Synthesis and Characterization of Carbon Fibers and their Application in Wood Composites

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Carbon fibers were synthesized using a low-cost, economical method. Fresh rubber wood fibers (*Hevea brasiliensis*) were burned using a furnace in an inert condition at 350 to 450 ^{o}C for 2-4 hours, and after that the fibers were ground at 18000 rpm for 20 to 40 seconds. The effect of carbon fibers as a reinforcement agent on mechanical, physical, and morphological properties was investigated. In the composite preparation, carbon fiber dosages (0, 0.1, 0.25, and 0.5 wt.%) were used as variable factors, along with a urea formaldehyde content of 10%. The morphology of the specimens was characterized using X-ray diffraction (XRD), Thermogravimetric analysis (TGA), and Field Emission Scanning Electron Microscopy (FESEM). The mechanical tests indicated that when carbon fibers were added, the modulus of rupture (MOR) and internal bonding strength (IB) improved significantly. From the TGA graph it was observed that the thermal stability of the composites based on carbon fiber was higher than composites without it. The thermocouple readings showed that at a higher loading of carbon, the core temperature of the board increased faster than for the control board.

Keywords: Urea-formaldehyde; MDF; Wood composite; Carbon fiber

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INTRODUCTION

Medium-density fiber board (MDF) is an engineered wood product composed of fine ligno-cellulosic fibers combined with a synthetic resin and subjected to heat and pressure to form panels (Irle and Barbu 2010). It is made up of wood fibers and can be used as a building material similar in application to plywood. It is stronger and much denser than normal particleboard.

The low thermal conductivity of wood fiber limits the production rates in existing production lines. The carbon fibers can be used as a filler to improve the thermal and mechanical properties of wood composite. Carbon fibers are widely employed in various fields because of their unique properties including adsorptive capacity, chemical stability, thermal conductivity, and electrical conductivity (Youssef *et al*. 2008; Yun *et al*. 2008; Kim *et al*. 2008; Roh *et al*. 2008).

Principles of composites demonstrate that for a given reinforcement and matrix, the properties of resultant composites are mainly dependent on interfacial adhesion, because a good interfacial adhesion guarantees effective transition of stress, and thus the reinforcement and matrix can take full action. However, a large number of studies have shown that the surfaces of carbon fibers exhibit inertness, and the interfacial adhesion

between carbon fibers and organic resin matrix is generally very weak (Meng *et al*. 2009; Xu *et al*. 2007; Lu *et al*. 2007). Hence, how to improve the interfacial adhesion between carbon fibers and resin matrix has been one of the most important topics of developing advanced composites.

Many theories or models have been proposed to explain or forecast the effect of surface modification of carbon fibers on the interfacial adhesion of resultant composites. To date, it is generally believed by many researchers that the chemical interaction between carbon fibers and polymeric matrix is necessary to improve the interfacial adhesion; however, some scholars have recently stated that good interfacial adhesion between carbon fibers and matrix can be also obtained by the formation of physical interaction. For example, Lu *et al*. (2007) employed air plasma to modify carbon fibers, and they concluded that mechanical interaction has a dominant effect on the interfacial adhesion of composites. The application of nano-based materials opens new aspects in the field of wood science, such as their use in solid wood, wood-based panels, *etc*. (Cai *et al*. 2007a, 2007b, 2008, and 2010). Mixing of carbon fibers has improved the thermal and mechanical properties of the wood composite boards.

This work was aimed at analyzing the influence of using carbon fibers as a reinforcing agent on the physico-mechanical properties of wood composites. Experiments were conducted to estimate the curing time at different weight concentrations of carbon fibers when mixed with resin and wood fibers. In the present work, the surface morphology and carbon fibers distribution in the composites have also been studied. The novelty of this work is the use of carbon fibers in wood composite for the first time.

MATERIALS AND METHODS

Materials

Wood Fibers

The lignocellulosic material used for this study was fresh rubber wood (*Hevea brasiliensis*) fibers obtained from Robin Resources Pvt. Ltd.

Urea formaldehyde

Urea-formaldehyde (UF) liquid resin used for this study was obtained from Dynea Malaysia Sdn. Bhd. The viscosity of the UF resin at 300 ^{o}C was 178 centipoises, pH 8.79, density 1.286 kg/m^3, and the gel time at 100 ^{o}C was 36 s.

Methods

Preparation of carbon fibers

The fresh rubber wood fibers (*Hevea brasiliensis*) were burned using a furnace in an inert condition at 350 to 450 ^{o}C for 2 to 4 h. After that the fibers were ground with a Retsch Chemical Grinder ZM 200 at 18000 rpm for 20 to 40 seconds. The purity of the carbon fibers was found to be 74.09% using an elemental analysis system (CHNS analyzer).

Mixing of carbon fiber with UF resin

A bench-top overhead mixer IKA®-WERKE, model RW 20 DZM, with a speed range of 72 to 2000 rpm was used for pre-mixing carbon fiber with the UF resin at 1800

rpm for 30 min. The weight fraction of carbon fiber and UF resin were based on the weight fraction of the oven dry wood fibers. After mixing the carbon fibers and UF resin in a bench-top mixer, the resin mixture samples were subjected to ultrasonic treatment with NANO-LAB Ultrasonic probe dispersion QS1 system for 1 h for uniform mixing of nanoparticles in UF resin. This procedure was used to prepare three samples with carbon fiber concentrations of 0.1%, 0.25%, and 0.5% by weight in the UF resin.

Preparation of MDF panels

The standard laboratory method was followed to manufacture 300 mm x 300 mm x 15 mm MDF boards of 800 kg/m^3. Rubber wood fiber in an amount of 750 g at 12% moisture content was used to prepare MDF. A rotary drum blender was used to blend the wood fibers, carbon fibers, and UF resin uniformly. The drum consists of steel dowels arranged in a zigzag pattern, which were intended to facilitate tumbling and mixing with a rotational speed of 18 rpm. The carbon fiber of various concentrations (0%, 0.1%, 0.25%, and 0.5%) mixed into 10% resin (all percentages are calculated from weight of wood fiber) was sprayed with a spray gun on the wood fibers. A puff mat of 300 x 300 mm was prepared from the resin added wood fiber, which was then pre-pressed at 1.5 MPa pressure. Finally, the pre-pressed panel was hot-pressed at 180 ^{o}C for 450 s with a target thickness of 15 mm; Fig. 1 shows the images of MDF samples. Except for the carbon fiber concentration, all the experimental parameters such as fiber moisture, resin loading, pressing time, and platen temperature were maintained the same in all experiments.

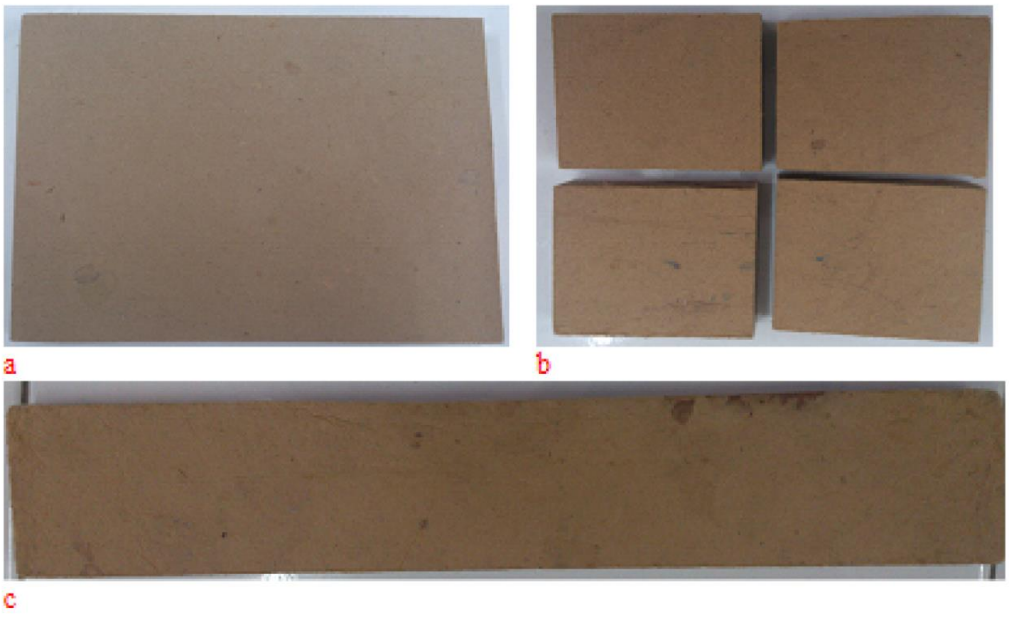

Fig. 1. Images of MDF samples (a) the 300mm x 300mm x 15mm MDF board, (b) for internal bonding, (c) for modulus of rupture

The MDF board was fabricated into two different sets as presented in Fig. 2. The first set of boards were used to find the temperature profile inside the core of mat during hot pressing and second set of boards were used to find the physical and mechanical properties. For the core temperature analysis, three replicates of each treatment, *i.e.* a

total of 12 boards, were prepared. For the board performance test, four replicates of each treatment, *i.e.* 16 boards, were prepared. Ten samples were prepared from each treatment to analyze MOR, IB, and TS.

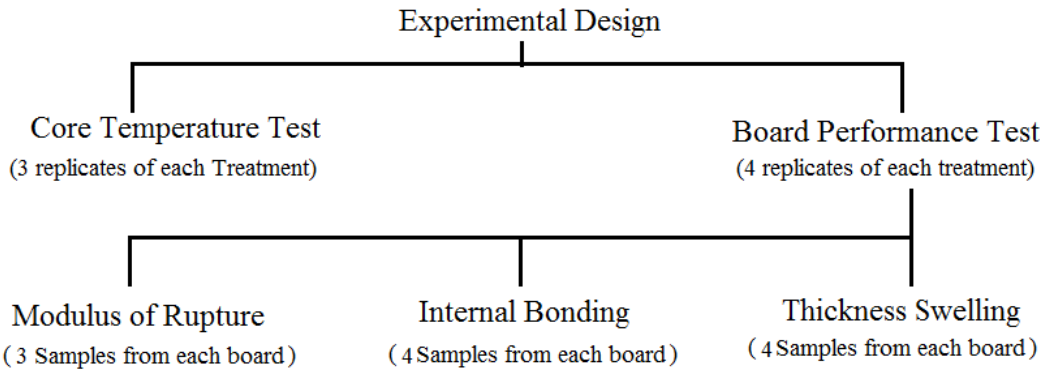

Fig. 2. Brief detail of the experiments performed

Measurements

Measurement of core temperature

Each wood fiber mat was pre-pressed to half-thickness of the loose mat. Two K-type thermocouple wires were inserted at the core of the mat from two opposite sides. The two thermocouple wires were 50 mm apart from each other. The minor deviations in the values from the two thermocouples were due to a slight difference in elevation of thermocouples location while hot pressing. The averages of the two values were taken as the mid-plane temperature of the mat. Three test specimens were prepared for each, by "board with 0.0%, 0.1%, and 0.5% weight concentration".

Mechanical properties measurements

The hardness for both treated and untreated samples was measured. The Modulus of Rupture (MOR) and Internal Bonding (IB), which are commonly used to evaluate MDF, for both treated and untreated samples, were measured according to standard testing method ASTM D 1037. Mechanical testing of the samples was done on a Shimadzu UTM AG-X plug series, and results were analyzed on Trapezium X-software.

A total of 10 samples for MOR and 10 samples for IB were tested for the final result analysis. IB, a tensile strength, is tested perpendicular to the plane of the boards with a cross head speed 1 mm/min. Flexure testing was done by a three-point static bending test to determine the MOR with a cross head speed 10 mm/min. Equations (1) and (2) were used to calculate the MOR and IB, respectively.

$$\text{MOR (N/mm}^2\text{)} = 3 \times P \times L / 2 \times b \times d2 \quad (1)$$

where P is the breaking load, L is the distance between knife edges on which the sample was supported, b is the average specimen breadth, and d is the average specimen depth.

$$\text{IB (N/mm}^2\text{)} = \frac{\text{Maximum force calculated at entire area (F)}}{\text{Surface area of the specimen (A)}} \quad (2)$$

Dimensional stability tests

The thickness swelling (TS) tests were conducted in accordance with ASTM D 1037. Before testing, the weight and dimensions, *i.e.* length, width, and thickness of each specimen were measured. Conditioned samples of each type were soaked in water at room temperature for 24 h. Samples were removed from the water, patted dry, and then measured again. Each value obtained represented the average of sample.

The values of the thickness swelling (TS) in percentages were calculated using the following equation,

$$TS\ (\%) = (T_2\text{-}T_1)*100/T_1 \quad (3)$$

where T_1 is the initial thickness of sample and T_2 is the thickness of wetted sample.

Morphological study

The extents of intercalation and exfoliation of carbon fibers inclusions in the matrix system were monitored by X-ray diffraction (XRD) and Thermogravimetric Analysis (TGA).

The TGA method measures the amount and rate of change in the weight of a material as a function of temperature or time in a controlled atmosphere. Measurements are used primarily to determine the composition of materials and to predict their thermal stability at temperatures up to 1000 °C. The technique can characterize materials that exhibit weight loss or gain due to decomposition, oxidation, or dehydration. The standard practice for calibration of temperature scale for thermogravimetry follows the ASTM 1582 method.

Thermal stability was investigated using non-isothermal thermogravimetry (TG, DTA) using a TA Instrument. Samples (6 + 0.2 mg) were placed in alumina crucibles. An empty alumina crucible was used as a reference. The samples were heated from 30 to 600 °C in a 20 cm^3/min flow of nitrogen with a heating rate of 10 °C/min.

X-ray Diffraction (XRD) measurements of boards containing carbon fibers and without carbon fibers were studied. The X-ray diffraction (XRD) was performed in a XRD analyzer. The samples were scanned over the 2θ range of 3 to 80° at a rate of 1 deg/min. The generator was operated at Cu/30 kV/15 mA. The inter layer spacing (d002) of carbon fiber was calculated in accordance with the Bragg equation: $2d \sin\theta = \lambda$.

The morphological structure of the composites was investigated by JEOL JSM-7500F Field Emission Scanning Electron Microscopy (FESEM), which provides narrower probing beams at low as well as high electron energy, resulting in both improved spatial resolution and minimized sample charging and damage.

RESULTS AND DISCUSSION

Core Temperature Progression

Figure 3 depicts the progression of core temperatures for the boards with carbon fibers of weight concentrations of 0.1, 0.25, and 0.5% in comparison to the control board (CB). The rapid rise in the core temperature can be attributed to the steep vapor pressure gradient (Gupta 2007; Bolton *et al*. 1989a) that developed during the pressing period of 15 s to 70 s. The 100 °C temperature reached the core in 72 s in 0.5 wt.% of carbon fiber

compared to the control where the temperature reached the core in 84 s, indicating that conductivity can be increased by using carbon fibers. In pressing time phase from 112 s to 172 s, a constant temperature in the central plane was observed; this could be ascribed to phase change occurring in the board. The vapor formed was observed to exit from the edges of the board due to higher vapour pressure formed at the core. From the time period of 172 s, a gradual rise in the central plane was observed, which is mainly due to heat conduction in the board. It could be observed that the conductivity of boards prepared with carbon fibers was increased.

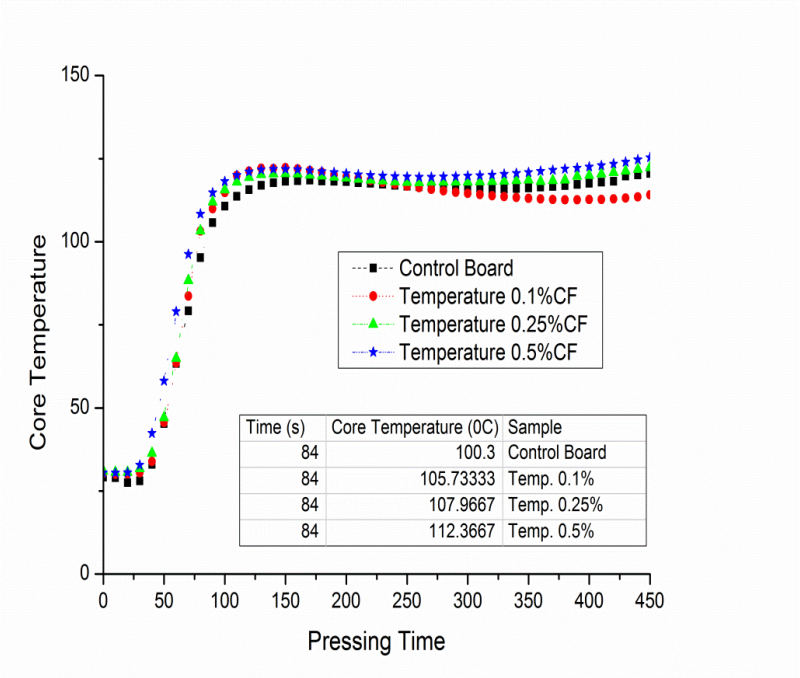

Fig. 3. Core temperature profile of mat during hot-pressing of MDF panels with different weight fraction % of carbon fibers

Enhanced Properties of the MDF

Internal Bonding (IB)

Figure 4 depicts the average internal bonding strength of composites made with various weight percentages of carbon fibers and 10% urea formaldehyde loadings. The bonding strength results show that the composites containing 0.1 wt% of carbon exhibited more internal bonding strength compared to samples without it. This could be possibly due to better adhesion between matrix (carbon fibers and urea formaldehyde) and wood fibers, which cause tension concentration in medium density fiberboards. It is seen from Fig. 4 that unlike 0.1 wt% of carbon fibers, internal bonding strength decreased with addition of carbon fibers, as can be seen in 0.25 wt% and 0.5 wt% of carbon fibers. Reduction of bonding strength with increasing carbon fibers can be related to the increase of the probability for agglomeration that creates regions of stress concentrations that require less energy to elongate the crack propagation (Eitan *et al*. 2003). This is consistent with the results reported by most authors studying the subject (Ashori *et al*. 2012; Tavasoli Farsheh *et al*. 2011; Ziaei Tabari *et al*. 2011). In general, the results for internal bonding strength test showed that medium density fiberboards, which contain 0.1 wt%, had the highest bonding strength value.

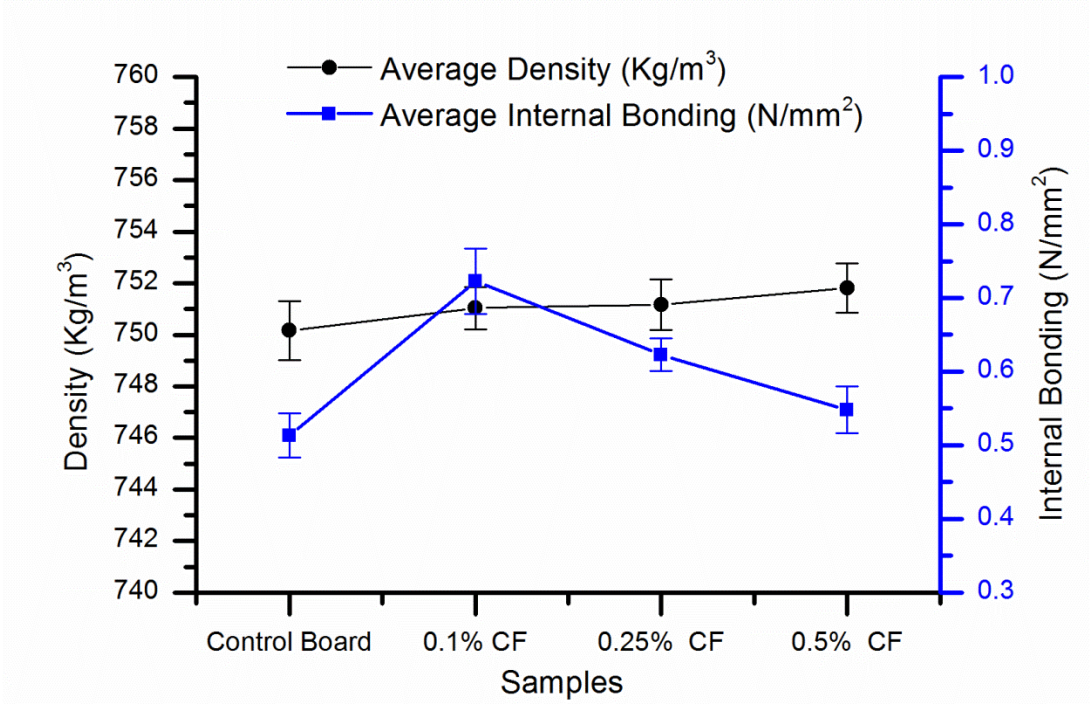

Fig. 4. Internal bonding results of MDF based on different ratios of carbon fibers and control board (without carbon fiber)

Modulus of Rupture (MOR)

The average values of MOR properties that were calculated and compared with the control specimen are shown in Fig. 5.

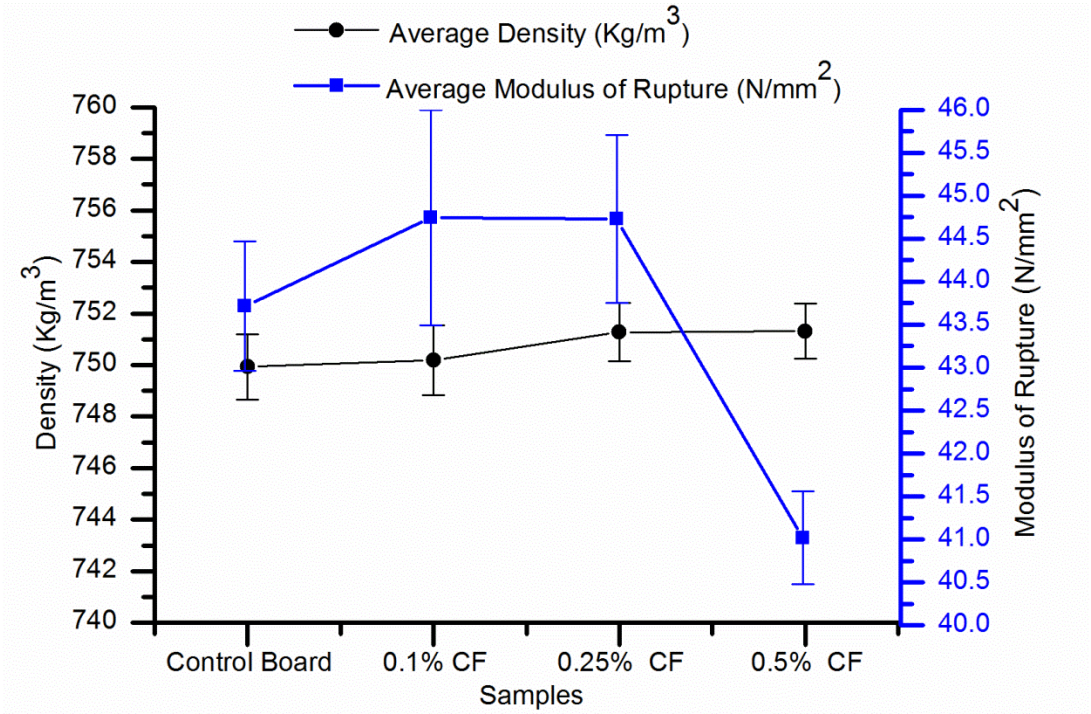

Fig. 5. Comparison of modulus of rupture (MOR) between control MDFs and MDF with different carbon fiber loading

There was no significant difference in MOR values. The MOR results showed that the MDF panels containing 0.1% carbon fiber exhibited higher MOR values as compared to MDF without carbon fiber (control board). This may be due to the improved adhesion between components in the MDF. The MOR of MDF at higher carbon fiber loading did not increase; this may be due to carbon fiber agglomerates. Tavasoli Farsheh *et al*. (2011) reported that modulus of composites at higher CNT loading may fail to increase because of CNT agglomerates. The MOR of MDF panels was increased when using 0.1 and 0.25 wt% carbon, and it was decreased when using 0.5 wt% of carbon fiber. There was improvement of MOR with the addition of 0.25 wt% of carbon fiber, and the maximum increase of MOR was observed in MDF panels with 0.1 wt% of carbon fibers, *i.e*. 44.7.

Thickness Swelling Test

Figure 6 shows the percentage of the water uptake for the composites after 24 h of immersion in water. It can be seen that thickness swelling was not improved by adding different percentages of carbon fibers as compared to panels without the addition of carbon fibers.

Thickness swelling can be reduced significantly with the addition of carbon fibers, but for this case, the TS was not improved. This may be due to the agglomeration of carbon fibers, which vary depending upon the carbon fibers and UF.

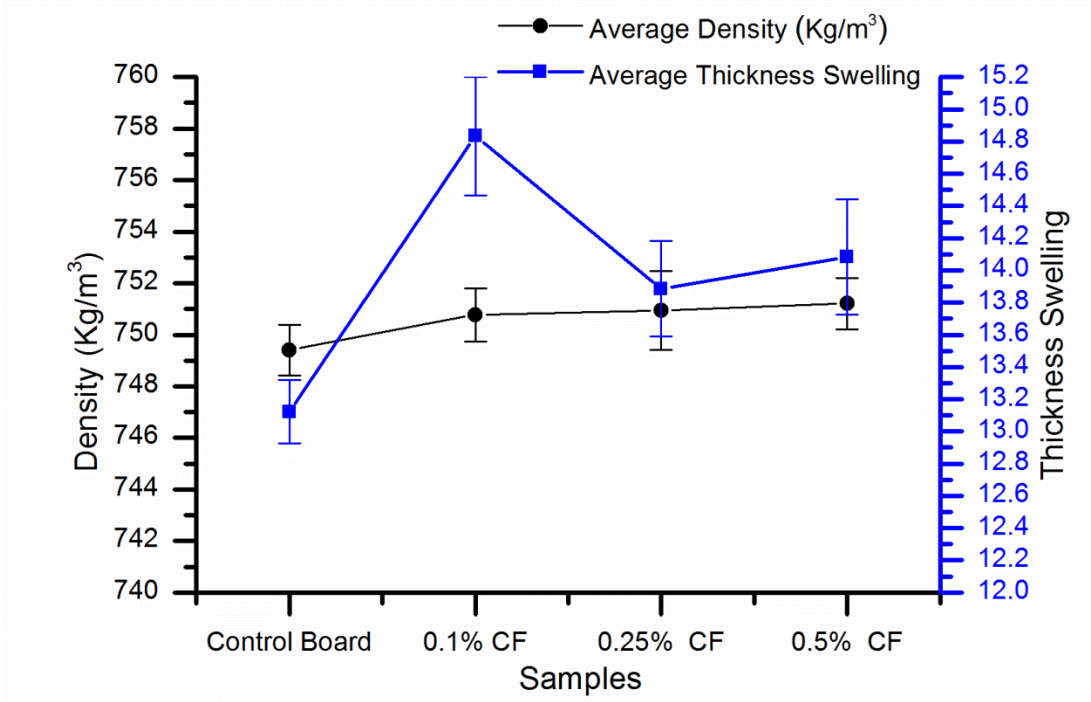

Fig. 6. Thickness swelling results of MDF based on different ratios of carbon fiber and control board (without carbon fiber)

Morphological Characteristics

Thermogravimetry (TGA)

TGA results of MDF based on different ratios of carbon fibers and control board (without carbon fiber) are shown in Fig. 7. The thermal stability of samples was

determined by using thermogravimetric analysis (TGA). In this test, the thermal stability was studied in terms of the weight loss as a function of temperature in a nitrogen atmosphere.

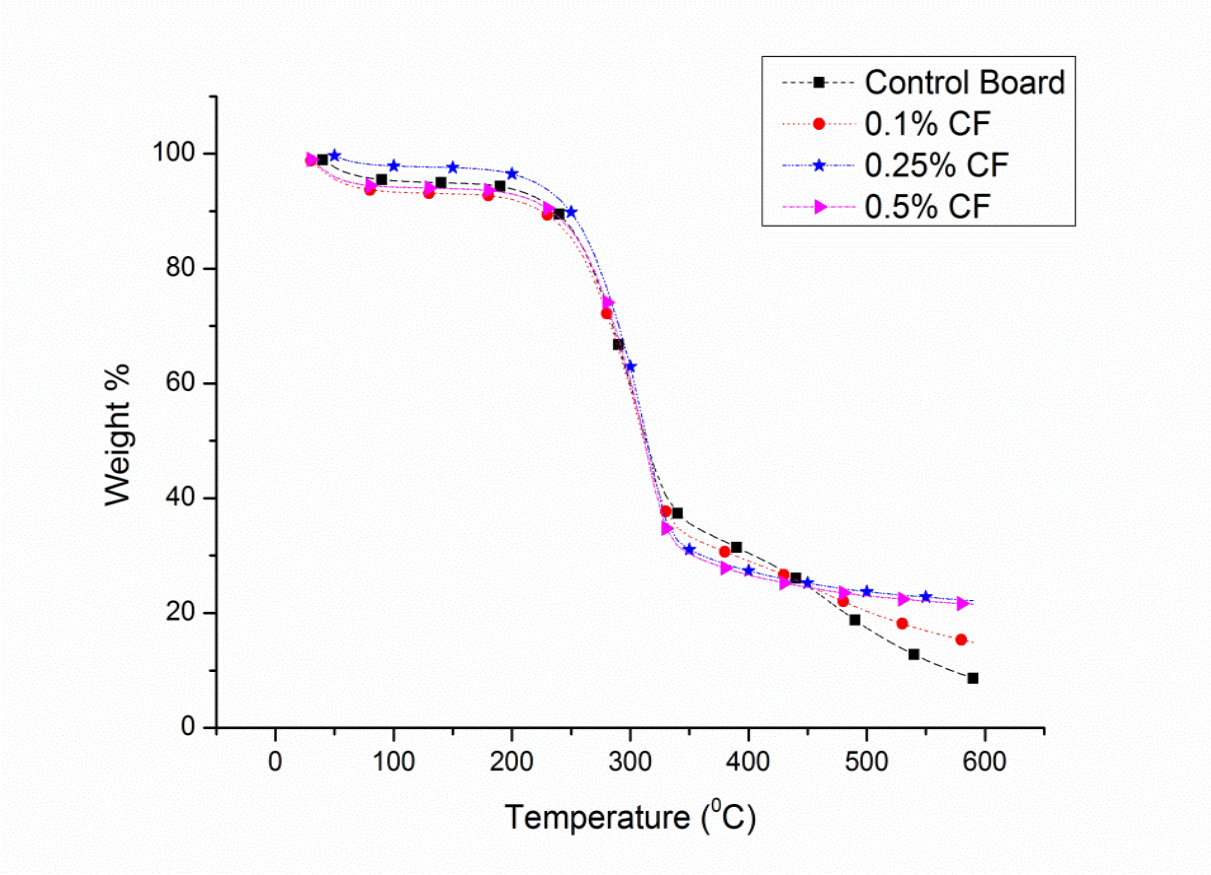

Fig. 7. TGA results of MDF based on different ratios of carbon fibers and control board (without carbon fiber)

The char yields at different temperatures are summarized in Table 1. It can be seen from the TGA curves that MDF based on different ratios of carbon fibers and control board exhibited two distinct stages of decomposition. The first stage of decomposition may be related to the vaporization of moisture in the composites. The second stage of decomposition may be related to degradation of fibers, urea formaldehyde, and its additives. It can also be seen that the major degradation for all samples occurred in the temperature range of 250 to 350 °C.

Table 1. Thermal Properties of MDF Containing Carbon Fibers and MDF without Carbon Fibers

Samples	Char yield at different temperature (%)						T_{max} (°C)
	100 °C	200 °C	300 °C	400 °C	500 °C	600 °C	
Control Board	95.31	93.9	59.76	30.45	17.41	8.629	253.16
CF 0.1%	93.34	92.04	58.98	29.08	20.34	14.91	257.53
CF 0.25%	97.87	96.5	62.93	27.35	23.74	22.15	265.68
CF 0.5%	94.18	93	60.6	26.64	23	21.55	266.66

From the amounts of residue at low temperatures from 50 to 240 °C, it can be seen that the presence of carbon fibers slightly increased the rate of sample degradation compared to that without carbon fibers. But, the maximum decomposition temperature (T_{max}) of the composites slightly changed after the addition of carbon fibers. The maximum decomposition temperature of MDF without using carbon fiber and with using carbon fibers with 0 wt%, 0.1 wt%, 0.25 wt%, and 0.5 wt% carbon fibers were 253.16, 257.53, 265.68, and 266.66 °C, respectively. However, at higher temperatures, MDF with carbon fibers performed better in thermal stability than without carbon fibers with a higher char residue of 22.15 wt% at 600 °C. It was reported in previous studies that the addition of nano-clay platelets would efficiently raise the char residue of polymers at high temperatures (Hwang *et al*. 2010; Madaleno *et al.* 2010; Chatterjee and Islam 2008; Ismail *et al*. 2008).

There was little difference between the curves of composites based on carbon fiber with 0.1, 0.25, and 0.5 wt% carbon fiber; such an effect can be attributed to the good dispersion in the polymer matrix. From Fig. 7 it is obvious that the thermal stability of the composites, based on carbon fiber, was higher than composites based on only UF, and it was improved by the addition of carbon fiber. By increasing the carbon fiber percentage, the thermal stability was enhanced.

XRD Analysis

XRD was applied to investigate the crystal structure of as-grown samples. Figure 8 shows the X-ray diffraction patterns of the (a) MDF containing only UF (control board), (b) MDF containing UF with 0.1 wt% of carbon fiber, (c) MDF containing UF with 0.25 wt% of carbon fiber, and (d) MDF containing UF with 0.50 wt% of carbon fiber. The diffraction pattern of the control board shows a high narrow diffraction peak at 2θ = 23.09°, corresponding to the C (002) reflection of a turbostratic carbon structure of MDF.

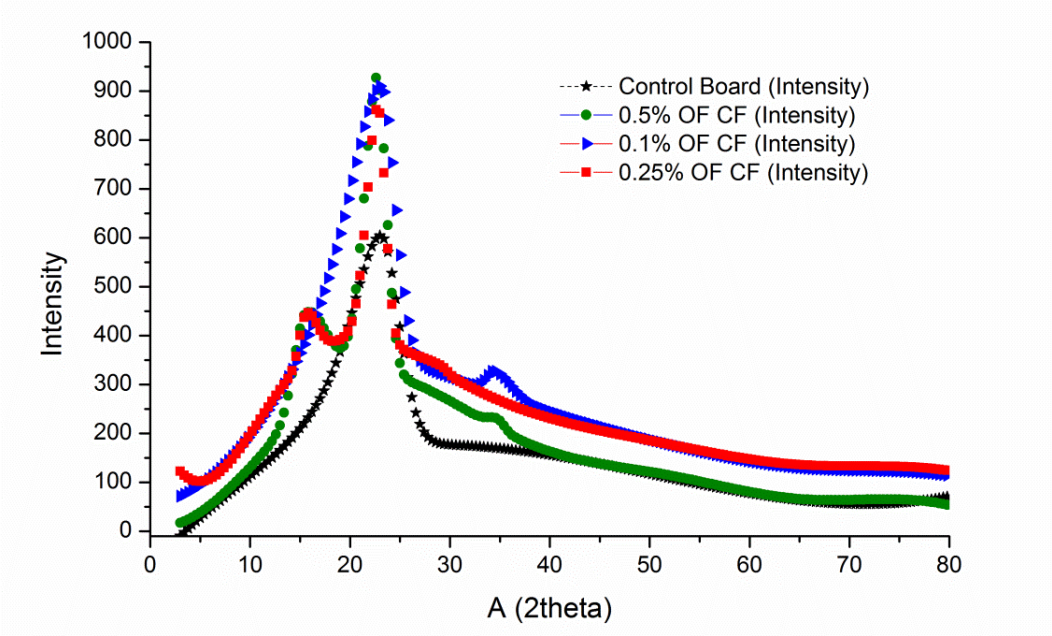

Fig. 8. XRD pattern of MDF containing different ratios of carbon fibers

After the growth of carbon fibers on the MDF, C(002) reflection become stronger, demonstrating that the structure order degree of the as-grown micro–nano carbon fiber in MDF increased significantly compared to the control board (MDF without carbon fibers). The average d-spacing (d0 0 2) values were calculated based on the Bragg equation ($n\lambda = 2d \sin \theta$), determining the interlayer distance of composite matrix. The broad asymmetric peak values for MDF without carbon fiber, MDF with 0.1 wt% carbon fiber, MDF with 0.25 wt% carbon fiber, and MDF with 0.5 wt% carbon fiber were located at 2θ = 23.09, 23.04, 22.80, and 22.70. Structural parameters for the materials investigated are given in Table 3. These show that they all contain disordered graphite microcrystallites, with inter-crystallite and intra-crystallite voids forming the pores. In fact, sharp narrow diffraction peaks show crystalline structures, while the broad peak corresponds to an amorphous structure. XRD pattern of MDF containing different ratios of carbon fibers indicate that changes in the network structure occurred in the amorphous region of the UF resin. In other words, the amorphous region was the important point of entry for moisture reaching the central layer of the compact solid carbon fiber composites.

Table 2. Peak List

2-*theta* (deg)	*d* (ang.)	Phase name
23.09(11)	3.849(18)	Control Board
34.46(18)	2.600(13)	Control Board
23.04(12)	3.86(2)	0.1 % CF
15.6(3)	5.68(11)	0.25 % CF
22.80(4)	3.897(6)	0.25 % CF
29.0(9)	3.07(9)	0.25 % CF
15.48(8)	5.72(3)	0.50 % CF
22.70(3)	3.913(5)	0.50 % CF
34.7(6)	2.58(4)	0.50 % CF

Field Emission Scanning Electron Microscopy (FESEM) Analysis

FESEM is an effective media for the morphological investigations of composites. Figure 9(a) corresponds to MDF without carbon fibers and with carbon fiber, which shows some evidence that fiber has been pulled out from the matrix. Therefore when stress is applied, it causes the fibers to leave the matrix easily and makes gaping holes. Besides this, there were some cavities in the surface that could absorb water and/or reduce mechanical properties. This indicates that the level of interfacial bonding between the fibers and UF in the composites without filler was weak. Figure 9(b) illustrates composite with 0.1 wt% carbon fiber; it was observed that there was no separation of the fibers from the matrix. Very good interaction between the components can be inferred from the image, where white arrows represent the presence of carbon fibers in composites. The strong adhesion that was observed at the interface has already been discussed in terms of the mechanical properties of the composites and is related to the coupling agent, which encapsulated the fibers in the matrix and facilitated strong bonding. The significant decrease in water absorption and thickness swelling of the treatment including 0.25 wt% carbon fiber and 0.5 wt% carbon fiber is further supported by Fig. 9(c) and 9(d), which show that when composite micro voids and the lumens of fibers were filled with carbon fibers, there was a smooth and uniform matrix without any holes and that penetration of water into the deeper holes and cavities of the composite was prevented.

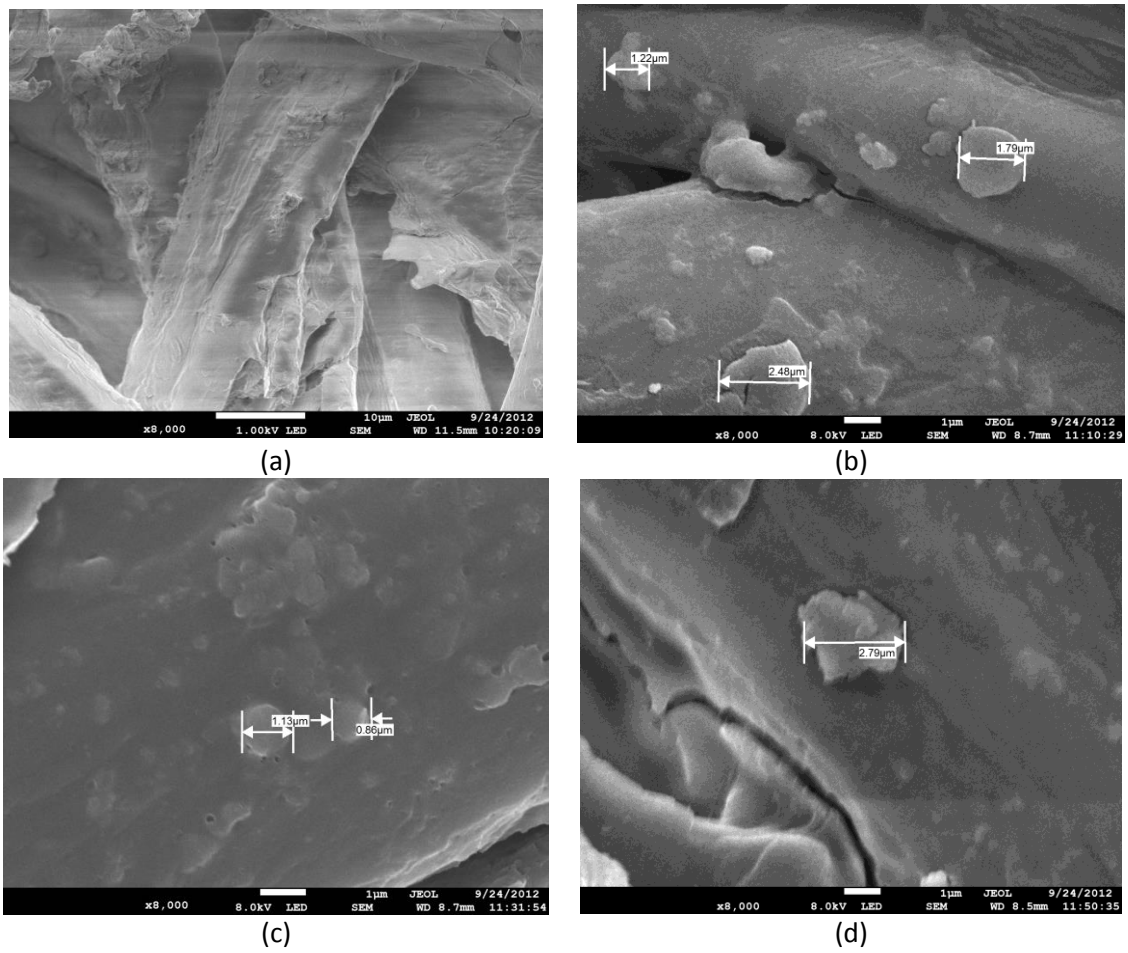

Fig. 9. FESEM micrographs at 8 kX magnification of (a) the surface of samples without carbon fiber, (b) with 0.1 wt% carbon fiber, (c) with 0.25 wt% carbon fiber, and (d) with 0.5 wt% carbon fiber

CONCLUSION

This study investigated the effect of carbon fibers as well as urea formaldehyde on the physical and mechanical properties of MDF boards. The mechanical properties of the MDF board were improved due to the stronger interfacial bonding between the fiber and the matrix polymer. Addition of carbon fibers improved the Internal Bonding (IB). The IB was increased by 0.5126 to 0.7227 (0.1 wt% carbon fiber). Among the various formulations, composites containing 0.1 wt% of carbon fibers gave the maximum improvement in both mechanical and physical properties. There was improvement in core temperature by using carbon fibers and the same effect was apparent as an increase in the conductivity of boards prepared with carbon fibers. From the TGA graph, it was observed that the thermal stability of the composites, based on carbon fiber, was higher than composites based on only UF. The scanning micrographs provided evidence of the smoother surfaces in the composites made with carbon fibers. This was attributed to the better encapsulation of fibers by the matrix polymer. This research combined high thermal conductivity carbon fibers as a means to promote resin cure, reduce pressing

time, and also increase the physical and mechanical properties. The findings may be useful for the wood composite industry in large-scale commercial production of MDF boards.

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