FEASIBILITY OF USING SALTCEDAR AS A FILLER IN INJECTION-MOLDED POLYETHYLENE COMPOSITES[1]

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Abstract.   Saltcedar (*Tamarix ramosissima*) was investigated for use as a filler in wood–plastic composites (WPCs). The mineral content, water-soluble extractive content, and thermal stability of saltcedar flour were compared with those of a commercial pine wood flour. The wood flours were compounded with plastic, and the viscosities of the composite melts containing the two species were compared. Injection-molded composites produced from the compounded material were evaluated for mechanical performance and weatherability. Saltcedar flour had more minerals and water-soluble extractives than pine flour, which resulted in lower thermal stability, but also lower melt viscosity when compounded with high-density polyethylene. Injection-molded WPCs made from unextracted saltcedar performed similarly to those made from pine in accelerated weathering tests, but their mechanical properties were generally lower. The flexural modulus of elasticity increased when extracted wood flour was used, especially for the saltcedar composites. However, color stability and flexural strength changed little. Producing WPCs from these composites is possible, although economically feasible applications that use the advantageous properties of these species and that can tolerate or address the less desirable ones need to be identified and demonstrated.

Keywords: Saltcedar, tamarisk, polyethylene, wood–plastic composite, WPC, filler, wood flour, injection molding.

INTRODUCTION

Invasive and small-diameter species have become more prevalent, contributing to a host of environmental and ecological problems, including increased fire danger, topsoil erosion, reduced groundwater, and reduced stream flows. Because these species are encroaching into natural indigenous ecosystems, prescribed burns and chemical, mechanical, and biological methods are being used to attempt to control them.

The Bureau of Land Management (BLM) has identified saltcedar (*Tamarix* spp.) as one such invasive species. Saltcedar is a shrub or shrub-like tree usually less than 6 m in height that tolerates an extreme range of environmental conditions. It has a deep, extensive root system, is very proficient at accessing limited water supplies, and has higher water-use efficiency than native riparian trees in both mature and postfire communities. Saltcedar accumulates salt in special glands in its leaves, which is then transferred to the surface layer of the soil when plants drop leaves. Germination and establishment of many native species have become impaired as surface soils become more saline, particularly along regulated rivers that are no longer subjected to annual flooding and scouring. Saltcedar may also be better adapted to the postfire environment than native species (Zouhar 2003).

Originally imported as an ornamental shrub or tree, saltcedar has spread primarily in the southwestern United States and Mexico. It is especially pervasive in Arizona and California,

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with specimens also found in Nevada, Utah, Colorado, New Mexico, Oklahoma, Texas, Kansas, Arkansas, New York, and Manitoba (Zouhar 2003). Saltcedar's aggressive growth can displace native plants and block streams, promoting flooding during periods of heavy rain. Also, its rapid evapotranspiration rate can seriously deplete ground water (Anon 2003).

Unfortunately, it is unlikely that land management agencies will have sufficient funds to pay for fuel reduction and forest restoration on the scale needed unless contractors are able to find commercial applications for salvaged material. If uses for these materials can be developed, restoration projects could provide economic benefits, including incomes, to nearby communities. Efforts could provide local employment in tree thinning, chipping and transporting the small-diameter material, and processing and developing value-added products.

This research investigates using saltcedar as a filler in wood–plastic composites (WPCs). Wood–plastic composites have been used in applications such as automotive paneling, furniture, and consumer products (Clemons 2002). However, the largest and fastest growing use of WPCs is in the construction industry (Morton and Rossi 2003). Over one-half of the WPCs produced in North America are used in decking applications, and the great majority of WPCs are in exterior building products such as deck boards, railings, and window and door profiles (Morton and Rossi 2003). There has been considerable interest lately in other applications such as signs, furniture, siding, and roofing as well as using WPCs in a variety of marine and construction applications requiring greater structural performance than demanded by current WPC products.

Although WPCs may provide an outlet for saltcedar, little information is available on their performance as a filler. Issues such as thermal stability and the effects of extractives will influence their desirability as fillers. If unique attributes, or at least approaches to mitigating any negative attributes, are identified, these fillers are more likely to be used.

We evaluated wood flour from saltcedar as a filler in WPCs. We investigated wood composition and the processing and performance of WPCs made from saltcedar and compared them with those made with commercial pine wood filler.

EXPERIMENTAL PROCEDURE

Materials

Small logs of saltcedar (*Tamarix ramosissima*), approximately 1.8 m long and about 5–13 cm dia, had been harvested previously from BLM lands on the lower Colorado River near Yuma, Arizona. The logs were shipped to the USDA Forest Products Laboratory (FPL) in Madison, Wisconsin, and were chipped and then hammer-milled successively using screens with 13 and 0.8 mm openings. The flour was then screened and the particle size fraction passing through a 40-mesh sieve, but not an 80-mesh sieve was used for the project. Logs were not debarked before processing. However, considerable bark was removed during hammermilling and screening. The final bark composition was less than 3% of the final screened wood flour. A western pine blend of wood flour (AWF 4020; American Wood Fibers, Schofield, WI) was obtained from a commercial supplier and used as a reference material. This material was also screened to a −40/+80 mesh before composite fabrication.

Two high-density polyethylenes (HDPE) were used as the matrix materials. One HDPE used was Exxon Mobil Chemical's HD6605 (Exxon Mobil Chemical Company, Houston, TX) and had a melt-flow index (MFI) of 5 g/10 min and a density of 948 kg/m^3. A second HDPE was used for extrusion capillary rheometry, a 0.8 g/10 min MFI HDPE (Petrothene LM 6007-00; Equistar Chemicals LP, Houston, TX).

Wood Characterization

The particle-size distributions of the wood flours were measured by image analysis using Image Tool, version 3.00 (University of Texas Health Science Center, San Antonio, TX).

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The projected area was used as a measure of particle size. The length (longest chord) and the thickness of each particle were also measured and used to determine the aspect ratio (length-to-diameter ratio). At least 500 particles of each species were analyzed.

To determine mineral content, wood flour samples were wet-ashed using nitric acid and hydrogen peroxide in a closed microwave bomb. Temperature and pressure were ramped from ambient to 140°C and 0.7 MPa in 10 min and then held at these conditions for 15 min. Elemental composition was then quantitatively determined by inductively coupled plasma-atomic emission spectrometry using a Jobin Yvon Ultima spectrometer (HORIBA Jobin Yvon, Inc., Edison, NJ).

Scanning electron microscopy on both the solid wood and WPC composites were performed using a Zeiss EVO40 scanning electron microscope (Carl Zeiss SMT, Inc., Thornwood, NY). Either secondary electrons or a combination of secondary and backscattered electrons were used in imaging specimens. Elemental composition of salt crystals was determined by energy-dispersive X-ray analysis using a Vantage-DSI X-ray Microanalysis System (Thermo Noran, Madison, WI).

Thermal stability was investigated by thermogravimetric analysis (TGA7; Perkin Elmer Corporation, Boston, MA). Wood flour was ground to less than 100 mesh using a grinding mill (Wig-L-Bug; Reflex Analytical Corporation, Ridgewood, NJ). The ground wood flour was then dried at 105°C for at least 4 h and then stored under desiccant. Approximately 6 mg of sample was used for each TGA test. Temperature scans from 100–600°C were performed with a heating rate of 10°C/min. Isothermal scans were also performed. Approximately 6 mg of each sample was held at 105°C for 10 min under nitrogen to remove any remaining moisture. The temperature was then quickly raised to the desired temperature and held for 45 min while the weight loss was monitored. A nitrogen purge of 20 mL/min was used for all TGA tests.

Composite Preparation and Characterization

Fifty percent by weight of pine or saltcedar wood flour (–40/+80 mesh) was compounded with HDPE using a 1 L thermokinetic mixer (K-mixer; Synergistics, Inc., St. Remi de Napierville, Quebec, Canada). Batch size was 120–150 g, the rotor speed was 5500 rpm, and the discharge temperature was 196°C. The discharged molten composite was cold-pressed, granulated, and dried in an oven at 105°C. To investigate the effects of water-soluble extractives on weathering performance, composites were also made with wood flour that had been extracted five times in water at 80–85°C (see Stark and Mueller 2008 for details) and then oven-dried at 105°C.

The MFI of various formulations was measured according to ASTM D 1238 (ASTM 2005a). Viscosity was more completely characterized by extrusion capillary rheometry using a Plasti-corder 19 mm single-screw extruder with capillary dies (C.W. Brabender Instruments, South Hackensack, NJ) following ASTM D 5422 protocols (ASTM 2005b). Straight lines were not found on the Bagley plots. Therefore, apparent viscosities and shear rates are reported from runs using a 3 mm dia and 12.3 mm long capillary over shear rates of approximately $50–500$ s^{-1}. Melt temperature in the die was maintained at 180°C.

The dry, compounded pellets were injection-molded into flexural specimens using a 33 t reciprocating-screw injection molder (Cincinnati Milacron, Batavia, OH). The barrel temperatures ranged from 182–191°C and the mold temperature was 99°C. Flexural tests were performed on the injection-molded composites according to ASTM D 790 (ASTM 2005c). Specimens were tested dry with at least five replicates. The tangent modulus of elasticity (MOE) and the flexural strength were calculated as per the standard. Student's two-tailed t-tests assuming unequal variance were performed at $\alpha = 0.05$ to compare mechanical properties of composites containing unextracted wood flour with those containing extracted wood flour.

Composite lightness (*L**) was measured following the CIE *L*a*b** color scale (Konica Minolta CR-400 Chroma Meter; Konica Minolta Sensing, Inc., Osaka, Japan) (Robertson 1977). CIE *L*a*b** is a 3-D color space measuring the lightness of the sample (*L**) and color coordinates (*a** and *b**). *L** ranges between 0 (black) and 100 (white). *L** was measured for five replicate samples.

The injection-molded composites were placed into a xenon arc-type weathering apparatus for 3000 h. The samples were mounted on a drum that rotated around a light source and subjected to a cycle of 108 min of light exposure followed by 12 min of light exposure and water spray according to ASTM D 2565 (ASTM 2005d). The xenon arc lamp was fitted with borosilicate inner and outer filters, resulting in a spectral irradiance distribution similar to solar radiation. WPC samples were removed after 1000, 2000, and 3000 h of weathering. During weathering, irradiance between 300 and 400 nm was measured. The removal of the samples corresponded with a radiant exposure of 148, 299, and 450 MJ/m^2, respectively. WPC samples containing hot water-extracted wood flour were removed after 3000 h of weathering. The composites were monitored for changes in flexural properties after drying and *L** values.

RESULTS AND DISCUSSION

Wood Characterization

Wood flour particle size and aspect ratio distributions were determined by image analysis and are summarized in Fig 1. Both species had similar particle size distributions. Although saltcedar had more particles with aspect ratios from 1.5 – 2.0, their overall distributions were still quite similar. Average aspect ratios of 4.0 and 3.2 were found for pine and saltcedar, respectively.

Because of the possible exposure of the composites to water when in exterior applications, the water-soluble extractive content was determined

by a 4 h Soxhlet extraction. The extractive content for saltcedar was 10.9%, more than twice that of the pine (5.0%). A room temperature water extraction according to ASTM D 1110 (ASTM 2005e) yielded a similar trend with extractive contents of 9.3 and 3.4% for saltcedar and pine, respectively. The increased extractive content may be the result of a higher extractive content in the wood itself or attributable to residual bark remaining in the wood flour after processing, which typically has a higher extractive content (Harkin and Rowe 1971). Because WPCs are not typically protected by sealing, painting, or staining, this suggests greater potential for extractive leaching and staining from WPCs made from saltcedar. The best protection against this would be careful processing and formulation (e.g., limiting wood flour content to 50% or less) or the use of additives to minimize moisture intrusion. Alternatively, the composites could be used in interior applications in which the potential for extractive staining would not be an issue.
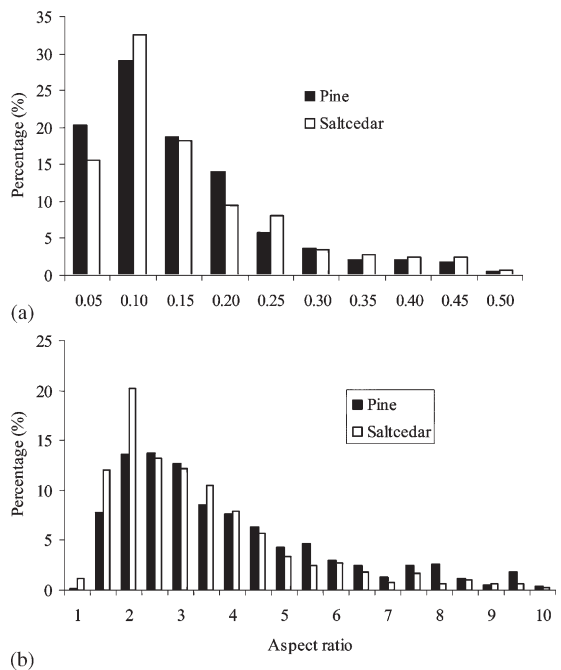

(a)

(b)

Figure 1.   Distributions of aspect ratios (length-to-diameter ratios) determined by image analysis of at least 500 fibers for each species.

Because saltcedar has the ability to accumulate salt in special glands in its leaves, we used scanning electron microscopy to check for the presence of salt in the wood itself. Salt crystals were readily apparent in many of the ray cells of the wood (Fig 2).

Results from inductively coupled plasma (ICP) emission spectroscopy for saltcedar showed a high mineral content in saltcedar, especially for sulfur and calcium, which were each nearly 1% by weight (Fig 3). Very small amounts of minerals were found in the pine flour. The total content of the minerals investigated was 2.5 and 0.2% of the total weight for the saltcedar and pine wood flours, respectively. Because this is the mineral content only, the total salt content would be considerably higher. Figure 4 shows the mineral contents of wood flour from saltcedar before and after a 4 h Soxhlet extraction with water. Nearly all the minerals, except for some of the sulfur and calcium, was removed during the extraction suggesting that a significant fraction of the material removed during the water extraction procedure is salt.

Thermal stability of wood fillers is important because WPCs are processed at temperatures as high as about 200°C. Wood's lack A of thermal stability limits the number of plastics that can be processed with wood and the number of applications in which WPCs can be used. For example, it is sometimes desirable to increase the processing temperatures of even low melting-point plastics to lower viscosity and decrease motor loads. Consequently, processors sometimes push the processing temperatures, which can create volatiles and cause discoloration or increase odor.

Figure 5 shows the weight loss rates for the wood flours as a function of temperature as determined by TGA. The large peak around 380°C is dominated by cellulose degradation, which does not begin until high temperature is reached but then quickly proceeds (Rowell and LeVan-Green 2005). Hemicelluloses typically degrade from about 225–325°C (Winandy and Lebow 2001). The faster degradation rate of saltcedar at low temperature suggests that it contains hemicelluloses or extractives that are less thermally stable.
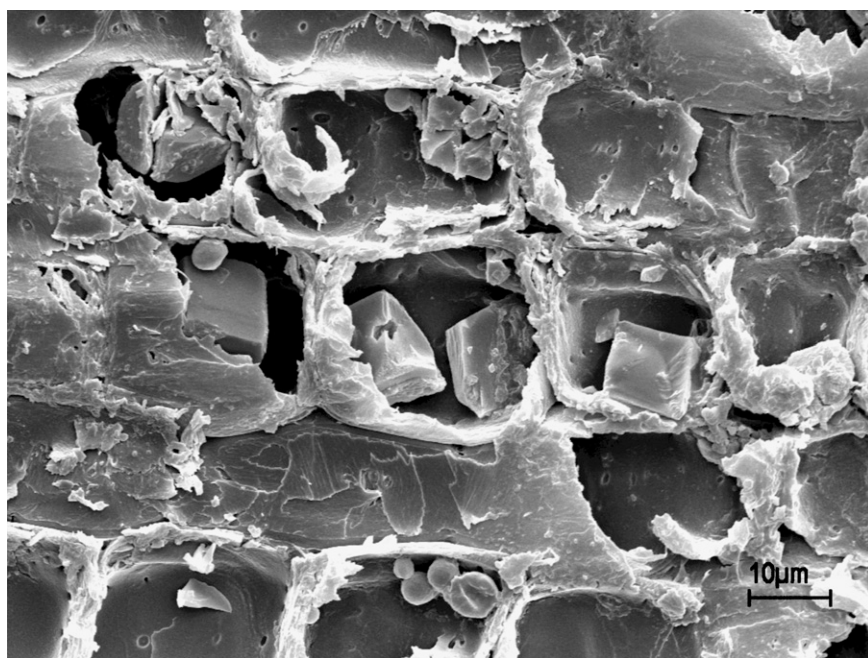

Figure 2.   Scanning electron micrograph of ray cells of saltcedar containing salt crystals.
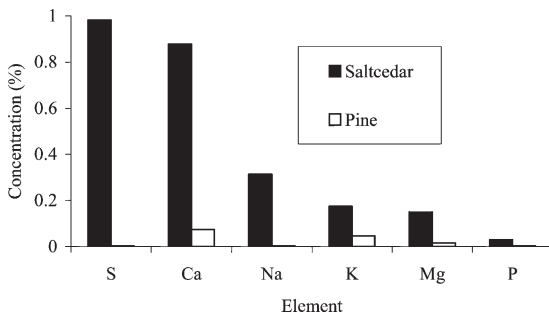
Figure 3. Mineral content determined by inductively coupled plasma (ICP) analysis of unextracted wood flour made from different wood species.
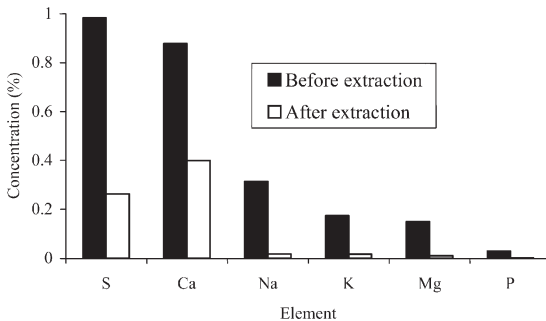
Figure 4. Mineral content determined by inductively coupled plasma (ICP) analysis of wood flour from saltcedar before and after a 4-h Soxhlet extraction with water.

To further investigate the relative thermal stability of the two species, isothermal tests were performed at temperatures typical of WPC processing. Figure 6a shows the weight loss curves at 200°C. Although weight losses were not large, even small ones can indicate the release of significant quantities of volatiles that can produce undesirable voids in the finished product. Saltcedar had larger weight losses than pine. In similar tests on Soxhlet-extracted wood flours (Fig 6b), lower weight losses were found overall and little difference was seen between the species suggesting that most of the weight loss difference was the result of extractives.

Melt Rheology of Wood–Plastic Composite Blends

Processing equipment such as extruders and injection molders relies on the ability of
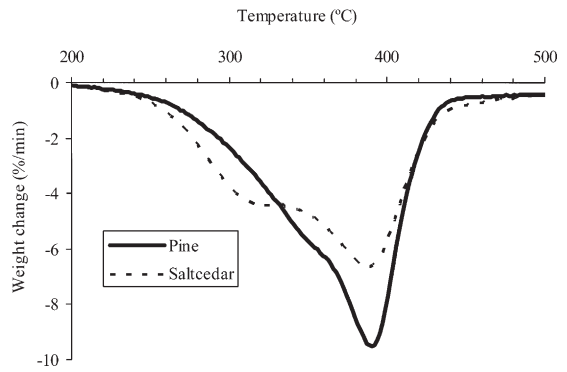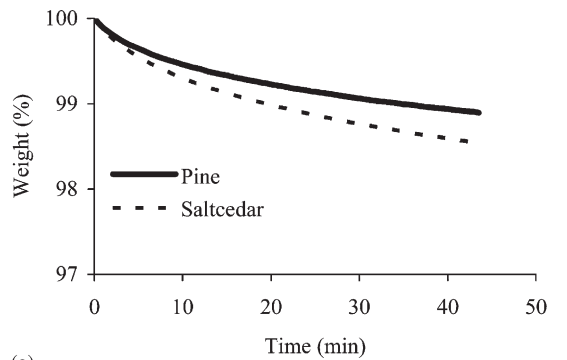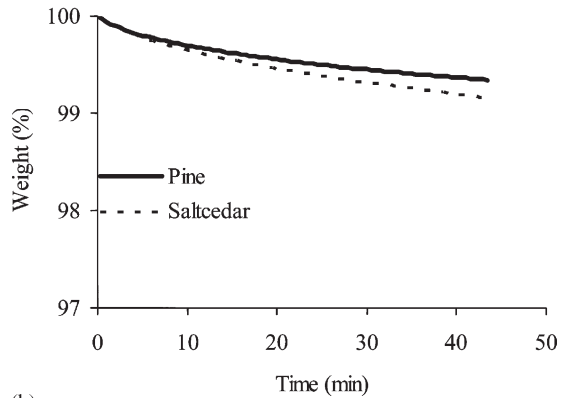
Figure 5. Weight loss rate with temperature for several species by thermogravimetric analysis (10°C/min heating rate under nitrogen).

(a)

(b)

Figure 6. Isothermal gravimetric analysis curves for several species at 200°C (a) before and (b) after a 4-h Soxhlet water extraction.

molten materials to flow through barrels, dies, and nozzles when blending constituent materials and forming them into useful products. Hence, the viscosity of the composite melt

is an important consideration. The viscosities of our WPCs were measured using several techniques.

The MFI is a crude but quick measure of viscosity that measures the amount of a melt that is forced through an orifice by a weighted piston (ASTM 2005a). For HDPE composites containing 50% wood flour, melt flow indices of 1.5 and 0.25 were found for saltcedar and pine composites, respectively. Because of its potential importance in WPC processing, this species effect on viscosity was more thoroughly investigated using extrusion capillary rheometry (ASTM 2005b). In capillary rheometry, the viscosity is measured over a variety of shear rates, which is important because different shear rates are found in different processing methods and rates. Viscosity decreased with increasing shear rate as is common for thermoplastics and their composites (Fig 7). Differences between the species were found over the entire shear rate range investigated, although the greatest differences were at low shear rates. For example, the viscosity of the saltcedar blend at 300 s^{-1} was about 20% lower than that of the pine blend, but was about 50% lower at 30 s^{-1}. This effect was the result of water-soluble extractives. When the extractives were removed from the saltcedar by Soxhlet extraction, this viscosity reduction disappears.

Melt rheology of WPCs is complex and many variables can influence behavior (Marcovich et al 2004; Li and Wolcott 2005). Many explanations for the effect of extractives on apparent viscosity are possible, including extractives acting as a lubricants or volatilization of thermally unstable extractive components resulting in voids that also can affect viscosity. More in-depth research is necessary to determine the mechanistic details of this viscosity reduction. The viscosity effect was not found with the pine flour because the viscosity changed little when extracted pine flour was used instead of unextracted pine flour.

Composite Characterization

A scanning electron micrograph of a microtomed composite specimen made with unextracted saltcedar is shown in Fig 8. The micrograph is a composite of secondary electron and back-scattered electron imaging. The wood flour particles can be clearly seen surrounded by the HDPE matrix. The bright salt crystals are present in some of the wood particles, presumably those containing ray cells in which salt would likely accumulate. Very few such salt crystals were found in similar composites made with hot water-extracted saltcedar. The X-ray spectra taken for one of the salt crystals is shown in Fig 9. Large amounts of sulfur and calcium were apparent, which supports the findings from the ICP analysis (Fig 4).

The mechanical performance and color fade over the lifetime of WPCs are important. Because weathering can adversely affect these properties, the lightness ($L*$), the flexural MOE, and the flexural strength of composites made from each wood flour were periodically assessed during exposure to ultraviolet (UV) and moisture in a xenon arc-type weathering apparatus.

Initially, the saltcedar composites were slightly darker than the pine composites (smaller value for $L*$, Fig 10). Although pigments and colorants can be added to WPCs to lighten the composites, the dark color from the saltcedar will limit the color range that can be economically achieved. Alternatively, a dark color may
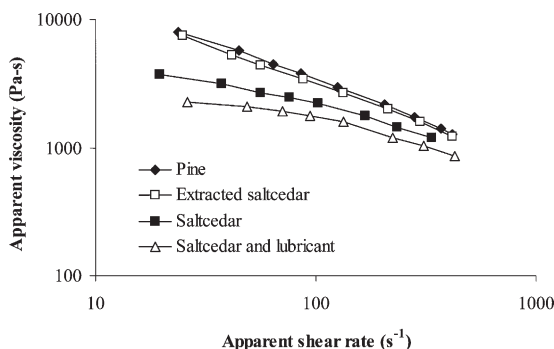

Figure 7.   Viscosity curves for selected high-density polyethylene composites with 50% wood flour by extrusion viscosity capillary rheometry.
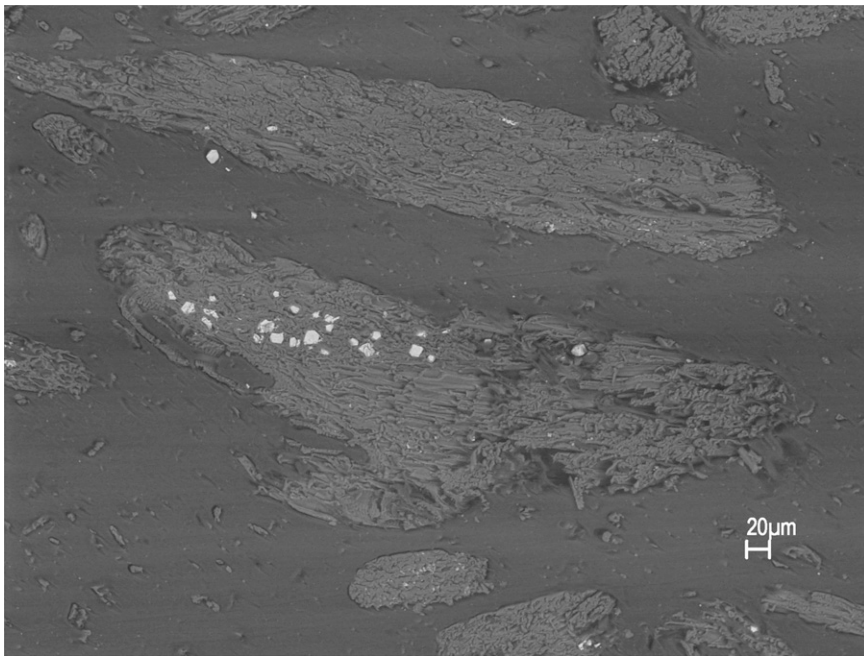
Figure 8. Microtomed specimen taken from injection-molded high-density polyethylene containing 50% unextracted saltcedar. The micrograph is a composite image of secondary electron and backscattered electron imaging. Bright areas are salt crystals.
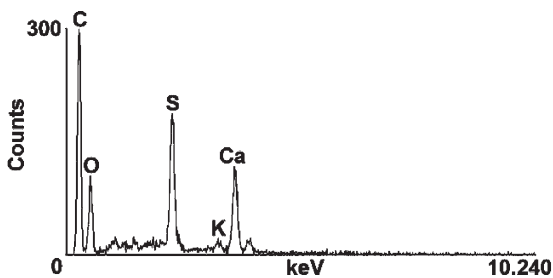

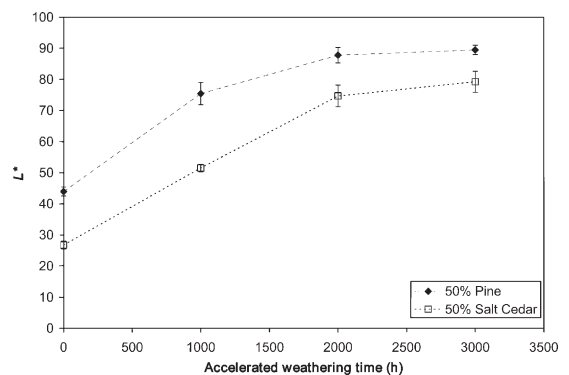Figure 9. X-ray spectra of a salt crystal.

Figure 10. Composite lightness (L^*) of injection-molded high-density polyethylene composites containing 50% wood flour during accelerated weathering.

be advantageous if such a color is desired and if the color is sufficiently stabilized. During weathering, L^* increased for both composites. The largest increases occurred early in the weathering period, with increases leveling off between 2000 and 3000 h of exposure. Overall, the saltcedar composites lightened slightly more than the pine composites with an L^* increase of 52 vs 46, respectively. However, the saltcedar composites remained slightly darker than the pine composites throughout weathering.

Both the flexural MOE and strength of the saltcedar composites were lower than the pine composites before weathering (Figs 11 and 12). During accelerated weathering, the MOE of the composites decreased through 2000 h. Further weathering resulted in a continued decrease for saltcedar composites and a slight increase
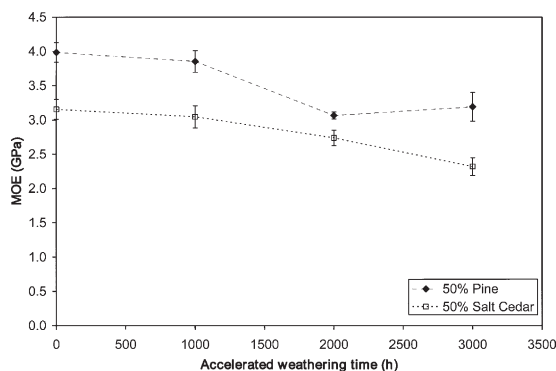
Figure 11. Flexural modulus of elasticity (MOE) for injection-molded high-density polyethylene composites containing 50% wood flour during accelerated weathering.
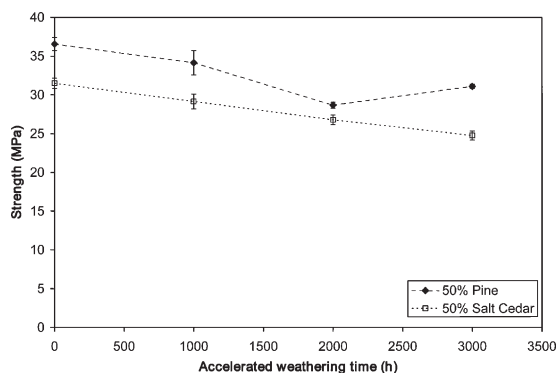

Figure 12. Flexural strength of injection-molded high-density polyethylene composites containing 50% wood flour during accelerated weathering.

Table 1. *Effect of wood flour extraction on weathering performance for high-density polyethylene—based wood–plastic composites.*

	$L*$[a]		MOE (GPa)		Strength (MPa)	
	Unexposed	3000 h	Unexposed	3000 h	Unexposed	3000 h
Pine	43.9 (1.5)[b]	89.5 (1.5)	4.0 (0.1)	3.2 (0.2)	36.5 (0.8)	31.1 (0.3)
Pine − extracted	40.5 (1.8)	89.7 (1.7)	4.4 (0.1)	3.6 (0.1)	37.6 (0.2)	33.5 (0.3)
Saltcedar	26.8 (1.3)	79.2 (3.4)	3.2 (0.1)	2.3 (0.1)	31.5 (0.7)	24.7 (0.6)
Saltcedar − extracted	36.0 (2.7)	81.5 (4.5)	3.8 (0.2)	2.9 (0.1)	31.9 (0.3)	26.3 (0.6)

[a] Stark and Mueller (2008).
[b] Numbers in parentheses represent one SD.
$L*$, lightness; MOE, modulus of elasticity.

for pine composites. Flexural strength generally trended downward as well for both composites. Similar to MOE, flexural strength of the composites decreased through 2000 h with further weathering resulting in a continued decrease for saltcedar composites and a slight increase for pine composites. After the 3000 h weathering, the MOE of the pine and saltcedar composites had decreased by 20 and 26%, respectively, and the strength had decreased by 12 and 18% (Table 1). These losses in mechanical properties are a result of synergistic effects of both UV radiation and water sorption resulting in material changes such as surface oxidation, changes in matrix crystallinity, and interfacial damage (Stark and Gardner 2008).

Previously, Stark and Mueller (2008) investigated the effect of removing water-soluble extractives on weathering performance of these composites. Composites were made from wood flour that had been extracted five times with hot water at $80-85°C$ and compared with those made with the unextracted wood flour. Results from the color measurements are included in Table 1. Using extracted pine in the composites resulted in slightly darker composites compared with unextracted pine, whereas extracted saltcedar resulted in lighter composites. After 3000 h of weathering, composites containing both unextracted and extracted wood flour lightened to the same value of $L*$ regardless of whether the wood species was pine or saltcedar.

The present investigation found that, unlike color stability, mechanical performance of the weathered composites was affected by wood flour extraction (Table 1). A statistically significant increase in MOE was reported when composites contained extracted wood flour compared with unextracted wood flour regardless of species or exposure. This increase was larger for the saltcedar composites than the pine composites. These changes might be expected

because saltcedar contains more extractives, which contribute little to the mechanical performance of dry wood. Adding extracted wood to HDPE effectively adds more of the structural components of wood (e.g., cellulose) than adding unextracted wood. This improves stiffness, which largely results from the relative quantities and stiffnesses of the constituent materials. Incorporated extracted wood flour also increased the flexural strength compared with unextracted wood flour. The increase was statistically significant for all except the unexposed composites containing saltcedar. However, the increase in flexural strength was overall a smaller percentage compared with the flexural MOE. Strength is more affected by the bonding between the wood flour and plastic as well as the aspect ratio of the wood flour. Extractives can influence the bonding between wood and plastics. For example, extractives can form a weak boundary layer, especially if the wood flour is oven-dried (Saputra et al 2004). In our work, the low aspect ratio of the wood flour greatly limits its ability to reinforce the matrix, despite any potential influence of extractives on wood–plastic adhesion; therefore, strength improvement was limited.

SUMMARY AND CONCLUSIONS

Small saltcedar logs, previously harvested from the lower Colorado River near Yuma, AZ, were made into wood flour and compared with a commercial pine flour. Saltcedar had a higher mineral content than the pine and contained twice the water-soluble extractive content. Small weight losses were found in isothermal TGA tests at temperatures typical of WPC manufacture. Saltcedar lost more weight than pine, which was likely the result of volatilization of water-soluble extractives as their removal eliminated these differences. These volatiles would need to be vented during composite production.

The wood flours were compounded with plastic, and the viscosities of the composite melts were found to depend on the water-soluble extractive content, especially at low shear rates.

Unextracted saltcedar yielded composite melts with lower viscosity than those with pine, which could be advantageous in some processing scenarios. However, the high extractive content could lead to extractive leaching in WPCs used in exterior applications if care is not taken in formulating and processing. Saltcedar composites were also darker than the pine composites. Flexural properties were generally lower for WPCs made with saltcedar, but the composites performed similarly to those made with pine in accelerated weathering tests. The flexural MOE increased when extracted wood flour was used, especially for the saltcedar composites. However, the color stability and the flexural strength changed little when extracted wood was used.

This was just an initial investigation on the use of saltcedar in WPCs and much more work is needed. Applications need to be identified that use the advantageous properties of these species and that can tolerate or address the less desirable ones. For example, a product that was compression-molded (a low shear rate process) might be able to take advantage of the low viscosity of WPCs containing saltcedar. If the product did not need to be extremely light in color or was covered, any difficulties with dark color of WPCs containing saltcedar would not be problematic and, in some instances, may even be preferable. The economic feasibility of saltcedar in WPCs will depend on many factors, including harvest costs, transportation costs, and costs associated with wood flour manufacture as well as local pricing of plastics and additives. The availability of suitable manufacturing facilities, markets for composite production, and identification of additional products from the harvested wood will also affect commercial feasibility

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