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Effect of Processing Method on Accelerated Weathering of Woodflour-HDPE Composites

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Abstract

Wood-plastic lumber is promoted as being a low-maintenance, high-durability product. However, it has been shown that wood-plastic composites (WPC) exposed to accelerated weathering may experience a color change and/or loss in mechanical properties. Different methods of manufacturing WPCs lead to different surface characteristics, which can influence weathering. In this study, 50 percent woodflour-filled high-density polyethylene (HDPE) composite samples were either injection molded, extruded, or extruded and then planed to study material with surface effects removed. Different processing methods produced composites with different surface components. Fourier transform infrared spectroscopy was used to chemically show these differences. The surface of planed samples exhibited more of a wood component than extruded and injection molded composites, respectively. Samples underwent accelerated weathering in a xenon-arc weathering apparatus and were removed after 1,000, 2,000, and 3,000 hours of accelerated weathering. The samples were analyzed for color fade, and loss of flexural modulus of elasticity and strength. It was shown that final lightness (i.e., a measure of reflected light) of the samples after weathering was not dependent upon the manufacturing method. However, mechanical property loss due to weathering was dependant upon the manufacturing method. Composites with more wood component at the surface experienced a larger percentage of total loss in flexural modulus of elasticity and strength after weathering. This was likely due to the greater effect of moisture on composites with the more hydrophilic wood at the surface.

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Introduction

In recent years, wood-derived fillers have made significant inroads into the thermoplastic industry. Much of this has been due to the emergence of wood-plastic composites (WPC) in the construction industry. Exterior non-structural or semi-structural building products such as decking, fencing, siding, window framing, and roof-tiles are being introduced into the marketplace. It is predicted that for building products alone, approximately 1.1 billion lb. (500,000 tons) of WPCs will be used in North

America in 2006 (1). Construction, transportation, industrial, and consumer applications are also on the rise. Currently, nearly 70 percent of WPCs are polyethylene matrix composites (1). Polypropylene and polyvinyl chloride are also common matrix materials used by WPC industry.

Wood-plastic lumber is promoted as being 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 exposed to outdoor environments. Ultraviolet (UV) durability is of particular concern. It has been shown that WPCs exposed to accelerated weathering experience color change (3-8) and a loss in mechanical properties (3-6,8). Changes in mechanical properties after weathering can be due to a combination of factors such as composite surface oxidation, matrix crystallinity changes, and interfacial degradation due to moisture absorption (9).

While the effect of weathering on WPCs has been consistently reported, the rate of change has not. Samples of 50 percent woodflour-filled high-density polyethylene (HDPE) have been tested for color fade after accelerated weathering. Some have reported that the majority of color fade occurs in the first 700 hours of 1,500 total hours of weathering (4), while others have shown that color fade continues through 2,000 hours of weathering (6). Even more disparity can be found in the literature regarding mechanical properties. Some have shown that for 50 percent woodflour-filled HDPE the flexural modulus of elasticity (MOE) and strength decrease during the first 1,000 hours of 2,000 hours total exposure time (3). Others showed that for the same formulation, the flexural MOE and strength change very little during the first 1,000 hours of accelerated weathering and the majority of change occurs during the second 1,000 hours of weathering (6).

Injection molding, compression molding, and extrusion are processing methods commonly used in the manufacture of WPCs. Temperature, pressure, and flow are some parameters that can change based on processing method. Both processing methods and processing variables greatly influence morphology and physical properties of the composite.

The method of manufacturing WPCs also affects moisture absorption. Clemons and Ibach (10) looked at 50 percent woodflour-filled HDPE composites manufactured via extrusion, compression molding, and injection molding. It was reported that extruded composites absorbed the most moisture while injection molded composites absorbed the least (10). It was presumed that this was due to a polymer rich surface layer and lower void content attributable to the higher density of injection molded composites (10).

Mechanical properties of woodflour-filled polymer composites are negatively affected by moisture (10,12-16). When the composite is exposed to moisture, the hydrophilic fiber swells. Cracks may form in the polymer matrix, which can also contribute to subsequent penetration of water into the composite (12). Exposing wood-filled polymer composites to moisture results in a drop in flexural MOE and strength by degrading the wood-polymer interfacial quality (12-14). The amount of moisture absorbed can be influenced by woodflour content and wood particle size (12,15).

Despite known effects of manufacturing methods on moisture absorption and mechanical properties of composites, research has not been extensively carried out to examine the influence of composite production methods on UV degradation. This study had two main objectives:

1. to characterize the surface of 50 percent woodflour-HDPE composites produced using different manufacturing methods, and
2. to understand how manufacturing methods affect the weathering of woodflour-HDPE composites.

Fourier transform infrared (FTIR) spectroscopy was utilized to study surface characteristics of woodflour-HDPE composites manufactured using different processing techniques because of its effectiveness to analyze functional groups present at the surface of a material. FTIR methods have been extensively used to study weathered polyethylene (17-22). In addition, FTIR has been employed to study functional groups present in wood (23-25).

The results of the study reported here will aid in the development of an understanding of how processing methods affect color fade and mechanical properties of woodflour-HDPE composites after weathering.

Methods

Materials

The materials used in this study were wood flour (WF) and high-density polyethylene (HDPE).

The WF was a 40 mesh ponderosa pine supplied by American Wood Fibers (Schofield, WI). The HDPE was a virgin material with a melt index (MI) of 0.72 g/10 min. and density of 0.963 g/cm^3 supplied by Solvay Polymers, Inc. (Fortiflex A60-70-162, Houston, TX). A lubricant was added to aid in extrusion (Struktol TR016).

Processing

Injection molded

The WF was dried for 24 hours at 105°C (221°F), and then the composites were dry-blended at 50 percent WE 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 (392°F) and the melt pressure was 2.96 MPa (430 psi). The pellets were dried at 105°C (221°F) for at least 24 hours prior to injection molding into flexural bar test samples. The composite samples were injection molded using a 33-ton (30 metric tones) Cincinnati Milacron (Batavia, OH) injection molder. The nozzle temperature was 204°C (400°F), and the injection pressure reached a peak of 12.4 MPa (1,800 psi). The American Society for Testing and Materials (ASTM) mold cavity used for flexural samples is 120 by 3 by 12 mm (26).

Extruded

Extruded samples were run at the University of Maine's Advanced Engineered Wood Composite Center. The composition of the composite was 49 percent by weight WF, 8 percent by weight lubricant, and the remainder HDPE. A Davis Standard 94 mm twin-screw extruder was used with a die that produced a 1.2 by 5.5 in. radius edge profile. The die temperature was 180°C (356°F) and the die pressure was 2.1 MPa (300 psi). Flexural samples (120 by 3 by 12 mm) were cut from the same side of all the deck boards to be used for testing (Fig. 1). The extruded surface was left in tact.

Planed

The surface of extruded samples were planed to remove any surface characteristics due to the extrusion processing method. Samples were cut from the deck board just below the planed surface to minimize any difference from the extruded samples that may result from the thickness of the board **(Fig. 1).**

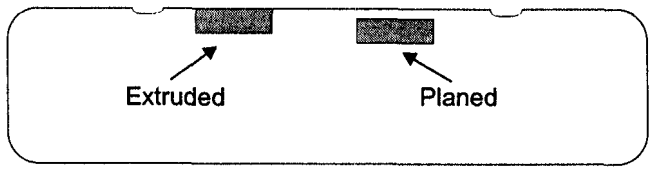

Figure 1. ~ *Extruded profile showing from where extruded and planed samples were cut.*

Testing and Analysis

Density

Unexposed samples were dried for 24 hours at 105°C (221°F) to remove moisture before being tested for density according to ASTM D792 (26). The sample size tested was approximately 60 by 3 by 12 mm. Twenty replicates for each processing method were tested.

Color measurement

A Minolta CR-200 Chroma Meter (Minolta Corporation, Ramsey, NJ) was used to measure color using the CIELAB color system. Lightness (L) was measured for at least five replicate samples. In the CIELAB color system, the value L can be thought of as a lightness factor. L represents reflectance of a sample. An L of 0 means the sample does not reflect light; an L of 100 means the sample reflects 100 percent of light. An increase in L means the sample is lightening (i.e., +DL for lightening and – DL for darkening)

Mechanical properties

Samples were ovendried at 105°C for 24 hours before testing. This ensured the same conditioning for samples before and after weathering. Flexural tests were carried out according to ASTM D790 (26) on an Instron Universal Testing machine. A three-point loading system was utilized with a crosshead speed of 1.3 mm/min. The exposed surface was placed away from the center load to place that part of the sample in tension. At least five replicate specimens were tested for each formulation. The modulus of elasticity (MOE) and maximum strength were calculated according to the standard.

Fourier Transform Infrared Spectroscopy

FTIR spectroscopy was conducted on a Mattson Genesis II spectrophotometer to provide knowledge of functional groups present at the surface of the samples. Penetration depth is dependent upon

wavelength and index of refraction of WF and HDPE and ranges from 0.5 to 3 µm. Scans were run at a resolution of 4 cm^{-1}. Each sample recorded consisted of 100 scans recorded in absorbance units from 4,000 to 700 cm^{-1}. Spectra were obtained using attenuated total reflectance (ATR). The surfaces of the samples analyzed were in contact with a ZnSe crystal that has a 45° angle of incidence. At least five replicate samples were analyzed.

Cellulose spectra have a strong peak due to hydroxyl groups at 1,023 cm^{-1} (23). A wood index was calculated using the following equation:

$$Wood\ index = \frac{I_{1023}}{I_{2912}} \times 100 \qquad [2]$$

where:

I = peak intensity.

Peak intensity was normalized using the peak at 2,912 cm^{-1}, which corresponds to alkane CH stretching vibrations of the methylene groups.

Weathering

Samples were placed in a xenon arc-type light exposure apparatus which was operated according to ASTM D2565 (27). Samples were mounted in four rows on a drum that rotated around the xenon arc bulb at 1 rpm. Each 2 hour weathering cycle consisted of 108 minutes W exposure and 12 minutes simultaneous water spray and W exposure (27). An irradiance sensor was used to measure light intensity for wavelengths from 300 to 400 nm. Irradiance was monitored and the voltage to the bulb was changed periodically in order to maintain a constant irradiance. The dosage, or amount of light energy the samples were subjected to, was calculated (irradiance × time). The dosage at 500-hour time increments is shown in Table 1. We can be confident that samples were exposed to consistent energy dosages for each thousand hours of weathering. To understand the effect of time on weathering, samples were removed for analysis after 1,000, 2,000, and 3,000 hours.

Statistics

To determine the effects of weathering on properties, Student's two-tailed t-tests were carried out at a = 0.05 for each blend, testing the data for significant differences.

Results and Discussion

Unexposed Samples

The WF-HDPE composites were manufactured by either injection molding or extrusion. Furthermore, extruded samples were either cut to leave the original extruded surface intact or the surface was planed to study material with surface effects removed. The physical properties of injection molded, extruded, and planed composites without W weathering are summarized in Table 2. Processing pressures during injection molding were much higher than during extrusion. The result was a higher density for injection molded samples than for extruded or planed samples.

Lightness of the composites also varied with manufacturing method (Table 2). Injection molded samples were darkest. Processing temperatures were higher for injection molded samples,

Table 1. ~ *The amount of light energy between 300 and 400 nm the samples were exposed to for 500-hour increments.*

Time increment (hours)	Dosage (kW-hr./m^2)
0 to 500	29.4
500 to 1,000	29.1
1,000 to 1,500	27.9
1,500 to 2,000	28.2
2,000 to 2,500	28.7
2,500 to 3,000	28.8

Table 2. ~ *Physical properties of unexposed 50% woodfour-filled HDPE composites.*[a]

Samples	Composite density (r_c) (g/cm^3)	Lightness	MOE (GPa)	Strength (MPa)
Injection molded	1.135 (0.002)	49.0 (0.7)	3.6 (0.2)	39.6 (0.7)
Extruded	1.087 (0.008)	57.0 (0.8)	3.3 (0.2)	24.5 (0.9)
Planed	1.085 (0.019)	62.1 (1.0)	3.4 (0.2)	27.2 (0.7)
HDPE	0.953 (0.001)	76.4 (0.2)	0.9 (0.1)	22.3 (0.2)

[a] Numbers in parentheses represent 1 standard deviation.

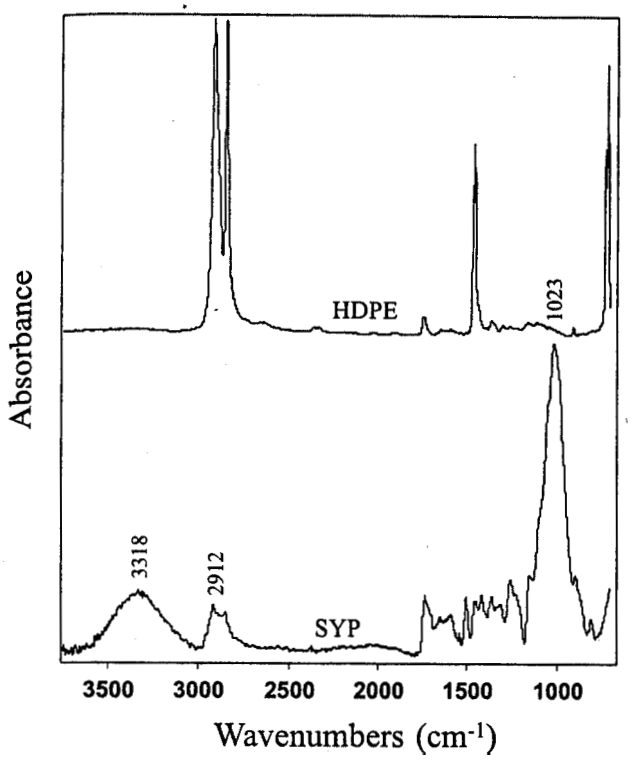

Figure 2. ~ *FTIR spectra of HDPE and SYP.*

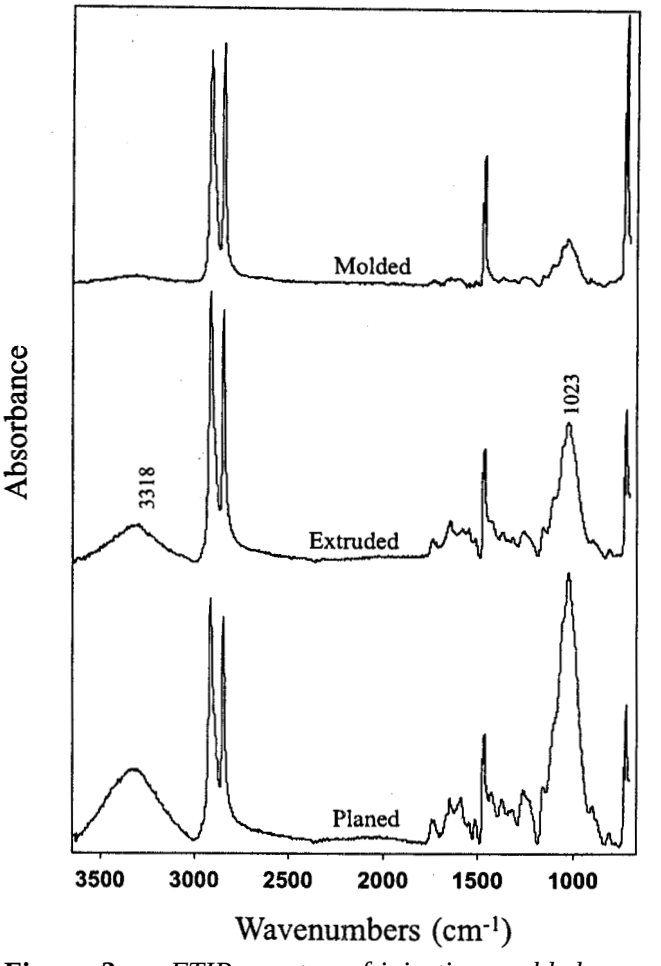

Figure 3. ~ *FTIR spectra of injection molded, extruded, and planed WF-HDPE composites.*

which resulted in a darker composite as the wood began to degrade. Comparing extruded to planed composites, planed composites were lighter in color. The polymeric surface layer of the extruded composite effectively wet the wood flour. When wetting occurs at a wood surface light is transmitted deeper in the wood cell, resulting in an apparent darkening of the wood (28).

Generally, the flexural MOE of the composites were similar. However, the flexural strength of injection molded samples was greater than for extruded and planed samples. This is most likely due to difference in densities. The higher densities of injection molded samples result in more intimate contact between the HDPE matrix and WF. The improvement in interfacial quality led to an apparently voidless composite and a more efficient stress transfer between the matrix and fiber, leading to increased strength.

The surface of each sample was analyzed using FTIR spectroscopy. Figure 2 shows two spectra, the top one of unfilled HDPE and the bottom one of a solid piece of southern yellow pine (SYP, *Pinus* spp). The broad peak at 3,318 cm^{-1} is associated with mixed hydroxyl groups originating mainly from cellulose. The peak at 2,912 cm^{-1} is due to the alkane C–H stretching of the methylene groups and appears as a very strong peak in HDPE and a much weaker peak in SYP. A relatively large proportion of HDPE is –CH2– groups compared to SYP. The peak at 1,023 cm^{-1} is assigned to a hydroxyl group associated with cellulose (23).

Figure 3 shows FTIR spectra obtained for injection molded, extruded, arid planed composites. The increase in hydroxyl groups at the surface was clearly seen as an increase in the broad peak at 3,318 cm^{-1} and the strong peak at 1,023 cm^{-1}. A wood index was calculated to relate the hydroxyl group peak at 1,023 cm^{-1} to the methyl group peak at 2,912 cm^{-1} (Eq. [2]). The results are shown in Table 3. Planed samples had the highest wood index, followed by extruded and injection molded samples. It was expected that planed composites would exhibit stronger peaks due to the wood component because wood particles were exposed during planing. The differences between injection

Table 3. ~ *Wood index determined from the cellulose peak at 1,023 cm^{-1}.*

Samples	Wood index[a]
Injection molded	0.24 (0.02)
Extruded	0.44 (0.09)
Planed	2.74 (1.39)
SYP	7.58

[a] Numbers in parentheses represent 1 standard deviation.

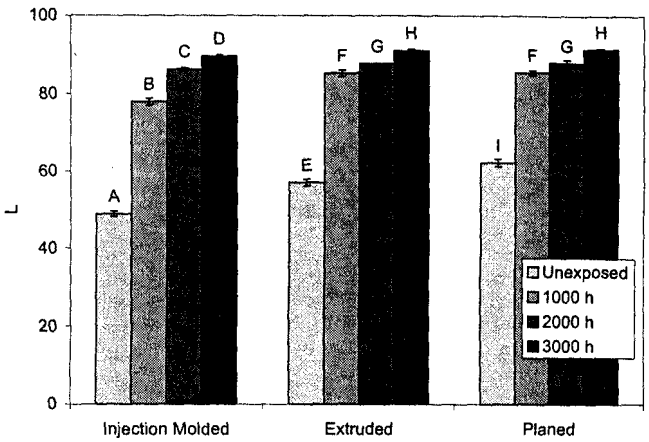

Figure 4. ~ *L as a function of processing method of WF-HDPE composites before and after 1,000, 2,000, and 3,000 hours of weathering. Statistically significant differences between composites are represented with separate letters.*

molded and extruded samples are probably due to processing differences. During processing, the higher pressures used in injection molding cause a thin layer of polymer to be formed at the surface. The increased pressure attributable to injection molding led to a composite with a thicker polymer rich surface layer.

Exposed Samples

Injection molded, extruded, and planed samples underwent accelerated weathering in a xenon-arc weathering apparatus. Samples were removed after 1,000, 2,000, and 3,000 hours of weathering. The composites were then tested for lightness, flexural MOE, and flexural strength. These results are shown in **Figures 4 through 6,** respectively. Each manufacturing method is shown on the x-axis. Statistically significant differences between composites were represented with separate letters in **Figures 4 through 6.** Bars with the same letter denotes that the difference between two means is not statistically significant.

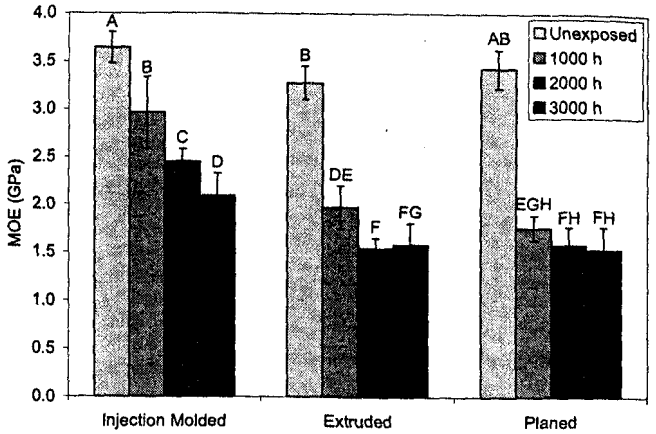

Figure 5. ~ *Flexural modulus as a function of processing method of WF-HDPE composites before and after 1,000, 2,000, and 3,000 hours of weathering. Statistically Significant differences between composites are represented with separate letters.*

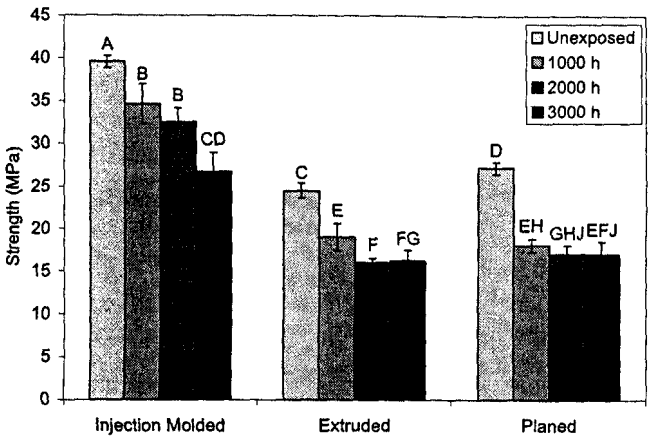

Figure 6. ~ *Flexural strength as a function of processing method of WF-HDPE composites before and after 1,000, 2,000, and 3,000 hours of weathering. Statistically significant differences between composites are represented with separate letters.*

The effect of weathering on composite lightness, L, is shown in Figure 4. It is clear that weathering resulted in a lightening of the composite. For each production method, the change in L after each time period was significant. Regardless of manufacturing method, the greatest increases in L occurred in the first 1,000 hours and all composites reached a similar lightness value after 3,000 hour of weathering. Lightening of the composite originates mainly from the wood flour bleaching, and is a composite surface phenomenon. Lundin (3) characterized this depth after 2,000 hours of weathering and found it to be 0.3 mm for 50 per-

Table 4. ~ *Loss in MOE and loss in strength after accelerated weathering for 1,000, 2,000, and 3,000 hours for 50% woodflour-filled HDPE composites compared with values found in the literature.*

Samples	MOE loss (%)			Strength loss (%)		
	1,000 hr.	2,000 hr.	3,000 hr.	1,000 hr.	2,000 hr.	3,000 hr.
Injection molded	19	33	43	12	18	32
Extruded	40	53	52	22	35	34
Planed	49	54	52	34	37	38
Injection molded (3)	29	33	--	17	20	--
Injection molded (15)	3	26	--	6	22	--

cent woodflour-filled HDPE. Because the final values of L are similar for composites regardless of manufacturing method, we can conclude that the lightened layer is deeper than the depth of the polymer rich layers in injection molded and extruded samples. Therefore production method has little effect on total lightness after 3,000 hours of weathering.

It is also evident that after 1,000 hours of weathering planed and extruded samples were closer to their final lightness than composites with more of a HDPE influence at the surface, the injection molded samples. After 1,000 hours of weathering, injection molded samples reached 71 percent of their total lightening after 3,000 hours, while extruded and planed samples reached 80 percent and 83 percent, respectively. This illustrates that samples with more wood component at the surface will experience a larger percentage of total lightening in the early stages of weathering.

Flexural MOE and strength generally decreased after accelerated weathering. The decrease in MOE for injection molded samples was significant for each 1,000 hours ofweathering **(Fig. 5).** For extruded samples, the decrease in MOE was not significant between 2,000 and 3,000 hours of weathering; and for planed samples, the decrease was not significant for 1,000, 2,000, and 3,000 hours of weathering.

The decrease in flexural strength for injection molded samples was not significant between 1,000 and 2,000 hours of weathering **(Fig. 6).** The same significant changes in strength after weathering were observed for extruded and planed samples as shown for MOE. After 3,000 hours of weathering, the MOE and strength were both similar for extruded and planed samples, and lower than injection molded samples. Unlike L after 3,000 hours of weathering, manufacturing method did appear to affect final flexural properties, with injection molded composites having higher flexural properties after weathering than extruded and planed composites. It is expected that increased weathering times would cause mechanical properties to reach similar values regardless of manufacturing method.

Similar to the trend observed for lightness, after 1,000 hours of weathering extruded and planed samples were closer to their final MOE and strength than injection molded samples. After 1,000 hours, injection molded, extruded, and planed samples reached 44, 77, and 88 percent, respectively, of their total MOE loss. Similarly, injection molded, extruded, and planed samples reached 38, 67, and 89 percent, respectively, of their total loss in strength after 1,000 hours of weathering. These results imply that composites with more wood component at the surface will lose a larger percentage of total property loss in initial weathering stages. Conversely, samples with a large polymer characteristic at the surface will lose a smaller percentage of their total property loss in the initial weathering stages.

This explains apparently conflicting data reported by other researchers. **Table 4** shows the loss in MOE and loss in strength for 50 percent woodflour-filled HDPE composites after weathering. Injection molded, extruded, and planed samples are compared to results found in two research papers where samples were also injection molded. Lundin (3) weathered 50 percent WF- HDPE composites for 2,000 hours. They found that the yield stress and bending stiffness decreased through 1,000 hours and then leveled off. Conversely, Stark et al. weathered 50 percent woodflour-filled HDPE samples for 2,000 hours (6). The samples were removed periodically. After 1,000 hours the MOE did not significantly drop. After 2,000 hours the drop was 26 percent. In addition to differences between the sample's surface differences, comparing

weathering effects based on weathering times may have an apparent error. The energy received by the samples also needs to be taken into consideration and may be different for each study.

The color fade of the composites is due mainly to the effect of UV exposure on wood flour. While it needs to be studied further, degradation of mechanical properties of WF-HDPE composites after weathering is thought to be largely due to moisture effects. This would most likely be due to a degradation of interfacial properties as a result of exposure to moisture. During weathering, samples cycle through environments of 35°C and 100 percent relative humidity during the water spray cycle, and 40°C and 30 percent relative humidity during the dry cycle. Moisture has been shown to adversely affect properties of WPCs. Injection molded HDPE composites filled with 40 percent wood fiber exposed to a water bath for 2,000 hours experienced a 39 percent loss in flexural MOE (13). Similarly, 30 percent WF-HDPE injection molded samples lost approximately 25 percent of their flexural modulus after being exposed to boiling water for 50 hours (16). The processing method has also been shown to influence moisture sorption properties. After soaking 50 percent WF-HDPE composites for 2 weeks, more dense injection molded composites absorbed only 4 percent moisture while less dense extruded composites absorbed 17 percent (10).

Summary and Conclusions

The FTIR-ATR method can be used to examine surface characteristics of WPCs manufactured using different processing techniques. By following peaks associated with functional groups present in polyethylene and cellulose, differences in surface chemistries between manufacturing methods become apparent. In this study, injection molded, extruded, and planed samples were analyzed. Injection molded samples had more of a polymer influence at the surface than extruded and planed samples. Planed samples exhibited a strong wood component as a result of removing polymer rich surface layer to expose wood particles.

The higher processing pressures injection molded samples experience result in a composite with a higher density than extruded and planed samples. The higher density results in improved strengths over extruded and planed samples through improved interfacial quality.

The manufacturing method of WPCs greatly influences their durability. Injection molded samples have more polymer component at the surface than extruded and planed samples. This results in increased color fade for extruded and planed samples in initial stages of weathering, which is due to a bleaching of wood fiber. However, after a sufficient weathering time period, the lightened degradation layer reaches deeper into the composite than the depth of the polymer rich layer. Therefore manufacturing method does not influence total lightness of the composite after 3,000 hours of weathering.

The retention of flexural. properties, however, is greatly influenced by processing method. Generally, injection molded samples show an improved retention of flexural MOE and strength over extruded and planed samples after 3,000 hours of weathering. In addition, extruded and planed samples lose a larger percentage of their total mechanical property loss during the first 1,000 hours of exposure than injection molded samples. This is likely due directly to a loss in properties due to moisture exposure. Planed samples provide a pathway for absorption of moisture due to a strong hydrophilic wood component at the surface.

Acknowledgments

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