Effect of Weathering Cycle and Manufacturing Method on Performance of Wood Flour and High-Density Polyethylene Composites

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Received 14 February 2005; accepted 17 May 2005
DOI 10.1002/app.23035
Published online in Wiley InterScience (www.interscience.wiley.com).

ABSTRACT: Wood–plastic lumber is promoted as a low-maintenance high-durability product. When exposed to accelerated weathering, however, wood–plastic composites may experience a color change and loss in mechanical properties. Differences in weathering cycle and composite surface characteristics can affect the rate and amount of change caused by weathering. In this study, 50% wood flour filled high-density polyethylene composite samples were injection molded, extruded, or extruded and then planed to remove the manufacturing surface characteristics. Composites were exposed to two accelerated weathering cycles in a xenon arc weathering apparatus. This apparatus exposed the samples to xenon arc radiation, which is a combination of UV, visible, and IR radiation that is similar to solar radiation. Composites were exposed to radiation with or without water spray. After exposure to radiation and water spray, composites with more wood component at the surface (i.e., planed samples) experienced a larger percentage of total loss in flexural modulus of elasticity and strength after weathering compared with the other composites. Composites exposed to radiation only did not experience as much change in properties as those exposed to radiation with water spray. The results of this study demonstrate that exposing wood–plastic composites to water spray in combination with radiation is more severe than exposing wood–plastic composites to radiation only. © 2006 Wiley Periodicals, Inc.* J Appl Polym Sci 100: 3131–3140, 2006

Key words: polyethylene; wood flour; extrusion; injection molding; weathering

INTRODUCTION

Wood-derived fillers have recently made inroads into the thermoplastic industry, primarily as wood–plastic composites (WPCs) become more prevalent in the construction industry. Exterior nonstructural building products made of WPCs, such as decking, fencing, siding, window framing, and roof tiles, are being introduced into the marketplace. For building products, it is predicted that approximately 500,000 T of WPCs will be used in North America in 2006.[1] Construction, transportation, industrial, and consumer applications for WPCs are also on the rise.

Wood–plastic lumber is being promoted as a low-maintenance high-durability product.[2] However, the use of WPCs by the construction industry has resulted in concern about the durability of these products after weathering. Several recent articles reported the weathering characteristics of polyethylene (PE)-based WPCs. When exposed to accelerated weathering, WPCs often fade[3–6] and lose mechanical properties.[3–7]

Although various assessments of WPC fading during weathering have been consistently reported, the quantitative rate of fading has not yet been determined. The lightness of 50% wood flour (WF)-filled high-density PE (HDPE) has been measured after accelerated weathering. Some researchers have reported that the majority of lightening occurs in the first 700 of 1500 h of weathering,[3] whereas others have shown that lightening continues through 2000 h of weathering.[4]

Even more disparity can be found in the literature in regard to mechanical properties. Some studies have shown that, after accelerated weathering, both the flexural modulus of elasticity (MOE) and strength of 50% WF-filled HDPE composites decreased during the first 1000 of 2000 h of total exposure time.[5] Others have shown that, for the same formulation, the flexural MOE and strength change very little during the first 1000 h of accelerated weathering and the majority of changes occurs during the second 1000 h.[4] This may

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Journal of Applied Polymer Science, Vol. 100, 3131–3140 (2006)
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be caused by differences in the accelerated weathering cycles of the various test methodologies as well as differences in WPC surface characteristics.

During accelerated weathering in the laboratory, variables can include exposure time, UV intensity, temperature, humidity, and water exposure. Each variable must be considered before comparing the results from different studies. A change in properties of WPCs after accelerated weathering has been frequently reported after a given exposure time,[3–7] but time of exposure may often be meaningless when comparing studies if the severity of exposure is not identical. To allow for better comparison between studies, it is recommended that the performance after weathering be reported after a specific radiant exposure, the time integral of irradiance.[8] In the current WPC weathering literature, the radiant exposure is not known; therefore, comparisons between studies are not meaningful. In addition, running accelerated weathering cycles with and without water spray can influence the performance of WPCs.

The method of manufacturing WPCs affects the composite surface characteristics.[6,9] This can influence weathering performance. Injection molding, compression molding, and extrusion are processing methods commonly used for manufacturing WPCs. The parameters that are affected by processing methods include the temperature, pressure, and flow. Processing methods and processing variables can both greatly influence the morphology and physical properties of the composite.

Clemons and Ibach[9] studied 50% WF-filled HDPE composites manufactured by extrusion, compression molding, and injection molding. After a 2-week water soak, the extruded composites absorbed the most moisture and injection-molded composites absorbed the least. The authors presumed that this result was caused by a polymer-rich surface layer and lower void content that was attributable to the higher density of injection-molded composites.

Stark and coworkers[6] studied injection-molded and extruded 50% WF-filled HDPE composites. Some extruded composites were also planed to remove surface characteristics. After examining the surfaces using Fourier transform IR spectroscopy, they found an increased presence of wood at the surface of the planed composites compared with the extruded and injection-molded surfaces. The manufacturing method, and its direct influence on surface characteristics, was again found to affect composite performance. Although the manufacturing method did not influence the final lightness after 3000 h of accelerated weathering, the samples with more wood at the surface became closer to their final lightness value after only 1000 h of weathering[6] compared with extruded and injection-molded samples. The mechanical properties followed a similar trend. The composites with more wood at the surface were closer to their final loss (3000-h exposure) in mechanical properties after the first 1000 h of weathering[6] compared with extruded and injection-molded samples.

Research to examine the influence of weathering cycles and composite surface characteristics on the weathering of WPCs has not been previously reported. This study had two main objectives: to understand how several aspects of the weathering cycle, such as exposure to radiation with water spray and to radiation only, influence the performance of WPCs and to determine how the manufacturing method interacts and itself affects the weathering of WF–HDPE composites exposed to the two weathering cycles.

EXPERIMENTAL

Materials

The materials used in this study were WF and HDPE. The WF was 40-mesh (0.420-mm openings) ponderosa pine supplied by American Wood Fibers (Schofield, WI). The HDPE was virgin material with a melt index of 0.72 g/10 min and a density of 0.963 g/cm^3 (Fortiflex A60-70-162, Solvay Polymers, Inc., Houston). A lubricant supplied by Struktol (TR016, density = 0.98 g/cm^3, Stow, OH) was also added to the extruded samples to aid in processing.

Processing

Injection molding

The WF was dried for 24 h at 105°C; HDPE and WF were then dry blended at 50% WF. Compounding was accomplished using a 32-mm Davis Standard (Pawcatuck, CT) twin-screw extruder to produce homogeneous WF–HDPE composite pellets. The melt temperature at the die was 200°C, and the melt pressure was 2.96 MPa. The pellets were dried at 105°C for at least 24 h just prior to injection molding into flexural bar test samples. The composites were injection molded using a 33-T Cincinnati Milacron (Batavia, OH) injection molder. The mold nozzle temperature was 204°C, and the injection pressure reached a peak of 12.4 MPa. The American Society for Testing and Materials (ASTM D 790) mold cavity used for the flexural samples was 3 × 12 ×120 mm.[10]

Extrusion

Extruded samples were run at the University of Maine Advanced Engineered Wood Composite Center (Orono, ME). The composite was 49 wt % WF, 8 wt % lubricant, and 43 wt % HDPE. A Davis Standard 94-mm twin-screw extruder was used with a die that produced a 30 × 140 mm radius edge profile. The die temperature was 180°C, and the die pressure was 2.1

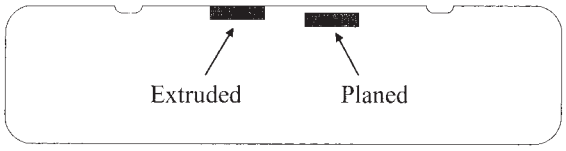

Figure 1 The extruded profile showing cutting sites for extruded and planed samples.

MPa. Flexural test samples (3 × 12 × 120 mm) were cut from deck boards (Fig. 1). The original extruded surface was left intact. The extruded samples included a lubricant to aid in processing, which may have migrated to the surface. This was not considered in comparisons between different responses to weathering cycles. However, this may have a minor influence on the initial property comparisons between injection-molded and extruded composites.

Planing

Less than 1 mm of the surface of the extruded samples was planed to remove any surface characteristics resulting from the extrusion processing method. Flexural test samples (3 × 12 × 120 mm) were cut from the deck board just below the original extruded surface (Fig. 1) to minimize any difference from the extruded samples associated with the board thickness.

Testing and analysis

Color measurement

A Minolta CR-200 Chroma Meter (Minolta Corporation, Ramsey, NJ) was used to measure color with the CIELAB color system. CIELAB is a three-dimensional color space that measures sample lightness (L) and color coordinates (a and b). The L value ranges between 0 and 100 (no reflected light and total light reflectance, respectively). An increase in L means the sample is lightening, and a decrease means the sample is darkening. The lightness was measured for five replicate samples.

Mechanical properties

Because WPCs can take long periods of time to equilibrate, samples were oven dried at 105°C for 24 h before testing. This ensured nearly the same moisture content for samples tested before and after exposure. Flexural tests were carried out according to ASTM D 790[10] on an MTS 810 Material Test System (MTS Systems Corp., Eden Prairie, MN). A three-point loading system was utilized with a crosshead speed of 1.3 mm/min, which corresponds with an outer fiber strain rate of 0.01 mm/mm/min. The exposed surface was placed away from the center load to place that part of the sample in tension. Five replicate specimens were tested for each formulation. The stress at maximum load and MOE were calculated according to ASTM D 790.[10]

Scanning electron microscopy

Molded surfaces were sputtered with gold and analyzed with a scanning electron microscope (JSM-840, JEOL USA, Inc., Peabody, MA) at a working distance of approximately 25 mm, a voltage of 15 kV, and a probe current of 1×10^{-9} A.

Weathering

The two accelerated weathering cycles to which the samples were exposed are shown in Table I. The composite samples were placed in a xenon arc exposure apparatus filtered with borosilicate filters (Weather-Ometer 65-WT, Atlas Materials Testing Technology, Chicago, IL). The filtered xenon arc radiation is similar to solar radiation, consisting of UV, visible, and IR radiation. In this article, exposure to radiation refers to xenon arc radiation. Composites were exposed to either radiation with water spray or radiation only. Samples were mounted on a drum that rotated around the xenon arc bulb at 1 rpm. If a water spray cycle was used, a 2-h cycle consisting of 108-min radiation exposure and 12-min simultaneous water spray and radiation exposure was followed. For samples weathered without water spray, the radiation exposure was continuous throughout the duration of weathering. For both cases, an irradiance sensor was used to measure light intensity for wavelengths from 300 to 400 nm (XenoCal, Atlas Materials Testing Technology, Linsengericht, Germany). The irradiance was monitored, and voltage to the bulb was changed periodically to maintain a constant irradiance. Irradiance follows an inverse square law with distance. The irradiance the samples received (I_2) was calculated using eq. (1):

$$I_2 = I_1\left(\frac{D_1}{D_2}\right)^2 \quad (1)$$

TABLE I
Points at Which Composite Samples Were Analyzed During Two Different Weathering Cycles

Weathering cycle	Radiant exposure[a] (J/m^2)	Water spray cycles	Time (h)
1	42	500	1000
1	81	1000	2000
1	122	1500	3000
2	41	—	1027
2	81	—	1991
2	123	—	2985

[a] The irradiance at 300–400 nm integrated with time.

where D_1 and D_2 are the distances to the sensor and the samples, respectively, and I_1 is the irradiance measured by the sensor. The equation assumes that the irradiance source is a point source. Although this is not the case in a xenon arc weathering apparatus, it does provide some correction for the different distances to the sensor and samples. The radiant exposure, or amount of light energy to which the samples were subjected, was then determined (irradiance integrated over time). To understand how properties change during a weathering cycle, samples were removed periodically for analysis. Monitoring the radiant exposure is preferable to monitoring the exposure time. Therefore, to allow for an improved comparison between weathering cycles, both the radiant exposure and exposure time were recorded (Table I).

Statistics

To determine the effects of weathering on the final properties, an analysis of variance was carried out to identify significant differences between the means at $\alpha = 0.05$. This was accomplished using Design Expert 6.0.0 software by Stat-Ease, Inc. (Minneapolis, MN).

RESULTS AND DISCUSSION

Unexposed samples

Micrographs of the surfaces of injection-molded, extruded, and planed composites are shown in Figure 2. The surface of the injection-molded composite [Fig. 2(a)] was relatively smooth, and polymer flow over wood particles was evident. The surface of the extruded composite [Fig. 2(b)] had many areas where the polymer failed to encapsulate the wood particles. The lower processing pressure and temperature during extrusion did not permit the polymer to flow at the surface of extruded composites as well as the processing conditions for injection-molded composites. Planed composites also had a relatively smooth surface [Fig. 2(c)]. However, it is visually apparent that wood fibers were exposed when the original extruded surface was planed. In a previous study, Fourier transform IR spectroscopy was used to characterize the composite surface.[6] A wood index was calculated that related the peak corresponding to hydroxyl groups with the peak corresponding to methylene groups. It was shown that the planed composites had more wood influence at the surface, followed by the extruded and injection-molded composites.[6]

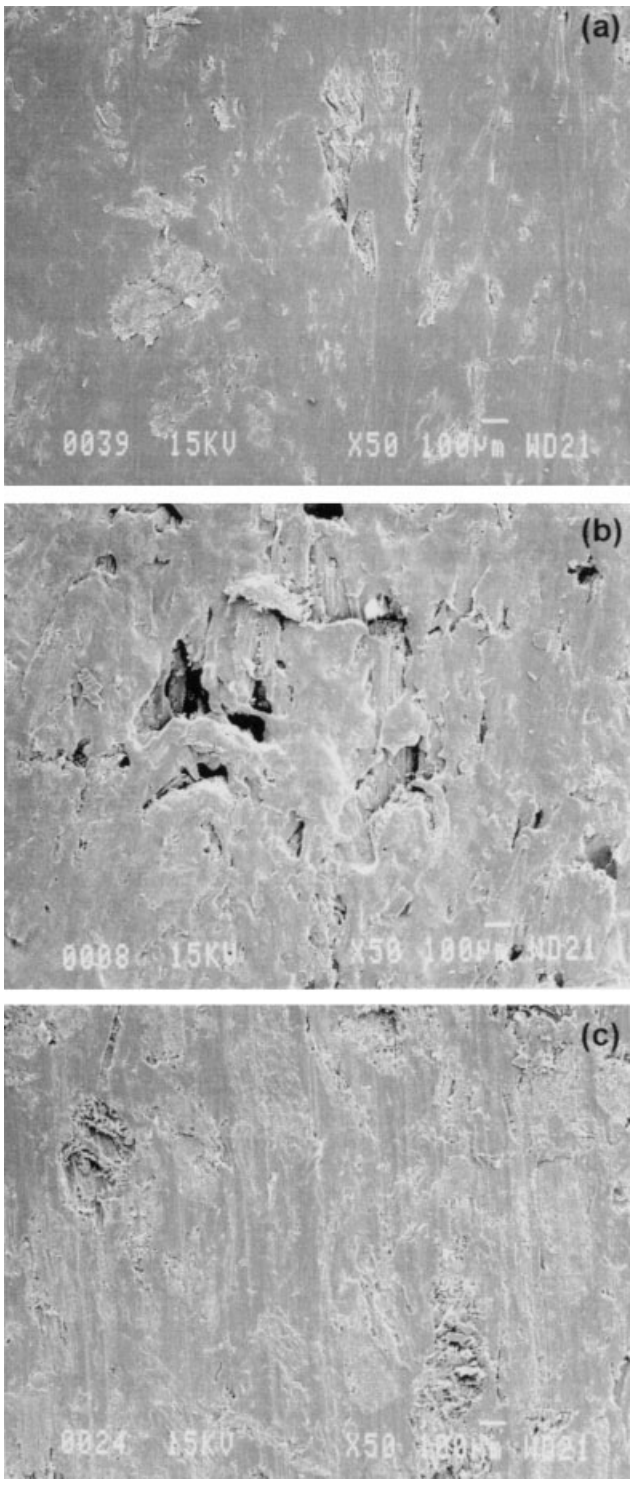

Figure 2 Scanning electron micrographs showing surfaces of (a) injection-molded composites, (b) extruded composites, and (c) planed composites.

Exposed samples

Figures 3–8 show the changes in composite properties as a function of radiant exposure throughout the weathering cycle. For each figure, the data points represent the average of five replicates and the error bars represent 1 SD. Table II shows the percentage changes in properties after the full weathering period.

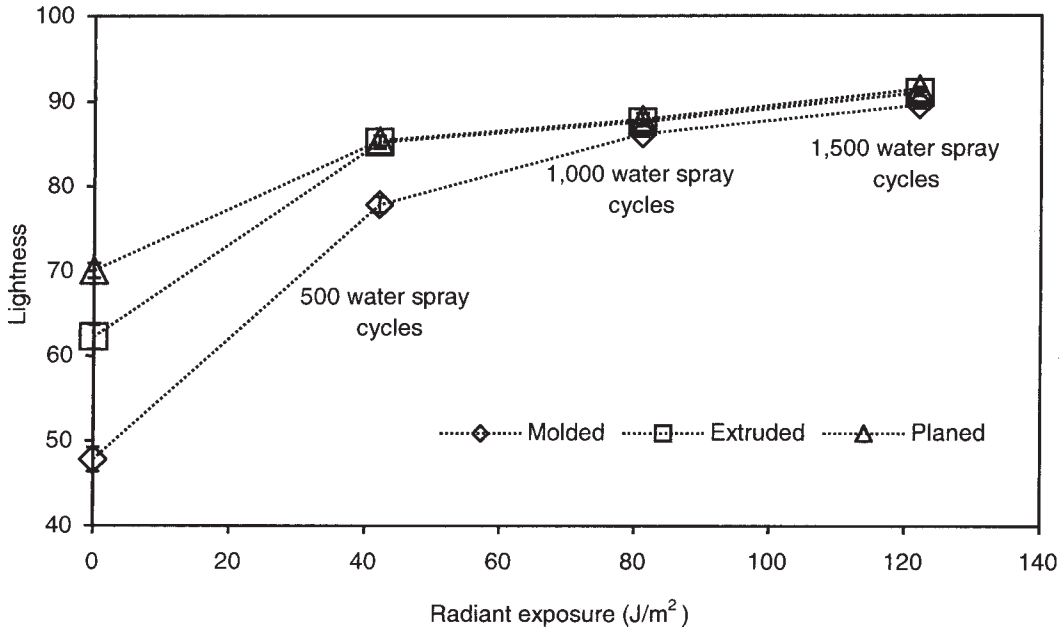

Figure 3 The lightness (*L*) as a function of radiant exposure during exposure of WF–HDPE composites to UV light with water spray. Error bars represent ±1 SD about the average.

Lightness

The color of WPCs is primarily reflective of the color of the wood component. The color of wood is dependent upon the thermal degradation imposed during high-temperature processing and the eventual interactions of each component with light. In virgin wood, light is not absorbed by cellulose or hemicellulose and lignin is pale yellow. Therefore, in virgin wood, the majority of colored substances are extractives.[11] Accordingly, the mechanisms leading to color change in wood during weathering are not well understood.

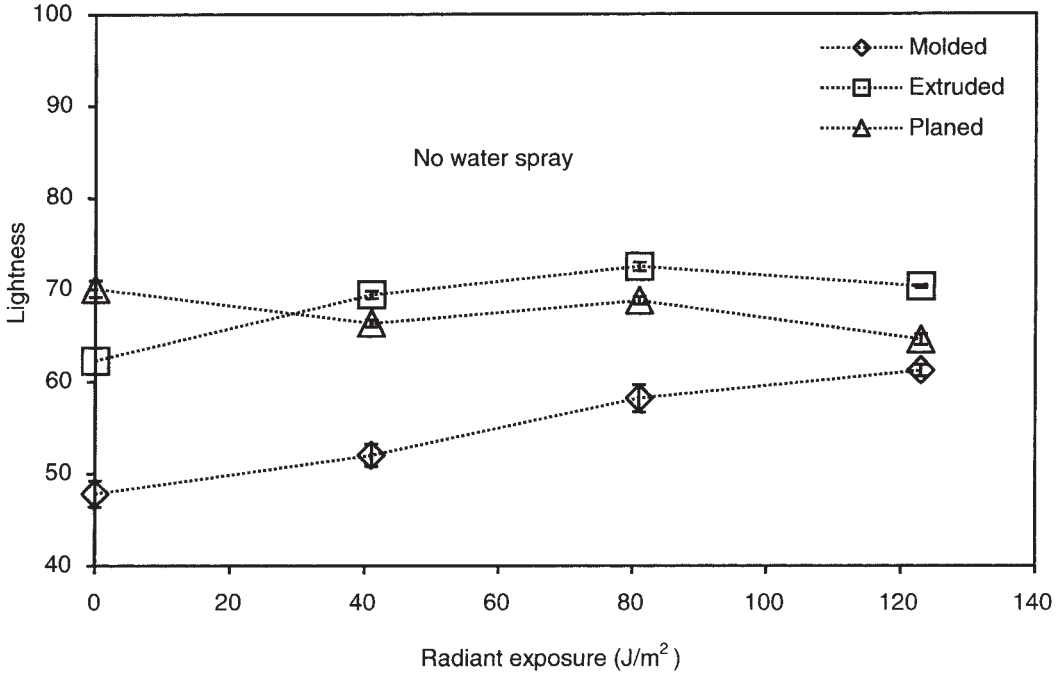

Figure 4 The lightness (*L*) as a function of radiant exposure during exposure of WF–HDPE composites to UV light. Error bars represent ±1 SD about the average.

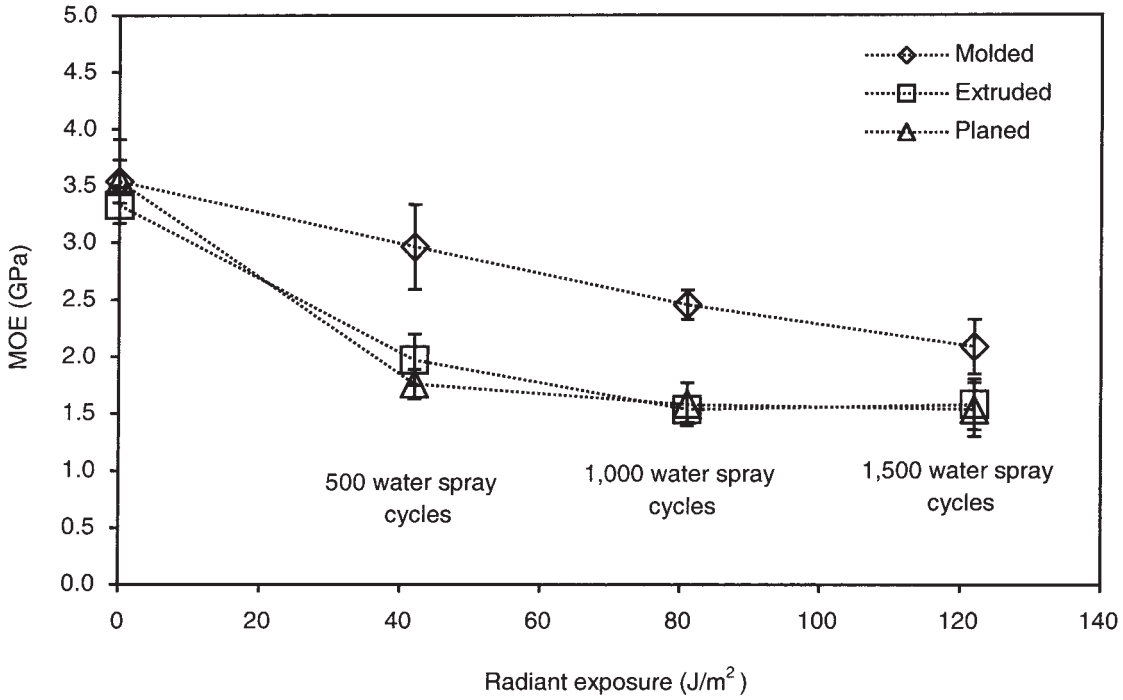

Figure 5 The modulus of elasticity (MOE) as a function of radiant exposure during exposure of WF–HDPE composites to UV light with water spray. Error bars represent ±1 SD about the average.

Initially, the injection-molded composites were the darkest. The processing temperature was higher for injection-molded composites (204°C), which resulted in a darker composite because the wood, especially hemicelluloses, began to degrade. Compared with the extruded composites, the planed composites were more faded (lighter). The polymeric surface layer of extruded composites effectively wet the WF, transmit-

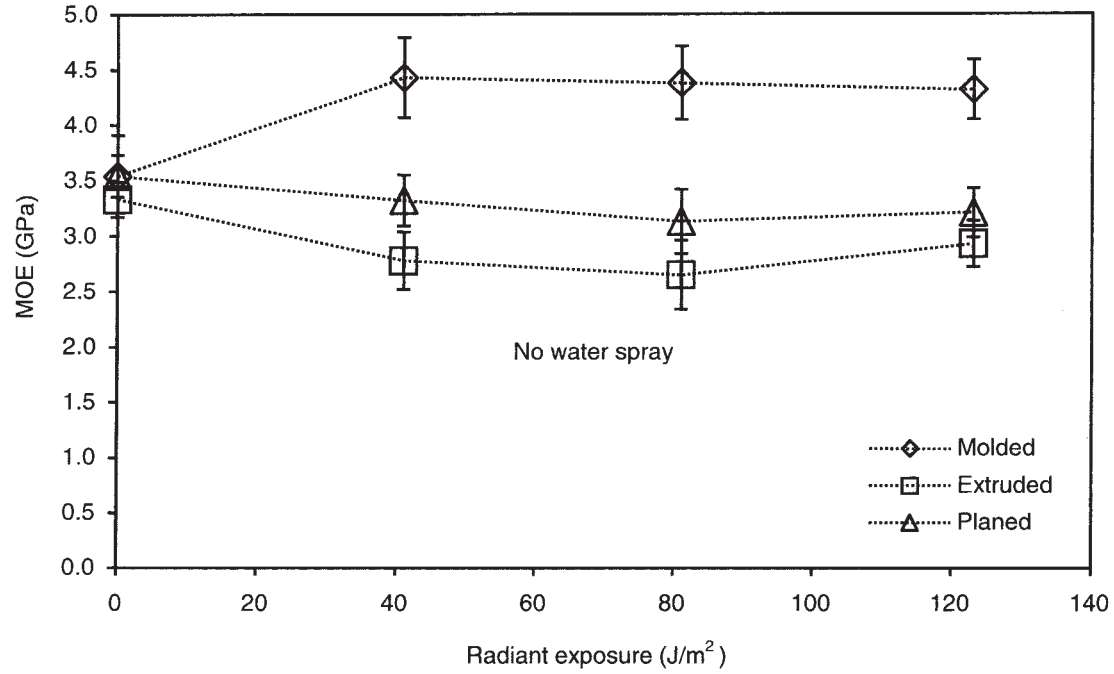

Figure 6 The modulus of elasticity (MOE) as a function of radiant exposure during exposure of WF–HDPE composites to UV light. Error bars represent ±1 SD about the average.

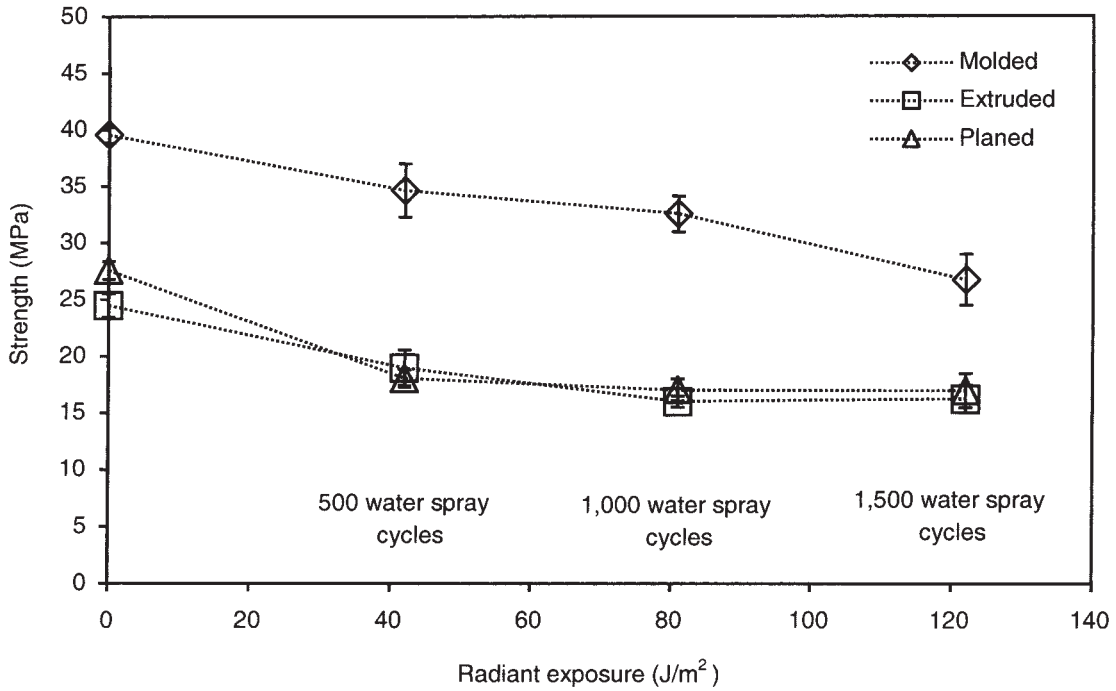

Figure 7 The strength as a function of radiant exposure during exposure of WF–HDPE composites to UV light with water spray. Error bars represent ±1 SD about the average.

ting light deeper in the wood cell, resulting in apparent darkening of the wood.[11]

The weathering effects on composite lightness (*L*) after exposure to radiation with water spray and radiation only are shown in Figures 3 and 4, respectively. Exposure to radiation with water spray clearly resulted in composite lightening (Fig. 3). Regardless of the manufacturing method, the greatest increase in *L*

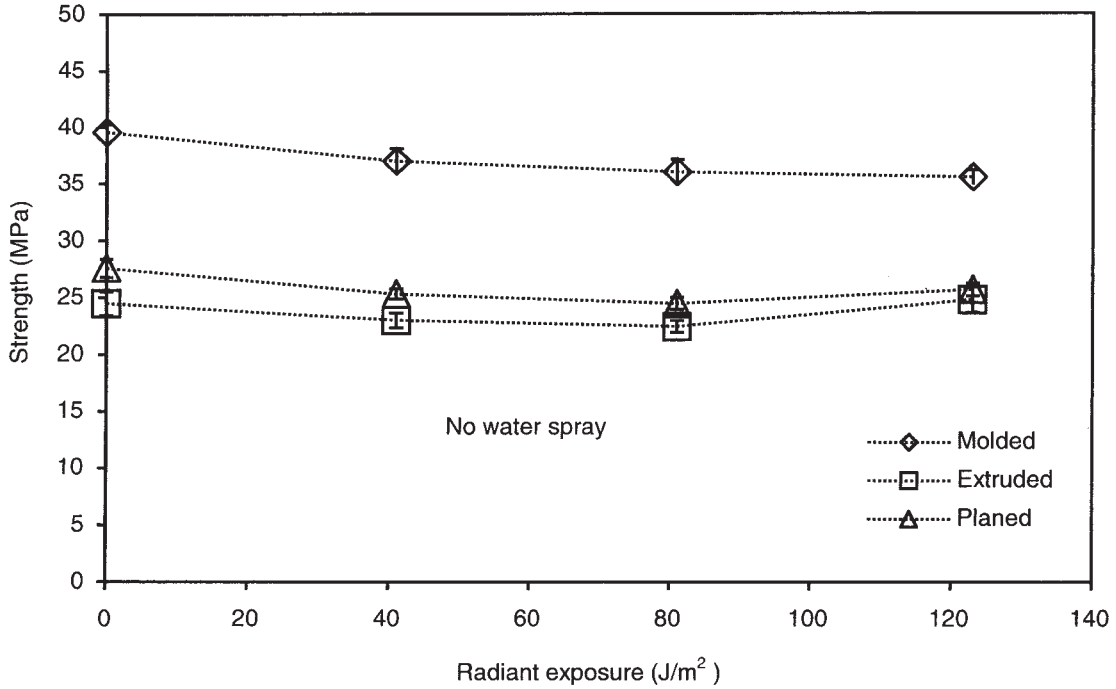

Figure 8 The strength as a function of radiant exposure during exposure of WF–HDPE composites to UV light. Error bars represent ±1 SD about the average.

TABLE II
Percentage Change in Property after Weathering with Radiation and Water Spray or Radiation Only

Composite type	ΔL (%)		ΔMOE (%)		Δ Strength (%)	
	Radiation + H_2O	Radiation only	Radiation + H_2O	Radiation only	Radiation + H_2O	Radiation only
Molded	87	28	−41	22	−32	−10
Extruded	46	13	−52	−12	−34	1[a]
Planed	31	−8	−57	−9[a]	−38	−7

[a] Differences between two means are not significant at $\alpha = 0.05$.

occurred in the initial stages of weathering and all composites reached a similar lightness after weathering. After exposure to approximately 40 J/m^2 and 500 water spray cycles, however, planed and extruded composites were closer to their final lightness than were injection-molded composites. Table II shows the total change in lightness as a percentage value. Because all three WPCs reached similar final lightness values, the initially darker injection-molded composites lightened at a higher rate of change.

The injection-molded and extruded composites also lightened after exposure to radiation only (Fig. 4). However, the planed composites darkened slightly. The change in lightness occurred throughout the entire weathering period, rather than the majority occurring during the initial weathering period. For each composite, the change in lightness was much less when the samples were exposed to radiation only. Table II shows that the lightness of the injection-molded composites increased 87% when exposed to radiation and water but only increased 28% when exposed to radiation. Similar observations can be made for the extruded and planed composites. This suggests that water spray has a large effect on composite fading.

Exposing wood to radiation degrades primarily the lignin component, leaving the cellulose at the surface, which may physically block radiation penetration. Exposure to a combination of radiation and water spray changes the mechanisms of water absorption. The degradation of hydrophobic lignin and increasing the amounts of hydrophilic cellulose at the surface have been shown to increase the surface wettability.[12] This may cause the surface to more readily absorb moisture. This is detrimental for two reasons. The first reason is that the presence of water in wood accelerates oxidation reactions that are a direct result of photodegradation. The second reason is that the wood cell walls swell when penetrated by water. This facilitates light penetration into the wood and provides sites for further degradation,[13] thereby accelerating the degradation effects.

The water spray cycle can also change the rate of discoloration of wood through physical mechanisms. Washing the degraded surface with water spray exposes new wood surfaces for further degradation. Therefore, weathering wood results in a cyclical erosion of the surface as the lignin is degraded and subsequently washed away, exposing more lignin to degradation. It can be quantitatively measured in wood. For example, the amount of degradation of ponderosa pine (*Pinus ponderosa*) after 10 years of outdoor exposure was 430 μm for late wood and 570 μm for early wood.[14] In addition, washing the surface can remove some of the extractives, which are the main components that impart color. The removal of the extractives is probably the main reason for the majority of color fade.

All these factors support the data, demonstrating that the presence of moisture and water spray cycles are critical factors in the lightening of wood and thus the lightening of WPCs, particularly in conjunction with exposure to radiation.

MOE

The flexural MOE decreased when the composites were exposed to radiation with water spray (Fig. 5). Although the initial MOE values for all the composites were similar, after weathering the MOE of the planed and extruded composites, which each have more wood exposed at the surface, decreased more than the MOE of the injection-molded composites (Table II). The decrease of the MOE after weathering was not a linear response. The MOE of each composite decreased more during the first exposure to 40 J/m^2 and 500 water spray cycles than during each subsequent exposure to 40 J/m^2 and 500 water spray cycles.

Exposing the WPCs to radiation only resulted in an increase in the MOE for the injection-molded composites, a small decrease in the MOE for the extruded composites, and no significant change in the MOE for the planed composites (Table II). The initial increase in the MOE of the injection-molded composites (Fig. 6) may have been caused by an increase in the crystallinity of the polymer-rich surface. It has been shown that, during the initial stages of accelerated weathering, the crystallinity of the polymer surface of injection-molded WF–HDPE composites increases.[15]

Previous reports demonstrated that moisture adversely affects the MOE of WPCs.[16–18] Injection-

molded HDPE composites filled with 40% wood fiber exposed to a water bath for 2000 h experienced 39% loss in flexural MOE.[16] Similarly, 30% WF–HDPE injection-molded samples lost approximately 20% flexural MOE after a 48-h water boil.[17] The large influence of water exposure on the MOE was expected, and there are several ways that moisture can influence the MOE of WPCs. When WPCs are exposed to moisture, the hydrophilic fiber swells. Cracks may form in the polymer matrix, which can contribute to increased water penetration into the composite[18] and a decrease in the MOE of the matrix material. The water spray also washes away the degraded surface layer. In this manner, the composite surface becomes increasingly vulnerable to further moisture penetration. Because wood at the composite surface provides an immediate pathway for moisture penetration into the composite, it was also expected that the composites with more wood at the surface (the planed composites) would be more susceptible to loss in the MOE after exposure to radiation with water spray.

Strength

The initial strength of the injection-molded composites was higher because of the increased density of the composite and the resulting increase in efficiency of stress transfer from matrix to fiber.[6] All composites exposed to radiation with water spray lost strength after weathering (Fig. 7). Similar to the trends observed for lightness and MOE, the strength decreased more during the first exposure to 40 J/m^2 and 500 water spray cycles than each subsequent exposure to 40 J/m^2 and 500 water spray cycles (Fig. 7). As expected, the planed composites lost a higher percentage of strength after exposure to radiation with water spray than the extruded and injection-molded composites (Table II). The composites with more wood at the surface were more susceptible to strength loss after exposure to radiation with water spray because of the increase in pathways for moisture penetration.

When the composites were exposed to radiation only, the strength of the injection-molded and planed composites decreased slightly but the strength of the extruded composites did not change significantly (Fig. 8, Table II). As with the change in flexural MOE, the change in strength continued through the duration of exposure to radiation. Exposure to radiation without water spray may not degrade the wood–polymer interface to such a degree that the flexural strength of the composite would be compromised.

Moisture penetration into the WPC seems to degrade the wood–polymer interface, which in turn causes a loss of strength as the stress transfer from matrix to fiber becomes less efficient. For example, when injection-molded HDPE composites filled with 40% wood fiber were exposed to a water bath for 2000 h, they experienced a 22% decrease in flexural strength.[16] The processing method can influence the amount of moisture the WPCs absorb, therefore influencing the amount of degradation to the wood–polymer interface. After 50% WF–HDPE composites were soaked for 2 weeks, the more dense injection-molded composites absorbed only 4% moisture whereas the less dense extruded composites absorbed 17%.[9]

During accelerated weathering, the composites were not continuously exposed to water spray. However, the increased wettability of the surface as a result of radiation exposure and surface erosion and the development of microcracks in the composite probably allowed for increased moisture penetration as weathering continued. Similar to the effects observed with the flexural MOE, the water spray can erode the surface, making new composite surfaces available for water penetration and photodegradation. As with the MOE, it was expected that the composites with more wood at the surface (the planed composites) would be more susceptible to loss in strength after exposure to radiation with water spray.

CONCLUSIONS

Each of the WPCs lightened more when they were exposed to radiation and water spray compared to radiation only. This suggests that water spray has a large effect on composite lightening. It is likely that several factors contribute to this. The presence of water both accelerates oxidation reactions and swells the wood cell wall, facilitating light penetration into the wood. The water spray also washes away the degraded layer and removes natural wood extractives.

In similar fashion, the changes in the MOE and strength were also much less when the composites were exposed to radiation only. This study showed that the majority of the loss in mechanical properties of WF–HDPE composites after weathering was caused by moisture effects. In addition to water absorption accelerating oxidation, the swelling of the wood cell wall compromises the interface between the wood and HDPE. Interfacial cracks contribute to the decrease in the MOE, and the less efficient stress transfer between the wood and HDPE contribute to the decrease in strength.

Differences in composite surface characteristics also affected the rate and amount of change in color fade and mechanical property loss caused by weathering. This study showed that composite performance improved and mechanical property loss was delayed when the composite had less wood at the surface. The wood provided readily accessible pathways for moisture to enter the composite, contributing to the destruction of properties.

The author gratefully acknowledges American Wood Fibers for supplying the WF.

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