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ORIGINAL RESEARCH ARTICLE

THE EFFECT OF FIBER SIZES ON MECHANICAL PROPERTIES OF MILLET HUSK (PENNISETUM GLAUCUM) FIBER FILLED HIGH DENSITY POLYETHYLENE COMPOSITES

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ABSTRACT

This study examined the effect of fiber sizes on the mechanical properties of millet husk powder (MHP) filled high density polyethylene (HDPE) thermoplastic composites. The MHP-HDPE composites were prepared by melt blending techniques, accompanied by compression molding process. Three different fiber sizes; 250µm, 500µm and 750µm were pulverized and the fiber loading was 10%, 20% 30% and 40% by weight. Tensile and impact properties were tested using universal testing machine (UTM). 1The results show that ensile strength increased at certain loadings while tensile modulus increased by increasing the millet fiber loading. While the impact strength decreases substantially with increase fiber loading the tensile strain considerably decreased at millet husk fiber application above 10%. Therefore, it was observed that composites with least size fiber indicated improvement with increasing strength as well as relative extension.

1.0 Introduction

Agro waste and natural fiber composites are not altogether a new phenomenon. The interest in superior material started to grow as a result of environmental safety and industrialization (Ester Rojo et al., 2015; Patel, 2012; Sdrobiş et al., 2012). Millet husk as waste are abundantly available and has the potential to be used as filler or reinforcement in plastic composites (Abubakar and Ahmad, 2010; Hammajam et al., 2014). Rapid increase in quantity and categories of agro waste as a result of rigorous agriculture in wake of population growth and improve standard of living is becoming dwindling problem as rotten waste emits methane and leachate, and open burning by farmers to clear lands generate carbon dioxide (CO_2) and other local pollutants (El-Sabbagh, 2014; Gloria, 2001; Hamzeh et al., 2013). However, improper management of these wastes is contributing toward the climate change, water and soil contamination, and air pollution (Chandak, 2010). Lignocellulosic thermoplastic composites have steadily achieved significance in most parts of the world recently (Azhar et al., 2015; Du et al., 2014; Hadjadj et al., 2016; Kumar and Bavan, 2010; Kwon et al., 2014). While wood play key role in recent time, but composites researchers continue to explore for new, cheaper and environmentally friendly lignocellulosic materials as substitute for wood and inorganic fibers (Crespo et al., 2010; El-Abbassi et al., 2015). Thus, these agro waste fibers such as millet husk, rice husk, flax straw, corn cobs, cotton husk, sugar baggage, kenaf, wheat straw, luffa fiber, banana stalk, barley husk and hazel shell are potentials precious raw materials in composites production.

Thus, these agro waste show better future applications due to its availability, annual renewability, light weight, low cost, and green environment component materials (Andrzej et al., 2010; Behzad, 2011; Sahari et al., 2013). Because of the recent environmental concerns and millet husk disposal difficulties, the use of MHP has become most important. Several studies indicate good and compatible properties of lignocelluloses materials (Bhattacharyya et al., 2015; Boopalan et al., 2013; El-Abbassi et al., 2015; Fan and Naughton, 2016). Other investigators have shown the upper hand for using cereal fibers in variety of polymer matrices. In this study, an attempt is made to utilize MHP-HDPE composites. The effects of fiber sizes on the mechanical properties of the composite were evaluated. These properties of the composites will determine its viability for different applications. Understanding these parameters is critical in strategizing composites material applications. Several studies were performed on the size effect of fibers on mechanical properties (Arib et al., 2006; Ratna Prasad and Mohana Rao, 2011; Vilay et al., 2008).

Millet husk is novel agro waste from cereal grain (pennisetum glaucum) in thermoplastic composites applications (Hammajam A .A et al., 2014; Patel, 2012). Fiber size and loading are critical parameters in thermoplastic composites applications due to its stress transfer function between the fiber and polymer matrix (Panthapulakkal et al., 2005a). Polyethylene plays crucial role in thermoplastic composites production because of it suitable characteristics such as temperature resistance, melt flow index and density. For effective utilization of cereal agro waste fiber filled/reinforced composites, certain parameters are paramount to be considered; strength, stiffness, and toughness (Rowell, 1997; Sapuan, 2001). Several studies have presented on natural fibers and their composites, however there were no attempt to characterize millet husk husk fibers. This investigation try to evaluate the effect of millet husk fiber sizes on the mechanical properties filled high density polyethylene composites.

2. Materials and Methods

2.1 Materials

Millet husk was collected from a farm site at Bulumkutu Kasuwa Maiduguri, Borno State Nigeria. Thermoplastic high density polyethylene (HDPE Titavene TITAN Petrokimia Nusantara, chemical divison Sdn Malaysia. HD5218EA) with the properties of matrix being density of 0.960 g/cm3 and melt flow index of 8.2 g/10 min (190 °C, 2.16 kg) were used.

2.2 Fiber Pulverizations

Millet husk fibers were pulverized into different sizes; 250µm, 500µm, and 750µm using Fritsch Pulverisette P15/P16 Pulvetic USA mill. The pulverized samples were subjected to 105°C temperature for 24hrs in an industrial oven to remove moisture before blending with the high density polyethylene. These pulverized parameters are shown in Table 1 while Figure 1 shows millet husks pulverized sizes.

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250 µm pulverized fiber

500 µm pulverized fiber

750 µm pulverized fiber

Unpulverized millet husk

Figure 1: Views of Millet Husks, a) 250 µm, b) 500 µm, c) 750 µm and d) unpulverized)

2.3 Composites Characterization

MHP and HDPE were compounded by melt blending techniques, followed by compression molding process. Internal mixer (Brabender GmbH) was used in the blending process at processing temperature of 170oC, for a period of 10 min, and rotating speed of 50rpm (EL-Shekeil et al., 2011). Labquip scientific 40ton compression molding machine was used in hot pressing the test specimens. 15cm × 15cm × 0.3cm steel mold was used for the compression process. The samples were pre–heated for 3 min at 170oC. Venting time was set to 3min and final press temperature of 170oC for 3min. Finally, the samples were cold pressed for 5min at room temperature. MHP–HDPE composites was formed from the three sample sizes with 0%, 10%, 20%, 30%, and 40% weight of fiber loading. Thus, this formulation can be seen in Table 2.

The tensile properties were tested according to ASTM D638 (ASTM Standard D638, 2010) by using Instron 3365 machine. Specimens for the tensile tests were specified according to type I ASTM standard test samples. Tensile testing was carried out at 5mm/min with load cell of 5kN. Thus, 3mm/min cross head speed for flexural tests with load cell of 5kN and span of 50mm according to (ASTM D790, 2010). Five samples were tested each and the average was taken for test.

The fiber-matrix dispersion analyses were carried out to understand the extent of fracture surface when the composites were subjected to tensile testing. The fiber dispersions were inspected using SEM. This process was examined using a scanning electron microscope (SEM

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model S-3400 N HITACHI, 20 kV). Samples of different fiber loadings were studied. The samples were coated with gold before the examination under the electron beam. The scanning electron micrographs were then used to analyze the composite filler-matrix interaction.

3. Results and Discussion

3.1 Effect of fiber size on tensile strength of MHP-HDPE composites

Figure 2 depicts the mean values of tensile strengths of different MHP–HDPE composites with different fiber sizes and loadings. These graphs did not follow any smooth pattern probably due to non-homogeneous dispersion of MHP fibers in the polymer matrix. Generally, in composites, there was a complicated interaction of the dynamics of the basic stages such as matrix, fiber and interfacial zone between the fiber and matrix (Sapuan and Bachtiar, 2012). The increment from 10–30% by weight of MH causes slight decreased in the tensile strength of MHP–HDPE composites when compare to 0% (HDPE) loading on the fiber size basis. It was observed that composites with 30% fiber loadings have variations of tensile strength of 23MPa, 15.6MPa and 18.8MPa for 250µm, 500µm, 750µm respectively; it is slightly below 0% HDPE by 28.8% for 500µm fiber sizes. It is noteworthy that fiber size has effect on the tensile strength of MHP–HDPE composites similar finding in Iftekhar and Prakash, (2010).

Furthermore, the decreases in the tensile strengths are due to poor MHP–HDPE interaction as a result of opposing separations of the MHP fiber and HDPE matrix. Figure 2 showed an increase in loading up to 40% has slightly increased the tensile strength of the composite for all the fiber sizes. This could be due to better dispersion and more interface in the HDPE matrix resulting in better bonding between MHP and HDPE. Thus, for MHP fibers with 250µm size, increased surface area might have allowed for better filler-matrix interaction hence increasing the chances to enhance the strength. However, in 500µm and 750µm sizes, improvements in tensile strength were very little and, in some instance, decreased. This might be also due to decreased surface area and ineffectiveness of MHP fibers as stress bearing centers and might, provided routes for stress release.

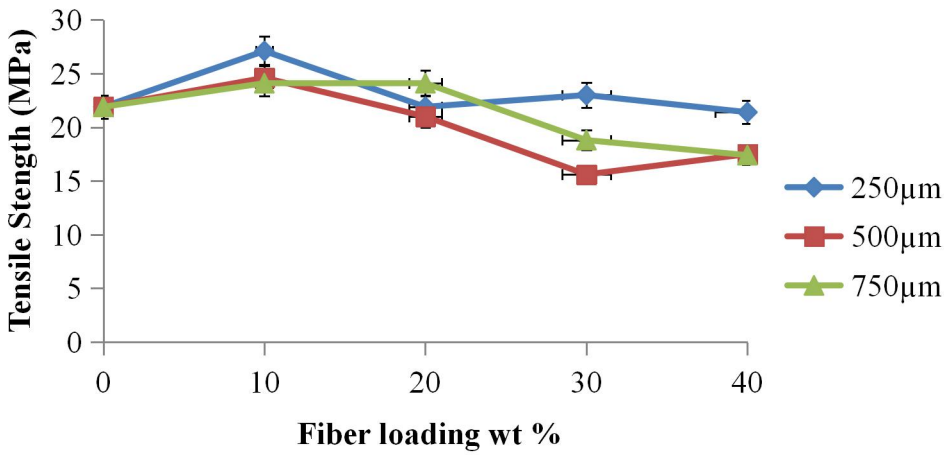

Figure 2: Tensile strength of MHP–HDPE composites at various fiber loadings and sizes

3.2 Effect of fiber size on tensile modulus of MHP-HDPE composites

Fiber size did not seem to affect the modulus but, fluctuations in the values of the modulus were observed. These fluctuations were more for highly filled HDPE as obvious from variations on graph in Figure 3. Non-homogeneity might be a reason for these fluctuations which became

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pronounced in cases of highly filled composites. From Figure 3, there were substantial increase in the tensile modulus of MHP–HDPE composites as fiber loadings increased. It was observed that the maximum value of the tensile modulus was 1103.6GPa at 40 % loading for 750µm. But at 30% loadings for 500µm and 750µm there were slight decrease in the tensile modulus.

From the study, it was still appropriate to argue that increasing MHP up to 40% in HDPE increases the rigidity of MHP–HDPE composites. It showed that the level of adhesion between MHP–HDPE hardly affect the tensile modulus. This is because modulus is processes that involve so small strain values and small stress may be produced with application of little value of elongation. The 250µm, showed somewhat longer relative elongations up to extent of MHP. Hence, it might be possible to get greater elongations by further reducing the sizes of the fibers. Also, from the SEM images of the fracture tensile test specimen showed the matrix separation had been originated at the interface of large fiber size, and no separation of matrix around small fibers. As the specimen is elongated, these would be a stress transfer from the polymer to the MHP through the interface if interfacial bondage is good. When there is an increase in the fiber size the void size also increases resulting in earlier failure and this might be the cause of poor relative elongation for higher fiber size. Another possible reason is the higher surface area provided by smaller fibers for interacting within the matrix polymer. Table 1 depict the millet husk fiber sizes used during the investigations.

Table 1: Millet Husk fiber Powder (sizes)

Sieve mesh number	Sieve diameter (µm)
20	750
40	500
60	250

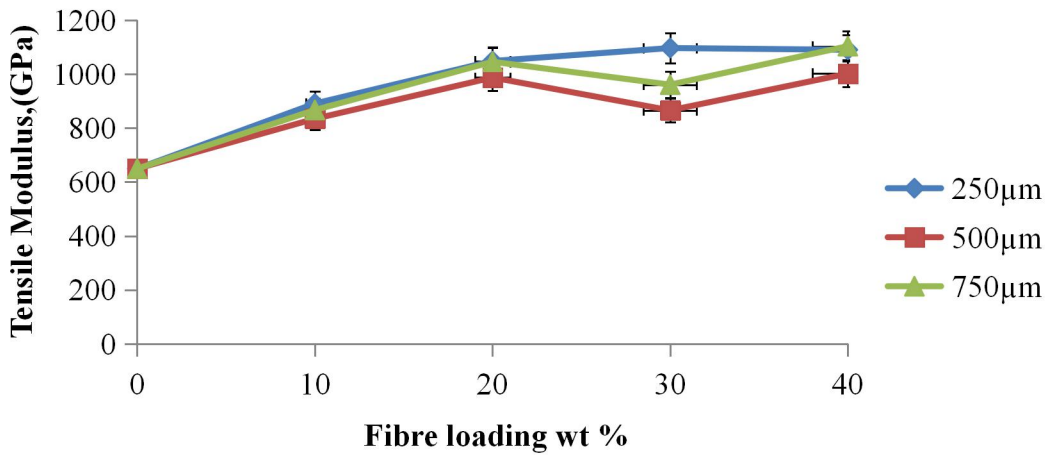

Figure 3: Tensile modulus of MHP–HDPE composites at various fiber loadings and sizes

3.3 Effect of fiber loadings and sizes on flexural strength of MHP-HDPE composites

Figures 4 show the variation in flexural strength with varying loadings of millet husk fiber of different particle sizes. It was observed that flexural strength increased as millet husk powder loading increased. The increase becomes more pronounced when loading went above 10%. A maximum of about 37% increase was observed from 25.9MPa for 0% HDPE to 41.1MPa for 40%

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loading of 250µm fiber size, 24.5MPa 500 µm and 32.4MPa 750µm. This could be attributed to the contribution provided by high strength millet husk powder. Thus, flexural strength increased with increasing fiber loading of millet husk. This again, is only possible if there is stress transfer from matrix to fiber through a fairly strong interfacial bond. Bond cleavage leading to failure is not obtained in bending 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 millet husk fibers and HDPE matrix had only served to transfer stress. As observed from the graph, the flexural strength of composites with 500µm and 750µm fiber sizes decreases at 30% and 40% fiber loadings. While the composites with fiber sizes of 250µm increase at 40% fiber loading.

Hence it is presumed that increase in fiber loading above 40% may lead to decrease in flexural strength for 500µm and 750µm fiber sizes and may also improve in strength for composites with fiber sizes of 250µm as expected from this study. It was observed that fiber with smallest size (250µm) showed better strength in 10% and 20% fiber loadings. This might be because of increased surface area exposed for interaction. Thus, increase the strength by using further small size of millet husk powder could be expected.

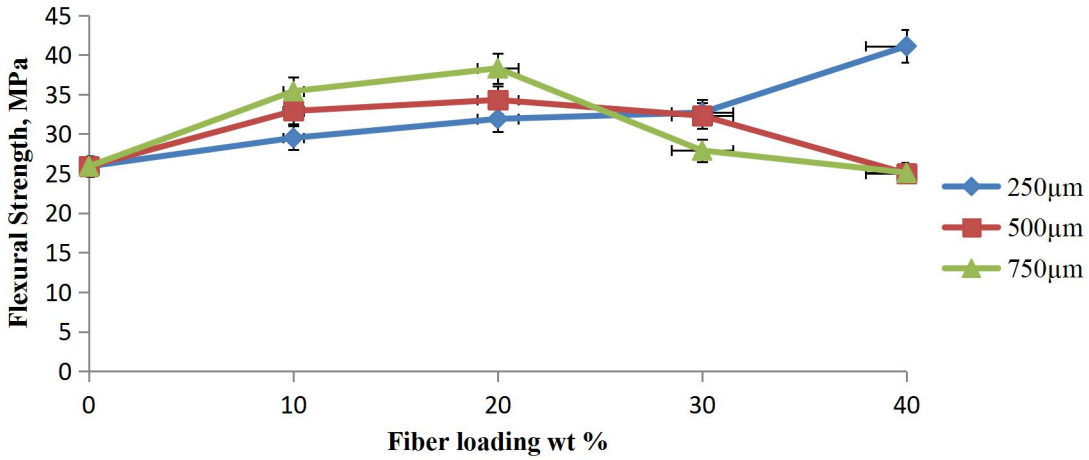

Figure 4: Effects of Fiber Loadings and Sizes on Flexural Strength of MHP-HDPE Composites

3.4 Effect of fiber loadings and sizes on flexural modulus of MHP-HDPE composites

As expected from the natural fiber composites trend, flexural modulus was found to increase with increasing millet husk powder loading as can be observed from Figure 5. Furthermore, this could be attributed to the contribution made by millet husk powder to impart its own property to the composite. From Figure 5 it was revealed that initially there was around 39% improvement in flexural modulus up to 10% millet husk powder loading for 750µm fiber sizes. The increase in the flexural modulus became pronounced for loadings greater than 10%. Maximum flexural modulus of about 1988GPa was achieved at 40% millet husk powder fiber loading of 250µm fiber sizes which amounted to about 64% more than 0% HDPE. The larger fiber size 750µm showed great increase in modulus at higher loadings, but had lower flexural modulus at loadings below 10%. As seen on this graph, increase in fiber loading to 40%, composites with fiber sizes of 500µm and 750µm decreases in flexural modulus. However, the composites with fiber size 250µm increase at 40%. Thus, it is expected that with further increase in fiber loading above 40%, it presumed increase in modulus of composites of 250 µm and decrease in modulus of composites with 500µm and 750µm. while Table 2 show the composite formulations of fiber and matrix.

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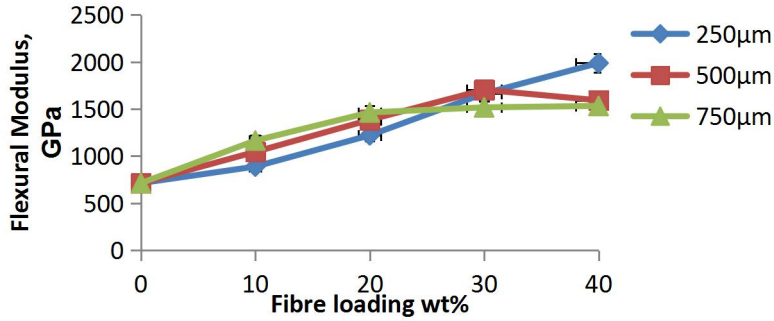

Figure 5: Effects of Fiber Loadings and Sizes on Flexural Modulus of MHP-HDPE Composites

Table 2: Composite Formulation

MHP (wt %)	HDPE (wt %)
10	90
20	80
30	70
40	60

3.5 Effect of fiber size on Impact strength of MHP-HDPE composites

Figure 6 indicate MHP-HDPE composites impact rates show that increase in fiber loading causes reduction in impact strength of the composites considerably. However, fiber size of 250µm indicates slight improvement in impact strength compare to rest. It is probably that this decrease is as a result of increased in fragility of the polymer matrix, because MHP fiber concentration increased, so also plastic behavior of polymer matrix decreases. 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. Additionally, weak adhesion between fiber and matrix within the composites as a results voids and fiber pull out can lead to failure as well as poor energy absorption.

It is probably that this decrease is as a result of increased in fragility of the polymer matrix, because MHP concentration increased, so the plastic behavior of polymer matrix decreases. Furthermore, increases in filler weaken the interaction ability which leads to reduction of filler to transfer that as applied to a test specimen. Therefore, the investigation reveals that toughness of MHP-PLA and MHP-HDPE composites are dependent mainly on the plastic behavior of composites.

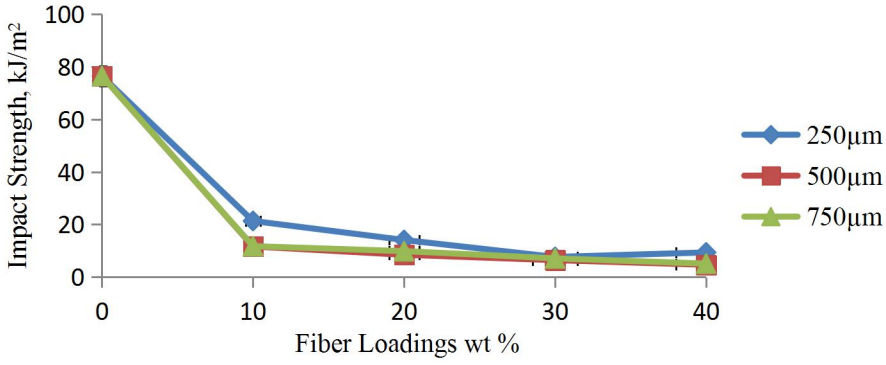

Figure 6: Effects of Fiber Loadings and Sizes on Impact Properties of MHP-HDPE Composites

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3.6 Microstructures observation

SEM images of tensile fractured surface of MHP-HDPE composites were depicted in Figure 7. The microstructure in figure (7a) revealed the formation of voids at the MHP-HDPE interfaces. When strained these voids or fiber pull-out as seen in figure (7b) coalesce and lead to fracture of break. The fiber pull-out advanced with MHP concentration hence decreasing the elongation at break. From the Figure (7c), it was revealed that small size fibers did not play important role in bringing about fracture. This fiber pull-out characteristic could make the fiber to undergo accumulation. Hence, these lead to reduction in the tensile strength of the composites mostly at higher fiber loadings. But at 10 % and 20 % loadings, there were better dispersion of MHP-HDPE formation with reduced fiber pull out and breakage. This is the main reason for improvement of tensile strength at these loadings for smaller fiber sizes.

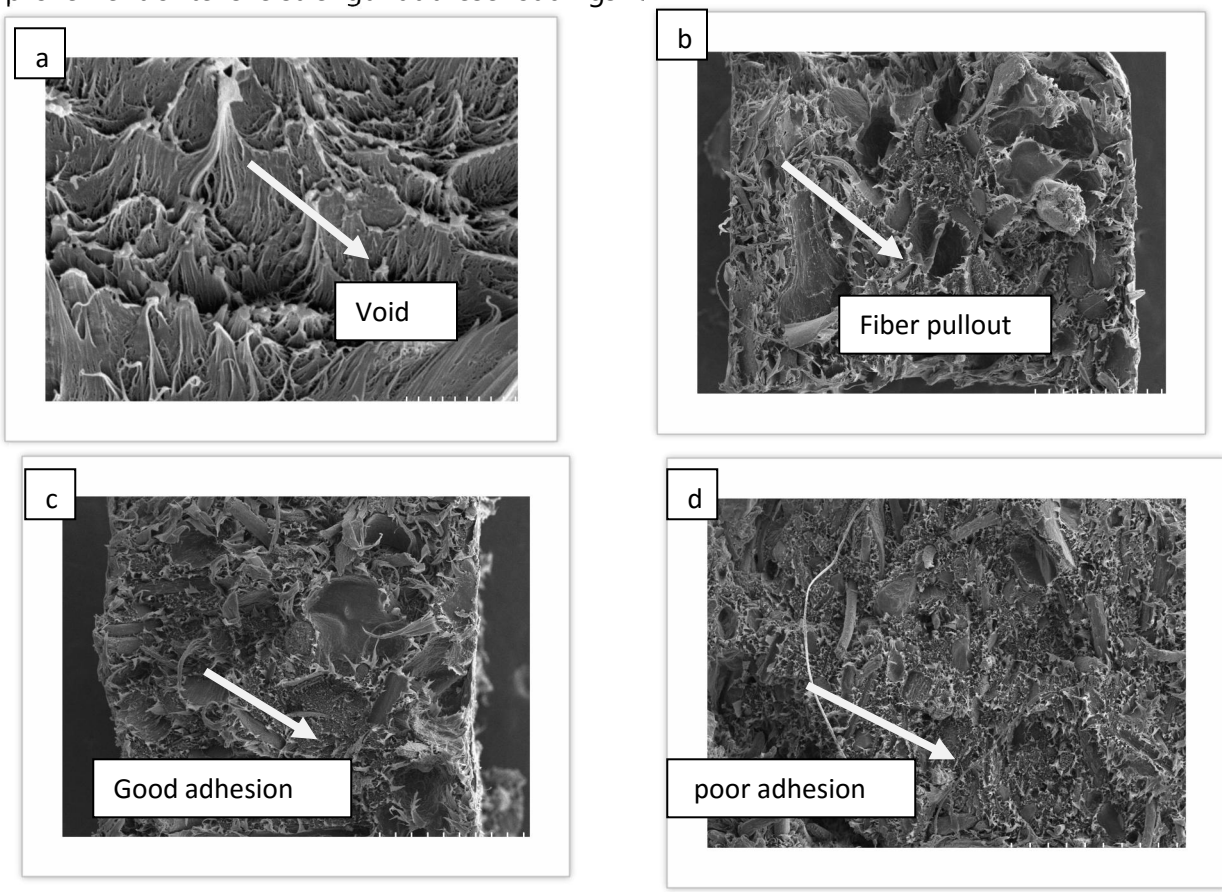

Figure 7: SEM fractomirographs for samples with various fiber loading; (a) 10 % (250 µm), (b) 30 % (500 µm), (c) 20 % (750 µm), and (d) 40 % (750 µm)

4. Conclusions

This study reveals that increasing fiber loadings lead to decrease in the tensile strength of composite. The plasticity of the polyethylene decrease with increase millet husk powder. Therefore, the elongation at break decreases but the flexural strength and modulus increase as loading increase. This shows that composites properties are brittle after the increase in fiber loading despite the sizes. It was also observed that increasing fiber loadings lead to increase tensile modulus of the MHP–HDPE composites with respect to fiber sizes. As shown from SEM microstructure, the presence voids and fiber pull out due to weak adhesion between MHP and HDPE may result to reduction in tensile and impact strength. These problems may be enhanced by fiber surface modifications. Therefore, it is concluded that husk powder sizes have insignificant influence on the mechanical properties of the composites.

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