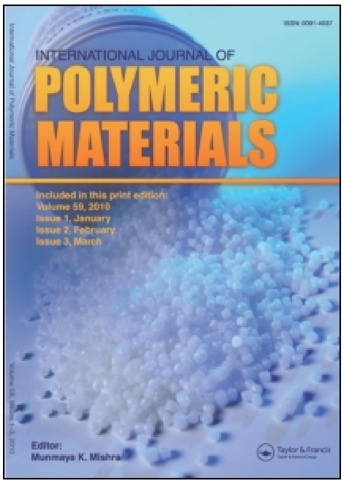

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Durability of Wood Flour-Thermoplastic Composites under Extreme Environmental Conditions and Fungal Exposure

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The main purpose of this research work was to investigate the response of wood flour reinforced polyethylene composites to 2 hours water boiling, five complete boiling and freezing cycles and fungal (Gloeophyllumm Traebum/Brown-rot fungus) exposure. Five composite formulations were manufactured and analyzed (0, 50, 60, 70 and copper carbonate treated 60% wood flour/polyethylene composites). The results showed that wood flour loading decreased the resistance of the composites to moisture and fungal environment. The exposure of the composites to 2 hours water boiling and five complete boiling and freezing cycles caused serious damage to the interfacial adhesion between wood flour and polyethylene matrix due to contraction and swelling stresses developed during cyclic exposure. The addition of 1 percent copper carbonate salt during compounding of wood flour and polyethylene prevented the colonization and proliferation of fungus on the surface of the composites, but had a negative effect on the water uptake and flexural properties of the composites.

Keywords: compression molding; durability; accelerated aging; polyethylene; wood flour

INTRODUCTION

Wood fiber-plastic composites are relatively new class of materials which are finding applications in various industries particularly in automotive parts as interior components, and more recently as non-structural building components, as well as substitutes for treated wood in exterior environments. The performance of wood under extreme

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environmental conditions is well documented. However, there is no long-term experience with wood fiber-plastic composites since their industry use is less than two decades old. In terms of field performance and biodegradation very limited research has been conducted on wood fiber-plastic composites [1–5]. Some concerns have been raised about the long-term field performance and weathering of wood fiber-plastic composites by Brooks and Beathy [6]. They suggested that areas of concern include response of these composites to accelerated aging and weathering. Expanding commercial production and marketing of wood fiber-plastic composites for outdoor applications have made an urgent issue for research concerning the performance of wood fiber plastic composites under extreme environmental conditions and microbial environment. The main objective of the present research project was to assess the resistance and performance of wood flour-polyethylene composites to physical and biological environments, and the effectiveness of copper carbonate salt in improving the biological resistance of wood flour polyethylene composites. Severe environment conditions were simulated by exposure of the composites to 2 hours water boiling, and five complete boiling and freezing cycles while biological environment was simulated by exposure of the composites to fungal environment.

EXPERIMENTAL

Materials

High density polyethylene (HDPE), Sclair 2909 from Dupont Canada Ltd. (density 0.960 g/cm3; melt flow index 13.5) was selected as matrix polymer. This polymer was selected because it can be processed below the decomposition temperature of lignocellulosic materials (about 220°C), it is widely used and disponsible at low cost.

Commercial wood flour (60–80 mesh fraction) with a moisture content of 7.3 percent, supplied by Northern Fibres Ltd, was used in the study as reinforcing agent.

Maleated polyethylene, Fusabond MB-110D (manufactured by Dupont Canada Inc.) was used as processing aid, to help the dispersion of fibers in the polymer matrix, to prevent the absorption of water in the fibers, and to improve the interfacial adhesion between the fibers and the matrix polymer.

Copper carbonate salt was used as a biocide to enhance the durability of the composites under fungal exposure. Copper carbonate salt has been reported to have an excellent biocide property [7].

Composites Manufacturing

Compounding

Wood flour were compounded with polyethylene matrix in the high intensity thermokinetic mixer (K-mixer, Werner and Pfleiderer Gelimat G-1) running at tip speed of 3,300 rpm, and automatically discharged at a pre-set temperature of 190°C. The amount of fibers in the compound was 0, 50, 60 and 70 percent by weight. About 5 percent of fusabond, based on wood flour weight, was used during blending. One of the main advantages of the Gelimat mixer is its ability to obtain optimum dispersion of fibers in a short time. High speed shearing action by the rotation of the rotor blade quickly brings the plastic to its melting point. The system does not require any external heating source other than heat generated by kinetic energy [8]. Copper carbonate salt was incorporated during compounding at 1 percent based on the total weight of the compound.

Compression Molding

Blends containing wood flour and polymer were compression molded in a laboratory Wabash hot press of 600 mm × 600 mm. The compounded material from the K-mixer was immediately hot pressed between the two hot platens maintained at 200°C. Mylar sheets were used to protect the metal platens and provide smooth flow surface. Steel bars were used to determine the thickness of the compressed panels. The compressed material was allowed to heat up for one minute at 10 tons of pressure. The sandwiched material thus obtained was removed from the press and masticated to thoroughly homogenize the material. After the mastication, the material was put back in the press and hot pressed for one minute at 10 tons, then for one minute more at 50 tons. After this period, the press was cooled to 50°C, the pressure was released and the compressed material was taken out of the press. Molded panels were cut to 1/2 inch wide and 5 inches long samples.

The samples were allowed to equilibrate at laboratory condition for two days. Compression molding was used in this study because of the difficulty that might occur in injection molding blends containing higher weight loading of wood flour.

2 Hours Water Boiling Test

Molded composites were subjected to 2 hours boiling water immersion following ASTM D 570. Molded samples were conditioned in an oven for 24 hours at 60°C, weighed and immersed. At the end of 2 hours boiling, the samples were removed from water, blotted with tissue paper to remove the excess water on the surface and weighed.

Boiling and Freezing Test

The boiling and freezing tests consisted of subjecting the molded samples to five complete boiling and freezing cycles. Each complete cycle consisted of the following three steps: step A-boiling in water for 2 hours, step B-freezing at −20°C for a week, and step C-soaking in water for a week at room temperature. The weight changes of molded samples were monitored at the end of each cycle. All samples were first conditioned according to ASTM D 570.

Decay Resistance Test

The decay tests were conducted according to ASTM G21 and ASTM D2017 with minor modifications. The tests with brown-rot fungus (Gloeophyllum Trabeum) were performed with molded composites in standard petri dishes over a period of 12 weeks at 28°C and 85% relative humidity. Molded specimens were conditioned in an oven for 24 hours at 100°C, boiled in water for 2 hours, and immersed in cool water for 24 hours. After immersion, the specimens were autoclaved at 250°C for 20 minutes and placed in sterile petri dishes containing solidified nutrient malt agar. The samples were then inoculated with the fungus culture at least in three different places; and incubated for 12 weeks. The only carbon source for the growth of the fungus was from the molded samples. The fungus growth was monitored by visual observations for the first four weeks, and the resistance of the composites was assessed by determining the weight and flexural properties changes after 12 weeks of incubation. The observed growth on samples were judged according to ASTM as follows: (0 = none); 1 = traces of growth (less than 10%); 2 = light growth (10–30%); 3 = medium growth (30–60%); and 4 = heavy growth (60% to complete coverage).

Mechanical Test

Flexural properties (3-point bending) of molded samples were determined before exposure, and after 2 hours water boiling, five complete boiling and freezing cycles, and 12 weeks fungal exposure. The flexural test was conducted according to ASTM D790 with a standard computerized Instron Tester (Sintech, Model 20) at the test speed of 2.8 mm/min. and a span of 4 inches. The results were the average of six specimens.

RESULTS AND DISCUSSION

Water Uptake After 2 Hours Water Boiling

Figure 1 shows the water uptake or water absorption of molded wood flour reinforced polyethylene composites as a function of wood flour loading when the composites were immersed in boiling water for 2 hours. The water uptake of the composites increased with increasing wood flour loading. The weight gain after 2 hours water boiling was 3% for composites containing 50 percent wood flour, 5% for composites with 60 percent wood flour, and 9% for composites with 70 percent wood flour. The increase in water uptake with wood flour loading may be explained by the poor encapsulation of the wood flour by the polymer matrix which acts as a barrier to water penetration to the system when the wood flour content is increased. However, the susceptibility of the wood flour reinforced polyethylene composites to absorb water is largely due to the formation of hydrogen bonds formed between the hydroxyl groups of wood flour and water.

The addition of a small amount of copper carbonate in the composites had a deleterious effect on the resistance of the composites to

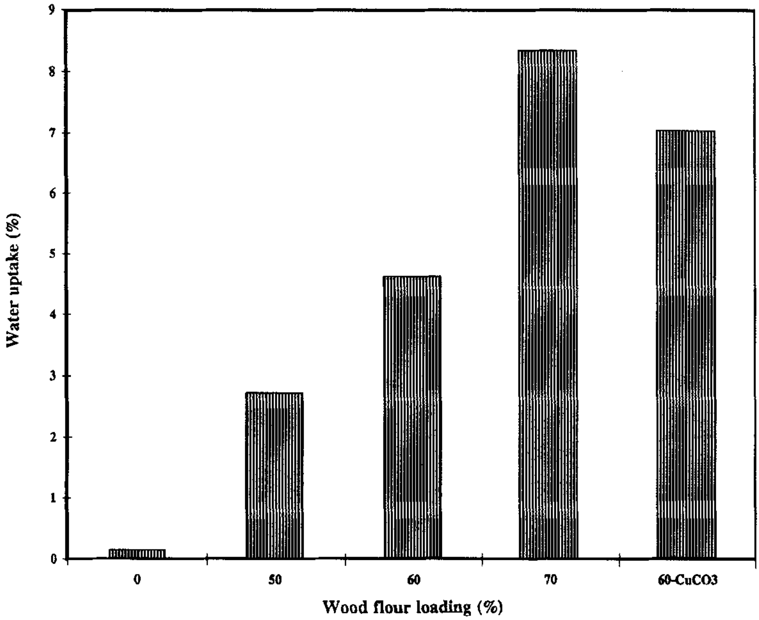

FIGURE 1 Water uptake of wood flour reinforced polyethylene composites after 2 hours boiling water.

moisture environment. Composites treated with copper carbonate absorbed more water than untreated composites at the same level of wood flour loading. The detrimental effect of copper carbonate on the water uptake of the composites may be explained by the poor miscibility of copper carbonate with other components in the system. It is thought that copper carbonate might have retained partial integrity as distinct phase, thus contributing to the degradation of the interfacial adhesion between wood flour and polyethylene matrix.

Water Uptake After Five Boiling and Freezing Cycles

The water uptake of wood flour reinforced polyethylene composites after five complete boiling and freezing cycles is shown in Figure 2. The water uptake increased gradually with the number of cycles up to the fourth cycle. After the fourth cycle the rate of water uptake decreased. The decrease in the rate of moisture uptake after the fourth

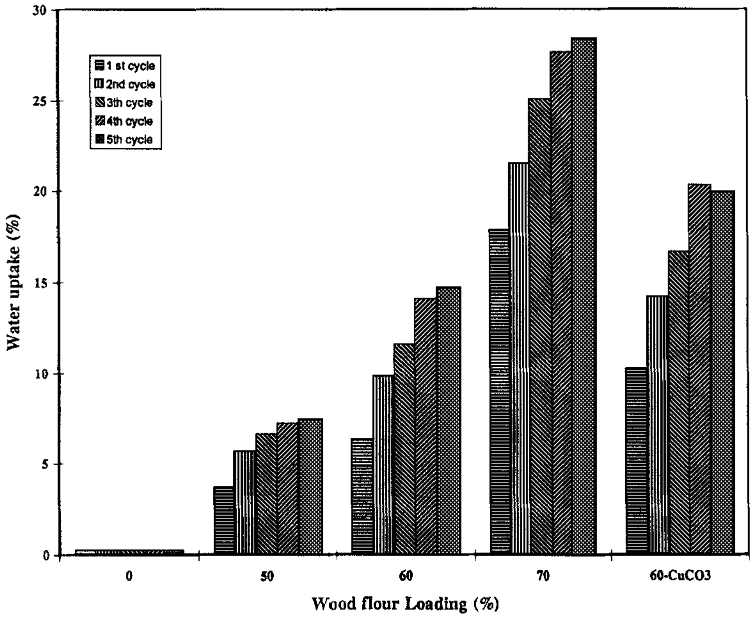

FIGURE 2 Water uptake of wood flour reinforced polyethylene composites after five complete boiling and freezing cycles.

cycle may be explained by the fact that the composites had reached a point of saturation, thereby limiting the capillary action of water in the composites. The water uptake increased substantially with increasing wood flour loading. The water uptake at the end of the five cyclic exposures was approximately 7% for composites with 50 percent wood flour, 15% for composites with 60 percent wood flour, and 28% for composites with 70 percent wood flour. The high moisture uptake of wood flour reinforced polyethylene composites containing 70 percent wood flour may have a detrimental effect on the resistance of the composites to microorganisms such as fungi. Levy [9] has indicated that the growth of microorganisms on wood or wood based composites surface depends on adequate amount of moisture. A moisture content around the fiber saturation point (25 – 30%) is often adequate for the proliferation of microorganisms and subsequent decay of wood based materials. These results suggest that to reduce the susceptibility of wood flour reinforced polymer composites to microbial degradation, it is desirable to keep the level of wood flour low in order to prevent moisture uptake above 20% of the dry weight of wood or wood based composites.

The treatment of wood flour with copper carbonate had a negative impact on the resistance of the composites to boiling and freezing exposures. At the end of the five complete cycles, the treated composites absorbed more than 19% water compared to 14% water for untreated composites at the same wood flour loading.

Decay Resistance

Table I summarizes the results of the biological exposure of wood flour reinforced polyethylene composites. There was no fungal growth on pure polyethylene during the first four weeks observations. The absence of any traces of fungal growth on pure polyethylene surface is mainly due to the absence of organic components in plastic materials and also to the low moisture holding of polymers, less than 5 percent, which is far below the adequate moisture for vigorous fungal growth.

The colonization of the composites by fungus increased with wood flour loading. After four weeks exposure, composites containing 50 percent wood flour were only slightly colonized by fungus while composites with 60 and 70 percent were completely covered by fungus. The treatment of the wood flour reinforced polyethylene composites with copper carbonate was effective in reducing the fungal colonization on the surface of the composites. After four weeks exposure, the treated

TABLE I Fungal growth on wood flour (WF) reinforced polyethylene (PE) composites

Composites	*Visual rating*				*Average weight loss* (%)
	First week	*Second week*	*Third week*	*Fourth week*	*Twelfth week*
Pure Polyethylene (PE)	0	0	0	0	0.0
50% WF/50% PE	1	1	1	1	+0.82
60% WF/40% PE	1	2	3	4	+0.32
70% WF/30% PE	2	3	4	4	−0.07
60% WF/40% PE/1% $CuCO_3$	0	1	1	1	−0.46

Visual observations according to ASTM (0 = none; 1 = traces of growth (less than 10%); 2 = light growth (10–30%); 3 = medium growth (30–60%); and 4 = heavy growth (60% to complete coverage).

composites were less than 10% covered by fungus compared to 60% to complete coverage for untreated composites at the same wood flour loading.

Table I shows also the average weight losses of the composites after twelve weeks exposure. As can be seen, no significant weight losses were observed. The slight weight-gain observed during exposure of the composites containing 50 and 60 percent wood flour may explain the high resistance of wood flour reinforced polymer composites to decay. The observed 0.07% weight loss in the case of the composites containing 70% wood flour may be ascribed to slight digestion of degraded organic components in wood flour. Nilson [10] suggested that Brown-rot fungus, which is a form of fungal decay caused by Basidiomycets, degrades the polysaccharide of wood through extensive depolymerization of cellulose and hemicelluloses and partial degradation of lignin at later and more advanced stages of decay. The weight loss of 0.46% observed after fungal exposure of copper carbonate treated wood flour-polyethylene composites may be due to leaching of copper carbonate during exposure.

The overall resistance of wood flour reinforced polyethylene composites to decay by brown-rot fungus (low weight losses) may indicate either the unfavorable conditions for vigorous fungal growth or decay in the petri-dishes chambers or the antimicrobial property of polymer matrix which encapsulate the fibers.

Flexural Properties

After 2 Hours Water Boiling

The flexural properties of the composites before and after 2 hours water boiling are shown in Figures 3 and 4. The flexural strength of the composites before exposure increased with increasing wood flour loading from 50 to 70%. The increase in flexural properties of the composites with wood flour loading may be attributed to the increase in available bonding area, and also to the greater homogeneity in fibers distribution. The moduli of the composites increased as the wood flour loading increased from 50 to 70%.

The exposure of the composites to 2 hours water boiling had a negative effect on the flexural strength of the composites. The flexural

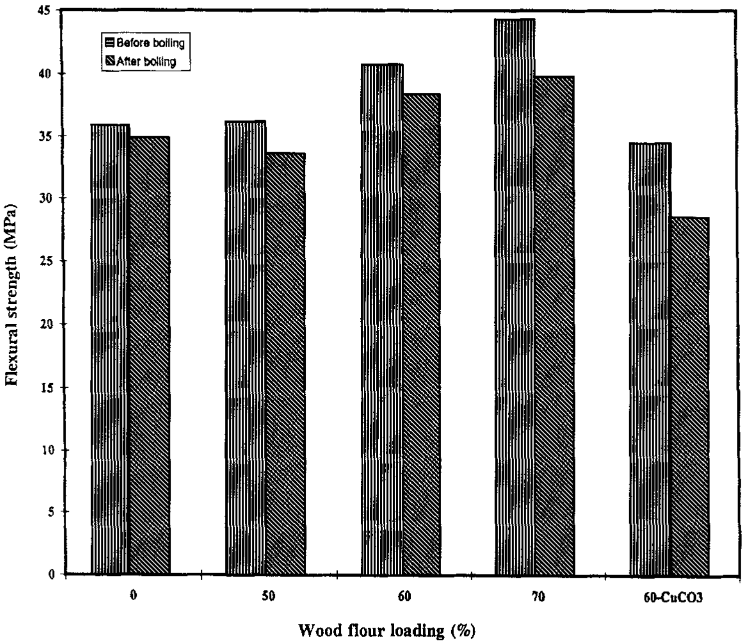

FIGURE 3 Flexural strength of wood flour reinforced polyethylene composites after 2 hours boiling water.

strength of the composites decreased by about 7% for composites with 50 percent wood flour, 6% for composites with 60 percent wood flour, 10% for composites with 70 percent wood flour, and 17% for copper carbonate treated composites with 60 percent wood flour. The reduction in the flexural modulus of the composites after 2 hours water boiling was greater than that in the flexural strength. The flexural modulus were reduced from original values by about 18% for composites with 50 percent wood flour, 26% for composites with 60 percent wood flour, 36% for composites with 70 percent wood flour, and 35% for copper treated composites. The loss in flexural properties of the composites after exposure to 2 hours water boiling may be due to the deterioration of the interface adhesion between wood flour and polyethylene matrix. It is well known that water and humidity are the main factors in offering multiple opportunities for structural degradation of interface. The decrease of the flexural properties of the composites after treatment with copper carbonate salt may be explained by the incompatibility of copper with wood and polymer. It appears that copper carbonate salt

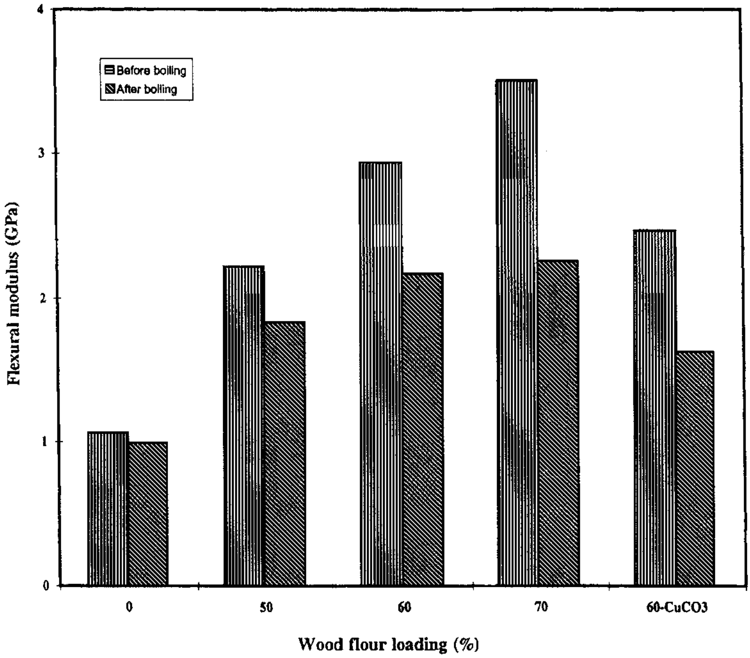

FIGURE 4 Flexural modulus of wood flour reinforced polyethylene composites after 2 hours boiling water.

tends to interface in the adhesion mechanisms between wood and polyethylene or acts as stress concentration points.

After Five Boiling and Freezing Cycles

The flexural properties of the wood flour reinforced polyethylene composites before and after the five complete boiling and freezing cycles are shown in Figures 5 and 6. The exposure of the composites to boiling and freezing cycles had a detrimental effect on the flexural properties of the composites. The flexural strength at the end of the complete five boiling and freezing cycles decreased by about 25% for composites containing 50 percent wood flour, 39% for composites with 60 percent wood flour, 59% for composites with 70 percent wood flour. The extent of flexural modulus reductions of the composites after the complete cycling was greater than that of flexural strength. There were approximately 40% for composites with 50 percent wood flour, 52% for composites with 60 percent wood flour, and 68% for composites with

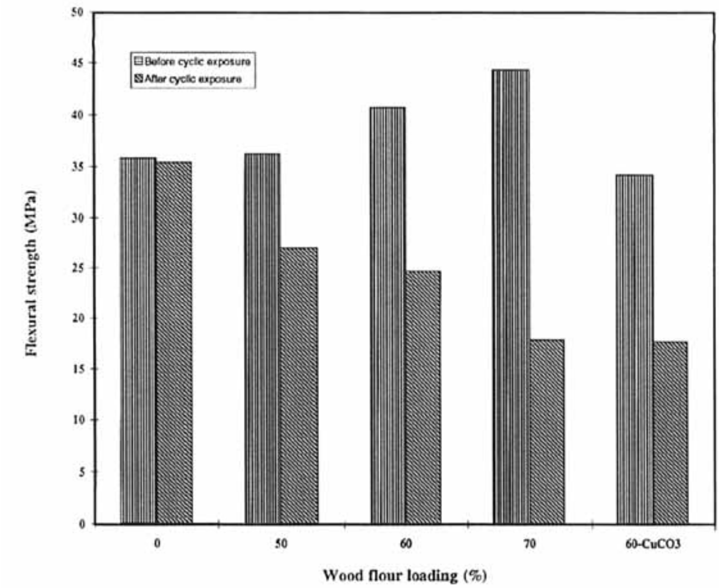

FIGURE 5 Flexural strength of wood flour reinforced polyethylene composites after five complete boiling and freezing cycles.

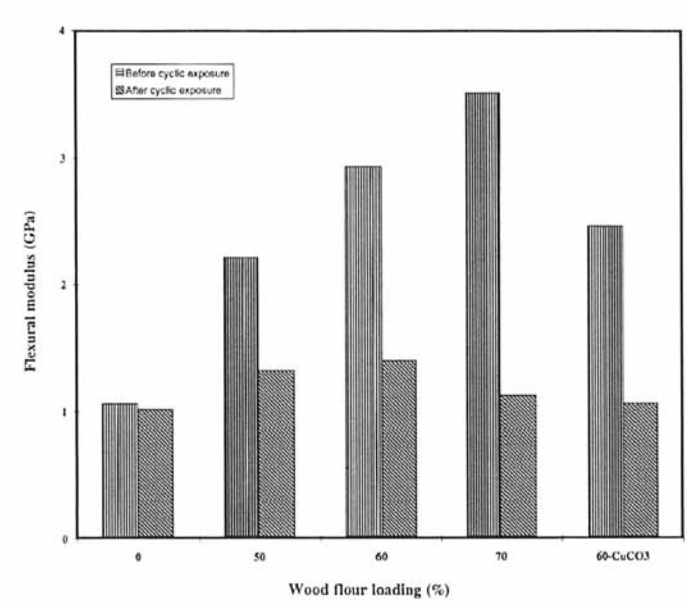

FIGURE 6 Flexural modulus of wood flour reinforced polyethylene composites after five complete boiling and freezing cycles.

70 percent wood flour. The observed reductions in the flexural properties of the composites may be the result of severe degradation of the interface adhesion between components due to contraction and swelling stresses developed during boiling and freezing exposures. The addition of copper carbonate in the composites had a negative effect on the resistance of the composites. The flexural strength of the treated composites was reduced by about 49% compared to 39% for the untreated composites at the same wood flour loading. The flexural modulus decreased by about 57% for the treated composites compared to 52% for the untreated composites.

After Fungal Exposure

The flexural properties of the composites after 12 weeks fungal exposure are shown in Figures 7 and 8. Compared to the exposure of the composites to five complete boiling and freezing cycles, the flexural properties of the composites were less affected after 12 weeks of fungal exposure. Except for composites containing 50 percent wood flour, the

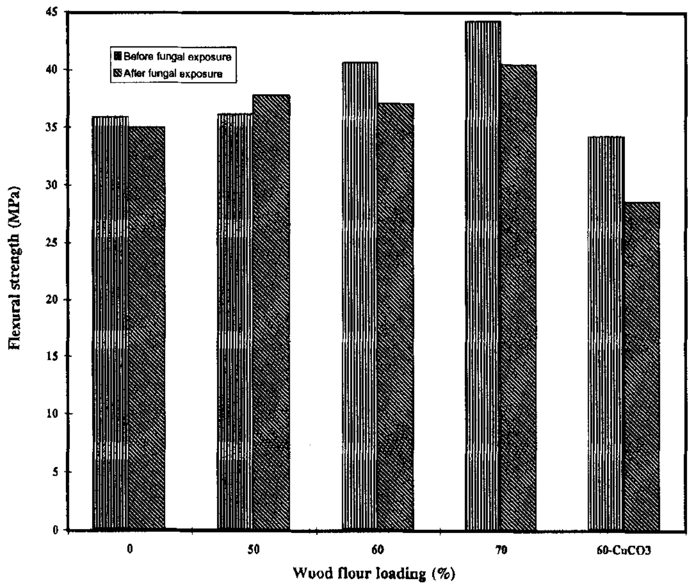

FIGURE 7 Flexural strength of wood flour reinforced polyethylene composites after 12 weeks fungal exposure.

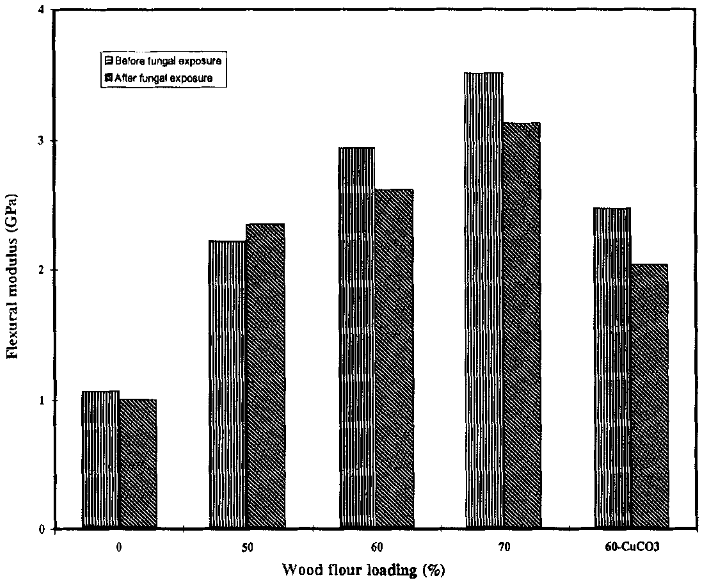

FIGURE 8 Flexural modulus of wood flour reinforced polyethylene composites after 12 weeks fungal exposure.

extent of reductions was about 9% for composites with 60 percent wood flour, 9% for composites with 70 percent wood flour, and 16% for treated composites. The loss in the flexural modulus of the composites was more pronounced. There were about 6% for composites with 50 percent wood flour, 10% reductions for composites with 60 percent wood flour, 11% composites with 70 percent wood flour, and 17% for treated composites.

CONCLUSIONS

The objective of this research work was to investigate the resistance of wood flour reinforced polyethylene composites to moisture and fungal exposure. In summarizing the results, the following conclusions may be drawn:

1. The susceptibility of wood flour reinforced polyethylene composites to moisture and colonization by fungus increased with increasing wood flour loading.

2. The water uptake of the composites was far greater after five complete boiling and freezing cycles than after 2 hours water boiling.
3. The flexural properties of the composites were more affected by cyclic exposure than either by 2 hours water boiling or fungal exposure.
4. The low weight losses observed in the wood flour reinforced polyethylene composites during fungal exposure indicate either the petri dishes chambers do not provide conditions favorable for decay or the antimicrobial property of polyethylene to brown-rot fungus.
5. The addition of copper carbonate to the composites was effective in preventing the infection of the composites by fungus during the first four weeks of incubation, but had a negative effect on water uptake and flexural properties of the composites.

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