# Physical and Mechanical Properties of Modified Wheat Straw-Filled Polyethylene Composites

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This study investigates the effect of modified wheat straw on the physical and mechanical properties of modified wheat straw/high-density polyethylene (MWS/HDPE) straw-plastic composites. Wheat straw fibers with particle sizes in the range of 0.25 to 0.50 mm were modified with caprolactam (CPL). A Fourier transform infrared spectroscopy (FT-IR) analysis of MWS showed that when the CPL level was 5%, the intensity of the hydroxyl (O–H) and carbonyl (C–O) absorption peaks noticeably decreased, indicating a corresponding decrease in the polarity of the fibers. A physical analysis of the wheat straw fibers indicated that after the modification, the characteristics of the fibers were closer to those of the HDPE polymer matrix, thus contributing to good compatibility and dispersion of the straw fibers within the matrix. The composites of the high-density polyethylene with modified wheat straw particles were successfully synthesized using the melt blend method. The prepared composites were characterized using scanning electron microscopy (SEM), and their mechanical properties were investigated. The MWS/HDPE composites showed superior mechanical properties because of a greater compatibility of MWS with HDPE. The modified WS fibers function as "biological steel," reinforcing the HDPE to produce bio-composites.

*Keywords: Wheat straw; Straw-plastic composites; Modification; Caprolactam; Mechanical properties*

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## INTRODUCTION

Over the last two decades, the use of natural fibers as reinforcements and blends in polymer composites has been steadily increasing. The use of lignocellulosic fibers and their composites has attracted attention because of their preferred properties, such as low density, low cost, their non-abrasive nature, their availability, biodegradability, renewability, world-wide distribution, high level of filler loading, good mechanical properties, and their ability to provide a safer working environment (Saheb and Jog 1999; Rowell *et al*. 2000). Natural fiber-reinforced composites from plant- and wood-based fibers have diverse applications in a range of products, including aerospace, automotive, and building materials (Panthapulakkal *et al*. 2006). Several types of natural fibers, such as wood fiber, sisal, jute, flax, abaca, banana, oil palm, pineapple leaf fiber, and bamboo, have been studied as reinforcements for making both thermoplastic and thermoset composites (Alemdar and Sain 2008), with wood being the most extensively and frequently used natural fiber reinforcement for polymers.

Although wood is a renewable source of fiber, it has a longer growing cycle compared to that of other sources of fiber such as agricultural residues, which can be

harvested annually (Hornsby *et al*. 1997a; Panthapulakkal *et al*. 2006). Agro-residues such as wheat straw, rice straw, corn-cobs, and corn stalks are currently being used as sources of cellulose-based fibers (Schirp *et al*. 2006; Reddy and Yang 2007). The abundant availability of these inexpensive agricultural residues makes their use especially suitable for the production of high-value fiber composites.

Wheat straw is one such agro-residue that has been used in polymer composites (Hornsby *et al*. 1997b; Panthapulakkal *et al*. 2006). The annual worldwide wheat straw production is estimated to be approximately 540 million tons (Lal 2005), while in China alone, approximately 126.6 million tons of wheat straw are produced and 39.6 million tons remain on fields unused, representing a large amount of biomass available for potential use (Jiang *et al*. 2012). Despite the abundance of agro-residues, their use as reinforcing agents in polymer composites is hindered by challenges from characteristics like high water absorption and impurities, causing poor particle dispersion and poor fiber loading, which restricts the use of agro-residues in the synthesis of natural fiber/polymer composites (Marcovich *et al*. 1998). Hence, improvement of these limitations associated with the use of agricultural residue fibers helps in assuring their application in composites with enhanced quality. In the present study, the effect of the modification of wheat straw fiber on the quality of wheat straw powder-filled polyethylene composites is analyzed.

## EXPERIMENTAL

### Materials

Wheat straw (WS) was collected in May 2014 from the Jiangsu Province of China. High-density polyethylene (HDPE, 7042), used as the polymer matrix, was obtained from the Sinopec Group, China. Caprolactam (CPL), which was of analytical grade, was supplied in granule form by Nanjing Biofunction Technology Co., Ltd., China. Calcium carbonate, of 400- to 600-mesh particle size, was obtained from Nanjing Jufeng Advanced Materials Co., Limited, Jiangsu, China.

### Wheat Straw Fiber Preparation and Modification

The wheat straw was dried to a moisture content of below 13% prior to grinding with a TQ-1000Y milling machine (Xulang Machinery Corporation, China). The straw fibers were fractionated by a vibration sieving instrument (Retsch-AS300, Germany) into particles with sizes in the ranges of < 0.25 mm, 0.25 mm to 0.5 mm, 0.5 mm to 1.0 mm, and > 1.0 mm. For the chemical component determination, the WS fibers of the 0.25 mm to 0.5 mm fraction were used, as measured according to the Van Soest (VST) method (Van Soest 1973). The WS fibers were dried overnight in an oven at 80 °C to a moisture content of below 5% before their use as a filler material. The oven-dried straw powder was then stir-compounded with various concentrations of CPL (0, 1, 3, 5, and 7 wt.% CPL), and 15 wt.% of calcium carbonate at 1500 rpm using a SHR-50A high-speed mixer (Grand Machinery Corporation, China), preheated to 110 to 130 °C for 30 min.

### Characterization of Straw Particles

The raw and modified wheat straw particles were microscopically characterized using an optical microscope (Olympus, Japan). The water content of the fibers was determined by a MA35M moisture analyzer (Sartorius, Germany). The bulk densities of both types of particles were measured according to reported methods (Liu and Yu 2002),

and the specific surface areas and average pore diameters were determined by use of an automatic analyzer (Beishide Instrument Corporation, China). Fourier transform infrared (FTIR) spectra in the range of 1200 to 4000 $cm^{-1}$ were recorded to study the polarity of the straw fibers after modification using a Perkin Elmer 100 Series FTIR 1650 spectrometer (USA). The spectra were obtained with an accumulation of 16 scans and with a resolution of 4 $cm^{-1}$.

### Processing of Composites

Varying proportions (30, 40, 50, and 60 wt.%) of modified wheat straw and unmodified wheat straw fibers were mixed thoroughly with HDPE granules and melt blended into composites using a two-spindled mechanical mixer, at a temperature of 195 °C for 10 min. The total weight of the mixture was 160 g. After mixing, the specimens were compressed by pressing into a SLB-25-D350 Carver hydraulic hot press (Suyan Science and Technology Corporation, China) at 190 °C for 20 min under a loading of 5 MPa; this pressed them into a steel mold of specific dimensions of 250 mm × 50 mm × 4 mm. The molded composites were then stored at room temperature conditions for 24 h before using them for mechanical and physical tests.

### Testing of Mechanical Properties

The flexural and tensile properties were measured on a UTM1422 standard electronic universal testing machine (Jinjian Testing Instrument Corporation, China). Flexural properties were measured according to ASTM D790 (2010), using the machine in the three point bending mode at a crosshead speed of 12.5 mm/min. The tensile properties were measured in accordance with the ASTM D638 (2010) procedure using the same testing machine mentioned above and at the same crosshead speed. A series of Izod impact tests followed the ASTM D256 (2010) procedure using a XJUD-5.5 pendulum type impact machine (Jinjian Testing Instrument Corporation, China). All the results were taken as the average value of five different specimens. The tests of all the mechanical properties were performed at room temperature.

### Scanning Electron Microscopy (SEM)

The surfaces of the fractured specimens under the tensile and impact tests were examined using a TM-3000 scanning electron microscope (Hitachi, Japan). All the tested specimens were sputtered, using a sputter coater (Leica, Germany), with a 10-nm-thick layer of gold prior to the SEM observations.

## RESULTS AND DISCUSSION

### Wheat Straw Analysis

The wheat straw particle size fractionation showed that particles 0.25 to 0.5 mm in size accounted for 68.3% of the particles, while particles less than 0.25 mm and greater than 0.5 mm in size accounted for 25.2% and 6.5%, respectively, as can be seen in Fig. 1. The wheat straw powder with a particle size of 0.25 to 0.5 mm was used as the basic reinforcing material of choice, as it was easy to process and can be obtained in large quantities, being the most abundant fraction.

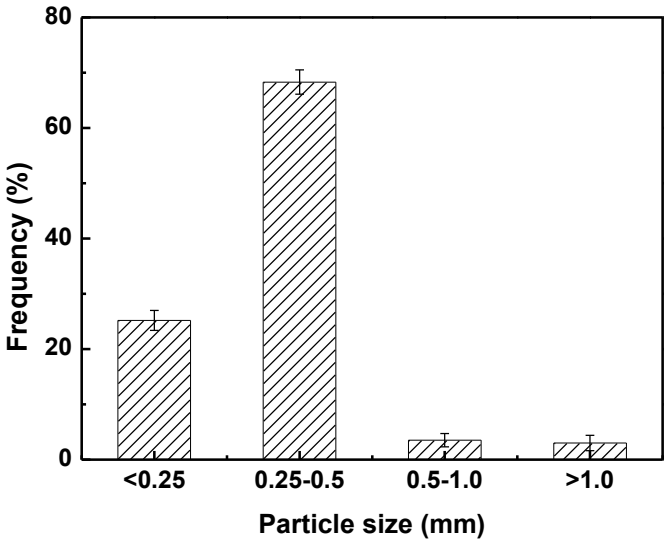


**Fig. 1.** The particle size distribution of ground wheat straw fibers

The main chemical components of plant fiber include cellulose, hemicellulose, lignin, and other extractives. These natural polymeric substances are interconnected and co-exist within the plant cell wall. Cellulose can improve the mechanical properties of the composites, while hemicellulose can have negative effects on the composite properties, and lignin can reduce the water absorption in composite materials to improve their thermal stability (Liu *et al*. 2014). The chemical analysis of the wheat straw powder revealed that cellulose is the dominant chemical component, accounting for 40.69% of the mass, followed by hemicellulose, lignin, and ash, accounting for 28.16%, 23.04%, and 3.53%, respectively, as shown in Table 1. The experimental and analytical methods were based on the Van Soest (VST) method (Van Soest 1973). The total of 63.73% cellulose and lignin content in wheat straw is higher than that of rice straw (Sun *et al*. 2000), and nearly equal to that of rape straw (Aicher *et al*. 2013). The cellulose and lignin can help improve the properties of the composites (Liu *et al*. 2014). Therefore, based on these observations, it is reasonable to use wheat straw as a natural material for preparing straw-plastic composites.

**Table 1.** Chemical and Physical Analysis of Wheat Straw Fibers

| Component | % of component in fiber (average) |
|---|---|
| Cellulose | 40.69 ± 10.45* |
| Hemicellulose | 28.16 ± 8.92 |
| Lignin | 23.04 ± 6.78 |
| Ash | 3.53 ± 1.41 |
| *Standard deviation | |

## The Effect of Fiber Modification

In the straw fibers, the cellulose is the skeleton, consisting of fibroblasts and thin-walled cells, with the surface containing a large amount of hydroxyl (-OH) groups, which are polar and hydrophilic in nature (Shukla *et al*. 1993). Many thermoplastic polymers are non-polar and hydrophobic; therefore, there are many difficulties in their complex

processing because of interfacial energy differences between the polar fibers and the non-polar polymer matrix. These differences can affect the dispersion and compatibility of the fibers and the matrix, ultimately being the main factor leading to a decrease in the physical and mechanical properties of the composites (Anandjiwala and John 2008). In addition, the hydroxyl groups within a cellulose molecular chain readily form hydrogen bonds with water molecules. Thus, the direct blending of plant fibers increases the water-absorbing tendency of the resulting composites (Sabo *et al*. 2013). The surface modification of fiber fillers with suitable modifiers was found to enhance the physical and mechanical properties of the composites by improving the compatibility between the two interfaces.

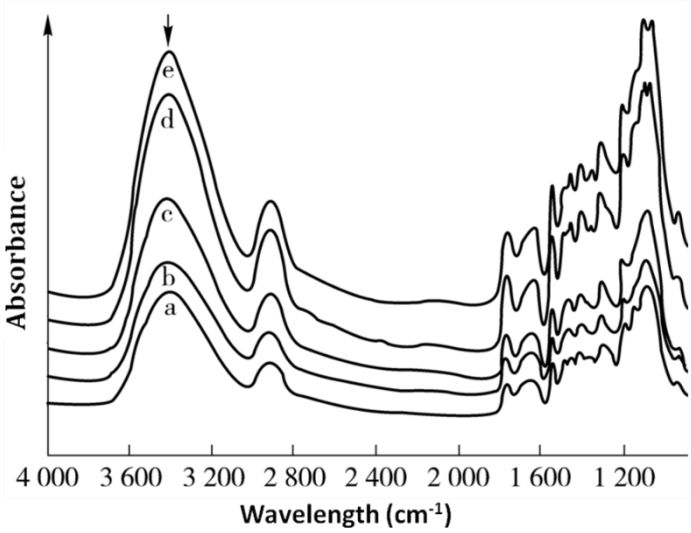


**Fig. 2.** FTIR spectra of modified wheat straw (MWS) fiber with various amounts of caprolactam (CPL) added: (a) WS+ 5% CPL; (b) WS+ 7% CPL; (c) WS+ 3% CPL; (d) WS+ 1% CPL; and (e) WS+ 0% CPL

The FT-IR spectra of wheat straw (WS) treated with various concentrations of CPL (0, 1, 3, 5, and 7 wt.%) are illustrated in Fig. 2. It can be seen in Fig. 2(e) that the FT-IR spectrum of WS is characterized by the absorption peak at 3400 $cm^{-1}$, which can be attributed to the stretching vibrations of O–H groups (the hydroxyl intensity). The C–H stretching vibration appears at 2940 $cm^{-1}$ (Faravelli *et al*. 2000). The absorption peak at 1735 $cm^{-1}$ in the WS is due to the aliphatic esters in lignin or hemicelluloses; The absorption band at 1646 $cm^{-1}$ is assigned to olefinic C=C vibrations (Qin *et al*. 2011). The weak absorption bands in the range 1400 to 1320 $cm^{-1}$ are probably due to stretching vibrations of phenolic O-H groups (Ismail *et al*. 2011). The region around 1100 $cm^{-1}$ illustrates C–O stretching and deformation bands from cellulose or lignin or hemicellulose (Dawy and Nada 2003).

The FT-IR spectra of the modified wheat straw (MWS) fibers show that the intensity of the peaks decreased with the percentage of CPL increasing from 1 to 7 wt.%. This band has a lower intensity compared to its intensity in the FT-IR spectra of WS. When the amount of CPL was 5%, the intensity of the hydroxyl (O–H) and carbonyl (C–O) absorption peaks decreased the most noticeably in this study. In addition, WS has no chemical interactions with the CPL, so there may be a physical interaction between the WS and CPL, since no new bands or any considerable shifts in bands occurred that may be indicative of the formation of new compounds. These results suggest that the CPL can

interact with the WS via the hydrogen bonding between the O–H groups available in the WS and as a "coat" that wraps around the apparent polar groups of the WS, so that the polarity of the WS is mitigated. The decreased amount of free surface hydroxyl groups after modification, which weakens the hydrogen bonds and lowers the polarity of the fibers, contributes to the good compatibility between the WS and the polymer matrix. When the content of the CPL was 7%, the peak intensities of the hydroxyl and carbonyl groups rebounded in the modified system, which may be due to the presence of excess CPL.

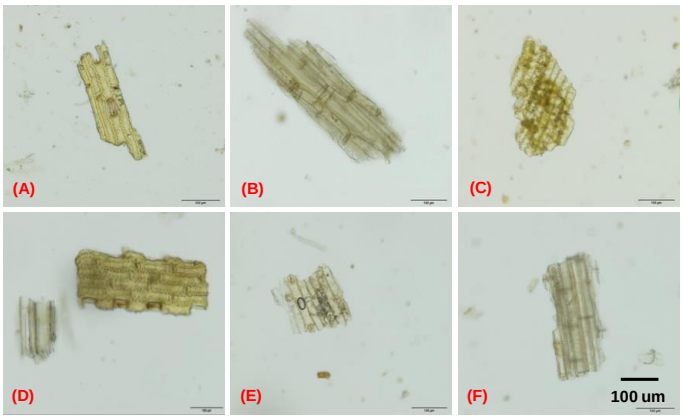


**Fig. 3.** Micrographs of unmodified wheat straw (0.25 to 0.5 mm) fibers: (A-F)

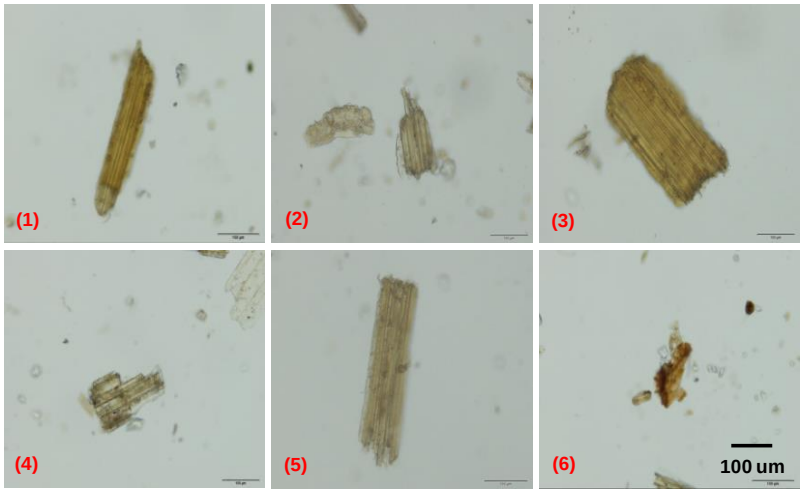


**Fig. 4.** Micrographs of modified wheat straw (0.25 to 0.5 mm) fibers: (1-6)

The microscopy observations performed using an optical microscope at 20x show different results for the WS (0.25 to 0.5 mm) fibers before and after modification. Figure 3 shows that the unmodified WS fibers consist of clearly arranged duct cells, the shapes of which are diverse and their color yellow or brown, with most of them having some surface roughness.

Figure 4 shows that the modified WS fibers, after being treated with 5% CPL at a temperature of 120 °C for 30 min, appear fuzzy near the fiber boundaries. The filler in the fiber particles can readily be seen, as smooth, with a dull color. These phenomena may be the result of the modification of the surface structure of the straw, with the embedding effect of the added CPL.

**Table 2.** Physical Parameters of Wheat Straw Before and After Modification

| Parameter | Before | After |
|---|---|---|
| Bulk density (g/cm$^3$) | 0.37 ± 0.004* | 0.93 ± 0.078 |
| Water content (%) | 10.24 ± 0.13 | 1.36 ± 0.115 |
| Moisture absorption (%) | 9.75 ± 0.31 | 3.74 ± 0.125 |
| Specific surface area (m$^2$/g) | 1.19 ± 0.137 | 0.96 ± 0.081 |
| Average pore diameter (nm) | 19.47 ± 0.836 | 32.78 ± 1.297 |
| *Standard deviation | | |

Table 2 shows the results of the physical characteristics of the unmodified WS fibers and MWS, after being modified with 5% CPL at 120 °C. Prior to modification, the bulk densities of the WS (0.37 g/cm$^3$) and HDPE (with a bulk density of approximately 0.9 g/cm$^3$) were quite unequal. However, the bulk density of the modified WS was 0.93 g/cm$^3$, closer to that of the plastic, which facilitates the dispersion of straw fibers in the plastic, so that subsequent compounded composites are more uniform in the granulation process. In addition, the water content and the moisture absorption rate of the modified WS were much lower than those of the unmodified WS, which helps enhance the water resistance of the straw-plastic composites and their products. In addition, the specific surface area of the modified WS became smaller than that of the unmodified WS, which is likely due to the filling of the internal pores of the straw fibers, thus reducing their number. After modification, the surface pore diameters of the WS increased, making it easier for the fibers to intersperse and adhere to the plastic, forming a complex cross-linked structure.

## Mechanical Properties

Plant fibers, because of their unique structure and composition, in general do not have good compatibility with the HDPE polymer matrix, whose surface is hydrophobic. The modification of the WS reduced the polarity of the straw fiber surface and improved the interfacial strength between the polymer matrix and the WS. The mechanical properties of the composites can be increased by improving the compatibility of the fiber-polymer interface (Zafeiropoulos *et al*. 2007). When the wheat straw fibers were modified with 5% CPL, there were different effects on the corresponding flexural strength, tensile strength, and impact strength of the composites with different amounts of modified WS, compared to the mechanical properties of the composites with unmodified WS.

Effects of the modified and unmodified wheat straw fiber loading on the flexural properties of the composites are shown in Fig. 5. It can be seen that the flexural strength of MWS/HDPE increased at first and then decreased when the amount of MWS increased from 30% to 60%, while the flexural strength of unmodified wheat straw/HDPE composites decreased linearly with increasing WS load. When the fiber loading was 40%, the flexural strength of MWS/HDPE reached a maximum of 45.31 MPa, while the flexural strength of the unmodified wheat straw/HDPE composite was only 16.76 MPa. Similar results have been reported by others (Mengeloglu and Karakus 2008b; Mengeloglu and Karakus 2012). Overall, the flexural strength of MWS/HDPE was higher than that of the unmodified wheat straw/HDPE composites. This is because MWS as the reinforcement material was compounded with polyethylene, while unmodified wheat straw was not very compatible with the polymer. When MWS reaches an appropriate proportion, the straw fiber and polymer have good bonding, which improves their mutual affinity and compatibility and forms stable blends, thereby resulting in good mechanical properties for the composites. When the MWS was added in excess of 40%, the flexural strength of the

composites decreased gradually. These observations might be due to the presence of excessive MWS, such that the particles tend to agglomerate into clusters, resulting in an uneven distribution within the polymer matrix. This is likely to cause stress concentration when an external force is applied, so that the composites might undergo fracture.

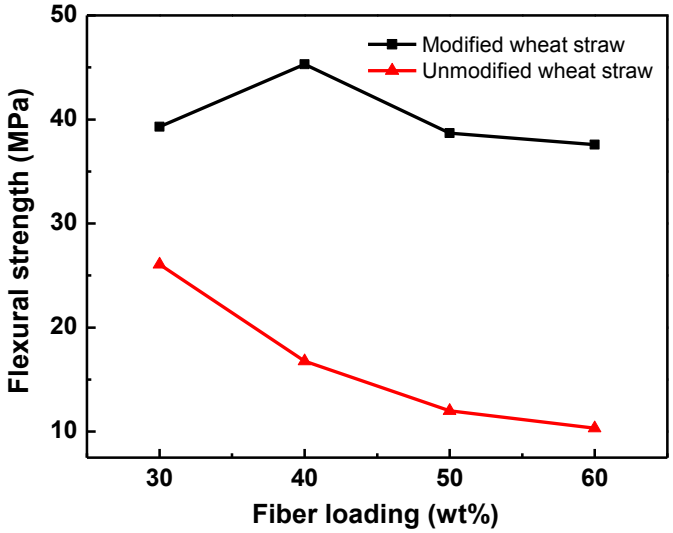


**Fig. 5.** Effect of the modified and unmodified wheat straw fiber loading on the flexural properties of the composites

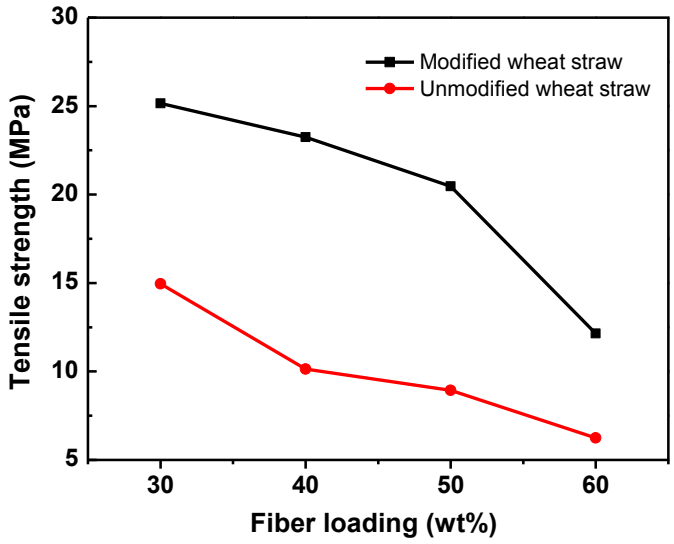


**Fig. 6.** Effect of the modified and unmodified wheat straw fiber loading on the tensile properties of the composites

The influence of fiber content on the tensile strength of MWS/HDPE and unmodified wheat straw/HDPE composites is shown in Fig. 6. As can be seen, with the increase of the fiber content from 30% to 60%, the tensile strength gradually decreased. The highest tensile strength values for MWS/HDPE and unmodified wheat straw/HDPE composites, which were reached at 30% fiber loading, were 25.16 MPa and 14.95 MPa,

respectively. The decrease in tensile strength with increasing amount of filler may be a result of the weak adhesion or interaction between fibers and HDPE that occurs due to agglomeration (Khandanlou *et al*. 2014). It was observed that the tensile strengths of the MWS/HDPE composites were remarkably higher than those of the unmodified wheat straw/HDPE composites. This might be due to reduced polarity of the MWS as a natural filler. It is well established that MWS enhances the interfacial adhesion between the wheat straw and the non-polar plastic matrix and ensures better encapsulation of the wheat straw particles by the plastic, which consequently results in better tensile properties.

Figure 7 presents the results of the notched Izod impact strength tests on the MWS/HDPE and unmodified wheat straw/HDPE composites. The experimental results indicate that the impact properties of MWS/HDPE were better than those of the unmodified composites. The impact strength values of the two composite types decreased with increasing fiber percentage in the mix composition. The maximum values recorded for the impact strength of MWS/HDPE and unmodified wheat straw/HDPE composites were 3.47 kJ/m$^2$ and 2.66 kJ/m$^2$, respectively. This difference was expected because the presence of fibers in the HDPE matrix provides points of stress concentrations, thus providing sites for crack initiation and potential composite failure (Farsi 2012). The MWS had an improving effect in the interfacial region due to bonding between wheat straw fibers and matrix. This was facilitated by the surface smoothness and regular cross section of these fibers. Furthermore, the additional fiber loading helps to form cellulosic fiber agglomeration that creates regions of stress concentrations that require less energy to elongate the crack propagation.

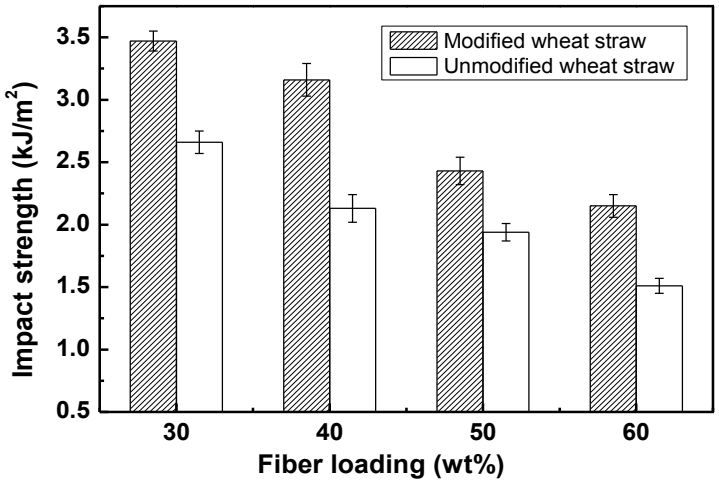


**Fig. 7.** Effect of the modified and unmodified wheat straw fiber loading on the impact strength of the composites

## SEM Analysis

Examination of the fracture surfaces by a scanning electron microscope at 400× gave information about the morphology of the straw-plastic composites containing 40% of the modified WS, as shown in Fig. 8. This SEM image shows that the fibers were well oriented in the polymeric matrix and are coated with polyethylene. The particles had an even distribution in the matrix and good adhesion at the fiber-polymer interface. These results are consistent with the mechanical properties of the composites.

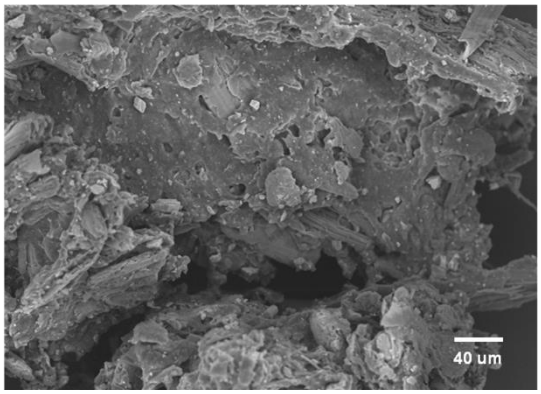


**Fig. 8.** SEM micrograph of a fracture surface of the composites at 400x

Based on the above results, a structural model of the straw-plastic composites can be proposed, as seen in Fig. 9. The modified straw fibers (1) as the reinforcement are filled and interspersed within the polymer (2), similar to a "cement and steel" structure, which strengthens the toughness of the composites. The additive (3), (CPL) can be seen modifying the surfaces of the fibers, permitting a closer interaction with the polymer. This model reflects the performance advantages of the subsequent products made with this composite.

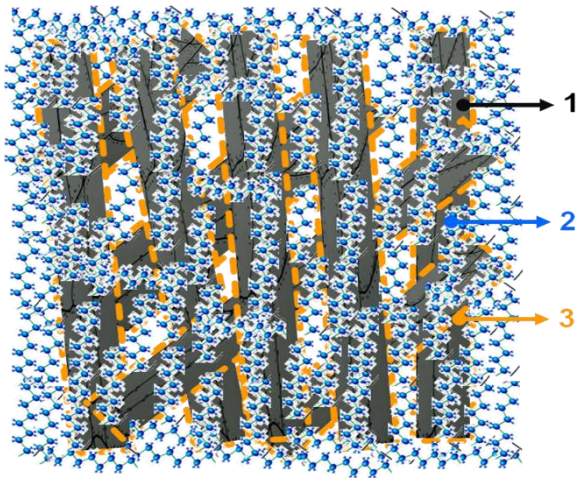


**Fig. 9.** A schematic illustration of the structural model of the straw-plastic composites; 1: modified straw fibers; 2: polymer; and 3: additive

## CONCLUSIONS

1. Wheat straw fibers have good lignocellulose content, and particle sizes in the range of 0.25 mm to 0.5 mm were modified with 5% caprolactam (CPL). A Fourier transform infrared spectrum (FTIR) of the modified wheat straw showed a decrease in hydroxyl groups and corresponding polarity in the fibers.
2. A physical analysis of the wheat straw fibers indicated that after modification, the characteristics of the fibers were closer to those of the HDPE polymer matrix, contributing to good compatibility and dispersion of the straw fibers within the matrix.

3. With respect to the mechanical properties, MWS/HDPE was superior to unmodified wheat straw/HDPE composites, because of the increased compatibility of the MWS with the HDPE. The SEM micrographs indicated good dispersion of the MWS into the matrix.
4. The flexural strength tended to decrease as the filler load of the modified wheat straw increased from 30% to 60%, with the maximum value (45.31 MPa) recorded at a 40% MWS load. The tensile strength and impact strength decreased linearly with the increasing MWS load. The maximum values recorded for the tensile strength and the impact strength were 25.16 MPa and 3.47 kJ/m$^2$, respectively. Applications of these composites will be illustrated in a future publication.

## ACKNOWLEDGEMENTS

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