International Journal of Automotive and Mechanical Engineering (IJAME)
ISSN: 2229-8649 (Print); ISSN: 2180-1606 (Online);
Volume 13, Issue 1 pp. 3238-3247, June 2016

DOI: http://dx.doi.org/10.15282/ijame.13.1.2016.10.0270

Alkaline treatment and thermal properties of Napier grass fibres

M. Haameem J.A.[1], M.S. Abdul Majid[1], M. Afendi[1], Haslan M.[2], I. Fahmi[1]

[1]School of Mechatronic Engineering, Universiti Malaysia Perlis, Arau, Malaysia
[2]Advanced Material Research Centre (AMREC), SIRIM Berhad, Kulim, Malaysia.
Email: mhaameem@gmail.com; shukry@unimap.edu.my

ABSTRACT

This paper presents an overview of a natural fibre, namely *Pennisetum purpureum*, which is commonly known as Napier grass. This paper's intention is to analyse Napier grass fibre to assess the effect of alkaline treatment on the tensile properties, thermal stability, and morphology of the fibres. These fibres were extracted by a water retting process from the internodes of Napier stems. The fibres were later subjected to alkaline treatments with NaOH at 5, 10, 15 and 20% concentrations for a period of 24 h at 25°C (RT). The tensile strength, thermal stability and crystallinity of the fibres were seen to upsurge upon conducting the alkaline treatments. Thus, the experimental results demonstrate that the 10% NaOH concentration has the strongest tensile test compared to untreated Napier grass fibre. The surfaces of the fibres after the treatment were observed with a scanning electron microscope (SEM), TM-3000. SEM investigation showed that the surfaces of the fibres become rougher after NaOH treatment. Moreover, from XRD, the amount of crystallinity is also higher in the 10% alkaline treated fibres, and DSC thermograms proved that they have better thermal stability. From this study, Napier grass fibres show potential to be used as reinforcing fibres in composite structures.

Keywords: Napier grass fibres; tensile; alkaline treatment; thermal properties; electron microscopy.

INTRODUCTION

Glass fibres are the most widely used to make reinforcement due to their low cost compared to carbon and aramid. However, these glass fibres have a significant environmental impact, meaning that they have serious drawbacks [1]. Presently, extensive research is being conducted to determine the potential of natural fibres in non-structural applications. Thus, it is fascinating to study the thermal stability of these natural fibres before applying them as reinforcement in thermal applications [2]. The physical properties of natural fibres are primarily classified by their chemical and physical composition, such as the structure of the fibres and cellulose content and by the degree of polymerization [3, 4]. Napier grass also contains hemicelluloses, cellulose and lignin. This content of hemicellulose and cellulose shows that this plant can be an alternative fibre to produce paper, as proposed by Daud et al. [5]. The biggest problem in the use of natural fibre composites is the fibre–matrix adhesion. The function of the matrix is to transfer the load to the fibres. This can be accomplished if a good adhesion bonding between the matrix and the fibres is ensured. Insufficient adhesion bonding at the interface means that their advantages cannot be fully utilised and that they are exposed to environmental attack [6]. Poor adhesion between hydrophobic polymers and hydrophilic fibres implies poor properties for the composites [7]. Nevertheless, these properties can be improved by physical and chemical treatments. Alkaline treatment is one of the most widely applied chemical methods to remove a certain portion of the lignin, wax and oils that cover the exterior surface of the fibre cell wall. The important alteration achieved with alkaline treatment is to breach the hydrogen bonding in the network structure, such that the surface roughness is increased , thereby enabling good bondings to be achieved [8, 9].

On the other hand, ligno-cellulosic fibres are submitted to intense heat during fabrication. Therefore, thermal analysis study is necessary to determine the influence of the treatments on the fibres to observe the degradation behaviour. Geethamma et al. [9] used 5% NaOH to remove surface impurities on oil palm fibres. Obi Reddy et al. [10] analysed and characterised Indian-grown Napier grass fibre when untreated, 2 and 5% NaOH treated and found that the NaOH treatment eliminated the amorphous hemicellulose component of the fibres. Joseph et al. [11] reported in their studies that the mechanical tensile strength for 5, 10, and 15% NaOH treatment was 9.95 MPa, 9.61 MPa and 8.86 MPa respectively for oil palm fibre. Joseph et al. [12] also concluded that the modification can either increase or decrease the strength of the fibres, thus what occurs structurally is of great importance. Valadez and colleagues [13] carried out research regarding kenaf bonding with resins. They concluded that alkaline treatment has two effects on the fibre, increasing both the surface roughness and the cellulose on the fibre surface. Both these effects can yield a better mechanical interlocking between the fibres. Recently, work conducted by Haameem et al. [14] showed that 10% NaOH treatment of Napier grass fibre demonstrates the highest tensile strength. Furthermore, the SEM observation suggests that NaOH treatment causes the fibres to become rougher, thus leading to increased friction in the matrix material. The aim of this paper is to investigate single fibre testing and the effects of different NaOH concentrations on Napier fibres. Furthermore, the thermal analysis is also studied by using digital scanning calorimetry (DSC) and thermogravimetry (TGA) to obtain their glass transition and melting temperature. X-ray diffraction (XRD) was also applied to determine the degree of crystallinity of the untreated and treated fibres.

MATERIALS AND METHOD

Fibre Preparation

Three to six month old Napier grass stems were collected from a farm in the Northern Malaysia region, Bukit Kayu Hitam, Kedah. This farm grows Napier grass to feed cattle. The Napier grass is then conventionally subjected to a water retting process to extract the fibres from grass its internodes. The stems are chopped at the internodes and crushed with a mallet, as presented in Figure 1(a). This process is to facilitate the process of separating the fibre strands. The chopped and crushed stems are then soaked in a tank as shown in Figure 1(b). The stems are left to soak for around 3 to 4 weeks. Water retting enhances the production of more identical and high-quality fibres. After soaking for 3 to 4 weeks, the stems were a processed in a roll-out machine to separate the fibres, as displayed in Figure 1(c). The separated fibres were washed carefully using running tap water to remove any impurities. Finally, the Napier grass fibre was sun-dried for a few days to remove the maximum moisture from the fibre, as shown in Figure 1(d). The fibres are weighed daily to ensure that they have completely dried and achieve a homogeneous weight of dried fibre. Colour change within the fibres also indicates the loss of moisture.

Figure 1. (a) Stem is beaten with a mallet, (b) soaked stems, (c) roll-out machine to separate fibres, and (d) extracted Napier grass fibre to be dried.

Alkaline Treatment of Napier Grass Fibres

The sun-dried fibres were then oven-dried to remove the moisture further within the fibres. The fibres were soaked in a solution of 5, 10, 15 and 20% sodium hydroxide (NaOH) at 28°C (room temperature) as shown in Figure 2(a). The fibre to NaOH solution ratio was kept at 1:10. The fibres were kept immersed in the NaOH solution for 24 hours to remove the cellulose [10]. After the soaking period, the fibres were repeatedly washed with distilled water as displayed in Figure 2(b). After washing carefully with distilled water, the fibres were sun-dried again for one week.

Single Fibre Tensile Test

The single fibre tensile test was determined using an INSTRON micro tester at a crosshead speed of 1 mm/min as stated in ASTM D3392. The gauge length was specified at 50 mm. In this test, Napier grass lengths of more than 100 mm were used and tabbing was done as shown in Figure 2(c). The tabbing was attached to the samples to make sure that the fibre end did not slip at the gripper during testing. A total of twenty samples were tested for this work and the average values of the tensile strength, tensile modulus, breaking tenacity, linear density and tensile strength were calculated. A digital optical microscope was used to measure the fibre diameters, as shown in Figure 2(d).

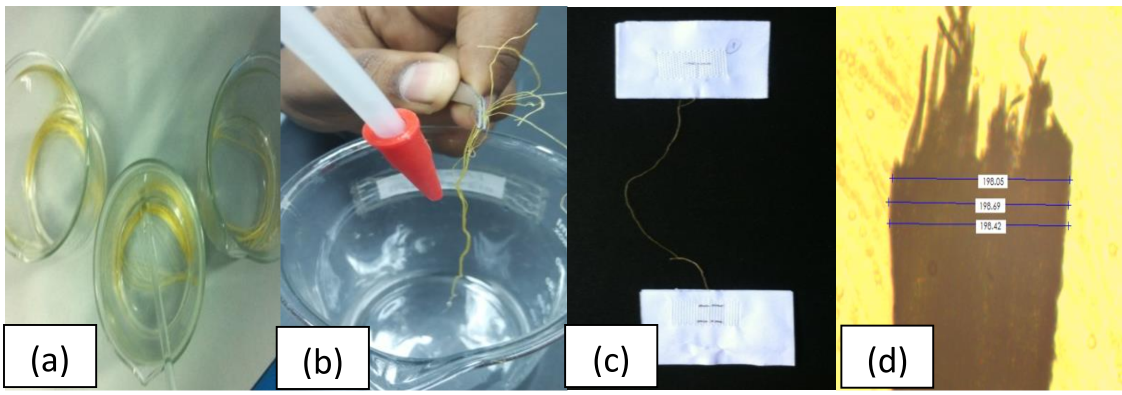

Figure 2. (a) Immersed fibre in NaOH solution, (b) washing fibre using distilled water, (c) fibre tabbing and (d) microscope measurements of fibre diameter.

X-ray Diffraction (XRD) Analysis

The X-ray diffraction (XRD) spectra of the Napier grass fibres were recorded on a Brucker D2 Phaser as shown in Figure 3(a). The XRD is a type of X-ray powder diffraction. The X-ray tube is the copper (Cu) type and generates at 30 kV and 10 mA. The diffraction machine was operated at a scan speed of 4°/min in steps of 0.05°. The samples were scanned from the range 2θ, varying from 5° to 45° for untreated and treated fibres.

Thermogravimetric analysis (TGA)

The samples used for this thermogravimetric analysis were extracted Napier grass fibre weighing 0.471 mg. After weighing, the samples were sun-dried in order to maximise moisture loss. The analyses were done at a heating rate of 20°C/min and inert nitrogen gas was used as the atmosphere.

Digital Scanning Calorimetry (DSC) Test

The DSC machine was used to find the glass transition temperature (Tg) and melting temperature (Tm). The rate of heating was maintained at 10^{o}C/min and data was taken until 350^{o}C.

Study of morphology

The morphology of the Napier grass fibre was analysed using a scanning electron microscope (SEM), TM-3000 with a magnified resolution of 100–200x.

RESULTS AND DISCUSSION

Single Fibre Tensile Test

The single fibre tensile test results are presented in Figure 3. From this, it is clearly seen that the highest strength obtained is for 10% alkaline treated Napier grass fibre with 106.2 MPa. The second highest is for 15%, followed by 5% and 20% alkaline treated Napier grass fibre. The lowest strength is 12.4 MPa recorded for untreated Napier grass fibre. This indicates that the 10% alkaline treated fibre had increased almost 85% from the untreated fibre. The alkaline treatment causes fibrillation, which is a process that causes the fibre bundle to break into smaller bundles. Smaller bundles help to distribute the load applied throughout the fibres [14, 15]. The most remarkable reduction of strength is seen at 15% and 20% alkaline treated; this is because at higher percentage concentrations, excess de-lignification of the Napier grass fibre occurs, weakening and thus damaging the fibres. Ridzuan et al. reported a similar observation, where the main structural components of the fibre were attacked, resulting in the formation of more grooves on the surface of the fibre. Consequently, this leads to further weakening of the fibre strength, resulting in a decrease in the ultimate tensile stress [16].

Table 1 summarises the single fibre parameters, comparing untreated and treated Napier grass fibre. The mass of the Napier grass fibre varies from 0.004 g to 0.0078 g. The diameter of the fibre varies from 154 µm to 259 µm. The table shows that the unit break is highest for 10% treated fibres, which also have the lowest linear density among other fibre conditions. The unit break is breaking tenacity over linear density. Linear density is a measure of mass per unit length. Thus, breaking tenacity is directly proportional to linear density. Therefore, the lower the linear density, the higher the breaking tenacity. The fibre diameter varied in the range ~150–250 µm. The results show that the 10% NaOH-treated fibre exhibited the highest breaking tenacity of >7000 N/tex. The untreated fibre exhibited the lowest breaking tenacity of 462 N/tex. The fibres were enhanced by the alkaline treatment because it increased their surface roughness and decreased their moisture content, particularly for the 10% NaOH-treated specimens. Subsequently, the breaking tenacity decreased from 7093 N/Tex to 6480 N/Tex and further reduced to 2117 N/Tex for the 10%, 15%, and 20% NaOH-treated samples, respectively.

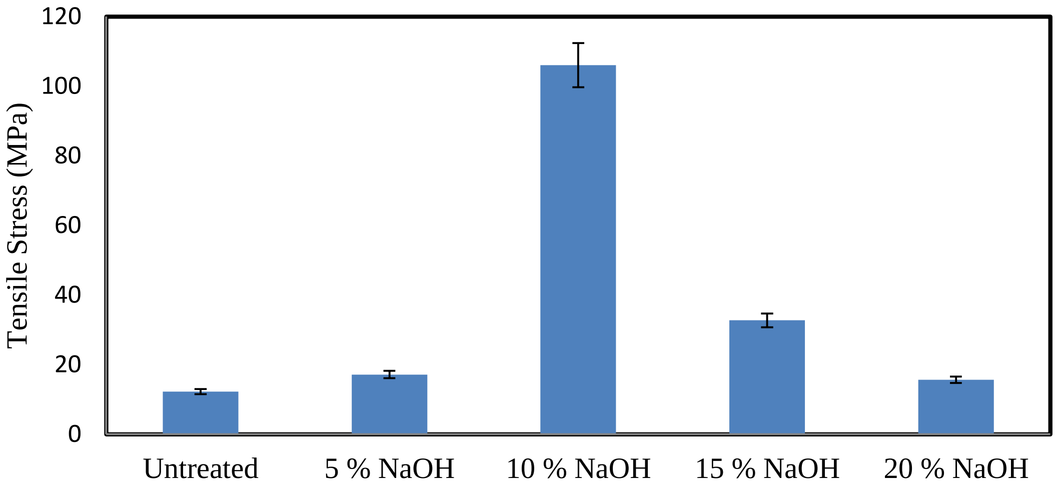

Figure 3. Tensile stress (MPa) versus against different NaOH percentage treatments.

Table 1. Single fibre average mass, length, diameter, area, linear density and unit break.

Sample type	0% Untreated	5% NaOH	10% NaOH	15% NaOH	20% NaOH
Average mass (g)	0.0041	0.0078	0.004	0.0041	0.0053
Average length (mm)	121.5	130.7	140.2	135.1	140.9
Average diameter (µm)	199	259	154	192	249
Average area (mm^2)	0.030	0.051	0.020	0.030	0.050
Linear density (kg/m)	0.000332	0.000589	0.00028	0.000298	0.000371
Unit break (N/Tex)	462.02	1621.61	7093.20	6479.57	2117.22

X-ray Diffraction (XRD) Analysis

XRD analysis was conducted to investigate the crystallinity of the Napier grass fibres. XRD diffractograms are given in Figure 4 and the % crystallinity was calculated according to the Segal empirical method as described in Equation (1):

$$\%\ Ic = \frac{I(002) - I(am)}{I(am)}\ \mathrm{X}\ 100 \tag{1}$$

where $I_{(002)}$ is the peak intensity counter reading at a 2θ angle close to 22° representing crystalline material and $I_{(am)}$ is the counter reading at a 2θ angle close to 16° representing amorphous material in the samples [17]. Therefore, the degree of crystallinity for the untreated fibres is 25.5% and for the 10% alkaline treated is 39.5%. This shows that the percentage crystallinity index of alkaline treated Napier grass fibres is 14% higher than that of the untreated Napier grass fibres. This increase in percentage crystallinity index indicates the improvement in the restructure of cellulose, and finally contributes to enhancing the tensile strength of the Napier grass fibres because of the realignment of cellulose molecules [17, 18]. Recently, Zhijia and Benhua found that the relative crystallinity of untreated bamboo and NaOH treated bamboo samples was 44.4% and 55.2%. The crystallinity is relatively higher because bamboo is 70% made up of cellulosic materials [19]. Moreover, alkaline treated fibres lose some of their amorphous constituents after treatment, which results in the treated fibres appearing to be more crystalline than the untreated ones [6].

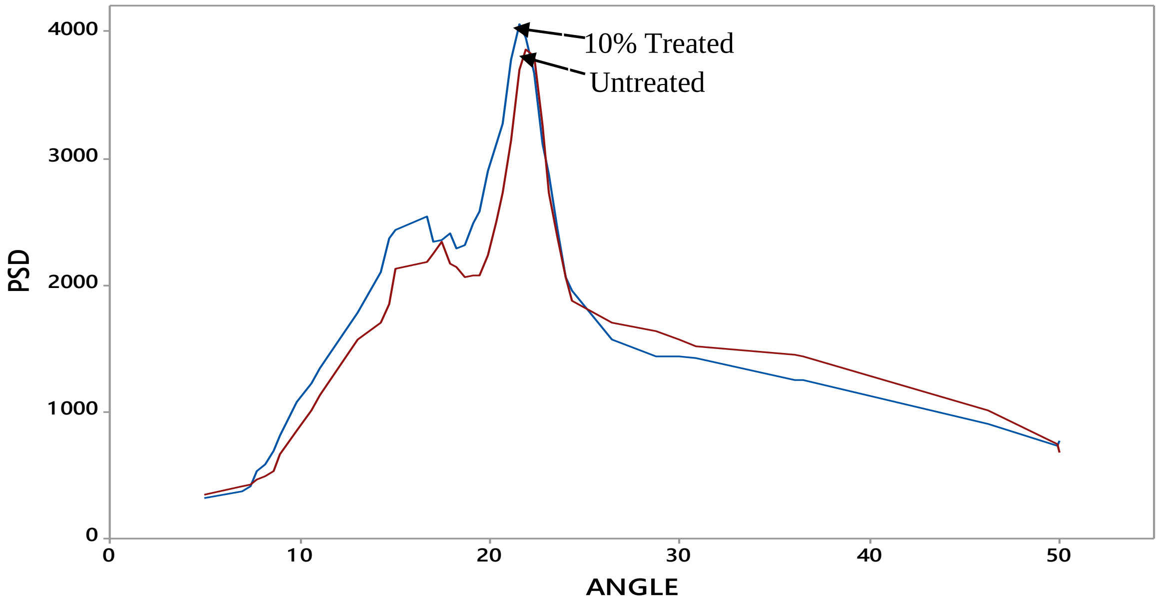

Figure 4. X-ray diffractograms of untreated and 10% alkaline treated Napier grass fibres.

Thermogravimetric Analysis (TGA)

The TGA thermogram is presented in Figure 5. The thermogram indicates that it takes a few stages of degradation for the untreated Napier grass fibre. Due to differences in the chemical structure, the components of the fibres usually decompose at different temperatures [20]. A slight weight drop was seen at 100°C. The weight is less than 10%, corresponding to the moisture depleted in the fibres. The first stage that takes place in the range of 230–300°C is associated with depolymerization of hemicelluloses and some of the lignin. The second stage occurs at 300–400°C and corresponds to the degradation of most of the alpha celluloses and lignin. This shows that the lignin is the hardest part to decompose as it requires high temperatures, as concluded earlier by Kabir et al [21]. Its decomposition extends over the entire temperature range. Based on the thermogram, the initial, 25%, 50% and final degradation calculated thus are summarised in Table 2. The initial and the final degradation occur at 250°C and 356°C respectively [8].

Digital Scanning Calorimetry (DSC) Test

Thermal transitions were observed from the DSC thermograms, as presented in Figure 6. The phases that can be seen in these thermograms are the glass transition, Tg. At the Tg, the Napier grass fibre molecules possessed enough energy to overcome the intermolecular forces and have a degree of freedom. The Napier grass fibre now becomes softer and flexible. The Tg for untreated fibre is around 40°C and 58°C for 10% alkaline treated fibre. Treated fibre shows a higher Tg compared to untreated fibre because of the hemicelluloses and part of the lignin that is removed through alkalization. Therefore, the thermal stability of the treated fibre increases compared to untreated fibres [21]. It was observed that 10% NaOH treated fibre showed higher thermal stability than other treated fibres, which indicates the improved hydrophobic nature of the fibre. It becomes more hydrophobic because the treated fibre has lost the hemicelluloses and a part of the lignin which contain moisture [13].

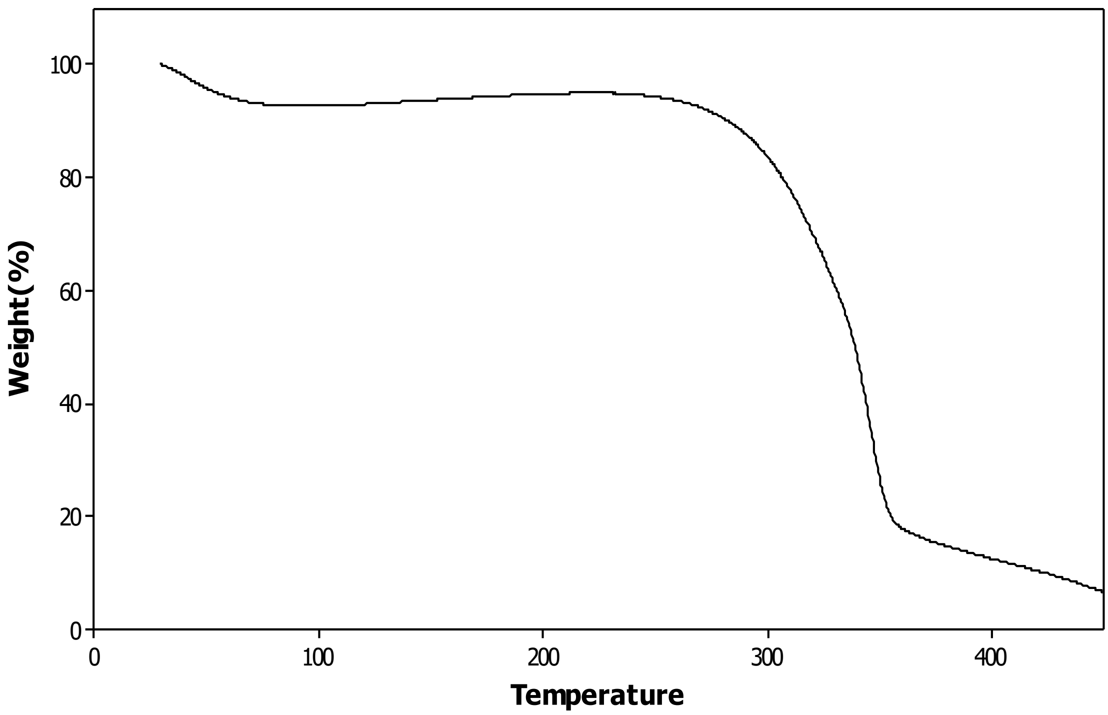

Figure 5. TGA thermogram of untreated Napier grass fibres.

Table 2. Thermal degradation parameters for untreated Napier grass fibres.

Degradation parameter	Temperature (°C)
Initial degradation	250
25% degradation	314
50% degradation	339
Final degradation	356

Morphology Study

Morphology examinations were conducted on the untreated and alkalized fibres to examine the changes that occurred before and after treatment. The SEM micrograph of the untreated Napier fibre bundles in Figure 7(a) shows the presence of wax, oil and surface impurities. Waxes and oils provide a shielding layer to the surface of the fibres [22]. The 10% alkaline treated Napier grass fibre in Figure 7(b) shows a rougher surface. The surface of the 10% alkaline treated fibre appears to be quite clean of waxes and oil, but in actuality is roughened by the chemical treatment. Such rough surfaces hopefully promote good interfacial bonding between fibres and the resin matrices, if the fibres are used as reinforcement [21, 23]. Fibrillation was also observed in the 10% alkaline treated fibre, increasing the numbers of fibrils to transfer the load applied amongst them [14, 15].

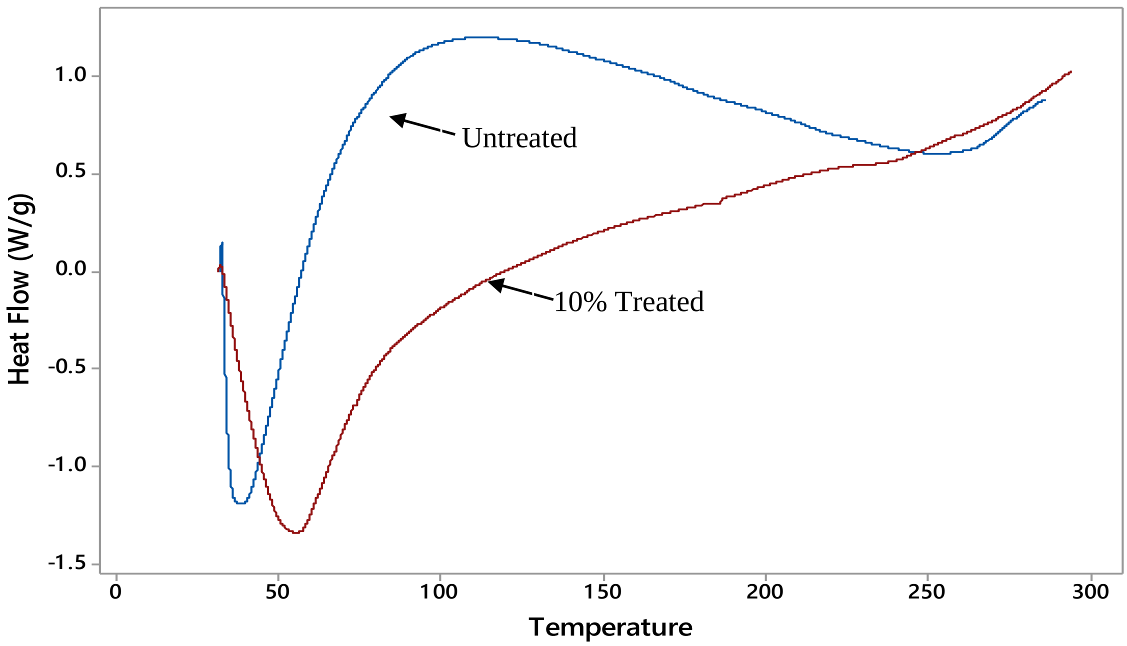

Figure 6. DSC thermogram of untreated Napier grass fibres.

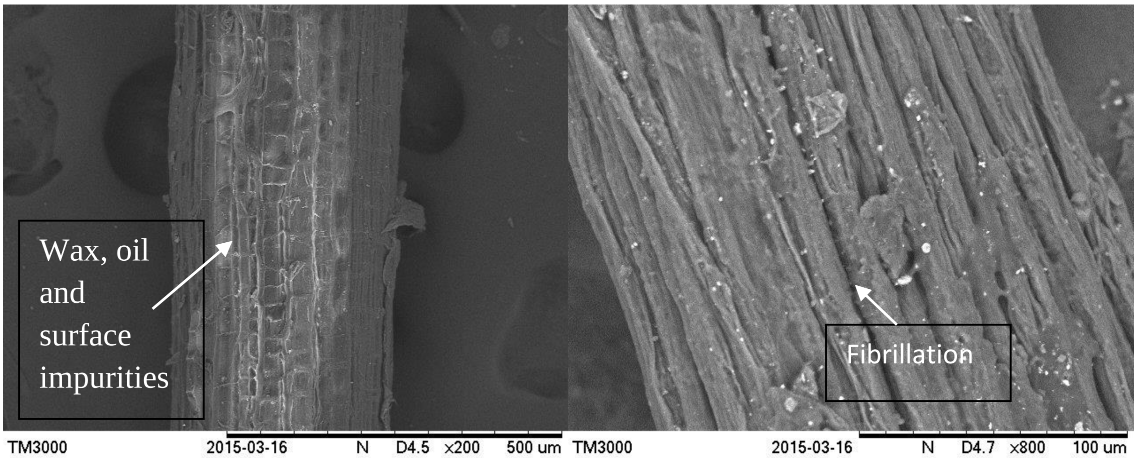

Figure 7. SEM of Napier grass fibres: (a) untreated and (b) 10% alkaline treated.

CONCLUSIONS

The Napier grass fibres were treated with different percentage alkaline concentrations. It can be seen that 10% alkaline treatment resulted in the highest tensile strength compared to untreated and other percentage concentrations. Moreover, from XRD analysis, the percentage crystallinity of treated Napier grass fibres is found to be higher than when untreated. The DSC test proves that the thermal stability increases for the 10% alkaline treated fibres as the glass transition value is higher by 18%. The SEM of the untreated and 10% alkaline treated fibres revealed that the fibre becomes fibrils, which helps the load transfer in the fibres. From this morphology it was also concluded that the fibres become roughened upon treatment, and unwanted substances on the fibres are eliminated. It is recommended that further research on Napier grass fibre should examine different types of chemical treatments. The study further supported the feasibility of exploiting Napier grass fibres as reinforcing materials in polymer composites.

ACKNOWLEDGEMENTS

The authors of this paper would like to thank Universiti Malaysia Perlis (UniMAP) for providing laboratory equipment and facilities for conducting this research.

REFERENCES

[1] Wambua P, Ivens J, Verpoest I. Natural fibres: can they replace glass in fibre reinforced plastics? Composites Science and Technology. 2003;63:1259-64.

[2] Joshi SV, Drzal LT, Mohanty AK, Arora S. Are natural fiber composites environmentally superior to glass fiber reinforced composites? Composites Part A: Applied Science and Manufacturing. 2004;35:371-6.

[3] Bledzki AK, Gassan J. Composites reinforced with cellulose based fibres. Progress in Polymer Science. 1999;24:221-74.

[4] Rejab MRM, Noor MM, Daud R, Kadirgama K, Sani MSM. Effect of adverse environment on buckling of composite laminates. Malaysian Technical Universities Conference on Engineering and Technology. Kuantan, Pahang, Malaysia.2009.

[5] Daud Z, Mohd Hatta MZ, Mohd Kassim AS, A. AM. Suitability of Malaysia's pineapple leaf and Napier grass as a fiber substitution for paper making industry. Enginering Conference Energy Environment2013. p. 1-4.

[6] Sapuan MM, Hamdan MM, Bachtiar HD. Flexural properties of alkaline treated sugar palm fibre reinforced epoxy composites. International Journal of Automotive & Mechanical Engineering 2010;1:12.

[7] Joseph PV, Rabello MS, Mattoso LHC, Joseph K, Thomas S. Environmental effects on the degradation behaviour of sisal fibre reinforced polypropylene composites. Compos Sci Technol. 2002;62:1357-72.

[8] John M, Anandjiwala R. Recent developments in chemical modification and characterization of natural fiber reinforced composites. Polymer Composites. 2008;29:11.

[9] Geethamma VG, Thomas Mathew K, Lakshminarayanan R, Thomas S. Composite of short coir fibres and natural rubber: effect of chemical modification, loading and orientation of fibre. Polymer. 1998;39:1483-91.

[10] Reddy KO, Maheswari CU, Reddy DJP, Rajulu AV. Thermal properties of Napier grass fibers. Materials Letters. 2009;63:2390-2.

[11] Sreekala MS, Kumaran MG, Joseph S, Jacob M, Thomas S. Oil palm fibre reinforced phenol formaldehyde composites: influence of fibre surface modifications on the mechanical performance. Applied Composite Materials. 2000;7:295-329.

[12] Joseph PV, Joseph K, Thomas S. Short sisal fiber reinforced polypropylene composites: the role of interface modification on ultimate properties. Composite Interfaces. 2002;9:171-205.

[13] Valadez-Gonzalez A, Cervantes-Uc JM, Olayo R, Herrera-Franco PJ. Effect of fiber surface treatment on the fiber–matrix bond strength of natural fiber reinforced composites. Composites Part B: Engineering. 1999;30:309-20.

[14] Haameem J, Abdul Majid M, Haslan M, Afendi M, Helmi E, Idris F. Effects of alkaline treatments on the tensile strength of Napier grass fibres. Applied Mechanics and Materials. 2014;695:4.

[15] Wong KJ, Zahi S, Low KO, Lim CC. Fracture characterisation of short bamboo fibre reinforced polyester composites. Materials & Design. 2010;31:4147-54.

[16] Ridzuan MJM, Abdul Majid MS, Afendi M, Aqmariah Kanafiah SN, Zahri JM, Gibson AG. Characterisation of natural cellulosic fibre from Pennisetum purpureum stem as potential reinforcement of polymer composites. Materials & Design. 2016;89:839-47.

[17] Groeneveld RA, Meeden G. Measuring skewness and kurtosis. The Statistician. 1984:391-9.

[18] Chai T, Ding J, Wu F. Hybrid intelligent control for optimal operation of shaft furnace roasting process. Control Engineering Practice. 2011;19:264-75.

[19] Li X, Tabil LG, Panigrahi S. Chemical Treatments of Natural Fiber for Use in Natural Fiber-Reinforced Composites: A Review. Journal of Polymers and the Environment. 2007;15:25-33.

[20] Monteiro SN, Calado V, Rodriguez RJS, Margem FM. Thermogravimetric Stability of Polymer Composites Reinforced with Less Common Lignocellulosic Fibers – an Overview. Journal of Materials Research and Technology. 2012;1:117-26.

[21] Kabir MM, Wang H, Lau KT, Cardona F. Chemical treatments on plant-based natural fibre reinforced polymer composites: An overview. Composites Part B: Engineering. 2012;43:2883-92.

[22] Haameem JAM, Abdul Majid MS, Afendi M, Marzuki HFA, Fahmi I, Gibson AG. Mechanical properties of Napier grass fibre/polyester composites. Composite Structures. 2016;136:1-10.

[23] Akhtar MN, Gaaz TS, Sulong AB, Raza R. Morphology and tensile properties of thermoplastic polyurethane-halloysite nanotube nanocomposites. International Journal of Automotive & Mechanical Engineering. 2015;12:13.