Highly Conductive Fiberboards Made with Carbon and Wood Fibers

Junfeng Hou,[a] Feng Fu,[a] Keyang Lu,[a,]* and Lanzhen Chen [b]

Carbon fibers (CFs) were mixed with wood fibers using the solution blend method to make highly conductive fiberboards. The microstructure, conductivity, shielding effectiveness (SE), and mechanical properties of fiberboards filled with CFs of various lengths and contents were investigated. The uniform distribution of CFs formed an excellent, three-dimensional conductive network. The CF-filled fiberboards exhibited evidence of percolation and piezoresistivity. A greater content of shorter CFs was necessary to realize the effects of percolation. The corresponding thresholds of fiberboards containing CFs of 2, 5, and 10 mm in length were 1.5%, 0.75%, and 0.5%, respectively. The volume resistance of fiberboards tended to be stable as the external pressure increased to 1.4 MPa. The volume resistivity of fiberboards reached equilibrium when the CF content was 10%. The fiberboards with greater than 10% CF content exhibited a SE of 30 dB above the average, yet they met the requirements for commercial application. The mechanical properties of fiberboards were investigated, and CFs were found to enhance the modulus of rupture (MOR) and modulus of elasticity (MOE). Therefore, it was concluded that fiberboards containing CF of 5 mm in length exhibited the best performance between percolation threshold and steady CF content.

Keywords: Highly conductive fiberboard; Percolation effect; Negative pressure coefficient of resistance; Three-dimensional conductive network; Shielding performance; Mechanical properties

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INTRODUCTION

Radiation from various devices may cause serious electromagnetic interference (EMI), which will significantly influence the performance realization of other electromagnetic (EM) devices (Togt *et al*. 2008; Alhusseiny *et al.* 2012). Electromagnetic interference can result in information-compromising emanations and potential health hazards to humans and other organisms. Electromagnetic shielding has been regarded as an effective method to prevent EM radiation from passing through blocking media (or shields) (Razavi and Halaj-Aminhosseim 2010). Currently, the products used for EM shielding are metals composed of some other materials by filling, coating, and laminating to form composite materials that are a mixture of polymers and carbons.

Wood-based EM shielding composites have attracted the public's interest because of their availability, cost, and renewability. In general, wood elements (*i.e.*, wood fibers, veneers, and flakes) are composited with conductive materials (*i.e.*, metallic conductive fillers and carbon conductive fillers) by filling (Liu *et al*. 2007; Yuan and Fu 2014), coating, laminating (Yuan *et al.* 2014), and electroless plating (Hui *et al.* 2014) to prepare wood-based composites with excellent shielding performance. Among the wood-based EM shielding composites, conductive material-filled fiberboards have become one of the main research trends because of their simplicity, performance dependability, and tremendous prospects in the engineering sector.

Carbon fibers (CFs) are characterized by their superior electrical properties, light weight, high strength, and high tensile modulus, and have been widely used in diverse multifunctional composites (Taipalus et al. 2001; Zhang and Liu 2009; Wang *et al.* 2013; Yuan *et al.* 2014). Several experiments have been conducted to investigate the mechanical properties (Matsumoto and Nairn 2009; Yang *et al*. 2012), electrical conductivity (Shi 2011), and shielding performance (Yuan *et al*. 2013) of CF-filled composites. In these studies, most of the research has focused on mechanical improvement by laminating the CF layer. There has been minimal research on the conductivity (Zhang *et al*. 2011) and EM shielding of CF-filled fiberboard. Zhang (2013) used CFs instead of powder to mix with isocynate resin. The SE of the charcoal composites reached 28.62 dB when the CF content was 50% of the fiberboard. However, it was also found that the CFs was not uniformly distributed and formed an excellent conductive network in the charcoal composites. No research has studied the effect of CF length and content on conductivity and shielding performance in CF-filled fiberboards. Therefore, research in this area needs to be conducted to predict and analyze CF-filled fiberboards with excellent conductivity and shielding performance.

Unlike previous research, which blended fiber directly with resin (Zhang *et al*. 2013), CFs and wood fibers in this study were blended using the solution blend method. The hybrid fibers were uniformly mixed with diphenyl-methane-diisocyanate (MDI) to prepare highly conductive fiberboards. The conductivity and shielding effectiveness (SE) of the CF-filled fiberboards of various lengths and contents were analyzed. The mechanical properties of fiberboards were also determined following the GB/T17657 (2013) standard. The purpose of this study was to develop a fiberboard product for the construction sector having excellent conductivity, shielding performance, and mechanical properties.

EXPERIMENTAL

Materials

Carbon fibers were obtained from the Weida Composite Material Co., Ltd, Nanjing, China. The lengths of the CFs were 2, 5, and 10 mm, and the diameters ranged from 7 to 10 μm. The density of the CFs ranged from 1.6 to 1.76 g/cm^3, and the volume resistivity was 1.5×10^{-5} Ω•cm. The tensile strength and modulus of CFs were 3.6 to 3.8 GPa and 240 to 280 GPa, respectively. Wood fiber was provided by the Fenglin Wood Industry Group Co., Ltd, Nanning, China. Diphenyl-methane-diisocyanate (MDI, I-BOND® MDF EM 4330) was provided by Huntsman, Shanghai, China. The density at 25 °C was 1.23 g/cm^3. The color of the resin was dark brown, and the viscosity at 25 °C was 275 cps. In addition, its solid content was 100%.

Methods

Surface pretreatment of CFs

The CFs was soaked in alcohol with a concentration of 75% for 8 h to dissolve the sizing agent on the surface of the fibers. Then, the CFs were rinsed using distilled water and oven-dried in a circulation oven at 103 ± 2 °C. The surface morphology of the CFs before and after the pretreatment was investigated by scanning electron microscope (SEM; S-4800, Hitachi Limited, Tokyo, Japan), as shown in Fig. 1. The number of grooves increased on the surface of the CFs after the pretreatment process. The depth and width of the grooves increased and resulted in a stronger interfacial bonding surface between the CFs and the MDI.

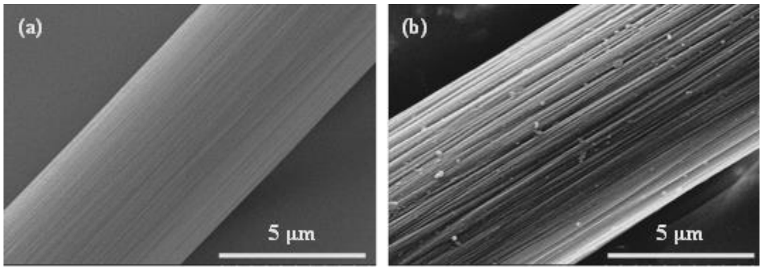

Fig. 1. The CF surface morphology (a) before and (b) after surface pretreatment

Preparation of hybrid fibers

A blender with a capacity of 200 L was used to mix the CFs and wood fibers uniformly. The uniformity of the hybrid fibers remarkably affected the fiberboard performance. Hybrid fibers were prepared at a rotational speed of 600 rpm for 20 min using the solution blend method (Guo *et al.* 2003). Then, the fibers were dried at a temperature of 103 ± 2 °C to a moisture content of 10% to 12%. The CF (2 mm) content in the hybrid fibers was 0.25%, 0.50%, 0.75%, 1.0%, 1.25%, 1.50%, 1.75%, 2.0%, 5.0%, 10%, and 20%. The CF content (5 and 10 mm) in hybrid fibers was 0.25%, 0.50%, 0.75%, 1.0%, 2.0%, 5.0%, 10%, and 20%. These hybrid fibers were used to prepare fiberboards, and their corresponding performances were investigated.

Preparation of highly conductive fiberboards

A standardized procedure was followed to prepare 360 mm × 340 mm × 3 mm fiberboards with densities of 0.65 g/cm^3. The hybrid fibers were held to between 10% and 12% moisture content. The MDI content was 10% (calculated using the weight of the hybrid fiber), and it was uniformly applied to the hybrid fibers. The fiberboards were hot-pressed at 150 °C for 6 min with a target thickness of 3 mm. The thickness of the fiberboards was determined using a thickness gauge.

Volume resistivity test

Figure 2 shows the setup for the insulation resistance and DC low-resistance tests. The insulation resistance of the fiberboards was investigated using a three-electrode method, according to the ASTM-D257 (2007) standard, and DC low-resistance was investigated using the four-electrode method, in accordance with the MIL-DTL-83528C (2001) standard. An insulation resistance tester (TH2683, Changzhou Tonghui Electronic Co., Ltd. Jiangsu Province, China) was used to measure the insulation volume resistance, ranging from 10^5 to 10^{13} Ω. A digital DC resistance tester (TH2512, Changzhou Tonghui Electronic Co., Ltd. Jiangsu Province, China) was used to measure the electrical resistance, ranging from 1.0 μΩ to 2.0 MΩ. The volume resistivity (ρ) in Ω.cm was determined using Eq. 1,

$$\rho = \frac{R \times A}{L} \tag{1}$$

where A is the smallest cross sectional area of a section of the sample between the probe electrodes (cm^2), L is the distance between the two electrodes in cm, or 2.45 cm, and R is the volume resistance of the specimens in Ω. Specimens with dimensions of 5 × 5 mm^2 were cut from the fiberboards, and four replicate specimens of each type were measured. Prior to testing, the entire specimens were conditioned at 20 °C and 65% relative humidity (RH) for 24 h, according to the ASTM D1037 (2006) standard. The external pressure selected in this study ranged from 0.2 to 2.1 MPa.

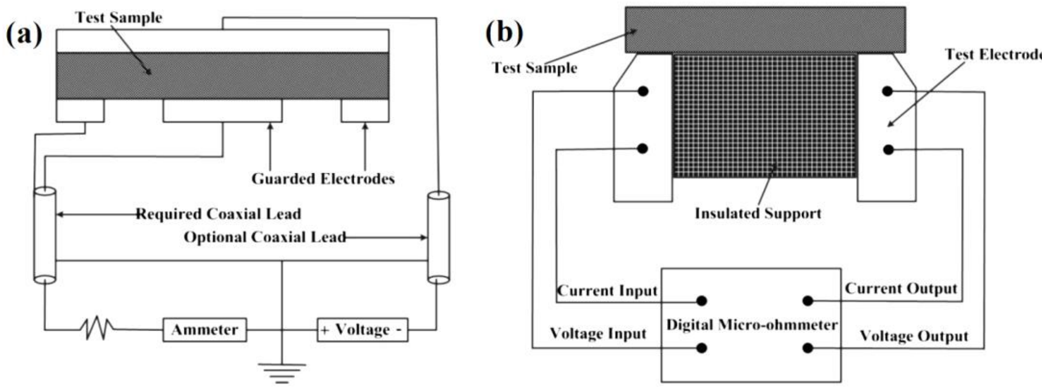

Fig. 2. Setup for measuring the (a) insulation resistance and (b) DC low-resistance

Electromagnetic shielding measurement

The SE was measured using the coaxial cable method (also referred to as transmission line method), according to Chinese standard SJ20524 (1995). The setup consisted of a vertical flanged test device with the input and output connected to a Hewlett-Packard (HP; USA) 7401A EMC Analyzer. The frequencies of the transmitted signals ranged from 100 kHz to 1.5 GHz. Standard circular specimens, with diameters of 115 ± 0.5 mm, were cut from the fiberboards (Fig. 3). Six replicate specimens of each type were prepared and tested.

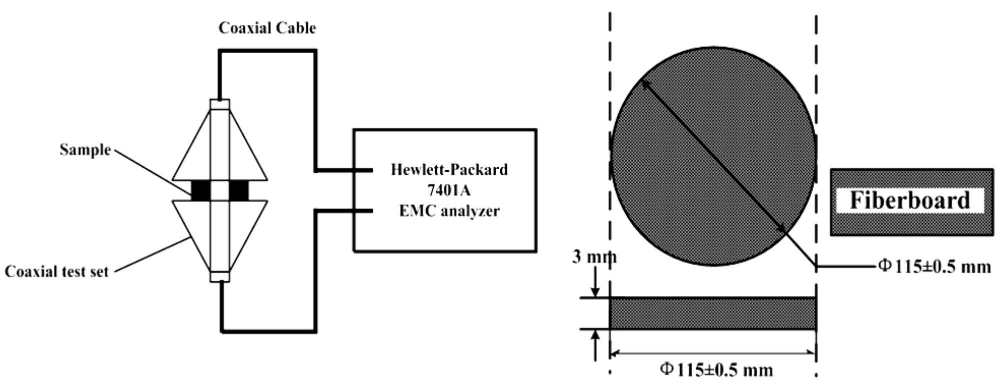

Fig. 3. The setup and specimen sizing for the electromagnetic shielding effectiveness test

Microstructural characterization

The CFs' contribution in the plane and cross-sections of fiberboards were investigated using a three-dimensional analysis system digital microscope (VHX-1000, KEYENG Corp., Itasca, IL, USA) and scanning electron microscope (SEM; S-4800, Hitachi Limited, Tokyo, Japan), respectively.

Mechanical and physical properties

The modulus of rupture (MOR), modulus of elasticity (MOE), and thickness swelling (TS) were measured in accordance with GB/T17657 (2013). Flexural tests were carried out using a three-point static bending test to determine the MOR and MOE of highly conductive fiberboards. Before testing, the dimensions (*i.e.*, length, width, and thickness) and weight of each specimen were measured. Prior to testing, the specimens were conditioned at 20 °C and 65% RH for 24 h. For the thickness swelling test, conditioned samples of each type were soaked in water at room temperature for 24 h. Specimens were removed from the water, patted dry and measured again. A total of six specimens for the bending tests (MOR and MOE) and eight specimens for the TS test were measured for the final analysis. The presented values for the MOR, MOE, and TS tests are the mean values.

RESULTS AND DISCUSSION

Microstructure Analysis of the Fiberboard

Figures 4 and 5 depict the CF distribution in the fiberboards. A two-dimensional conductive network was observed in the fiberboards with CF contents ranging from 0.5% to 10%, and a three-dimensional conductive network was seen as the CF content increased to 20%. Results reported by Chiarello *et al.* (2005) showed a similar conclusion in the electrical conductivity of self-monitoring, CF-reinforced cement (CFRC).

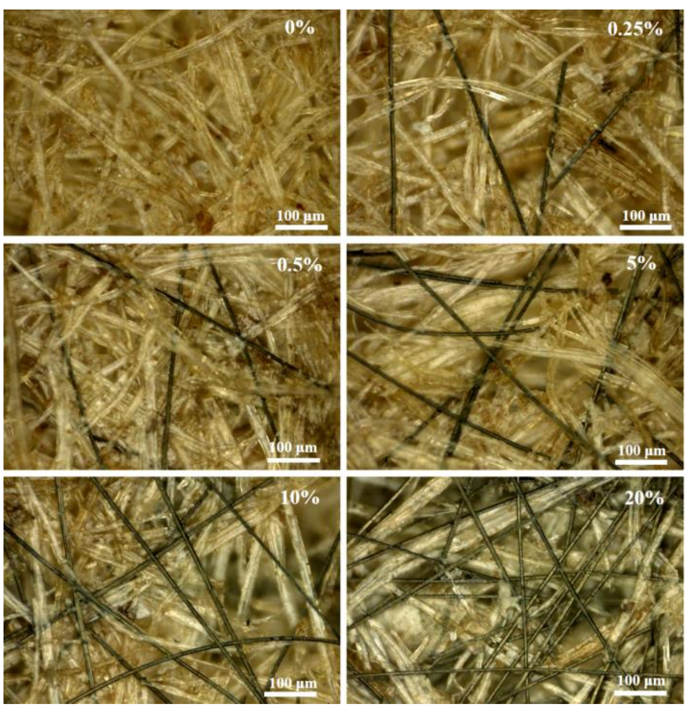

Fig. 4. CF distribution in the plane of fiberboards. The black fibers are CFs, and the brown fibers are wood fibers

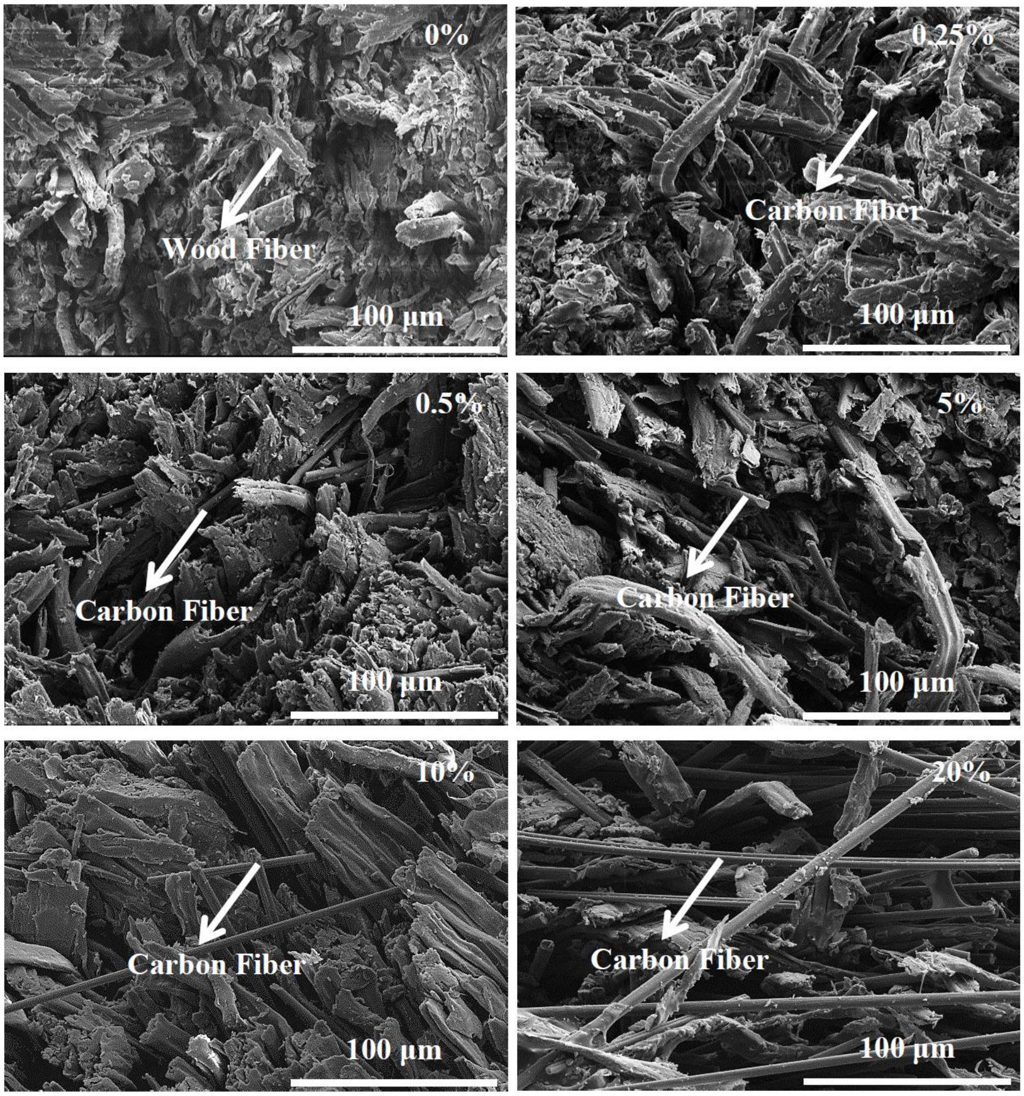

Fig. 5. CF distribution in the cross-section of the fiberboards

Piezoresistive Behavior of the Fiberboards

The volume resistance of the CF-filled fiberboards (10 mm, 5%) under various external pressures is shown in Fig. 6. CF-filled fiberboards exhibited a phenomenon of negative pressure coefficient of resistance in piezoresistivity. In general, the electrical conductivity of the highly conductive fiberboards was determined using the conductive network formed throughout the matrix. The conductive network depends markedly on the external loading (Wang and Chung 2000). The results reported by Wen and Chung (2007b) exhibited a similar conclusion. The distance between CFs decreased as the external pressure increased, especially in the low external loading area. The contact number and region of the CF increased, which contributed to a decline in the volume resistance, especially in the low-pressure region. When the external pressure was above 1.4 MPa, the volume resistance declined and reached a plateau; there was no further decrease with increasing external pressure. The same conclusion was obtained for other CF-filled fiberboards. A continuous CF polymer-matrix composite was prepared by Wang *et al*. (2013), and it exhibited Piezoresistivity when tension was applied to the fiber orientation.

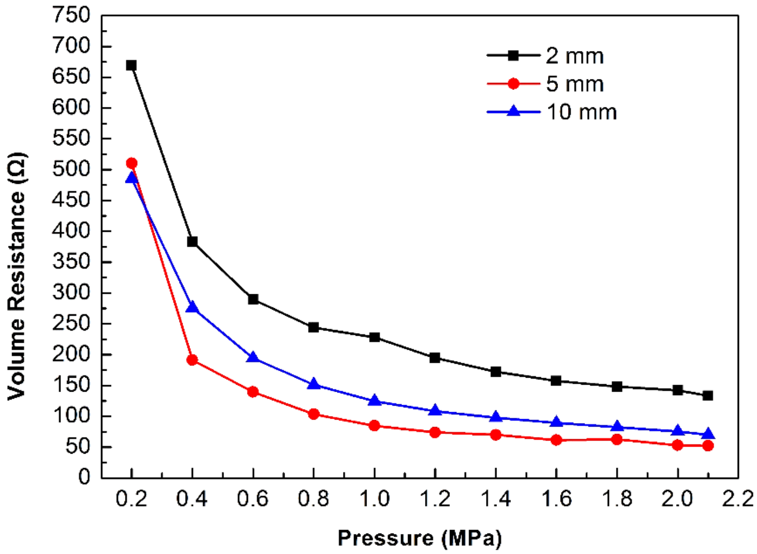

Fig. 6. The volume resistance of fiberboards under external pressure

Conductive Uniformity of the Fiberboards

Figure 7 shows the normality analysis for the volume resistivity of the CF-filled fiberboards (2 mm, 5 mm, and 10 mm; the CF content was 5%).

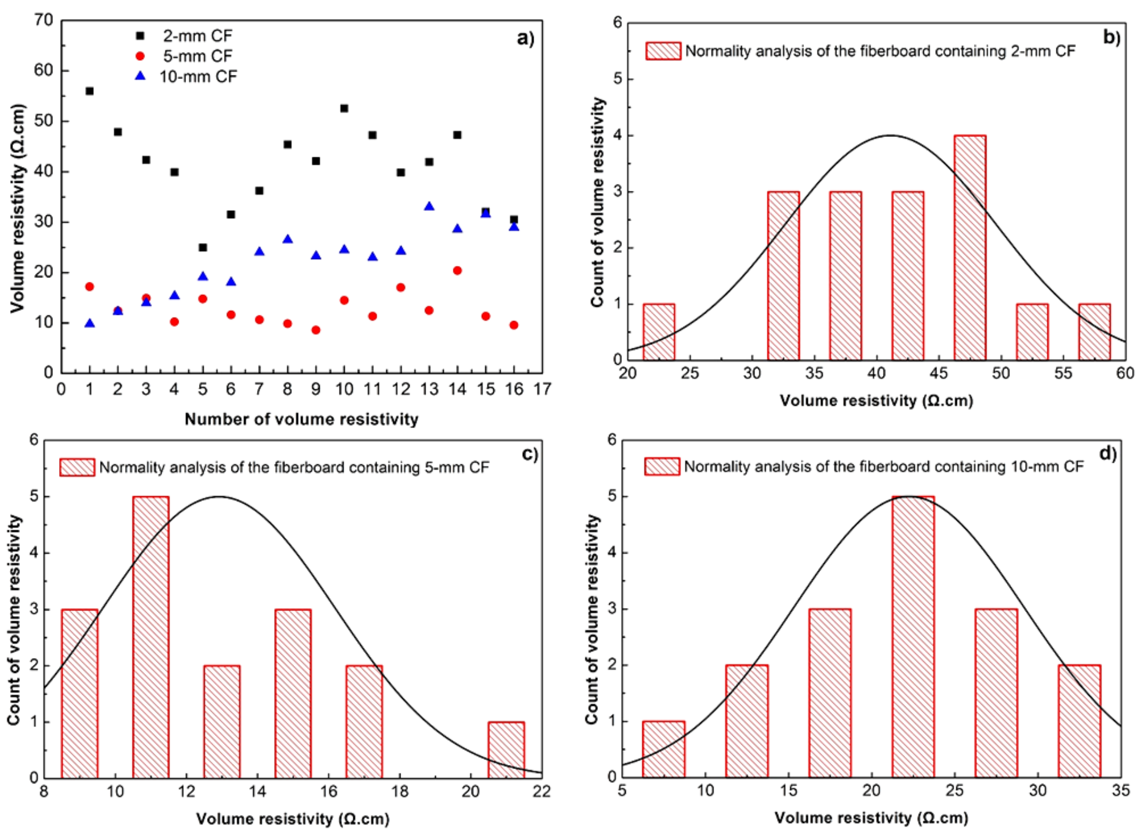

Fig. 7. Normality analysis for the volume resistivity of the fiberboards: a) Volume resistivity of the fiberboards; b) Normality analysis of the fiberboard containing 2-mm CF; c) Normality analysis of the fiberboard containing 5-mm CF; and d) Normality analysis of the fiberboard containing 10-mm CF

The corresponding normality test result of volume resistivity of the fiberboards is shown in Table 1. As depicted in Fig. 7, the volume resistivities of the fiberboards followed a normal distribution. Additionally, the normality test result (Table 1) revealed that the uniform distribution of the volume resistivity for the fiberboards was significantly at 0.05 levels. Therefore, the fiberboards exhibited good uniformity with respect to conductivity. Additionally, the electrical conductivity of the fiberboards was dependent upon the conductive network of the CFs formed in the fiberboards (Chung 2012). The uniformity of electrical conductivity of the fiberboards indicated the uniform distribution of CFs in the fiberboards. This indicated that CFs and wood fibers were blended uniformly using the solution blend method.

Table. 1. Normality Test Result of Volume Resistivity of the Fiberboards

Length of CF/mm	Volume resistivity/(Ω.cm)	DF	Statistic	Prob<W
2	41.11±8.46	16	0.97643	0.92888
5	12.92±3.36	16	0.92915	0.23632
10	22.24±6.92	16	0.96235	0.70466

Volume Resistivity of the Fiberboards

Figure 8 shows the effect of CF length and content on the volume resistivity of the fiberboards. The conductive network formed by the CFs affected the electrical conductivity noticeably (Chung 2012). When a sufficient amount of CF was uniformly dispersed in the fiberboards, a continuous network could be established. The continuous network greatly affected the electrical conductivity and turned the fiberboards conductive. The formation of a conductive network could be detected as a dramatic decrease in the volume resistivity of the fiberboards as the CF addition increased to a specific content, described as the percolation effect (Rejõn *et al*. 2000; Wen and Chung 2007a). The specific content of CF was the threshold. In general, the volume resistivity changed sharply as the CF content increased beyond a threshold value. Above the threshold level, conductivity reached a plateau and did not increase sharply, even with further addition of CFs.

As depicted in Fig. 8, the threshold for the case of fiberboard containing CFs of 10 mm was 0.5%. Moreover, the 5-mm and 2-mm fiberboards exhibited a percolation threshold of 0.75% and 1.5%, respectively. As expected, the shorter CF-filled fiberboards with a larger fiber length were more conductive at a smaller content of CF; this resulted in a smaller threshold value. It was for this reason that the fiberboards with a greater CF length were more prone to overlapping and formed a better conductive network with a lower CF content. A greater content of shorter CFs was necessary to form the conductivity networks. In another study by Chiarello *et al.* (2005), it was observed that the effect of CF length on the threshold value of CFRC was similar.

It was found that electrical conductivity of the fiberboards increased with increase of CF contents. As the CF content increased to 10%, a moderately complete conductive network formed (Fig. 4 and 5), and the volume resistivity decreased slightly because the conductive pathways did not show a large increase (Fig. 4 and 5). The volume resistivity of the fiberboards decreased because of the increasing using of CF, which reached equilibrium by 10%. It was also noted that increasing the length of the CF from 2 mm to 5 mm decreased the volume resistivity of the fiberboards. And the volume resistivity of the fiberboards increased as the length of CF increased to 10 mm from 5mm. This may have been due to the formation of 10-mm CF agglomerates. And the 5-mm CFs formed better conductive network at the same CF content (Guo *et al*. 2003).

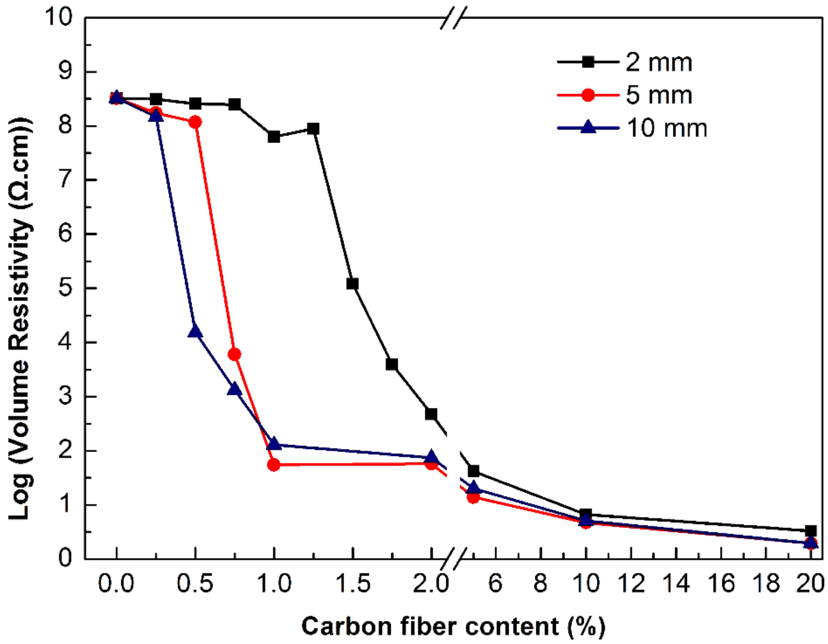

Fig. 8. The effect of CF length and content on the volume resistivity of fiberboards

Shielding Effectiveness of the Fiberboards

The SE is a measurement of the attenuation of an electromagnetic signal through the shielding material. It is commonly used to evaluate electrical shielding performance. When the electromagnetic signal reaches the surface of the shielding material, it will be reflected, absorbed, and refracted, whereby a significant amount of energy from the electromagnetic signal will be lost. The SE (dB) of a material under the interference of an ambient electromagnetic signal can be expressed according to Clayton *et al*. (2006), using Eq. 2,

$$\mathrm{SE} = R + A + B \tag{2}$$

where R is the reflection loss in dB, A is the absorption loss in dB, and B is the correction factor resulting from multiple reflections within the material. For a plane electromagnetic field, the reflection loss (R) and the absorption loss (A) can be calculated using Eqs. 3 and 4, respectively,

$$R = 168 + 10\log_{10}(\sigma_r / [\mu_r f]) \tag{3}$$

$$A = 0.131\, t\, (f\, \sigma_r\, \mu_r)^{0.5} \tag{4}$$

where σ_r is the electrical conductivity relative to copper, μ_r is the permeability relative to free space, f is the frequency of the signal in Hz, and t is the thickness of the material in mm. When neglecting the effect of the multiple reflections (B) within the material, Eq. 2 can be rewritten as Eq. 5:

$$\mathrm{SE} = 168 + 10\log_{10}[\sigma_r/(\mu_r f)] + 1.7\, t\, (f\, \sigma_r\, \mu_r)^{0.5} \tag{5}$$

The SE of fiberboards with various CF lengths and contents is presented in Fig. 9. Fiberboards without CFs had almost no SE because of the high volume resistivity (3.25×10^{8} Ω.cm). After the CF content reached the threshold, the CF started to form a two-dimensional conductive network (Fig. 4 and 5) that shielded the electromagnetic waves efficiently. As described in the volume resistivity analysis, the conductivity of the fiberboards increased with the further addition of CFs (Fig. 8). Reflection, absorption, and SE losses increased, according to Eqs. 3, 4, and 5, respectively. Therefore, SE of the fiberboards increased with the further addition of CFs (Yuan *et al*. 2013). Results determined by Lu (2007) showed a similar conclusion for the SE of a conductive film filled with copper fibers.

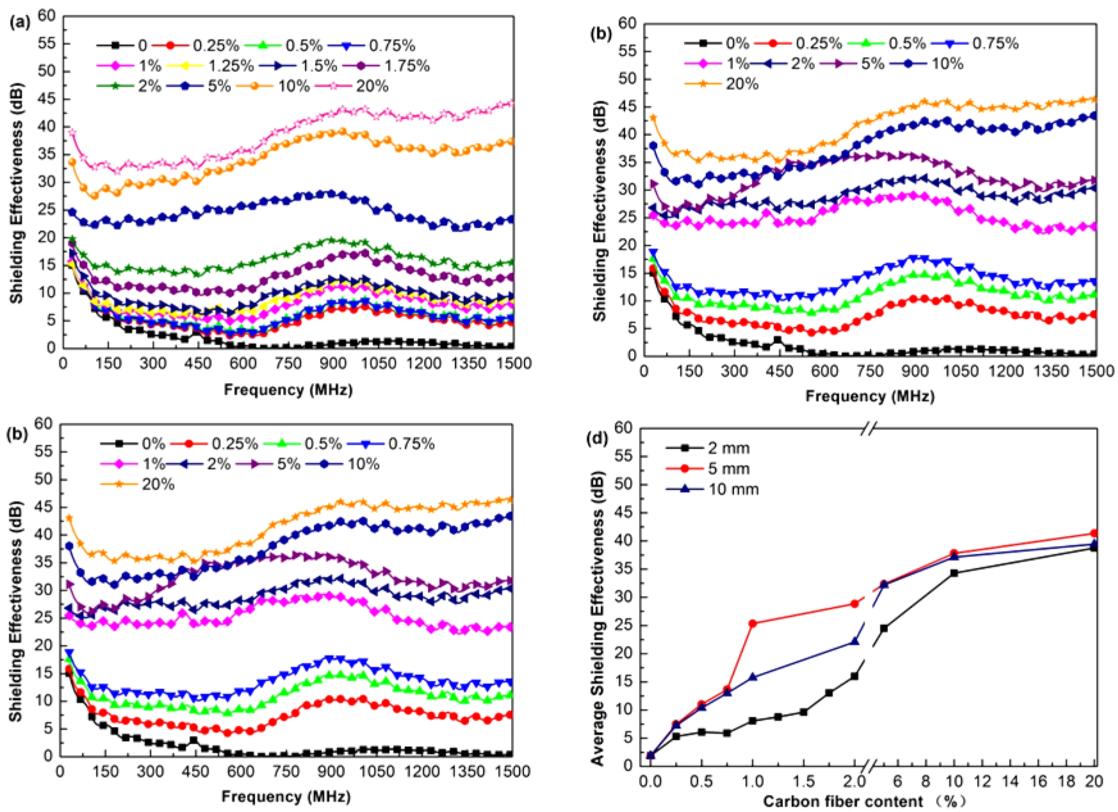

Fig. 9. SE of fiberboards with various CF lengths and contents: (a) CF length of 2 mm; (b) CF length of 5 mm; (c) CF length of 10 mm; and (d) mean SE of the fiberboards

The mean SE (test frequency: 30 MHz to 1.5 GHz) of the fiberboards was calculated, as shown in Fig. 8. Fiberboards containing CFs of 5 mm in length exhibited obvious SE advantages over those containing 2-mm and 10-mm CFs between the percolation threshold (CF content: 0.75%) and steady-state value (CF content: 10%). As described in the analysis of volume resistivity, the conductivity advantage of the CF-filled fiberboards of 5 mm in length was obvious between the percolation threshold and steady-state value (Fig. 8). The conductivity advantage of fiberboards filled with CF contents lower than 0.75% or greater than 10% was small. The SE of the fiberboards was mostly dependent on the conductivity, increasing with increasing conductivity; the corresponding SE mirrored the conductivity value.

CF-filled fiberboards (fiber lengths: 5 and 10 mm) of more than 10% CF content exhibited a SE of 30 dB and above, meeting the requirements for commercial application (30 to 60 dB). However, fiberboards that contained CFs of 2 mm in length with 20% CF content exhibited a lower conductivity. At the CF content of 20%, the SE of the fiberboards with increasing lengths was between 31.96 and 44.43 dB, 36.67 and 46.65 dB, and 34.07 and 44.90 dB, respectively.

Mechanical and Physical Properties of the Fiberboards

Modulus of elasticity (MOE) and modulus of rupture (MOR)

The mean values for the MOE (a) and MOR (b) are shown in Fig. 10. The MOE increased with increasing CF content. This may have been due to the formation of CF agglomerates. Ghasemi *et al*. (2012) and Khan *et al*. (2013) reported the same conclusion.

Fiberboards containing 20% CFs exhibited greater MOE and MOR values, as compared to the fiberboards without CF (control boards). The MOE was 1.963, 2.545, and 2.176 GPa for 2, 5, and 10 mm lengths, respectively, and the MOR was 10.98, 13.58, and 9.49 MPa, respectively. This may have resulted from the generation of three-dimensional conductivity (Fig. 4 and 5) and an excellent adhesion capability between the CF and MDI in the fiberboards.

The bridging and pull-out effect that occurred in the fiberboards also played an important role in the improvement of the MOE and MOR (Tatsuya and Mototsugu 1991). The addition of the CFs delayed the rupture that occurred in the fiberboards because of the excellent tensile strength (3.6 to 3.8 GPa).

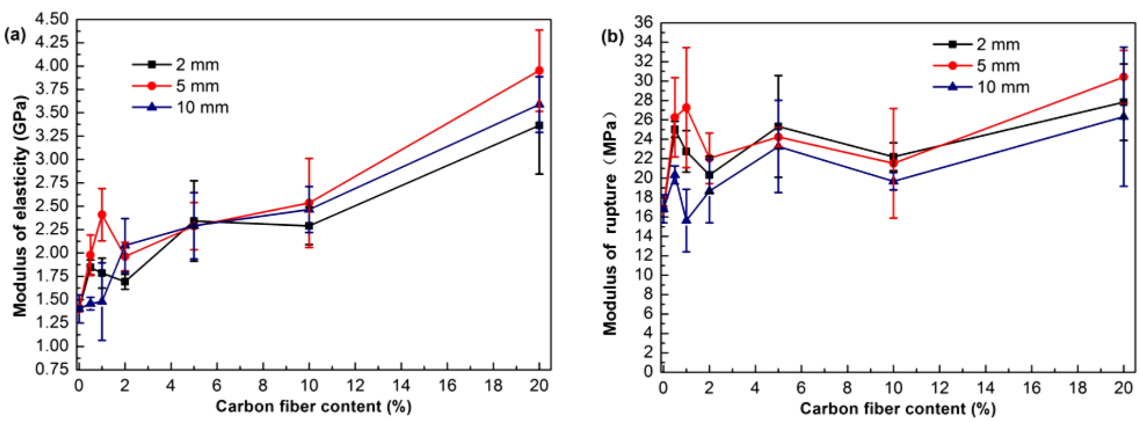

Fig. 10. (a) MOE and (b) MOR of fiberboards with various CF lengths and contents

Thickness swelling (TS)

Figure 11 illustrates the TS of the fiberboards after 24 h of immersion in water. It was found that the TS rate increased with the addition of CFs. The fiberboard containing CFs of 2 mm in length illustrated the lowest TS rate in the study. The increase in the TS rate could have resulted from the poor interfacial adhesion of the hybrid fibers with the further addition of CFs. Therefore, as CFs agglomerate for longer periods of time, it becomes increasingly difficult to form excellent bonding interfaces between the CFs in the fiberboard, resulting in an increase in the TS rate.

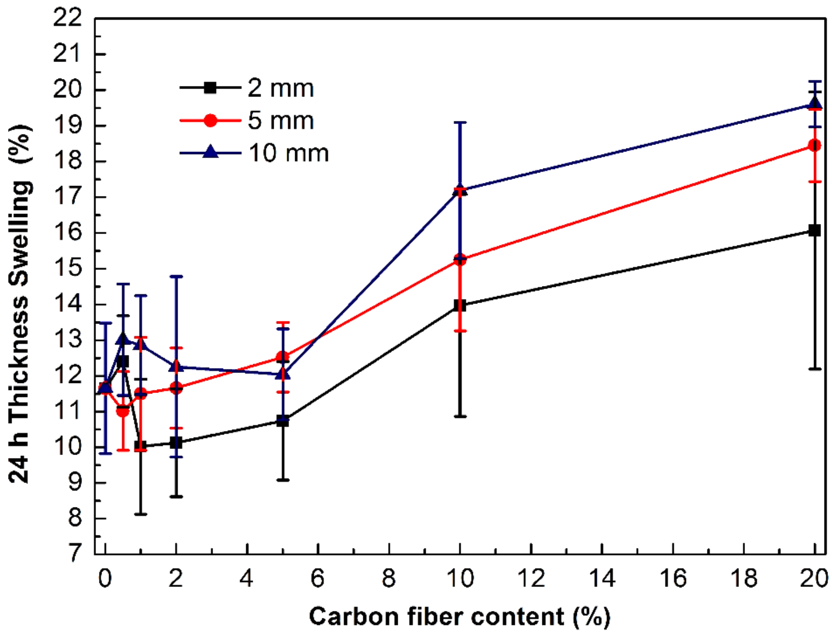

Fig. 11. Thickness swelling of fiberboards with different lengths and contents of CF

CONCLUSIONS

1. A three-dimensional conductive network was observed in fiberboards as the CF content increased to 20%. This was attributed to the improvement in electrical conductivity, electromagnetic shielding effectiveness, and mechanical properties of the fiberboards.

2. The volume resistance of the fiberboards depended on the external pressure and exhibited the phenomenon of negative pressure coefficients of resistance in piezoresistivity. As the external pressure increased, the volume resistance decreased from 0.2 to 0.8 MPa and then reached a plateau as the pressure increased to 1.4 MPa.

3. The electrical conductivity of the fiberboards was dependent upon the conductive network of the CFs formed in the fiberboards. The uniform distribution of the volume resistivity for the fiberboards is significantly at 0.05 levels. The CFs and wood fibers were blended uniformly using the solution blend method.

4. The CF-filled fiberboards exhibited percolation. Fiberboards containing CFs of 10 mm in length showed a threshold value of 0.5%, and the threshold values of CFs of 5 mm and 2 mm doubled and quintupled, respectively. Additionally, the volume resistivity of the fiberboards decreased with increasing CF content, and reached equilibrium by 10%.

5. The SE of fiberboards containing a CF of 20% was 31.96 to 44.43 dB (2 mm), 36.67 to 46.65dB (5 mm), and 34.07 to 44.90 dB (10 mm), respectively. These values met the requirements for commercial application (30 to 60 dB).

6. The mechanical properties of the fiberboards were improved because of the addition of CFs. When the CF content increased to 20%, the fiberboards exhibited the maximum enhancement of the mechanical properties. Furthermore, the MOE

increased by 139.96% (2 mm), 181.78% (5 mm), and 155.86% (10 mm), respectively, compared to that of fiberboards without CF. The increase in MOR was 65.21% (2 mm), 80.69% (5 mm), and 56.36% (10 mm), respectively. Moreover, the TS of the fiberboards increased with increasing of CF contents and lengths.

7. Fiberboards containing CFs of 5 mm in length exhibited better electrical conductivity and shielding performance than those of 2 and 10 mm in length, between the percolation threshold and steady-state value of CF content.

ACKNOWLEDGEMENTS

The authors thank the National Special Funds for Scientific Research on Public Welfare of Forestry (NO. 201104004) for supporting this paper. Additional thanks to Professor Z. Cai (USDA Forestry Products Laboratory) and Neil Gribbins (USDA Forestry Products Laboratory) for their comprehensive review and suggestions for enhancing this paper. We also greatly appreciate Professor Youming Yu (Zhejiang Agriculture & Forestry University) for his guidance in the microstructure analysis.

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Article submitted: March 31, 2015; Peer review completed: June 10, 2015; Revised version received and tentatively accepted: July 9, 2015; Further revision and final acceptance: July 23, 2015; Published: August 5, 2015.
DOI: 10.15376/biores.10.4.6348-6362