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The influences of filler loading on tensile properties and water absorption properties of carbonized sugarcane bagasse filled high density polyethylene (HDPE) composite

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Abstract. This study synthesized carbonized sugarcane bagasse (CSB) filled high density polyethylene (HDPE) composites. The effect of pre-treatment reagent of SB with different concentration of sodium hydroxide (NaOH) was examined. The untreated, treated and carbonized SB were characterized by proximate analysis and functional group analysis. By considering all the characteristics, 7 wt% is the optimum concentration which gave lowest moisture content and highest fixed carbon (FC) content. It was chosen to undergo carbonization process at temperature of 600 ˚C to produce CSB and used as filler in composites. The filler with different loading was mixed with HDPE via extrusion process. The tensile properties, surface morphology and water absorption behaviour of the bio-composites were evaluated. As filler loading increased, the Young's modulus was increasing gradually. Meanwhile, the tensile strength and elongation at break were increasing up to 5 wt% but then decreasing as filler content increased due to the formation of filler agglomeration in HDPE matrix. The deterioration of the interfacial adhesion of these composites was confirmed by the SEM observations. The percentage of water absorption is gradually increased with increasing the filler loading. In short, the optimum filler loading at 5 wt% imparts good tensile and water absorption properties of composites.

1. Introduction

In recent years, most of the preceding effort on lignocellulosic fibres in thermoplastics has focused on the wood-based flour or fibres. Many investigators have completed important improvements which proved natural fibres is an attractive substitute as support of polymer patterns. The major benefits of such fibres are their performance of mechanical that really good, renewability, biodegradability, requirements in cost and comparatively little machine scratch compared to another fibre such as glass fibres [1]. It is also had the benefits of little cost, low density and are biodegradable.

There are roughly downsides when will be used in composites. For instance, lowly compatibility through dissimilar patterns, high in absorption of moisture and swelling possessions that allow creation of crashes in breakable matrices [2]. The hydrophilic fibres tend to agglomerate and not disperse well in the polymer matrix is the main issue for natural [3].

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Plenteous research institutes acknowledged the challenge of developing and improving the existing composites [4]. Most of the research are focusing on the downsides of natural fibres reinforced mixtures which is the dissimilarity between polymer matrix and natural fibres because of hydrophilic nature of natural fibre and inherent polar and non-polar properties of the most thermoplastics [5]. Thus, pre-treatments are being considered for modifying the fibre apparent characteristics which expected to improve the bond between the surface of fibre and polymer matrix [6]. Alkaline pre-treatment has widely been studied among all the pre-treatment methods [7]. It can modify the surface connections of fibre/matrix by forming strong chemical bonding and thus the mechanical properties of the composites can be enhanced [2].

Many studies have been carried out on carbon black (CB) from biomass as filler in composite such as bamboo, oil palm shell [8], rice husk [9], coconut shell [10] and wood apple shell [11]. Carbonization process of sugarcane bagasse can remove volatile matter and the moisture content [12]. In fact, the potential of high carbon content of carbonized sugarcane bagasse can be used as a filler material in the polymer composite for innovative composites with better dimensional stability, best stiffness and chemical resistance are needed [13].

The objective of this paper was to evaluate the effect of chemical and carbonization modification on
mechanical properties of carbonized sugarcane bagasse filled composites. The modification the fibres were evaluated by proximate analysis and Fourier Transformed Infrared (FTIR). These carbonized sugarcane bagasse (CBS) which acts as filler was mixed with HDPE and subsequently the tensile properties and water absorption properties of carbonized bagasse filled HDPE composites were evaluated to determine the optimum filler loading.

2. Experimental

2.1 Materials

Sugarcane bagasse was collected from a sugarcane juice seller after being crushed to extract the juice in area Padang Besar, Perlis, Malaysia. It was washed, dried in oven at 80 ℃, 48 hours and ground into fine size (1-125 µm).

The ground SB was pre-treated with NaOH at different concentration (1, 3, 5 and 7 wt%) for 2 hours, 60 ℃. The quantity of every concentration of alkaline solution used for soaking followed a ratio of 15 ml of alkaline solution to 1 g of sugarcane bagasse fibres. It was neutralized with acetic acid until the pH level reached 7 as indicated by pH meter and rinsed with distilled water to cleanse them from NaOH excess on the surface then followed by drying up in an oven at temperature of 80 ℃, 24 hours. The optimum alkali pre-treatment (7 wt%) of SB was chosen for carbonization process. The treated SB was carbonized in muffle furnace at carbonization temperature 600 ℃ at heating rate of 15 ℃ min^{-1} and was held around one hour. After this period, carbonized sugarcane bagasse was cooled down to room temperature.

2.2 Characterization of filler

2.2.1 Fourier Transform Infrared Spectroscopy (FTIR). The structure of chemical of untreated, treated, carbonized SB was evaluated by FTIR spectroscopy (Perkin Elmer). The samples were prepared by mixing the respecting material and potassium bromide (KBr) in 0.5 mg to produce a pellet. The spectra were recorded in the range between 400 – 4000 cm^{-1} wave numbers.

2.2.2 Proximate analysis. Moisture, volatile matter and ash contents were determined experimentally in proximate analysis and then the fixed carbon content was calculated by subtraction the sum of moisture, volatile matter, and ash contents from 100.

2.3 Preparation of Composites.
The carbonized sugarcane bagasse was mixed and extruded using twin screw extruder at different filler loading (5, 10, 15, 20 wt%) at temperature of 180 °C. The specimens were moulded using an automatic hot press machine. The pellets were pre-heated, pressed and cooled at 180 °C for 3 minutes, 5 minutes and 5 minutes respectively. The moulded sheets were then cut into dumb-bell shape by using level cutter press machine and ready to be tested.

2.4 Measurement of properties of composites

2.4.1 Tensile properties. The tensile properties measurement experiments were performed using universal tensile testing machine. In accordance with ASTM D638-02 type IV, the load cell and crosshead speed for all composites are 50 kN and 20 mm/min respectively. Five samples were tested for each type of composite.

2.4.2 Morphological properties. Scanning electron microscopy (SEM) were used to observe the tensile fractures of the samples at an accelerating voltage of 15 kV and to study the surface morphology of the carbonized sugarcane bagasse filled HDPE composites samples. The specimens were air-dried and coated with 10-nm-thick platinum sputtering for clear visibility of the surface morphology.

2.4.3 Water absorption properties. The water uptake measurement was performed according to D5790-98 in a distilled water bath for 24 hours at 23 °C shall rest on edge and entirely immersed to measure percent water absorption of the fine size of carbonized sugarcane bagasse filled HDPE composites. The specimens were removed from the water one at a time, all surface water was wiped off with a dry cloth, and weighed to the nearest 0.001 g immediately. The weight change of the specimens was intermittently measured during the test period. The percent water absorption was calculated.

3. Results and discussion

3.1 Characteristics of Sugarcane Bagasse

3.1.1 Functional groups of filler. The functional groups present in the natural fibre can be determined by Fourier Transform Infrared Spectroscopy (FTIR). The spectra of untreated, treated and carbonized sugarcane bagasse are tabulated in Table 1.

Table 1. Functional group of untreated, treated and carbonized sugarcane bagasse.

Wavenumber (cm^{-1})	Functional group	Bond
3470	Hydroxyl	O-H
2899	Hydrocarbon	C-H
1650	Carboxylic Acid	C=C
1430	Carboxylic Acid	C=C
1250	Ether	=C-O
1050	Hydroxyl	C-O

The broad band ranging from 3200–3600 cm^{-1} was assigned to the O-H stretching vibration correspond to hydroxyl groups. Table 1 simplified main functional groups present on untreated, treated and carbonized sugarcane bagasse surface which were hydroxyl, ether, hydrocarbon and carboxylic acid. The broad band ranging from 3200–3600 cm^{-1} was assigned to the O-H stretching vibration correspond to hydroxyl groups. The absorption bound at 1430 cm^{-1} was related to the presence of cellulose. As shown in figure 1, the band around 2899 cm^{-1} is assigned to C-H stretching (CH and CH_2) in cellulose and hemicellulose. It can be observed that it

decreases due to increasing concentration of alkaline treatment and removed due to decomposition for hemicellulose after carbonization process.

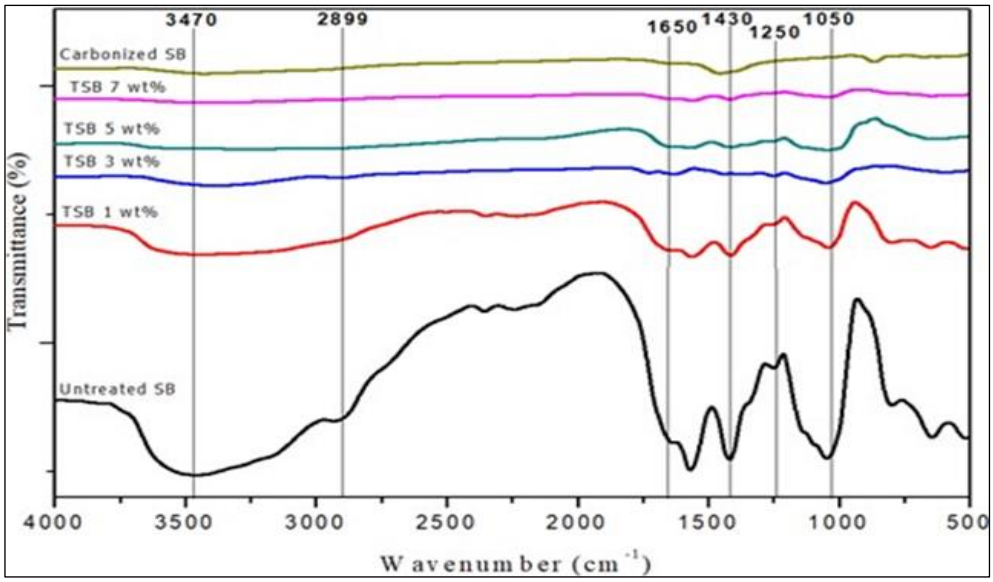

Figure 1. FTIR spectra of the untreated, treated and carbonized sugarcane bagasse.

3.1.2 Proximate analysis of filler.

From overall observation as shown in table 2, the highest fixed carbon (FC) was selected to be filler in order to allow better adhesion between the fibre and polymer matrix [11] at the same time enhance the mechanical properties of the composites. Increasing concentration of NaOH tends to reduce the content of moisture of SB which allowed the reducing of hydrophilic nature of SB which is the polar hydroxyl (OH) group in lignin [14]. A similar trend was observed by [15] in which SB have a low percentage of ash content which is in the range of less than 2 %. This in par with the result showed the lowest moisture content and volatile matter were attributed by CSB. Also, the highest ash content is 7.37 % which result the highest FC, 54.05 %. This have the same trend with finding by [16] as the range of FC of the bagasse is between 11.9 to 58.6 %. To conclude, CSB is suitable to be filler of the polymer matrix to form composite which in par with many studies that have been carried out on carbon black from biomass used as filler in composite.

Table 2. Proximate analysis of sugarcane bagasse.

Material	Moisture Content (M %)	Ash Content (A %)	Volatile Matter (VM %)	Fixed Carbon (FC %)
USB	8.16	1.23	75.11	15.50
TSB-1 wt%	7.51	1.91	65.72	24.86
TSB-3 wt%	7.41	2.22	59.48	30.88
TSB-5 wt%	6.47	2.77	56.55	34.21
TSB-7 wt%	5.98	3.68	54.03	36.31
CSB	1.60	7.37	36.99	54.05

3.2 Properties of CSB filled HDPE composites

3.2.1 Tensile properties of composites. The effects of filler loading on the tensile properties of CSB filled HDPE composites were evaluated as shown in figure 2. The tensile strength and Young's modulus were improved after adding the filler which due to a better interfacial bonding between the fibre and polymer matrix [17]. The composite reinforced with CSB had a higher tensile strength compared to the neat HDPE matrix composite, which had the lowest tensile strength. However, increasing the filler loading causes the decreasing of tensile strength of CSB filled HDPE composites. The decrease of the tensile strength at 10, 15 and 20 wt% may be due to the microstructural defects or imperfections existing between the fibre and the matrix, resulting from the insufficient encapsulation of the HDPE matrix surrounding individual CSB fibres.

The Young's modulus increases against the increasing percentage of fibre content. This comes from the high Young's modulus of the CSB, and naturally, the more CSB content, the higher the stiffness of the composite. This might due to the better distribution of CSB with the HDPE matrix, contributed by the alkaline treatment and carbonization process.

Meanwhile, the elongation at break decreases when the filler loading of CSB increases. when the fibres were inserted in the matrix, the rigidity was strongly increased. However, the addition of fibres caused failure between fibre and matrix and this failure depending on the stress transference, could cause a beforehand rupture, and thereby the elongation at break was decreased.

Thus, 5 wt% is the optimum filler loading which resulted the highest tensile strength of the composites by 8.3 MPa. The elongation at break was found to be decreasing significantly for the filler loading of 10, 15 and 20 wt%.

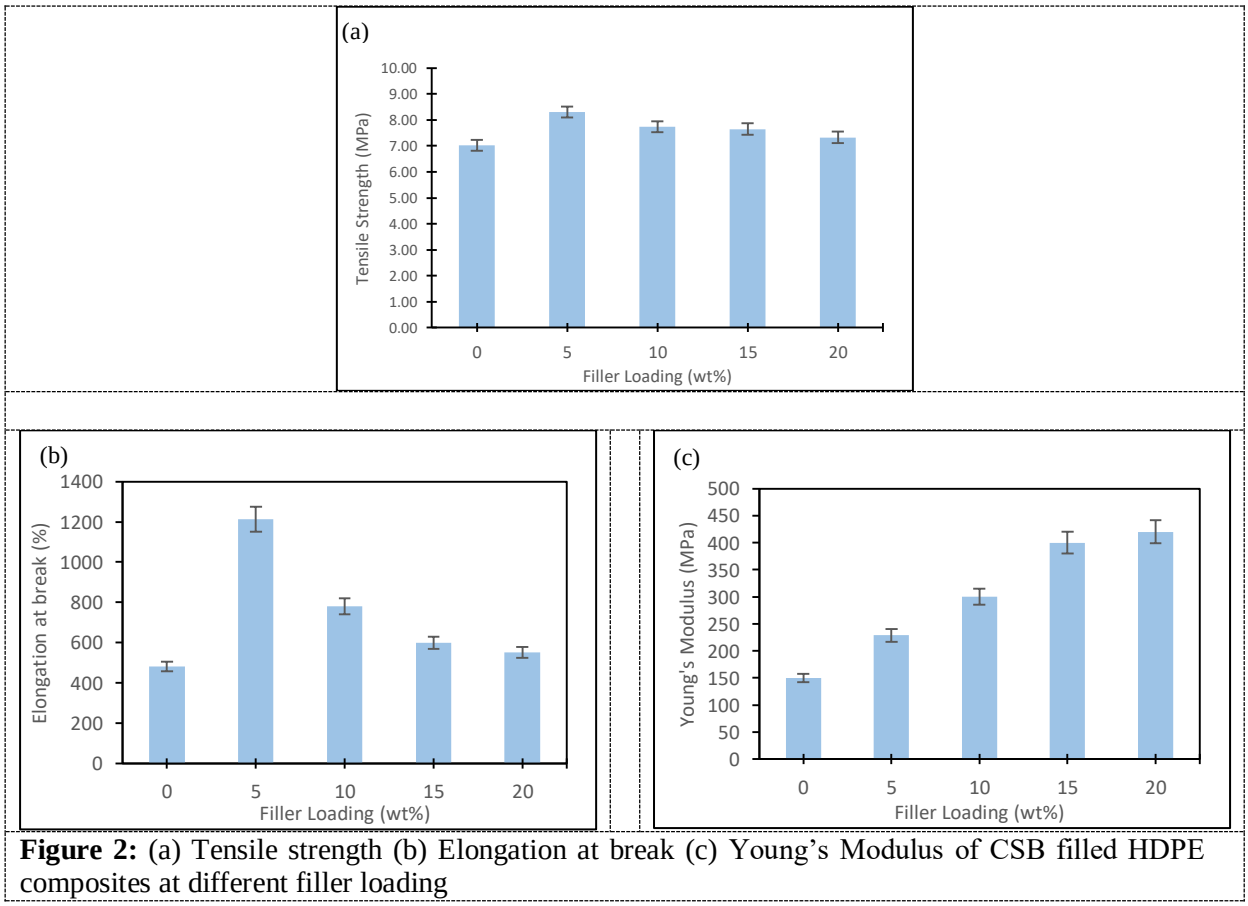

Figure 2: (a) Tensile strength (b) Elongation at break (c) Young's Modulus of CSB filled HDPE composites at different filler loading

3.2.2 Morphological study of composites. It can be observed in figure 3 (a) & (b) that the neat HDPE matrix has homogenous, smooth and uniform fractured surface with minimal tearing lines. Meanwhile figure 3(c) & (d) show the tensile fracture of composite with 5 wt% filler loading, the more fibres can be found which morphological change in the microstructure occurred and the narrower gaps between CSB fibres and HDPE matrix which indicated the better compatibility. This showed good interfacial adhesion as the CSB fibres adhere better to the surface of the HDPE matrix.

However, increasing the filler loading caused the reducing of tensile strength of composites. Figure 3 (e) & (f) presented the tensile fracture surfaces for 20 wt% filler loading. The observation indicated that fibres appeared to be free of any matrix interactions as well as voids were found in figure 3(e). Also, the fibres tend to agglomerate with polymer matrix as shown in figure 3(f). Increasing the filler loading causes the filler to accumulate at one place only and lead to the absence of physical contact between CSB fibres and HDPE matrix due to poor interfacial adhesion compatibility between hydrophilic cellulosic fibres and hydrophobic polyolefin [18].

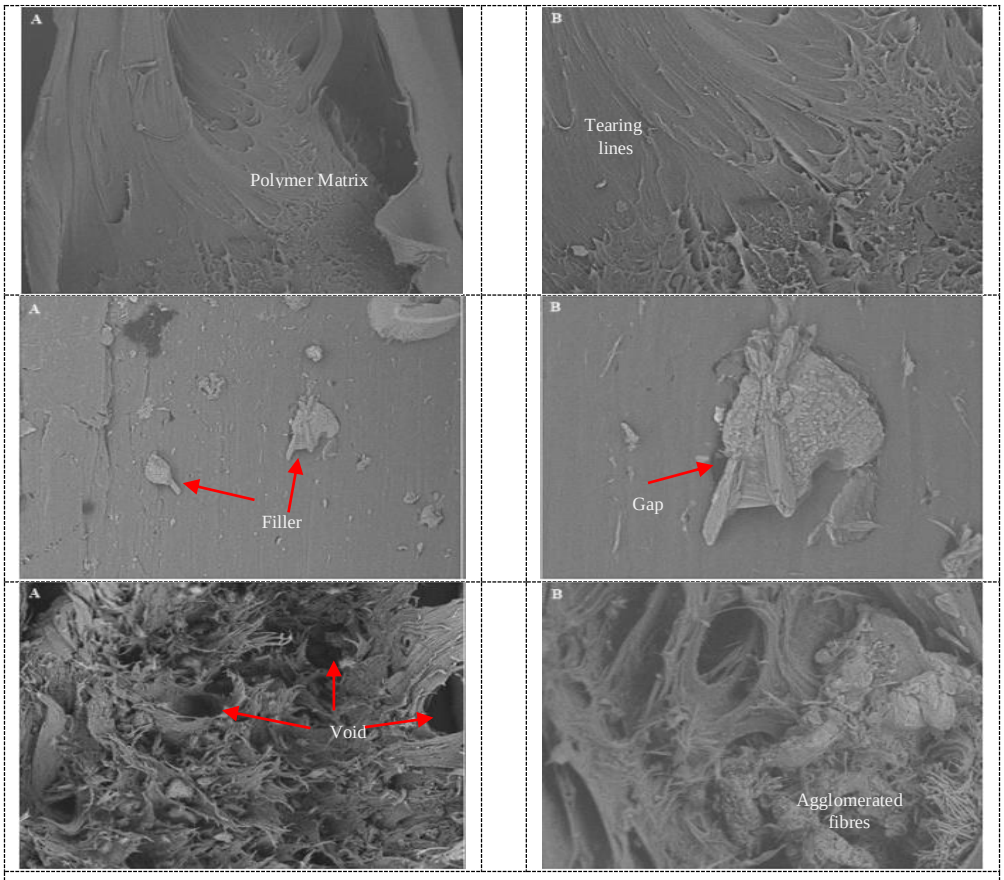

Figure 3. SEM images of tensile fractured surfaces of neat HDPE **(a)** at magnification 100x **(b)** at magnification 200x, 5 wt% of CSB filled HDPE composites **(c)** at magnification 300x **(d)** at magnification 1000x and 20 wt% of CSB filled HDPE composites **(e)** at magnification 100x **(f)** at magnification 200x

3.2.3 Water absorption properties of composites.

The percentage of water absorption increased with increasing amount of carbonized sugarcane bagasse powder at constant time of immersion. When the filler loading of CSB powder was 20 wt%, the water absorption of composites was the highest (0.31 %) as compared to other filler loadings which can be observed in figure 4. The water absorption of neat HDPE is less than 0.1 % which is 0.03 % and it is the lowest percentage out of all filler loading. This results can be attributed to the hydrophobicity of polymer matrix is diminished by the joining of filler and indicates that the CSB-HDPE composite is sensitive to moisture due to the hydrophilic character of natural fibre [19].

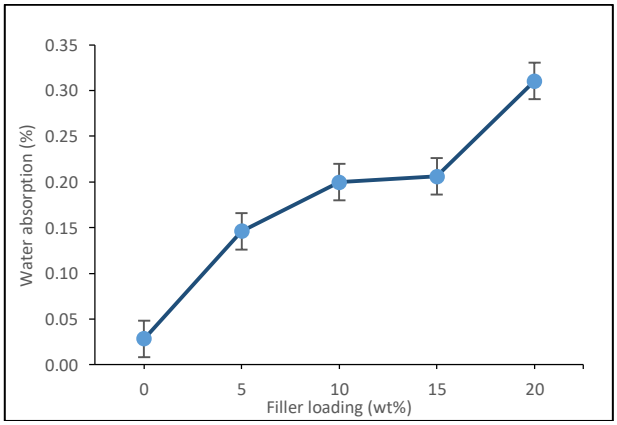

Figure 4. Water absorption of CSB filled HDPE composites at different filler loading.

4. Conclusion

The CSB filled HDPE composites were successfully synthesized. Chemical modification and carbonization of sugarcane bagasse fibres were performed to demonstrate the effect of modification on the mechanical properties of the composites and to study the attainability of processing these agro-residue with thermoplastics. By considering all the characteristics, 7 wt% is the optimum concentration to be a good filler as it gave lowest moisture content and highest fixed carbon (FC) content. The modification of fibres from sugarcane bagasse was successfully accomplished and it was verified that 5 wt% of CSB filled HDPE composites is the optimum filler loading which effectively improves the tensile strength and water absorption properties in comparison to the pure polymer.

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