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Thermal, Composition and Morphological Properties of Untreated and Alkali-Treated Napier Grass Fibres

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Abstract: The effect of alkali treatment on thermal, composition and morphological of Napier grass fibre were investigated. The fibres were treated with 5, 10 and 15% sodium hydroxide wt% concentration for 24 h. The fibres were subjected to Thermo-Gravimetric Analysis (TGA), Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscopy (SEM). The moisture content of the treated fibres decreased as the concentration of the alkali increased. The FTIR shows that the composition of Napier grass fibre has change with increase of alkali concentration. The morphological observation demonstrated that as the alkali concentration increased, the fibre becomes more compressed due to collapse the cellular/lumen structure and the void content decreased.

Key words: Napier grass fibre, thermo-gravimetric analysis, infrared spectroscopy, morphology, collapse

INTRODUCTION

Natural fibres are promising alternative raw materials to be used as reinforcement materials in polymer composites. The advantages of natural fibres compared to synthetic fibres are flexibility of the processing, renewability, sustainability, highly specific stiffness, low cost, no impact on global warming, biodegradability (Fowler *et al*., 2006). There has been a tremendous growth in polymer composites containing many natural fibres has shown a good promises.

Alkali treatment was found to have good potential effects on the natural fibres. The resulted rougher surface of the fibres provides better mechanical interlocking with the resin system hence, stronger interfacial strength between them. Through study conducted by many researchers, they have reported improvements in mechanical properties of natural fibres when undergoes alkalization process for different soaking periods and at different concentrations. The alkali treatment also breaks the hydrogen bonds and increases the number of free hydroxyl groups of the fibre, thus increasing the fibre reactivity (Ray *et al*., 2002). The alkali treatments of various lignocellulosic fibres such as jute, hemp, banana (Zuluaga *et al*., 2009), kapok, coir (Gu, 2009) sisal (Mwaikambo *et al*., 2002) and Napier grass (Reddy *et al*., 2009) have been previously investigated. Reddy *et al*. (2009) reported degradation in thermal and tensile properties of alkali treated (up to 5%) Napier grass fibres. Rao *et al*. (2010) later reported the investigation on the tensile properties of Indian grown Napier grass fibres extracted through chemical modification and water retting processes. Recently we have studied the effect of concentration and soaking exposure of alkali treatment on the tensile strength of Napier grass fibres (Ridzuan *et al*., 2015a, b). Reddy *et al*. (2012) which determined that the maximum ultimate tensile stress of Napier fibre was achieved with 5% alkali treatment. The modulus of jute fibres improved by 12, 68 and 79% following 4, 6 and 8 h of alkali treatment, respectively. The tenacity of the fibre improved by 46% following alkali treatment for 6 and 8 h and the breaking strain was reduced by 23% following an 8 h treatment (Ray *et al*., 2001). Liu *et al*. (2004) and Rao *et al*. (2010) demonstrated that the natural fibres exhibit great potential for use as an alternative to glass and carbon fibres during the production of thermosetting or thermoplastic composite. In this study, thermal, composition and morphological characterization of untreated and treated Napier grass fibres are reported.

MATERIALS AND METHODS

Extraction of Napier grass fibres: Napier plants were supplied from a local plantation at Bukit Kayu Hitam,

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Table 1: Summary of Thermo-Gravimetric Analysis (TGA) of Napier grass fibre

Sample	Initial Degradation IDT (°C)	Wt. loss (%)	Final Degradation FDT (°C)	Wt. loss (%)	Final residue (%)
Untreated	91.5	5.26	400.06	73.38	26.62
5% treated	104.0	4.13	365.50	52.70	47.30
10% treated	104.0	3.62	385.40	49.39	50.61
15% treated	104.0	3.02	385.40	49.00	51.00

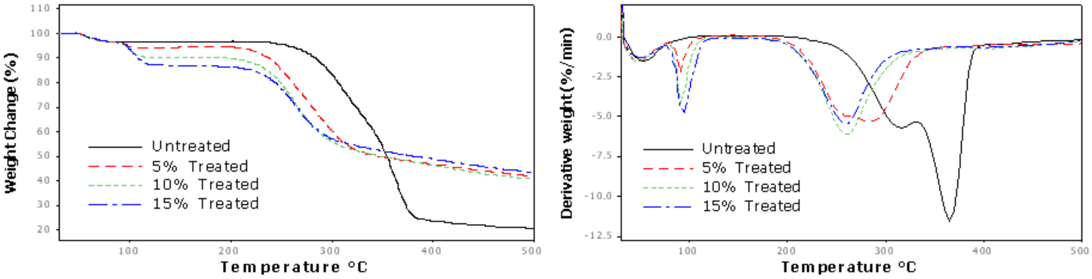

Fig. 1: Thermo-Gravimetric Analysis (TGA) curves and derivative of thermo-gravimetric (DTG) of untreated and treated Napier grass fibres

Kedah in Northern peninsular Malaysia. The fibres were manually extracted from the stem internodes through water retting process (Haameem *et al*., 2014). The stems were initially cleaned and crushed into small pieces with a mallet. To facilitate the separation process, the short plant stems were immersed under running tap water for a few weeks. Finally, the extracted Napier fibres strands were cleaned with distilled water and dried under the sun to remove the moisture content.

Alkali treatment: Napier fibres were treated using alkali (sodium hydroxide) solutions with concentration of 5, 10 and 15 wt.% for 24 h at room temperature. A liquor ratio of 40:1 was used for this study. Lastly, the distilled water was used for cleaning the fibres and sun-dried the fibres for a few days.

Thermo-Gravimetric Analysis (TGA): Thermo-gravimetric Analysis (TGA) of the Napier fibre was recorded using the Gas Controller GC 200 STAR System analyser. To prevent oxidation, the TGA analyses were performed under a nitrogen atmosphere at a flow rate of 20 mL min^{-1}. The Napier fibres were crushed and placed in an alumina crucible to avoid any temperature variation in the thermocouple measurements. The heating rate was maintained at 10°C/min during heating between 30-500°C (Reddy *et al*., 2009).

Fourier Transforms Infrared Spectroscopy (FTIR): The Perkin Elmer Spectrum 400 FTIR spectrophotometer was used to derive the FTIR spectra of the untreated and treated Napier fibres. All the spectra were recorded in the wavenumber range of 650-4000 cm^{-1}, operating in ATR (attenuated total reflectance) mode. The fibres were dried in an oven to remove absorbed moisture prior to FTIR measurement.

Surface morphology of Napier grass fibre by Scanning Electron Microscopy (SEM): The cross-sectional morphology of the untreated and treated Napier fibre strands were examined using an SEM (Model TM3000). The scanning images were obtained with an accelerating voltage of 15 kV and magnification of 1000-1200.

RESULTS AND DISCUSSION

Thermo-Gravimetric Analysis (TGA): The Thermo-gravimetric Analysis (TGA) and Derivative of Thermo-Gravimetric (DTG) curves for the untreated and alkali-treated Napier fibres are presented in Fig. 1. It can be observed that the thermal degradation of Napier fibres was divided into three stages. The first, second and third stages were found in the temperature ranges of 60-115, 200-300 and 300-400°C, respectively. The purpose of this analysis is to observe the thermal degradation curves of the hemicelluloses, cellulose and lignin. The first stage indicated moisture and other volatiles were decomposed. The second stage related with degradation of hemi cellulose and some amount of the lignin. The final stage in the high temperature range was related with degradation of lignin and cellulose. The TGA-DTG curves for all the Napier fibres demonstrated a reduction in the

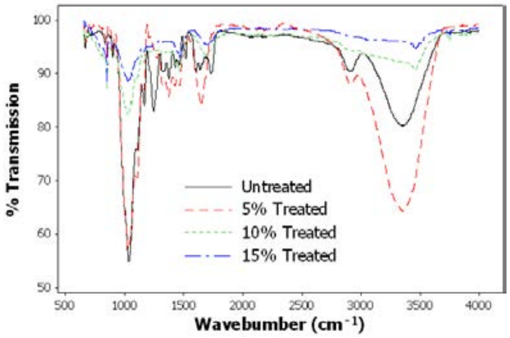

Fig. 2: FTIR spectra of untreated and alkali-treated napier grass fibres

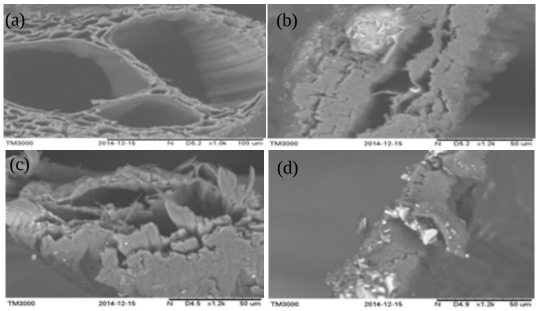

Fig. 3: a-d) Morphologies of Napier grass fibre

moisture content and residue following the burning of the fibres. Table 1 shows the summary of TGA and DTG data for the untreated and alkali-treated Napier fibres. From this table, it is clearly evident that the moisture content was reduced when treated with higher concentration of alkali solution.

FTIR analysis: The FTIR spectra of the untreated and alkali-treated fibres are presented in Fig. 2. From the figure, it can be observed that the untreated fibre demonstrate well-defined bands at approximately 665, 897, 1033, 1160, 1242, 1371, 1425, 1512, 1604, 1729, 2905 and 3345 cm^{-1} within its spectra. The small peaks at 665 cm^{-1} associated with the C-OH out of plane bending (Bezazi *et al*., 2014) and the peak at 897 cm^{-1} can be attributed to the presence of β-glycosidic linkages between the monosaccharides (Rosa *et al*., 2010). The band, centred at 1033 cm^{-1} is associated with the C-O stretching modes of the hydroxyl and ether groups in the cellulose (Paiva *et al*., 2007). The peak at 1160 cm^{-1} is associated with the C-O-C stretching vibration of the pyranose ring in the polysaccharides (Yang *et al*., 2007).

The absorbance peak centred at 1242 cm^{-1} is owed to the C-O stretching vibration of the acetyl group in the lignin (Liu *et al*., 2006), whilst the peak at 1371 cm^{-1} is attributed to the bending vibration of the C-H group of the aromatic ring in the polysaccharides (Troedec *et al*., 2008). The absorbance at 1425 cm^{-1} is associated with the CH_2 symmetric bending (Sgriccia *et al*., 2008). The next peak at 1512 cm^{-1} is attributed to the C = C stretching of the benzene ring of the lignin (Sgriccia *et al*., 2008). The peak centred at 1604 cm^{-1} indicates the C = C aromatic stretching with conjugated C-C bond and this peak is attributed to lignin content of the fibre (Indran *et al*., 2014). The absorption band centred at 1729 cm^{-1} can be attributed to the C = O stretching vibration of the acetyl groups in the hemicelluloses (Biagiotti *et al*., 2004). The peak at 2905 cm^{-1} is a characteristic band for the C-H stretching vibration of CH and CH_2 in the cellulose and hemicelluloses components (Gu, 2009). The last peak at 3345 cm^{-1} is owed to the presence of O-H stretching vibrations and the hydrogen bond of the hydroxyl groups (Yang *et al*., 2007).

FTIR is used to determine the compositional changes that occur during the alkali treatment. The bands for the 5% alkali-treated fibre is at approximately 665, 897, 1031, 1158, 1368, 1650 and 3342 cm^{-1}. The bands for the 5% alkali-treated fibre are relatively similar to those of the untreated fibre with little difference. This indicates that compositional changes had occurred. The FTIR spectra for the 10 and 15% alkali-treated fibres with bands are at 665, 847, 1019, 1459, 1692 and 3458 cm^{-1}. The bands are almost completely different to those of the untreated fibre.

Surface morphology: Figure 3 shows the cross-sectional morphologies of the untreated and treated Napier grass fibres. As mentioned above, the Napier grass fibre shows a multi-cellular structure which indicates a porous structure. A hollow cavity known as a lumen exists inside the unit of the fibre; this can be observed in Fig. 3a. Figure 3b-c demonstrates that the cellular/lumen structures disappear as the percentage of alkali treatment increases. The fibres treated with 15% alkali are shown in Fig. 3d. They show signs of a compressed cellular/lumen structure without a void content. Furthermore, the alkali treatment destroyed the cellular/lumen structure of the fibre and hence reduced the void content of the fibres. This can result in lower water absorption and explain the reduction of diameter for alkali-treated fibres. Therefore, the alkali treatment improves the mechanical and physical properties of the Napier grass fibre and thus facilitates its applications in composite structures.

CONCLUSION

The thermal, compositional and morphological properties of Napier grass fibres were investigated. The untreated Napier grass fibres exhibited the highest moisture content as a result of their multi-cellular structures. Following an increase in the concentration of the alkali treatment, there is a reduction in the moisture content because the alkali treatment compresses the multi cellular structures and reduces the void contents of the fibres. This was confirmed by the observation of the cross-sectional morphologies. The cross-sections of the Napier fibres were observed. The untreated fibres exhibited large lumen structures and there was a change in the structure following the alkali treatment. The TGA-DTG results indicate that the alkali treatment removed the hemicelluloses of the fibres. This was further supported by the FTIR spectrometry analysis. The 15% alkali treatments demonstrated the damaged compress textures. The study further supported the Napier fibres can be used as reinforcing materials in polymer composites.

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REFERENCES

Bezazi, A., A. Belaadi, M. Bourchak, F. Scarpa and K. Boba, 2014. Novel extraction techniques, chemical and mechanical characterisation of Agave Americana L natural fibres. Compos. Part B. Eng., 66: 194-203.

Biagiotti, J., D. Puglia, L. Torre, J.M. Kenny and A. Arbelaiz *et al*., 2004. A systematic investigation on the influence of the chemical treatment of natural fibers on the properties of their polymer matrix composites. Polym. Compos., 25: 470-479.

Fowler, P.A., J.M. Hughes and R.M. Elias, 2006. Biocomposites: Technology, environmental credentials and market forces. J. Sci. Food Agric., 86: 1781-1789.

Gu, H., 2009. Tensile behaviours of the coir fibre and related composites after NaOH treatment. Mater. Des., 30: 3931-3934.

Haameem, M.J.A., M.A. Majid, M. Haslan, M. Afendi and E.A. Helmi *et al*., 2014. Effects of alkaline treatments on the tensile strength of napier grass fibres. Appl. Mech. Mater., 695: 340-343.

Indran, S., R.E. Raj and V.S. Sreenivasan, 2014. Characterization of new natural cellulosic fiber from Cissus quadrangularis root. Carbohyd. Polym., 110: 423-429.

Liu, W., A.K. Mohanty, L.T. Drzal, P. Askel and M. Misra, 2004. Effects of alkali treatment on the structure, morphology and thermal properties of native grass fibers as reinforcements for polymer matrix composites. J. Mater. Sci., 39: 1051-1054.

Liu, Z., S.Z. Erhan, D.E. Akin and F.E. Barton, 2006. Green composites from renewable resources: Preparation of epoxidized soybean oil and flax fiber composites. J. Agric. Food Chem., 54: 2134-2137.

Mwaikambo, L.Y. and M.P. Ansell, 2002. Chemical modification of Hemp, Sisal, Jute and Kapok fibers by Alkalization. J. Applied Polym., 84: 2222-2234.

Paiva, M.C., I. Ammar, A.R. Campos, R.B. Cheikh and A.M. Cunha, 2007. Alfa fibres: Mechanical, morphological and interfacial characterization. Compos. Sci. Technol., 67: 1132-1138.

Rao, K.M.M., K.M. Rao, A.V.R. Prasud, 2010. Fabrication and testing of natural fibre composites: Vakka, sisal, bamboo and banana. Mater. Des., 31: 508-513.

Ray, D., B.K. Sarkar, R.K. Basak and A.K. Rana, 2002. Study of the thermal behavior of alkali-treated jute fibers. J. Applied Polym. Sci., 85: 2594-2599.

Ray, D., B.K. Sarkar, A.K. Rana and N.R. Bose, 2001. The mechanical properties of vinylester resin matrix composites reinforced with alkali-treated jute fibres. Compos. Part A. Appl. Sci. Manuf., 32: 119-127.

Reddy, K.O., C.U. Maheswari, D.J.P. Reddy and A.V. Rajulu, 2009. Thermal properties of Napier grass fibers. Mater. Lett., 63: 2390-2392.

Reddy, K.O., C.U. Maheswari, M. Shukla and A.V. Rajulu, 2012. Chemical composition and structural characterization of Napier grass fibers. Mater. Lett., 67: 35-38.

Ridzuan, M.J.M., A. Majid, M. Afendi, K.S.N. Aqmariah and M.B.M. Nuriman, 2015a. Effects of alkaline concentrations on the tensile properties of Napier grass fibre. Appl. Mech. Mater., 786: 23-27.

Ridzuan, M.J.M., M.A. Majid, M. Afendi, K. Azduwin and S.A. Kanafiah *et al*., 2015b. The effects of the alkaline treatment's soaking exposure on the tensile strength of napier fibre. Procedia Manuf., 2: 353-358.

Rosa, I.M.D., J.M. Kenny, D. Puglia, C. Santulli and F. Sarasini, 2010. Morphological, thermal and mechanical characterization of okra (*Abelmoschus esculentus*) fibres as potential reinforcement in polymer composites. Compos. Sci. Technol., 70: 116-122.

Sgriccia, N., M.C. Hawley and M. Misra, 2008. Characterization of natural fiber surfaces and natural fiber composites. Compos. Part A. Appl. Sci. Manuf., 39: 1632-1637.

Troedec, L.M., D. Sedan, C. Peyratout, J.P. Bonnet and A. Smith *et al.*, 2008. Influence of various chemical treatments on the composition and structure of hemp fibres. Compos. Part A. Appl. Sci. Manuf., 39.

Yang, H., R. Yan, H. Chen, D.H. Lee and C. Zheng, 2007. Characteristics of hemicellulose, cellulose and lignin pyrolysis. Fuel, 86: 1781-1788.

Zuluaga, R., J.L. Putaux, J. Cruz, J. Velez and I. Mondragon *et al.*, 2009. Cellulose microfibrils from banana rachis: Effect of alkaline treatments on structural and morphological features. Carbohydr. Polym., 76: 51-59.