matured 4-year-old culms of cultivated tropical bamboo *Bambusa vulgaris* was studied and analyzed. The analysis using the Fourier Transform Infrared Spectroscopy and Thermal Gravimetric Analysis revealed the presence of basic functional groups in the bamboo which consists mainly of ester, carbonyl, and hydroxyl groups. The chemicals present in the bamboo vary depending on the location of the samples taken from the bamboo culms. The moisture content and extractive were omitted in the kinetic study since they constituted less than 10% of the overall chemicals in bamboo and observed below 100°C. Low reactivity of lignin components and hemicellulose in bamboo occurred due to the peculiarities of the chemical structure and composition. The mechanism of the decomposition

reactions taken as a three-step reaction which involved the activation energies and dynamics related to volatile fractions of hemicellulose, cellulose, and lignin. Activation energies of 46, 100, 105, 127, and 236 kJ/mol applied for all of the bamboos. The activation energy carried could provide better insight into the thermal decomposition process. It provides more information on critical energy needed to start a reaction. The decomposition activation energy range obtained could assist in understanding the thermal decomposition stability of the bamboo fibers and application in natural fiber reinforced polymer composite industry.

Keywords: Cultivated *Bambusa vulgaris*, thermal degradation, chemical constituents, Fourier transform infrared spectroscopy, thermal gravimetric analysis.

1. Introduction

Plant-based materials often used as durable goods and part of construction materials due to their robust chemical and excellent physical properties (Wahab *et al*., 2007; Wahab *et al.* 2005)). These materials often referred to as lignocellulosic-based on their main chemical components, cellulose, and lignin (Wahab *et al*., 2016). At high temperature, these lignocelluloses materials are subject to thermal degradation. This action depending on the temperature and environment they are exposed can take different forms. The Fourier transform infrared spectroscopy (FTIR) is an essential equipment for studying the bamboo decay, as well as to characterize the chemical composition of bamboo. It is also helpful to analyze chemical and structural changes that occur in bamboo components due to a different temperature. The thermal gravimetric analysis (TGA) used in studying the thermal stability of bamboo species. The measurements in TGA can perform during a rising in temperature or under a temperature program. The TGA is one of the thermal analysis techniques used to quantify weight changes and thermal decomposition of the sample. The thermal behavior affects the major factors in biomass chemical composition, heating rate, temperature, and inorganic substances.

The culms wall of bamboo principally made of the polymer cellulose in the form of microfibril, hemicellulose, and lignin. The composition of these four constituents of the wall in bamboo tissue varies between species and variate in the physical structures. The variation in parts within bamboo materials has an impact on the thermal-mechanical and thermos dielectric properties. Information on the thermic stability of materials is necessary to determine their thermomechanical or thermoelectric properties. Tropical bamboo species of *B. vulgaris* analyzed for thermal degradation. The process of degradation of bamboo material through thermal analysis was studied and tried to know the influence practiced by the constituents of the wall. The thermal characterizations of selected bamboo species are performed by Fourier transform infrared spectroscopy (FTIR) and Thermal gravimetric analysis (TGA).

2. Materials and Methods

2.1 Samples Preparation

Four (4) years old bamboo culms *B. vulgaris* collected from the Kepong Botanic Garden in the Forest Research Institute Malaysia (FRIM). The selected bamboo culms harvested having

a diameter of 10-18 cm at diameter at breast height (DBH). The bamboo culms segregated into internode and nodes. The epidermis of each strip was slightly scraped off with a blade. The strips then divided based on volume into inner, middle and outer layers along the radial direction. A 15 cm long fresh bamboo sections cut from each culm locations were dried in an oven at 50 ℃ for 72 h. Some parts of the bamboo reduced into chips using a chipper, screened to the size of 40 mesh and 60 mesh. Technique develops by Wahab *et al.* (2014), Wahab *et al.* (2010a) and Wahab *et al.* (2010b) were followed in the samples preparation.

2.2 Fourier Transform Infrared Spectroscopy (FTIR) Measurements

A Nicolet AVATAR 360 spectrometer was used for the FTIR tests, taking 32 scans for each testing sample with a resolution of 4 /cm. The bamboo pounded in a mortar and ~1 mg of the obtained powder dispersed in 100 mg of KBr. The bamboo dried before dispersion in KBr, and the mixed powder pressed into a disk that was immediately analyzed.

2.3 Thermal Gravimetric Analysis (TGA) Measurements

A Shimadzu (Model 30) thermal analyzer with heating rates 20 ℃ per min and the temperature, ranged from 30 to 1000 ℃ under nitrogen was used to analyze the simultaneous and differential thermogravimetric. The bamboo mass of about 5 mg and particle sizes below 0.18 mm (using sieve No. 80) were distributed evenly in an open platinum crucible. The experimental conditions ensure a negligible spatial gradient of the temperature inside the samples with negligible effects on the thermal decomposition of *B. vulgaris.* A duplicate sample of TGA taken for each test to cross check if the two curve overlaps on each other.

3. Result and Discussion

3.1 Fourier Transform Infrared Spectroscopy (FTIR) Analysis

Figures 1 and 2 shows the FTIR spectra of *B. vulgaris* internodes and nodes respectively. Irrespective of the site of the fiber taken, the chemical compositions of the bamboo fiber are similar in all aspect. No significant differences in intensity of the absorption bands noted in the fibers taken from three (3) different locations of the internodes or the nodes. Absorbance at 897, 1049, 1161, 1430, 1734, 2906, 3411 per cm was associated with the characteristic absorption of lignocelluloses materials (El Oudiani *et al.*, 2009; Yang *et al.*, 2008). These absorbances are present in all the FTIR of the bamboo studied irrespective of the location of the fiber. The FTIR spectra dominated by the peaks at 3411 and 1048 per cm indicating stretching vibrations of OH and CO, present in the bamboo constituents. The chemical compositions of bamboo fiber have shown to contain mainly, cellulose, hemicellulose, and lignin-like other common lignocelluloses fiber like wood, empty fruit bunch fiber and coir fiber (Wahab *et al*, 2014; Liu *et al.*, 2008). Table 1 shows the IR absorptions of the wavelength of peaks used for FTIR analysis and corresponding functional groups.

Table 2 shows the main IR peaks and their corresponding functional groups for the internode and node of *B. vulgaris* species. This spectra are typical style of lignocellulosic fiber. The absorption at 2899 per cm and 3412 per cm are attributed to the stretching vibration of OH groups and CH stretching, respectively, corresponding to the aliphatic moieties of

hemicelluloses, cellulose, and lignin. The band at 1733 per cm is ascribed to stretching vibration of the carbonyl group (CO) of the cellulose and hemicelluloses in the bamboo fiber (El Oudiani *et al.,* 2009; Herrera Franco and Valadez-Gonzailez, 2005).

The absorbance at 1335, 1459, 1510 and 1603 per cm, corresponds to the aromatic skeletal vibrations and ring breathing, with CO stretching in the aromatic ring of the lignins (Ganan *et al*., 2008; Yang *et al*., 2008; Sun *et al*., 2002). The bands in the 1604 and 1640 per cm region may attribute to CO stretching vibration of the alpha keto carbonyl in the cellulose component of bamboo (Silverstein *et al.,* 2005). The bands in the region between 1049 to 1248 per cm attributed to CO stretching vibrations of aliphatic primary and secondary alcohols in cellulose, hemicellulose, and lignin (Wang *et al.*, 2009; El-Meligy *et al.,* 2004; Sun *et al*., 2002). The second most prominent and strong band at 1046 per cm attributed to CO stretching in cellulose, hemicelluloses, and lignin or COC stretching in cellulose and hemicelluloses (Liu *et al.,* 2008; Sun *et al.,* 2002). The peak at 833 per cm was due to b-glucosidic linkage while the peaks at 664 and 607 per cm are due to out-of-plane bending vibration of inter-molecular H-bonded OH group and out-of-plane OH bending (Wahab *et al*., 2014; El-Meligy *et al.,* 2004).

Table 1. Wavelength of peaks used for FTIR analysis and corresponding functional groups.

Wavenumber per cm	Phenomenon	Functional Group
1200-1070	CO stretch	Ethers
1670-1615	CC stretch	Alkenes
1800-1680	CO stretch	Carbonyls groups
1465-1440	CH3	Methyl groups
1475-1400	CH2	Methylene groups
3000-2850	CH stretch	Alkyl groups
3550-3000	NH stretch	Primary amines
3600-3200	OH stretch	Alcohols, water

Table 2. The main IR peaks and their corresponding functional groups for the internodes and nodes.

Frequency [cm^{-1}]			Frequency [cm^{-1}]				
Internode			Node				Remark
Outer (A)	Middle (B)	Inner (C)	Outer (A)	Middle (B)	Inner (C)	Assign	
834	830	828	834	833	832	CH	Stretching of β-glucosidic linkage
1050	1044	1046	1050	1054	1052	COC	Asymmetric stretching of the ester group
1426	1428	1427	1426	1426	1426	OH	Plane bending vibration of hydroxyl
1513	1509	1501	1513	1513	1514	CC	Stretching frequency of aromatic group
1736	1733	1732	1736	1735	1732	CO	Stretching frequency of carbonyl group
2889	2903	2928	2889	2913	2933	CH	Stretching vibration
3412	3414	3413	3412	3411	3415	OH	Stretching frequency of hydroxyl

Table 3. The thermal degradation temperatures and residue weight of *B.vulgaris.*

Location / Position		Moisture	T_{10} (℃)	T_{30} (℃)	T_{50} (℃)	T_{70} (℃)	Residual weight % at 800 ℃
Internode:	BIA (outer)	7.5	268	347	375	659	26.1
	BIB (middle)	6.4	216	327	365	387	7.6
	BIC (inner)	7.4	195	325	367	401	14.1
Node:	BNA (inner)	7.1	238	327	362	387	11.2
	BNB (middle)	4.9	241	330	363	390	13.5
	BNC (outer)	6.0	216	329	364	711	28.3

Table 4. Decomposition temperatures for *B. vulgaris* at the internodes and nodes samples.

Bamboo samples	First decomposition	Second decomposition	Max. temperature
The outer, middle and inner location *B. vulgaris* of fiber at node and internode.	Between 210 ℃ and 390 ℃ (mainly cellulose and hemicellulose)	Between 390 ℃ and 800 ℃ (decomposition of lignin).	800 ℃. (residual weight loss).
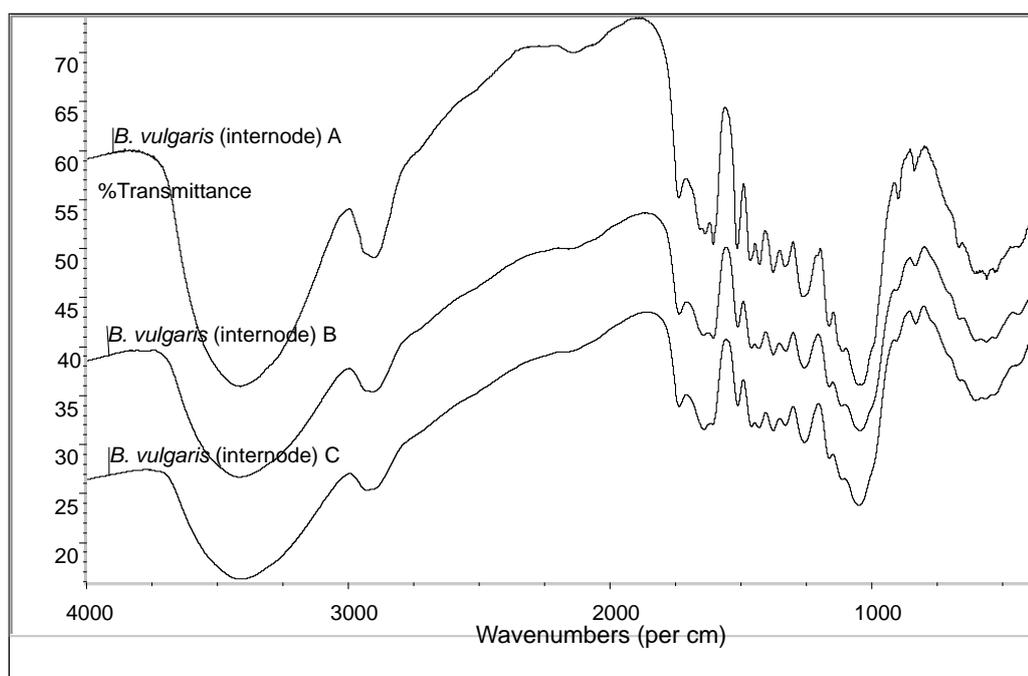
Fig. 1. FTIR spectra of *B. vulgaris* internodes

Comparing all the spectra at different location and position in all species, the peaks at the frequencies 1051 and 3410 per cm are the dominant feature in the spectrum. The strong and broadband at 3410 per cm originates from OH stretching. On the other hand, the sharp and strong band at 1049 per cm is attributed to CO stretching in cellulose, hemicelluloses, and lignin or COC stretching in cellulose and hemicelluloses (Rana *et al.,* 1997). Also, the intense band at 1643 per cm was assigned to HOH bending of absorbed water. The small absorbance at 1335, 1447 and 1520 per cm corresponds to the aromatic skeletal vibrations and ring breathing with CO stretching of the lignin components of various bamboo species (Sun *et al.,* 2001).

The sharp band observed at 1733 per cm is due to the absorption of the carbonyl stretching of ester and carboxyl groups, which are the most abundant in bamboo hemicelluloses (Mui *et al.,* 2008). The band in the region of 1059-1248 per cm involves the CO stretching vibrations of the aliphatic primary and secondary alcohols in the cellulose, hemicellulose, and lignin (El Meligy *et al.,* 2004; Valadez Gonzalez *et al.,* 1999). The lignocellulosic fibers can experience significant weight loss due to partial dissolution of hemicelluloses, lignin and when treated with alkali pectin (Ganan *et al.,* 2008; Mwaikambo and Ansell, 2002). The CO absorption band of the carbonyl group at 1733 per cm disappears El Meligy *et al.,* 2004; on alkali treatment of natural fibers which is evidence that this band is due to hemicellulose component of natural fiber (El Meligy *et al.,* 2004). A small sharp band at 903 per cm arises from α-glucosidic linkages between the sugar units in hemicelluloses and celluloses (El Meligy *et al.,* 2004; Valadez-Gonzalez *et al.,* 1999).
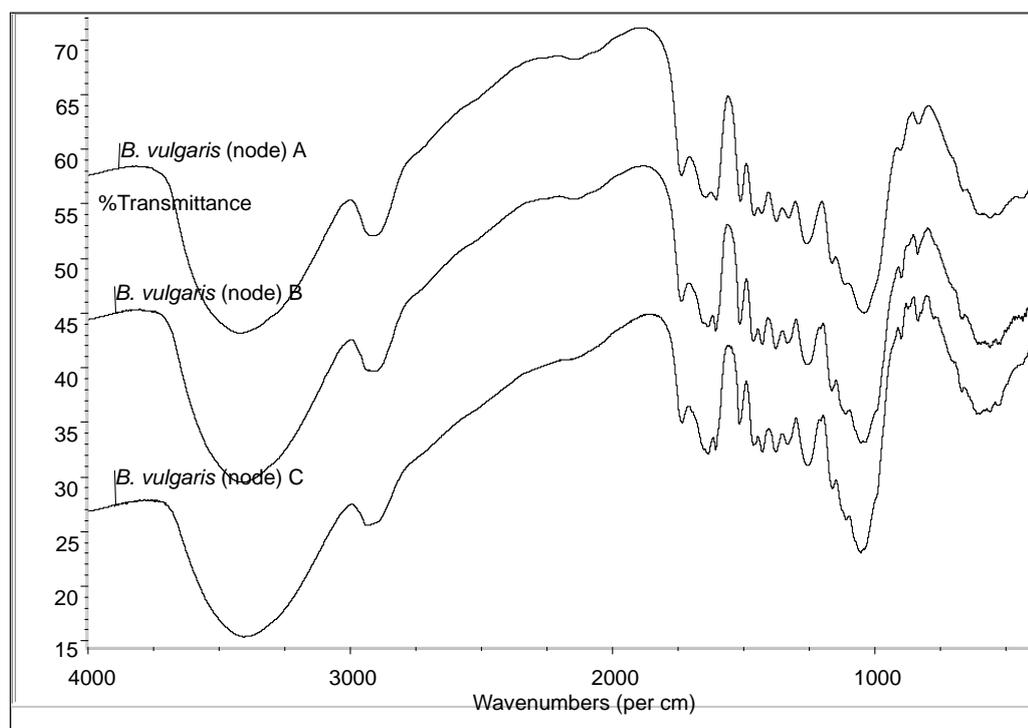
Fig. 2. FTIR spectra of *B. vulgaris* nodes

3.2 Thermogravimetric (TGA) Analysis

The TGA curves of the outer, middle and inner layer of *B. vulgaris* internodes shown in Figures 3 and 4. Thermogravimetric analysis (TGA) was used to determination of the quantitative of the degradation behavior and the composition of the natural fiber. The samples were designated as BIA (outer), BIB (middle) and BIC (inner) layer of *B. vulgaris* internodes respectively. The BNA (inner), BNB (middle) and BNC (outer) designated for layers of *B. vulgaris* nodes. The TGA curves show a slight weight loss before 100 ℃, attribute to the evaporation of water. The slight weight loss ranged between 6.4 to 7.5% in which the middle

position recorded the least value. The lower values of moisture content (see Table 3) recorded might be due to a reduction in hydrophilic tendency associated with the reduction of the free hydroxyl of the phenolic group present in the cellulosic and lignin component (Sun *et al*., 2002).

The *B. vulgaris* degradation (both at the internode and the node) observed under nitrogen atmosphere revealed striking similarities of the shape of the TG and DTG curves. Similar observations were made by Xie *et al.* (2007) and Mui *et al.* (2008) during the investigation of the degradation of this wood. The weight losses and the rates of thermal at the different stages of the thermal degradation (de-volatilization and combustion steps) changed with each different fiber and at any particular location on the plant (Jeguirim *et al.,* 2010). The *B. vulgaris* internodes samples are stable up to 210 ℃ and after that starts to decompose. The decomposition temperatures, of the outer, middle and inner site of the *B. vulgaris* fiber (at both the node and internode) decomposed in two stages due to cellulose, lignin, and hemicellulose (Table 4). The decomposition temperatures for the outer layers at different time intervals are higher than those for the middle and inner layer in both internode and node. The decomposition temperature occurred at 375, 365 and 367°C for the outer, middle and inner sample of *B. vulgaris* internodes for the 50% weight loss. The decomposition becomes more visible at 70% weight loss.
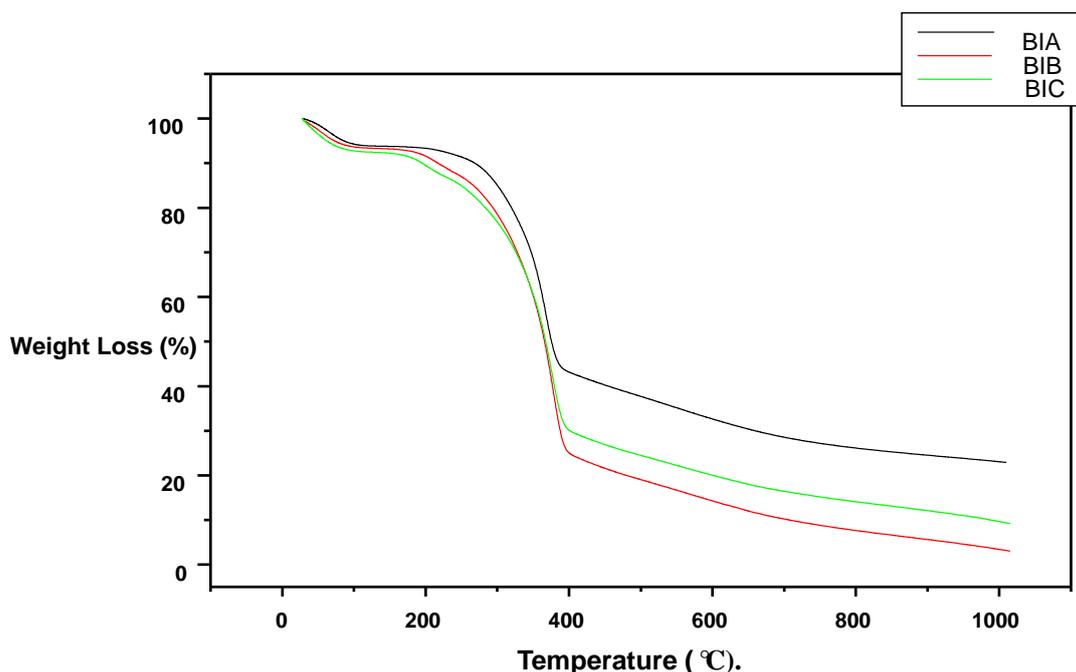
Fig. 3. Thermogravimetric analysis of *B. vulgaris* internode under nitrogen.

The increasing trend of decomposition temperature indicated the thermal stability of the inner layer which is higher than of the outer layer at the internode. The thermal stability of the middle layer recorded the least stability. The weight loss at a temperature above 800 ℃ corresponds to the formation of volatile products which arose from random chain scission and intermolecular transfer involving tertiary hydrogen abstractions from the hemicellulose,

cellulose, and lignin (Remiro *et al.,* 2002). The outer samples of *B. vulgaris* nodes and internodes showed the highest residual weight at the internodes and nodes respectively.

The thermal decomposition of almond shells at 10 °C per min under nitrogen, resulting in two not entirely separate DTG peaks, with one centering at around 310 °C and the other centering at around 368 °C (Font *et al.* (1991). Font *et al.,* (1991) recorded one major peak for the degradation of cotton stalk, sugar cane bagasse, and rice straw, under nitrogen, while the same author reported two peaks for the degradation of these bio-resources under oxygen. The changes in the experimental condition such as the heating rate and atmospheric condition may sometimes merge the two peaks into one very broad peak (Barneto *et al.; Font et al.*, 1991; Liu *et al.,* 2002). This could be responsible for the wide peak with a shoulder around 300 °C observed in Figures 3 and 4.
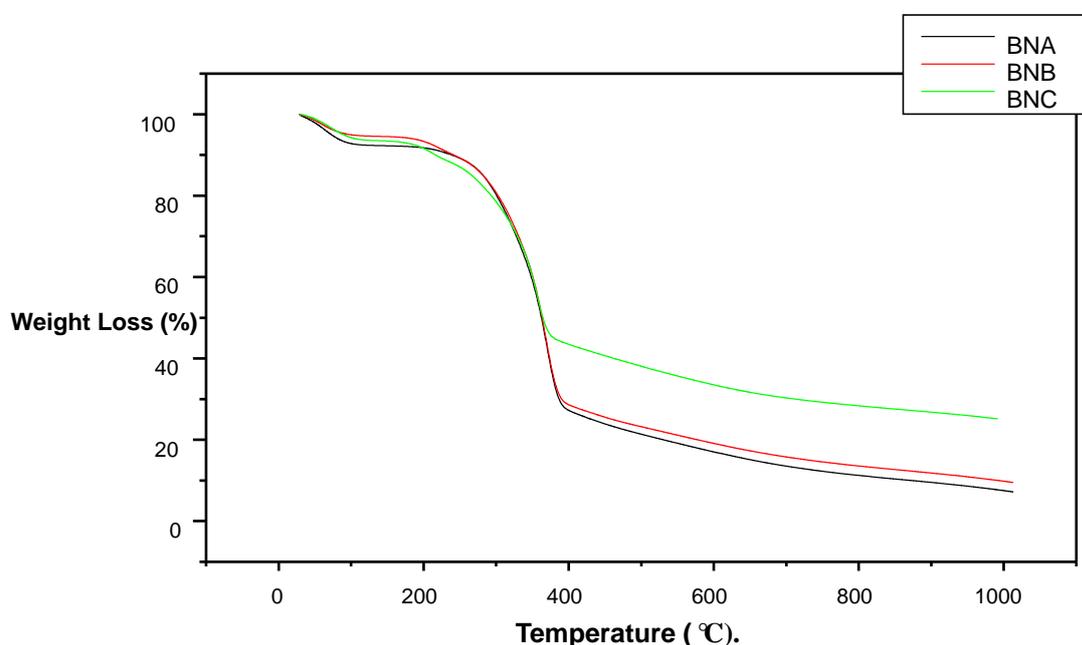
Fig. 4. Thermogravimetric analysis of *B. vulgaris* nodes under nitrogen.

The DTG curves in Figures 5 and 6 indicated that two major processes took place when the bamboo decomposed. The curves show minor weight loss occurred below 100 °C, with peaks between temperatures of 25 °C and 105 °C for BIA, BIB, and BIC respectively. These weight losses are associated with moisture evaporation (Munir *et al.,* 2009). A very prominent degradation peak also observed followed by the appearance of a long steep tail. The main DTG peak corresponds to hemicellulose, and cellulose decomposition (Yang *et al.,* 2008), Contributions from the decomposition of lignin are visible as wide tail emerging from the main de-volatilization domain which may occur due to the vast decomposition range of these components. The mass loss of small biomass sample at in nitrogen low to moderate heating rates usually produce one to two major distinct DTG peaks, respectively associated with hemicellulose and cellulose pyrolysis.
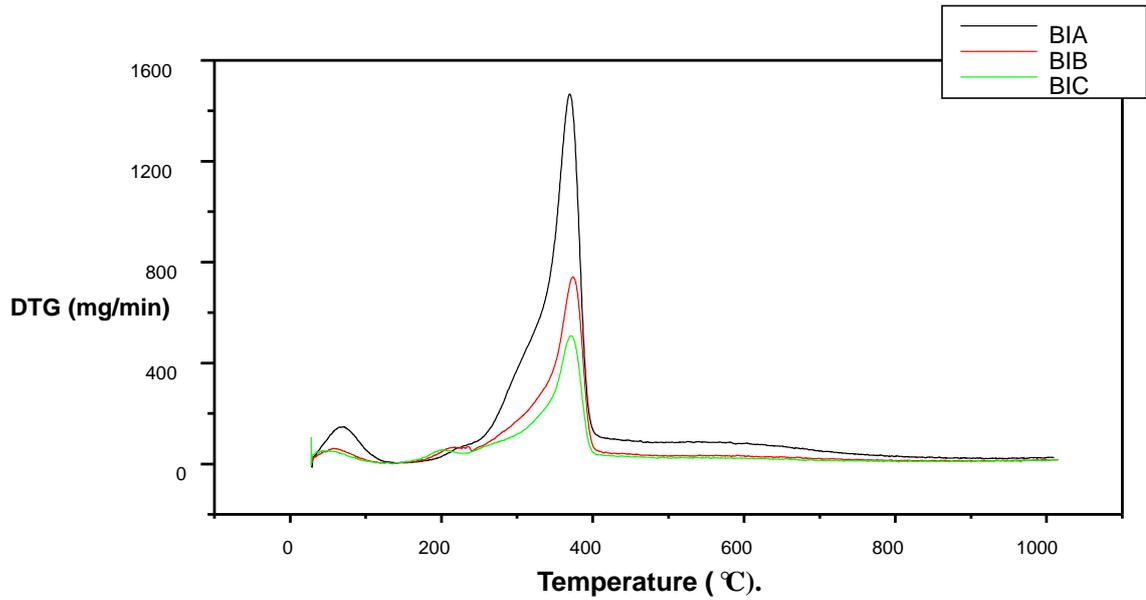
Fig. 5. Derivative thermogravimetry (DTG) analysis of *B. vulgaris* internodes
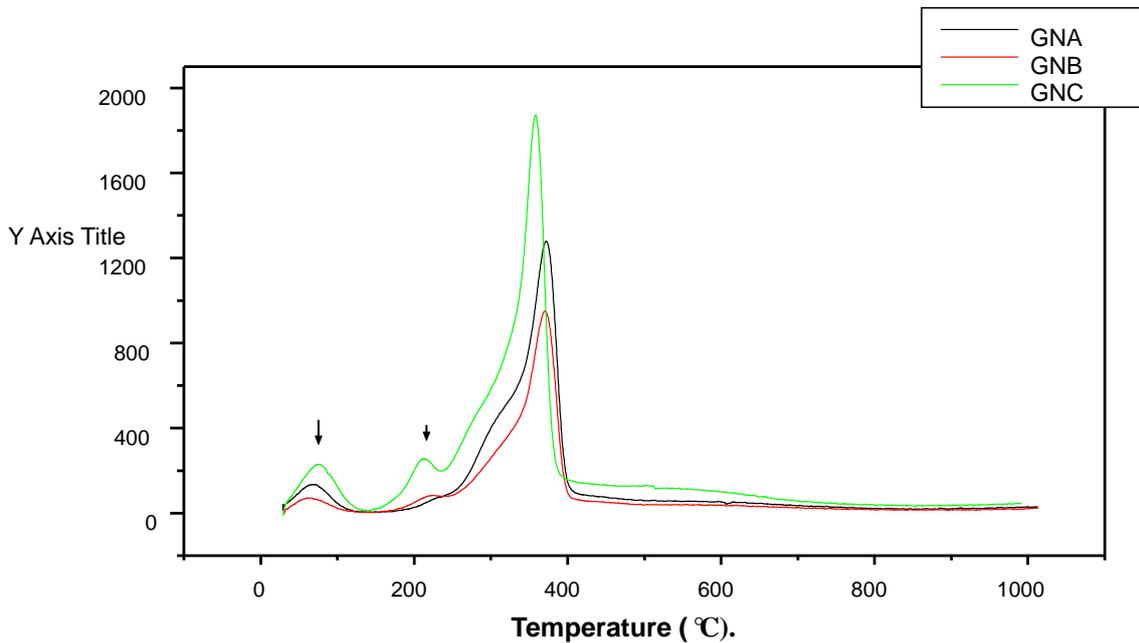
Fig. 6. Derivative thermogravimetry (DTG) analysis of *B. vulgaris* nodes.

4. Conclusion

The FTIR analysis confirmed the presence of basic functional groups in *B. vulgaris* especially the four (4) year-old culms. These groups are mainly composed of ester, carbonyl, and hydroxyl. The DTG analysis also showed qualitatively similar finding, apart from a higher overlap between the hemicellulose and cellulose zones for the bamboo culms. The analysis

shows the lowest reactivity of lignin components and hemicellulose probably because of peculiarities in their chemical structure and composition. The decomposition reaction of the bamboo can be described well by a simple mechanism of decomposition reaction taken as three-step reactions involving hemicellulose, cellulose, and lignin with activation energies and dynamics of the related volatile fractions in good agreement with the previous literature.

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