Characterization of Millet (*Pennisetum glaucum*) Husk Fiber (MHF) and Its Use as Filler for High Density Polyethylene (HDPE) Composites

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Mechanical properties were investigated for millet husk (MH) fiber filled high density polyethylene (HDPE) composites. The chemical and thermal attributes of the fibers are also studied. The fibers were pulverized to 250 µm size. The composites were prepared by a melt blending technique using a Brabender® internal mixer, accompanied by hot compression. Composite formulations were based on; 10%, 20%, 30%, and 40% wt fiber loadings with 170 °C temperature, 10 min flow time, and 20 rpm rotational speed. Mechanical properties were obtained according to ASTM D3039, ASTM D790, and ASTM D256 for tensile, flexural, and impact test, respectively. Microstructures of fracture tensile test specimens were observed by SEM. Fiber chemical compositions were determined using acid detergent, neutral detergent, and acid detergent lignin to evaluate the cellulose, hemicelluloses, and lignin contents correspondingly. The percentages were 50.4% cellulose, 23.7% hemicelluloses, and 13.2% lignin with remains of other chemical constituents. Thermogravimetric analysis showed that the highest stable temperature was 245 °C. The tensile and flexural strength of the composites decreased with increasing fiber loading, while their modulus increased with increasing fiber loading. The impact strength was reduced drastically as fiber loading was increased. Therefore, it was concluded that millet husk fiber has potential to be used as raw material in composites applications.

Keywords: Millet husk fiber; Cellulose; Cereal; Composite; Filler; Polyethylene; Lignin

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INTRODUCTION

Agricultural wastes are often becoming environmental problems. The use of natural fibers for composite material manufacturing has received great attention. Problems associated with the use of synthetic materials are prompting legislation to protect the environment in many countries. Most natural fibers from these wastes can be used as reinforcements or fillers in polymer composites for creating technology innovative materials as a function of cost (Cruz- Estrada *et al.* 2007; Treffler 2007; Hammajam *et al.* 2014b; Carbonell-Verdu *et al.* 2015). High technology industries are pressured to seek alternative materials to replace the conventional synthetic materials due to environmental and health related issues (Bhattacharyya *et al.* 2015). The use of non-renewable fibers such as glass or carbon is facing stiff competition from natural fiber materials that are environmentally friendly. Natural fibers have some advantages over the conventional materials in terms of renewability, cost effectiveness, availability, low density, and biodegradability, to mention a few. Disadvantages of natural fibers include moisture absorption and poor adhesion between fibers and the matrix.

Thermoplastic high density polyethylene (HDPE) has been used in several composite materials industries such as household items, automobile, and toy components. It has good and varied properties. Therefore, it can be formulated and fabricated with natural fibers to achieve superior properties in term of stiffness and strength (Panthapulakkal and Sain 2007; Babaei *et al.* 2014; Ahmad *et al.* 2012; Ashori *et al.* 2013; Kraiem *et al.* 2013).

Millet (*Pennisetum glaucum*) is an annual and biannual cereal crop that grows in different climatic conditions and is native to Africa and Asia (Dominique *et al.* 2012). Millet has become a vital product in sub-Saharan Africa and India, where it is used in several consumables such as food and beverage industries (Hammajam *et al.* 2014a,b). Millet is a cereal crop that can be produced within a very short period of time. Several reasons may be responsible for the growing of millet at large scale, but population growth and climate change necessitate the search for drought-resistant crops with high yield as a panacea for food security globally. The husks are left on the farm site after harvest. This resource is costless and has zero competition in terms of consumption and usage in Nigeria (Abubakar and Ahmad 2010). When left in the field, it becomes home for microorganisms that shelter diseases. Millet husks are among the millions of tons of agricultural wastes seeking ways of disposal (Rosa *et al.* 2009; Heuze and Tran 2012). Attractive attributes of millet husk include renewability, biodegradability, light weight, and cost effectiveness.

Similarly, various researchers (Steward 2007; Ortega-Leyva 2008; Rosa *et al.* 2009; Cruz-Estrada *et al.* 2010) had investigated the use of lignicellulosic fibers as reinforcement for thermoplastic matrices to develop natural fiber composites for a variety of applications. The effects of fiber sizes, fiber treatments, and fiber loadings on the mechanical, morphological, chemical, biodegradability, and thermal properties were studied. The outcomes of the findings were generally successful and promising. Great success has been reported for applications of high density polyethylene in composites made with combinations of long and short fibers (Adhikry *et al.* 2008; Korol *et al.* 2015; Carbonell *et al.* 2015). So far there has been no attempt to develop composites from millet husk fibers based on the search of the literature. In this study, development and characterization of millet husk fiber and effect of fiber loading on the composites mechanical properties is presented.

EXPERIMENTAL

Materials

Millet husk (MH) was obtained from a farm site where the millet cereal was milled and the waste husk was dumped; the farm was located in the Bulumkutu Kasuwa Area of Maiduguri Borno, Nigeria. The matrix, high density polyethylene with brand name HD5218EA, was procured from PT.TITAN Petrokimia Nusantara Malaysia. The properties of the HDPE are: melt flow rate of 0.5 g/min at 190 ^{o}C, density at 23 ^{o}C is 950 kg/m^3, and melting point of 127 ^{o}C. While MAPE with brand name KT-12 has the following properties; grafting rate of 1.0% to 11.5% and density of 945 kg/m^3 at room temperature. The MAPE were also obtained from the same company as the HDPE.

Characterization

Fiber chemical compositional analysis

These analyses were carried out in the Agriculture Chemical Analysis Laboratory (D 10), Food and Agriculture Analysis Laboratory Programme Technical Services Centre, MARDI Malaysia. The methods used were in-house methods for dry fiber analysis.

Lignocelluloses content

Acid detergent fiber (ADF) analysis was used to determine the hemicelluloses content of the fiber *via* the following methods: D10/M12 in-house method and D10/M5 in-house method. For acid detergent lignin (ADL), as a measure of the lignin content of fiber, the following methods were adapted: D10/M13 in-house method and D10/M7 in-house method.

The NDF determination makes use of Hot Extraction Unit (Fibertec System, USA). Three grams of the dried solid millet husk (MH) specimens were dispensed into the unit and then added with 100 mL pre-heated neutral detergent solution. The mixture was heated for two and one half hours at the solvent boiling point. After that the solution was allowed to cool and then filtered. Then the remains was washed for 2 h using hot distilled water and shifted to a Cold Extraction Unit (Fibertec System, USA). The mixture was washed again for 2 h, this time using acetone, and then dried in a vacuum. Furthermore, another drying process was carried out at a predetermined temperature of 120 ºC over a period of 2 h. The initial and final weight difference gives the expected percentage required. Hence, NDF can be deduced from the equation. These processes were applied to NDF and ADL.

$$\text{NDF} = \frac{W_1 - W_2}{W_0} \times 100 \qquad (1)$$

Fiber fundamental analysis

The elemental (fundamental) analysis was determined using an instrumental method for the determination of solid biomaterial of the total content of carbon, hydrogen, and nitrogen. However, oxygen content was determined using D10/M11 in-house method. The same method was used as in the chemical analysis above.

Millet Fiber Thermogravimetric Analysis

Thermogravimetric analysis (TGA) was conducted using a temperature range from 30 ºC to 900 ºC with a heating rate of 10 ºC/min under a10 mL/min nitrogen flow. The test specimens of 5 to 10 mg were exposed to the TGA instrument model TGA/SDTA 851e from Mettler Toledo (Switzerland).

Alkaline Treatment of Fibers

The millet husk fibers needed to be clean in order to obtain desirable fiber-matrix adhesion and to enhance homogeneity of the overall polyethylene composite. The needed cleaning was carried out using an alkaline treatment method. A coupling agent (maleic anhydride polyethylene (MAPE) was applied to enhance adhesion. The fibers were treated with a 4% w/v NaOH solution for 1 h at 25 ºC; afterwards, the fibers were washed with distilled water until all the NaOH was removed (*i.e.*, until the filtrate pH was no longer alkaline). Subsequently, the washed fibers were dried at 105 ºC for 48 h.

Composites Preparation

Mixing: Rule-of-mixtures (ROM)

This process involved the formulations of fiber and matrix in composites fabrication. In this study, the selection of test conditions was based on the capacity of the twin rotor internal mixer (Brabender GmbH) to blend and extrude the composite. The volumes of the fiber and plastic matrix were governed by the ROM expressions for composites,

$$V_f + V_m = 1 \qquad (2)$$

$$V_{mf} + V_{hdpe} = M_{cw} \qquad (3)$$

where V_f is the fiber volume, V_m is the plastic matrix volume, V_{mf} is the millet husk fiber volume, V_{hdpe} is the high density polyethylene volume, and M_{cw} is the extruder mixer capacity. Thus, specimens were prepared using 0, 10, 20, 30, and 40 wt.% fiber loadings. MHF-HDPE blends using the above recipes were prepared with the use of the extruder mixer and the required capacity. These blends were prepared by mixing the raw materials for a period of 3 to 5 min. The millet husk fiber powder (250 µm particle size) was blended with the HDPE. The rule-of-mixtures (ROM) was employed throughout the process. Tables 1 and 2 show the blend extruder parameters and the composites formulations, respectively.

Table 1. Brabender® Twin Rotor Internal Mixer Condition

Condition	Value
Temperature (ºC)	170
Time (min)	10
Rotor Speed (rpm)	50
Sample weight	Base on rule-of-mixtures(ROM)

Table 2. Formulation of MHF/HDPE

Sample Name	MHF Content (wt.%)	HDPE Content (wt.%)	MAPE (wt.%)
Pure HDPE 100%	0	100	2
MHF-HDPE 10%to40%Loading	10,20,30,and40	60,70,80,and90	2

Compression Molding Process

The composite test specimens were made using a hot press (hot compression molding process) with the heating zones set at 165 ºC (Table 3). The mechanical properties of test specimens, which were prepared from the extruded pellet samples from the internal mixer, were determined in this study. Composite test samples were compressed molded into 15 cm x 15 cm flat plates by the molding process. The molding was performed at a temperature of 165 to 170 ºC and at an estimated compression force of 25 kN for about 10 min. The test specimen thickness was based on the specifications defined by ASTM D638-99 ("Standard Test Method for Tensile Properties of Plastics") and ASTM D790-99 ("Standard Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials") procedures. Subsequently, the plates were machined using a band saw to the dimensions specified in these ASTM standards for particular strength tests.

Table 3. Compression Molding Condition for Formed Test Composites

Parameter	Value
Temperature (ºC)	170
Molding Time (min)	3
Cooling time (min)	5
Sample weight	76 g (per molded sample)

Mechanical Properties Characterization

Samples were prepared for the mechanical properties tests of the composites. The tensile strengths of the composite specimens were measured using an Instron 3365 tester in accordance with ASTM D3039-99. The flexural strength properties of the samples were measured using an Instron 3365 tester in accordance with ASTM D790-99, and the impact strength properties were measured using an Instron CEAST 9050 tester in accordance with ASTM D256-99 (“Standard Test Method for Izod Impact Resistance of Plastics”) and with ISO 180A (“Determination of Izod Impact Strength”).

The composite specimens were conditioned in accordance with ASTM D618-99(“Standard Practice for Conditioning Plastics for Testing”) prior to strength testing in order to standardize the moisture content contained in the samples. Specimens were conditioned at room temperature (23 ºC) and 50% relative humidity for at least 40 h prior to strength testing and assessment. Tensile strength measurements were carried out at 5 mm/min and 3 mm/min cross head speed for flexural tests with a load cell of 5 kN and a span of 50 mm. Un-notched Izod impact strength measurements were performed in accordance to ASTM D256-99 and ISO 180A standard test procedures by the Instron CEAST 9050 impact tester, which is similar to the impact test methods described by Sapuan and Bachtiar (2012). The impact measurements, (with dimensions of 80 mm x 12.7 mm x 3 mm) were tested. For all the tests, five samples were tested, and an average was reported in this study.

Moisture Content

In this study, the moisture content of the fiber was determined in accordance with the ASTM D5229 test standard. A balance with a sensitivity of ±0.01 g was used. A sample specimen was weighed initially, and the mass value was recorded as the wet mass. Afterwards, the sample was dried in an oven at 105 ºC until constant mass was obtained. The sample was allowed to cool. The cooled sample was weighed again, and the mass was record as the dry mass. The moisture content (M_c) was calculated:

$$M_c = \frac{initialmassoffiber - ovendrymassoffiber}{ovendrymassoffiber} x\ 100\% \tag{4}$$

Morphology

The microstructure observations were carried out using a scanning electron microscope (SEM) model HITACHI S-3400N, 20 kV. The microstructure test was carried out by using the fractured surfaces after tensile tests were studied. All the specimens were coated with layers of gold before SEM examination.

RESULTS AND DISCUSSION

Moisture Content

The results for the millet husk fiber moisture content were determined 24 h after oven-drying. It was found that the entire moisture contents were low (0.39%) *i.e.* less than 0.5%. This moisture uptake was due to changes in weather and humidity before the test. One of the major setbacks in the use of natural fiber has been moisture uptake. The moisture absorption can lead to dimensional instability in composites as a result of fiber swelling, which causes micro cracks between the fiber and matrix, thus leading to weak mechanical properties of composites. Therefore, there is less moisture uptake by fiber, the better interfacial interaction with the matrix, leading to enhanced mechanical properties when exposed to environment.

Fiber Chemical Compositions Analysis

Chemical compositions of millet husk fiber are shown in Table 4. It was observed that the cellulose content of the millet husk was 50.4%. This value was different from the 32.7% value reported by Petchwattan and Covavisaruh (2013). The husk's starch content was 13%. It was also observed that the husk's hemicelluloses and lignin contents were 23.2% and 13.2%, respectively, whereas the protein content was 5.9%. However, other constituents of the fiber – pectin, waxy, and fats (3.3%) – contributed to the remaining 7.3%. The general observations in natural fiber was expected that moisture uptake in fiber takes place mainly by hemicelluloses, starch, lignin and surface of cellulose content. Lignocellulosic cereal fiber contains fat and protein; these components are covalently bonded together, and act as a fiber coating. Hence, there are slight variations in composition for cereal byproducts, as has been reported by Andrzej *et al.* (2010).

Table 4. Chemical Composition of Millet Husk Fiber

Component	Content (%)
Cellulose	50.44
Hemicellulose	23.17
Lignin	13.19
Protein	5.90
Fat	3.32

Elemental Compositions of the Millet Husk Fiber

Table 5 shows the elemental composition of the millet husk fiber. The fibers consisted mainly of carbon and oxygen with smaller amounts of other elements. In natural fibers, the percentage of oxygen and carbon play a vital role in compatibility with matrix. In this study, millet husk fiber presents a good percentage of carbon of 19.6%. The high level of carbon in fiber is a result of the presence of a hydrocarbon-rich waxy coating on the fiber surface (Pathapulakkal and Sain 2007). Other elements that are found in the fiber may influence the properties of the fiber and matrix in composites. In natural fiber, the binding energy of carbon and oxygen is usually high, which is coherent with a fiber's surface energy (Williams *et al.* 2011). The change in level of oxygen to carbon ratio O:C was observed to be evidence of the presence or removal of hemicelluloses and lignin which play an important role in fiber-matrix interaction in composites (Liu *et al.* 2007; Williams *et al.* 2011). The oxygen-to-carbon ratio in millet husk was evaluated.

Table 5. Elementary Compositions of Millet Husk Fiber

Element	C	O	N	H	O/C
Percentage (%)	19.62	39.00	3.32	8.80	1.99

Millet Husk Fiber Thermo Gravimetric Analysis

Figure 1 depicts the thermogram of the millet husk fiber (MHF). The MHF mass slowly decreased as the temperature was raised from room temperature to about 245 ºC. The gradual mass loss at this temperature is presumably due to the loss of moisture absorbed from the environment and the release of low molecular weight compounds from the fibers. It was also observed from the thermogram over the 245 to 370 ºC range that there was significant weight loss. This finding revealed the degradation of cellulose and hemicelluloses. Also, the appearance of carbon residue coincided with the degradation temperature. Similar trends have been observed by Gani and Naruse (2007). From 370 ºC, sample weight loss gradually occurred again, showing a certain trend to stabilize. Above 370 ºC, the lignin and carbon residues from the husk fibers were observed to degrade. Hence, the results from the thermograms suggested that the maximum processing temperature recommended when using millet husk as filler in composites should not be higher than 245 ºC. Similar findings have been reported by others (Gani and Naruse 2007; Andrzej *et al*. 2010; Cruz-Estrada *et al*. 2010; Rozita *et al*. 2011). It was observed that there was little difference between the cellulose and hemicelluloses degradation at lower temperature. It was also observed from the thermal analysis that moisture content was removed before 100 ºC. Therefore, weight loss at 100 ºC is considered as initial weight and degradation of fiber was defined at 1% weight loss. A similar finding was reported by Patel (2012).

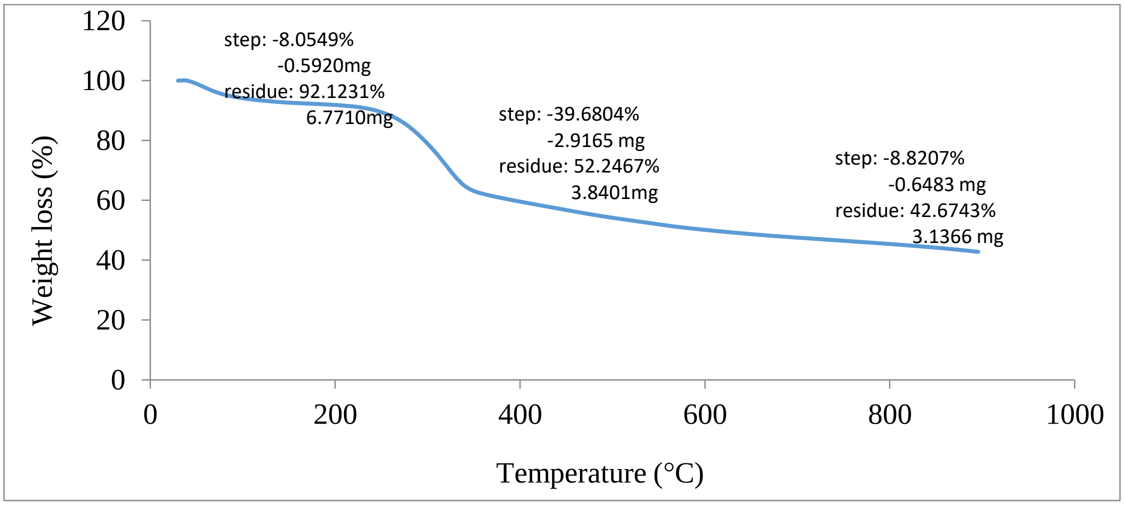

Fig. 1. Thermographic profile of millet husk fiber

The effects of temperature on the fiber are pre-determined to ascertain the degradation of the fiber and its ability to act as fillers or reinforcements in polymer composites. Therefore, when the thermal degradation of millet husk fiber becomes higher than 245 ºC, there are residues that comprise a small amount of ash, leading to poor interfacial adhesion between fiber and matrix in the composite. This can be seen from the thermogram. Accumulation the volatile compound with fiber residues can lead to voids during composites fabrications that can result in low mechanical properties.

Figure 2 presents the derivative thermogram curve of millet husk fiber. Additionally, the position marked with an “X” on the derivative thermogravimetric (DTG) curve reveals the start of cellulose decomposition; this temperature was higher when compared to other lignocellulosic agro-waste, but is similar to results reported by Panthapulakkal and Sain (2007).

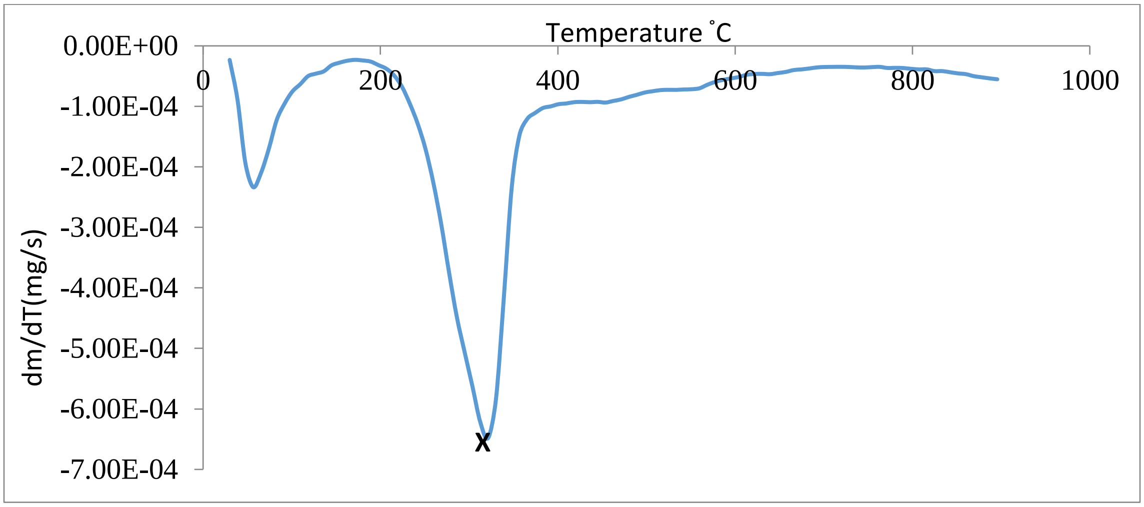

Fig. 2. Derivative thermographic (DTG) curve of millet husk fiber

Effect of Alkali Fiber Treatment and Processing on HDPE Composites

The millet husk fibers were subjected to alkali treatment to clean impurities from the fiber surface. The fibers were first washed with water and then soaked in 40g/L NaOH solution for 24 h at 25 °C. The resulting fibers were separated from the NaOH solution and washed with several 100 mL portions of water until the filtrate had a neutral pH. These alkali-treated fibers were dried for 72 h. The chemical treatment of the lignocellulosic fibers by NaOH, which is also known as mercerization, is used to modify the crystalline structure of the cellulose (Ortega-Leyva 2008). The alkaline treatment disrupts the orientation of highly crystalline cellulose structure to form amorphous regions. These amorphous regions provide greater access for chemicals to penetrate the cellulose portions of the husk fibers (Liu *et al.* 2007; Andrzej *et al.* 2010). Therefore, fiber surface treatment play an important role in fiber-matrix interaction in composites.

Mechanical Properties of the Composite

Tensile

Figure 3 shows how the tensile strengths of various MHF-HDPE composites changed with different MHF loading levels. The trend in this figure did not follow any smooth pattern, which probably indicated the heterogeneous dispersion of the MHF filler within the polymer matrix. In natural fiber polymer composites, there are different stages in the fiber-matrix interactions. Several factors are responsible for these attributes. The types of the fibers, matrix, environment, and fabrications techniques play important roles in these dynamics. The increase of the MHF loading to 10% by weight resulted in an increase in the tensile strength of the MHF-HDPE composite when compared to 0% filler loading (*i.e.*, 100% HDPE). However, as the fiber loading increased, the composite’s tensile strength decreased. However, it was observed that at 30% fiber loadings there was loss in strength of 18.8 MPa relative to the strength of neat HDPE. The phenomenon may be due to the presence of voids in composites, and accumulation of these voids leading to early failure. It was also observed at 40% fiber loading that there was a slight increase in the composite tensile strength. Hence, the decreases in the observed tensile strengths were attributed to poor MHF-HDPE interaction as a result of weak adhesion between the fiber and matrix. While, the increases in tensile strength is due to better fiber dispersion as well as bonding at the interface fiber and matrix.

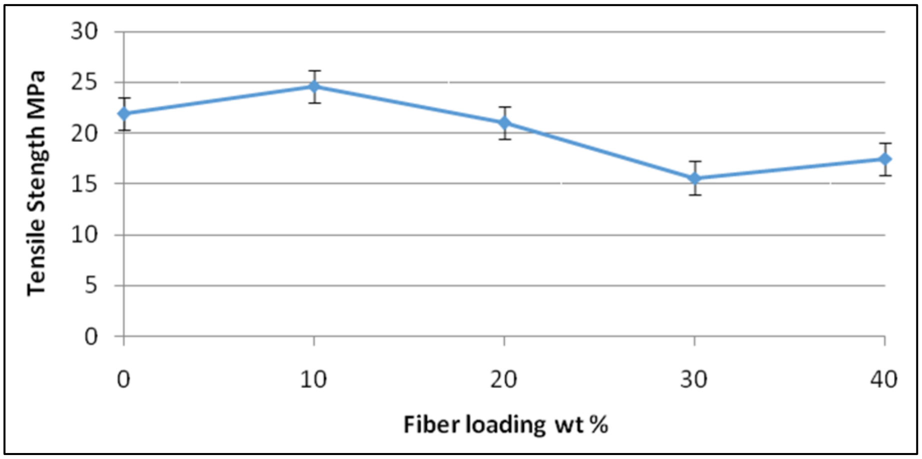

Fig. 3. Effects of MHF loading on tensile strength of MHF-HDPE composites

Figure 4 shows that there were substantial increases in the tensile moduli of the MHF-HDPE composites when the MHF loadings were increased.

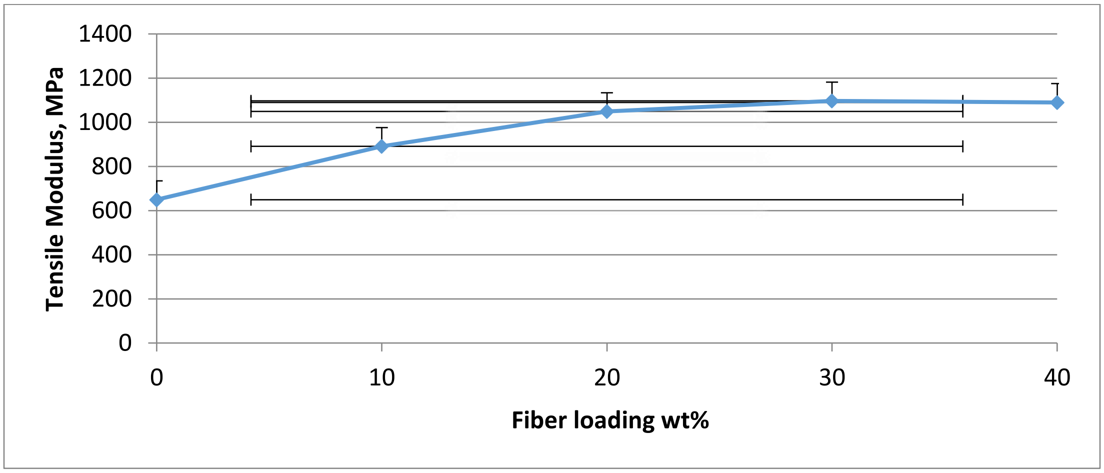

Fig. 4a. Effects of MHF loadings on tensile moduli of MHF-HDPE composites

It was observed that the maximum tensile modulus value was 1103.6 MPa, which occurred at 40% MHF loading. However, at 30% MHF loading, there was a slight decrease in the tensile modulus. But there were variations in the values of the modulus, as can be observed from the figure.

These variations were more for high fiber loadings of the composites. Heterogeneous distribution of MHF filler in the polymer matrix might be a reason for these observed fluctuations, which became pronounced in the cases of highly-filled composites. From this study, increasing the MHF loading up to 40% in the HDPE matrix increased the rigidity of the MHF-HDPE composites. The level of adhesion between MHF filler and the HDPE matrix hardly affected the tensile moduli of the resulting composites.

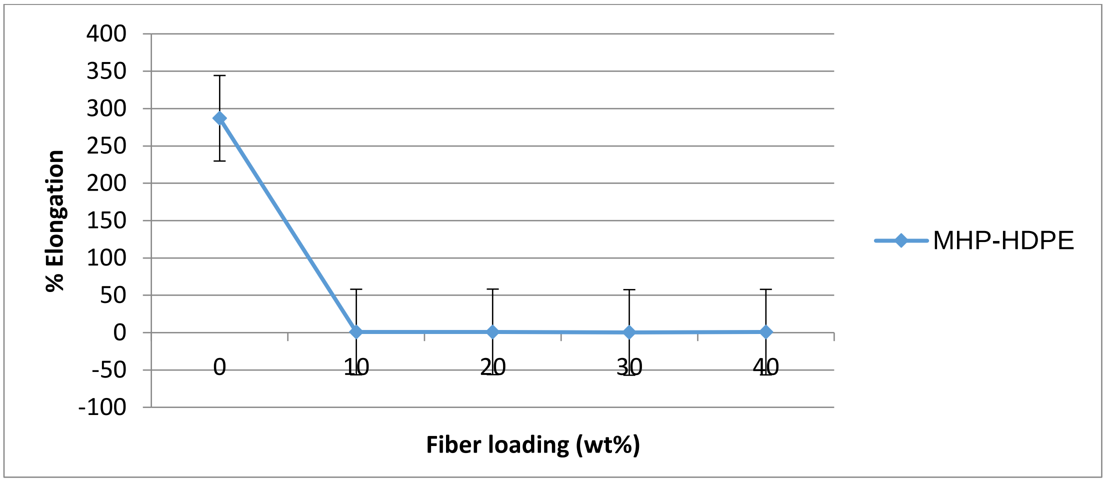

Fig. 4b. Elongation at break of MHF-HDPE composites

Flexural Properties

Figure 5 shows the flexural strength with different MHF loadings. It was observed that flexural strength increased as MHF loading was increased. The increase became more pronounced when the filler loading was greater than 10%.

A maximum increase in flexural strength of about 37% was observed (*i.e.*, from 25.9 MPa to 41.1 MPa) when the filler loading in the composite was increased from 0 to 40%. This observation could be attributed to the contribution provided by the high strength millet husk fibers.

There was an increase of 63% at 40% fiber loading to neat HDPE and 72% at 40% fiber loading to 10% loading. The strength difference of 15.2 MPa between neat HDPE and 40% loading indicate steady increases. Therefore, fiber did not completely reduce the ability of fibers and matrix to interact. The increased adhesion led to better interactions between the fibers and matrix, which allow stress transfer from the matrix to fiber and translate into improve flexurability.

The increase became more prominent when fiber loadings went above 10% as a result of good surface adhesion between the fiber and matrix and increased surface area exposed for interaction. This again, is only possible if there is better stress transfer from matrix to fiber through a fairly strong interfacial bond. Bond splitting leading to failure is not possible in a flexural test, as the maximum strain applied is only 5%. It could be inferred that 5% strain was insufficient to cause a failure. Hence, in the case of flexural strength, interfacial interaction between fibers and matrix had only served to transfer stress. These differences in flexural strength could be attributed to the contribution provided by high strength fibers.

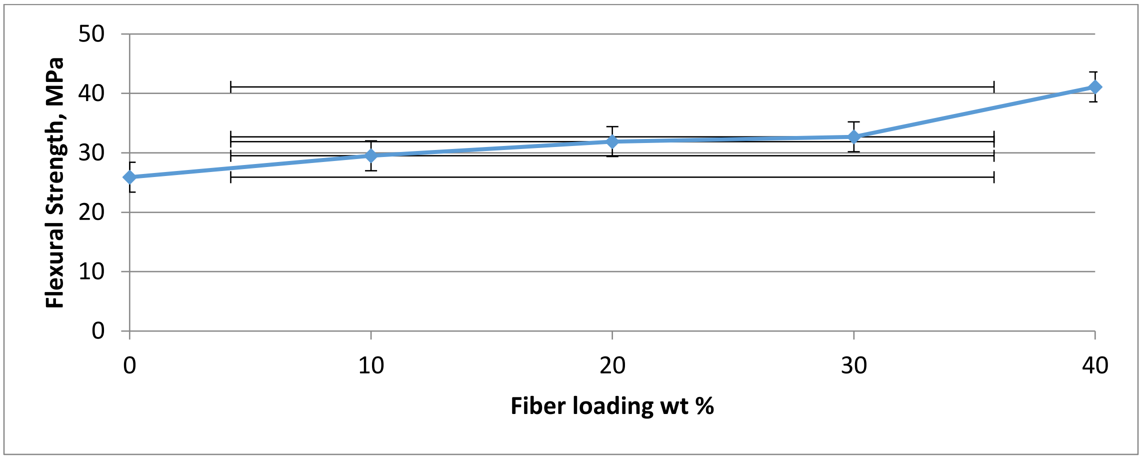

Fig. 5. Effects of MHF loadings on flexural strength of MHF-HDPE composites

The flexural moduli of the composites were found to increase with increasing millet husk fiber loading, as depicted in Fig. 6. The investigations revealed a steady increase in the modulus values as the fiber loading was increased from 712.9 GPa for neat HDPE to 1987.5 GPa at 40% MHP loading of HDPE composites. This could be attributed to the contributions from the fibers.

It was noted that there was initially about a 39% improvement in flexural modulus when the fiber loading was increased from 0 to 10%. Further improvements in flexural moduli became more pronounced for loadings greater than 10%. A maximum flexural modulus of about 1988 MPa was achieved at 40% fibre loading. Therefore, the increases in the modulus of these composites were due to the higher initial modulus of the fibers acting as a main building block of composites and improvement of interfacial bonding between the fibers and matrices. Furthermore, this could be attributed to the contribution made by fibers to contribute its own mechanical properties to the composite. It can be seen that surface treatment played a vital role in modulus strength in all the composites.

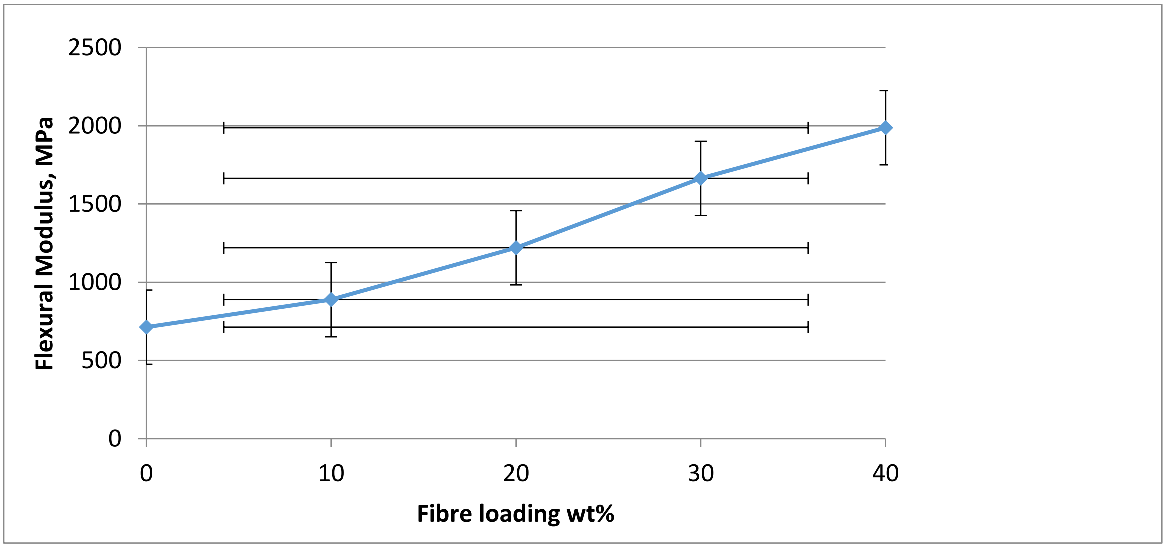

Fig. 6. Effects of MHF loadings on flexural moduli of MHF-HDPE composites

Izod Impact Strength Properties

Figure 7 presents the impact stress as a function of fiber loading in treated fiber composites. The stress steadily decreased as the fiber loading was increased. The maximum absorbed energy of neat HDPE was 76.5 kJ/m^2, whereas the value was 9.37 kJ/m^2 at 40% fiber loading of HDPE composites. There was a 67.1 kJ/m^2 difference between neat HDPE value and 40% fiber loading value, indicating a 82% decrease from the neat value, while there was a 11.96 kJ/m^2 difference between 10% and 40% fiber loading showing 12% decrease. Although in general the impact strength of the composites was poor, the treatment of the fibers did not improve the strength. Increasing the fiber loading in the composites implies a decrease of the thermoplastic matrix content, which absorbed the impact energy more efficiently than the fiber. In this way, the higher the fiber content in the composites, the less efficient they are at absorbing impact energy, and the highest susceptibility to fracture, resulting in lower rates of impact resistances. Also weak adhesion between fiber and matrix within the composites as a result of voids and fiber pull out can lead to failure as well as poor energy absorption. This decrease appeared to be the result of an increase in fragility of the polymer matrix, because MHF concentration increased, so the plastic behavior of polymer matrix decreased. Similar results have been reported by Bouafif *et al.* (2009).

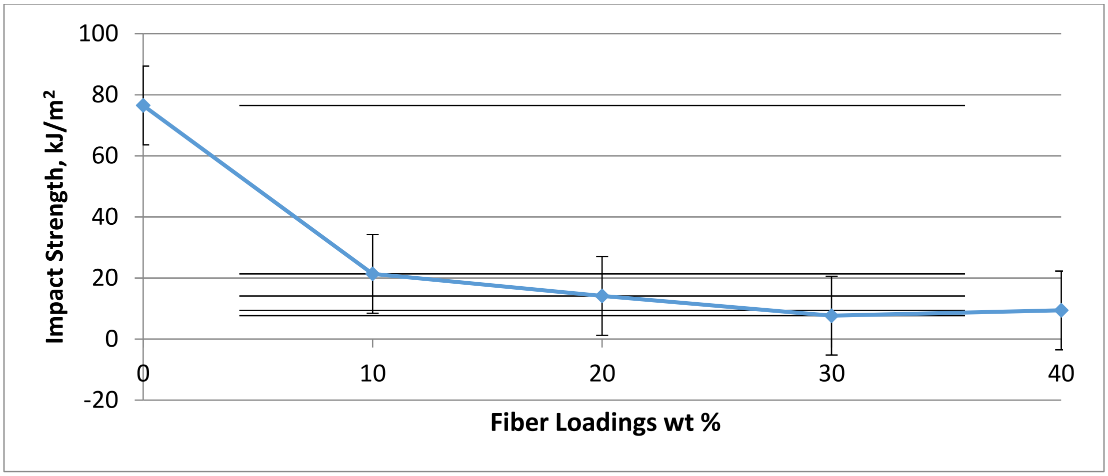

Fig. 7. Effects of MHF loadings on Izod impact strength of MHF-HDPE composites

Microstructure Observations with Scanning Electron Microscopy

SEM images of the fractured surfaces of MHF-HDPE composites are depicted in the micrographs of Fig. 8. Figure 8(a) shows that there was better adhesion between the fibers and the polymer matrix. As a result, at 10% fiber loading, there was an increase in tensile strength of the composites. Figures 8(b) and 8(c) show fiber pull-out from the fractured matrix surface; this fiber pull-out characteristic could make the fiber agglomerate. Hence, this led to reduction in the tensile strengths of the composites as the fiber loadings became greater in value. The microstructure revealed the formation of voids at the MHF-HDPE interfaces (Fig. 8 (d)). When the composite is strained, these voids or fiber pull-out merged and resulted in fracture of break. The fiber pull-outs increases as the fiber loadings increased, therefore decreasing the elongation at break. However, at 10% loadings, there was better dispersion of the MHF filler within the HDPE matrix, which reduced fiber pull-out and breakage.

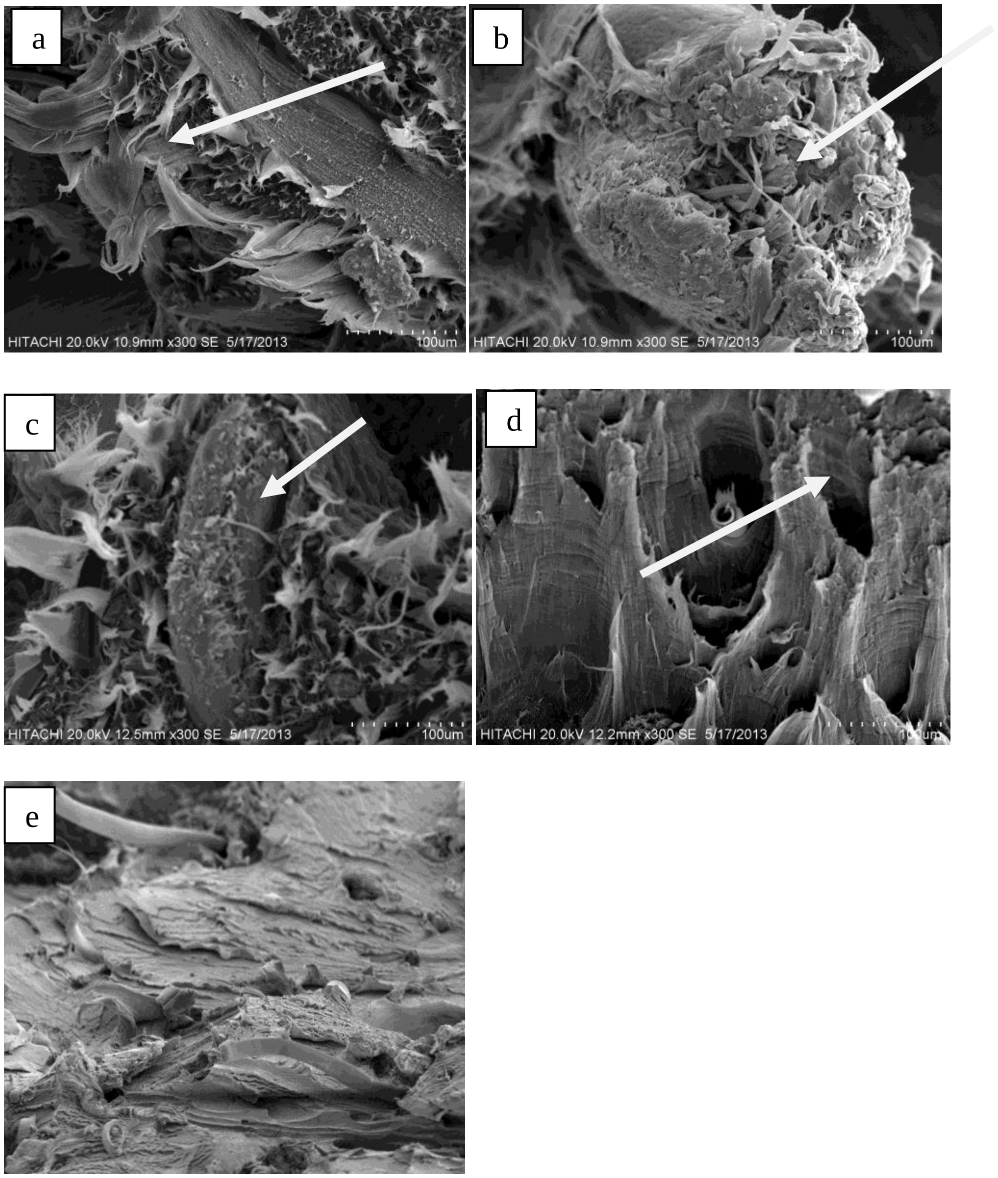

Fig. 8. SEM micrographs of samples with various MHF loading: (a) 10% with arrow showing fiber-matrix dispersion; (b) 20% with arrow showing fiber pull out; (c) 30%, with arrow showing fiber pull out; (d) 40% with arrow showing void; and (e) Neat HDPE

CONCLUSIONS

- Millet husk fiber (MHF) possessed high amounts of cellulose (50.4%), along with hemicelluloses (23.8%) and lignin (13.2%).
- The maximum allowed processing temperature for MHF filler in HDPE composites should not be higher than 245 ºC.
- Due to the high cellulose content of MHF, the amount of moisture absorption by this filler increases.

- Increasing the MHF loading level in the composite led to decreases in the tensile strengths of the composites, whereas the tensile moduli increase.
- The plasticity of the HDPE matrix decreased with increasing MHF loading; therefore, the elongation at break decreased.
- The flexural strengths and moduli increased as loading increased. This showed that MHF-HDPE composites exhibit brittle behavior when increasing the MHF loading level.
- Therefore, millet husk fibers with proper surface treatment/modification could represent promising raw material filler for polymer composites.

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