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Use of Saltcedar and Utah Juniper as Fillers in Wood–Plastic Composites

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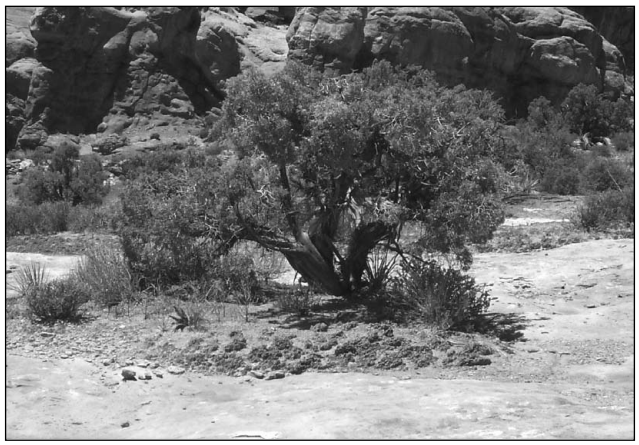

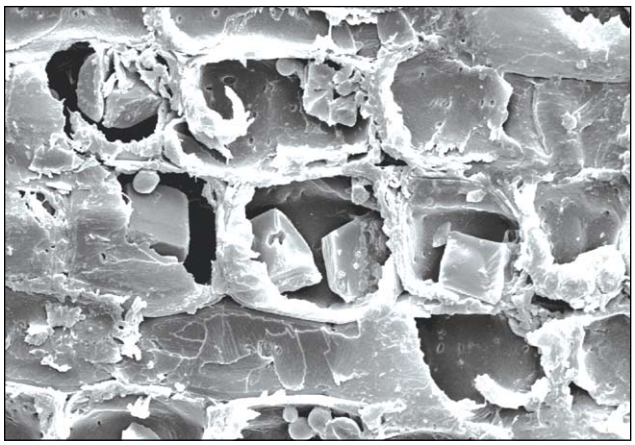

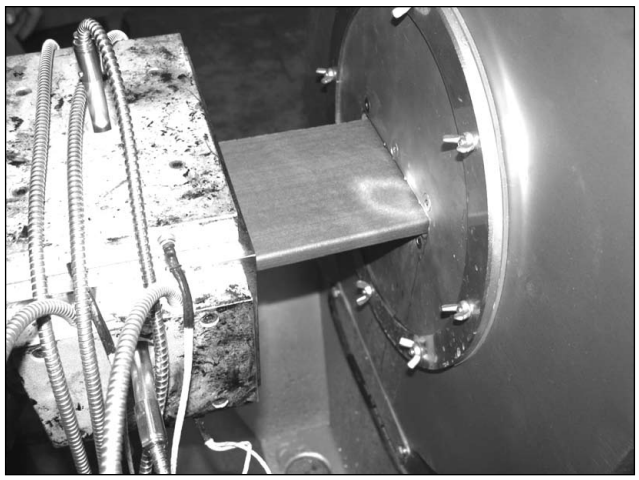

Abstract

Invasive and small-diameter species have become more prevalent, creating numerous environmental and ecological problems. One potential method to control and eliminate invasive species and thereby promote natural rangeland restoration is developing new, value-added uses for them. Saltcedar (*Tamarisk ramosissima*) and Utah juniper (*Juniperus osteosperma*) were investigated for use as fillers in wood–plastic composites (WPCs).

The chemical composition and thermal stability of wood flours from both invasive species were compared with those of commercial pine wood flour. The wood flours were compounded with plastic and additives, and the viscosities of the composite melts containing the different species were compared. Composites produced from the compounded material by profile extrusion and injection molding were evaluated for mechanical performance, appearance, and weatherability.

Saltcedar wood flour had the most minerals and water-soluble extractives, which resulted in the lowest thermal stability and the lowest melt viscosity when compounded with high-density polyethylene. Injection-molded WPCs made from saltcedar or juniper were both considerably darker than those made with pine but performed similarly in accelerated weathering tests. Their mechanical properties were generally lower than those of the composites made from pine, but appropriate application selection and proper design could help compensate. Extruded WPCs were successfully made with each of the species.

Producing WPCs from these composites appears technically feasible, although continued formulation development and durability evaluation are needed so that informed decisions regarding applications can be made. 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, juniper, wood–plastic composite, WPC, filler, wood flour

Cover photo, top left: *Juniperus osteosperma*. Copyright 2001 by J.S. Peterson @ USDA–NRCS PLANTS Database, used by permission.

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Use of Saltcedar and Utah Juniper as Fillers in Wood–Plastic Composites

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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 these species. However, it is unlikely that land management agencies will have sufficient funds to pay for fuels 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 adding value to harvested small-diameter invasive species by incorporating them into wood–plastic composites (WPCs). Wood–plastic composites are currently one of the fastest growing markets of composites in the United States and are increasingly being used in exterior building applications such as deck boards and railings. The Bureau of Land Management (BLM) identified two problem invasive species for this investigation: saltcedar or tamarisk (*Tamarix ramosissima*) and Utah juniper (*Juniperus osteosperma*). They are discussed in more detail below.

The objective of this research was to evaluate saltcedar and Utah juniper wood flours as fillers in WPCs. Wood composition and the processing and performance of WPCs made from these species were investigated and compared to those made with commercial pine wood filler. Though WPCs may provide an outlet for invasive wood species, little information is available on their performance in this role. Issues such as thermal stability, effects of extractives, and economics 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.

Saltcedar

Saltcedar is a shrub or shrub-like tree usually less than 6 m (20 ft) 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 (Zlatnik 1999). Saltcedar accumulates salt in special glands in its leaves, which is then transferred to the surface layer of the soil when plants drop their 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 (Zlatnik 1999). Saltcedar may also be better adapted to the postfire environment than are native species (Zlatnik 1999).

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, New Mexico, western Texas, Nevada, and Utah but is also widespread in southern California, the Rocky Mountain states, the western plains states, and parts of Oregon and Idaho. Saltcedar is considered by some to be one of the most widely distributed and troublesome nonnative, invasive plants along water courses in the southwestern United States. Its presence reduces recreational use of parks, national wildlife refuges, and other riparian areas for camping, hunting and fishing, boating, bird watching, and wildlife photography (Zlatnik 1999).

Utah Juniper

Utah juniper grows less than 8 m (26 ft) and is often only 3 to 4.5 m (10 to 15 ft), with a trunk 10 to 30 cm (4 to 7.5 in.) in diameter at breast height. The species is widely distributed throughout the arid West. The tree occurs occasionally in southern Idaho, southern Montana, and western Wyoming and is common in Colorado, Utah, Nevada, New Mexico, Arizona, and southeastern California (Zlatnik 1999).

Where Utah juniper has occurred historically, it plays a significant role in native ecosystems. However, in the past

100 years, Utah juniper has been encroaching on shrubland and grassland communities in Utah and elsewhere in the West as a result of overgrazing and fire suppression. In the absence of fire or other disturbances, trees eventually crowd out herbaceous and shrub species (Utah State University 2006; BLM 2005)

Junipers also compete more efficiently for soil moisture than do herbaceous understory plants (Utah State University 2006). The Bureau of Land Management (BLM) has identified loss of shrubland and grassland communities to juniper encroachment as one of the most important fire-related ecological issues in Utah (BLM 2005).

Wood–Plastic Composites

Traditionally, inorganic fillers such as fiberglass, calcium carbonate, and talc have been added to plastics to improve their performance or to reduce cost. More recently, wood flour has been added to plastic, leading to the development of wood–plastic composites (WPCs). Wood flour fillers are advantageous because they are inexpensive, renewable, lighter, and less abrasive to processing equipment compared with inorganic fillers. However, issues such as moisture removal and low thermal stability need to be addressed when using wood flour fillers.

Wood fibers that have higher aspect ratios than wood flour have been shown to result in greater reinforcement if properly bonded to the polymer (Stark and Rowlands 2003). However, these fibers often have low bulk density and are difficult to meter and feed into conventional plastics-processing equipment. Because most WPCs in the United States are used in building applications where stiffness is more important than the strength provided by reinforcement, the vast majority of manufacturers use wood flour that can be easily fed and metered. Products usually contain approximately 50% to 60% wood, though a few contain as little as 5% or as much as 70%. The relatively high bulk density and free-flowing nature of wood flour compared with wood fibers or other longer natural fibers, as well as its low cost, familiarity, and availability, are attractive to WPC manufacturers and users.

Though wood flour has historically been used in various products including soil amendments, extenders for glues, and absorbents for explosives, its large-scale use in common commodity thermoplastics has only occurred in the past several decades. Wood flour for WPC manufacture is typically 10- to 80-mesh size and derived from byproducts of wood processors. Wood flour typically costs about $0.11 to $0.22/kg ($0.05 to $0.10/lb), and common species include pines, oaks, or maples. Many reasons are given for species selection including slight color differences, regional availability, and familiarity. Some species, such as some of the red oaks, can contain extractives that can be leached out from the wood in the composite by rain and dew if moisture is allowed to penetrate the composite. When the water evaporates, it can leave stains from the extractives behind.

Because of its low thermal stability, wood flour is usually used as filler only in plastics that are processed at temperatures lower than about 200°C (392°F). Polyethylene (PE) is the most often used plastic, although polypropylene (PP), polyvinyl chloride (PVC), and to a much lesser extent, polystyrene (PS) are also used.

Wood flour is commonly supplied in multi-walled paper bags (approximately 23 kg, or 50 lb), bulk bags (typically 1.5 m^3 or 55 ft^3), or bulk trailers at moisture contents between 4% and 8% and must be dried before use in thermoplastics.

Processing

Wood and plastic are not the only components in WPCs. These composites also contain additives, materials that are added in small amounts to affect processing and performance. Although formulations are often proprietary, additives such as coupling agents, light stabilizers, pigments, lubricants, fungicides, and foaming agents may be used in commercial applications.

To produce WPCs, wood and additives are mixed into a molten plastic in a process called compounding. Many options are available for compounding using either batch or continuous mixers. The compounded material can be immediately pressed or shaped into an end product or formed into pellets for future processing. Some product manufacturing options for WPCs force molten material through a die (sheet or profile extrusion), into a cold mold (injection molding), between calenders (calendering), or between mold halves (thermoforming or compression molding).

The majority of WPCs are manufactured by profile extrusion, in which molten composite material is forced through a die to make a continuous profile of the desired shape. Extrusion lends itself to processing high-viscosity, molten blends of wood and plastic and to shaping the long, continuous profiles common to building materials. These profiles can be a simple solid shape or a highly engineered hollow one and are produced on extrusion lines at rates of about 500 to 1,000 kg/h (roughly 1,000 to 2,000 lb/h). If larger outputs are desired, additional extrusion lines are added.

Other processing technologies such as injection molding and compression molding are less often used to produce WPCs but are of increasing interest. These alternative processing methods have advantages when processing a continuous piece is not desired or a more complicated shape is needed. Composite formulation must be adjusted to meet processing requirements (the low viscosity needed for injection molding can limit the wood content). Various press-forming methods are also common in automotive applications.

Specialized equipment has been developed for many aspects of WPC processing, including materials handling, drying and feeding systems, extruders, dies, and downstream equipment (equipment needed after extrusion, such as

cooling tanks, pullers, and cut-off saws). Additionally, toll compounders specializing in producing wood and other natural fibers mixed with thermoplastics supply preblended, free-flowing pellets that can be reheated and formed into products by a variety of processing methods. The pellets are useful to manufacturers who do not typically do their own compounding or do not wish to compound in-line (for example, most single-screw profilers or injection-molding companies).

Markets

Wood–plastic composites have been used in automotive paneling, furniture, and consumer products such as cosmetic cases (Clemons 2002). However, the largest and fastest growing use of WPCs is in the construction industry (Morton and Rossi 2003). Over 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 is demanded by current products.

Our Research

We evaluated wood flour from two invasive species identified by the BLM, saltcedar (tamarisk) and Utah juniper, as fillers in WPCs. We investigated wood composition and the processing and performance of WPCs made from these species and compared them to those made with commercial pine wood filler.

Experimental

Materials

Small logs of saltcedar, approximately 1.8 m (6 ft) long and about 5 to 13 cm (2 to 5 in.) diameter, had been harvested previously from BLM lands on the lower Colorado River basin near Yuma, Arizona. The logs were shipped to the USDA Forest Products Laboratory (FPL) in Madison, Wisconsin, and chipped (Fig. 1). Utah juniper chips were sent from BLM lands in Utah. Because the chips had a high moisture content (about 88%), the juniper was dried overnight in a drum dryer at 105°C (221°F) to a moisture content of about 5%.

The saltcedar and juniper chips were hammermilled twice, first using a screen with 13-mm (½-in.) openings and then using one with 0.8-mm (1/32-in.) openings. The particle-size distribution was then further refined by screening the hammermilled wood. Two screens were used: a 40 U.S. standard mesh with 420-µm openings and a 80 U.S. standard mesh with 180-µm openings. Either a single 40-mesh screen was used to remove the largest particles (overs) or both the 40- and 80-mesh screens were used to remove the largest (overs) and the smallest (fines) fractions. These screened

Figure 1—Saltcedar logs (top) and Utah juniper chips (bottom) supplied by the Bureau of Land Management.

wood fractions are designated –40 mesh and –40/+80 mesh in subsequent discussions. A western pine blend of wood flour (AWF 4020, American Wood Fibers, Schofield, Wisconsin) was obtained from a commercial supplier and used as a reference material.

Three polymers, two high-density polyethylenes (HDPE) and a PP, were used as the matrix materials. One HDPE used was Exxon Mobil Chemical's HD6605 (Exxon Mobil Chemical Company, Houston, Texas) and had an melt-flow index (MFI) of 5 g/10 min and a density of 0.948 g/cm^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, Texas). The PP homopolymer had a MFI of 20 g/10 min and a density of 0.905 g/cm^3 (Certene PHM-20AN, Muehlstein, Norwalk, Connecticut).

Various additives were also used for the extruded composites depending on the formulations desired. The lubricant used was a modified fatty acid ester compatible with maleated polyolefins (TPW 113, Struktol, Stow, Ohio). The coupling agent used was a maleated polyethylene with a MFI of 5 g/10 min and containing 1% maleic anhydride (Polybond 3009, Chemtura Corporation, Middlebury, Connecticut). To enhance light stability, a UV package consisting of a 2:1 weight ratio of colorant (Holcobatch Brown 936755, Holland Colors Americas, Richmond, Indiana) and UV absorber (Tinuvin 328, hydroxyphenylbenzotriazole UV

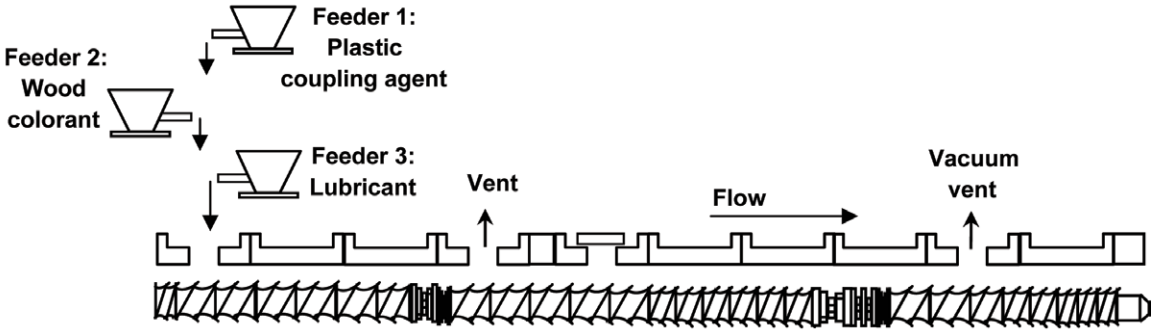

Figure 2—Screw design and setup for twin-screw compounding.

absorber Ciba Specialty Chemicals, Tarrytown, New York) was used.

Wood Characterization

The particle-size distributions of the –40/+80 mesh wood flour fractions were measured by image analysis using Image Tool, version 3.00 (University of Texas Health Science Center, San Antonio, Texas). The projected area was used as a measure of particle size. The length (longest chord) and the thickness of each particle were measured and used to determine the aspect ratio (length divided by diameter). At least 500 particles of each species were analyzed.

Wood flour samples (–40/+80 mesh) were hydrolyzed and the sugars were quantified by anion chromatography following procedures described in Davis (1998). Acid-insoluble (Klason) lignin was determined using the following procedure. Approximately 100 mg of dried wood flour (–40/+80 mesh) was hydrolyzed with 1.00 ml 72% (w/w) H_2SO_4 for 1 h at 30°C (86°F). Samples were then diluted to 4% H_2SO_4 in distilled H_2O, and a secondary hydrolysis was performed for 1 h at 121°C (249.8°F). Hydrolyzed samples were then immediately filtered through tared Gooch porcelain crucibles containing Whatman 934-AH (Whatman, Brentford, Middlesex, UK) glass fiber filters. The filtrate was washed three times with 5-ml distilled H_2O, six times with 10-ml hot distilled H_2O, dried, and weighed. Klason lignin values were then calculated and corrected for ash content. Ash content was quantitatively determined after subjecting the lignin to 575°C (1,067°F) for at least 3 h.

To determine mineral content, wood flour samples (–40/+80 mesh) were wet-ashed using nitric acid and hydrogen peroxide in a closed microwave bomb. Temperature and pressure were ramped from ambient to 140°C (284°F) and 0.7 MPa in 10 min, then held at these conditions for 15 min. Elemental composition was then quantitatively determined by inductively coupled plasma-atomic emission spectrometry (ICP-AES) using a Jobin Yvon Ultima spectrometer (HORIBA Jobin Yvon, Inc., Edison, New Jersey).

Scanning electron microscopy on both the solid wood and WPC composites was performed using a Zeiss EVO40 scanning electron microscope (Carl Zeiss SMT, Inc., Thornwood, New York). 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, Wisconsin).

Two methods were used to quantitatively measure the water soluble extractive content of –40/+80 mesh wood flour: (1) a 48-h ambient water soak according to ASTM D 1110 (ASTM 2005c) and (2) a 4-h Soxhlet extraction with a reflux cycle of about 5 min.

Thermal stability was investigated by a thermogravimetric analysis (TGA) system that included a thermogravimetric analyzer (TGA7, Perkin Elmer Corporation, Boston, Massachusetts) and related equipment. Wood flour (–40/+80 mesh) was ground to less than 100 mesh using a grinding mill (Wig-L-Bug, Reflex Analytical Corporation, Ridgewood, New Jersey). The ground wood flour was then dried at 105°C (221°F) 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°C (212°F) to 600°C (1,112°F) were performed with a heating rate of 10°C/min under nitrogen. Isothermal scans were also performed. Approximately 6 mg of each sample was held at 105°C (221°F) 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

Wood flour was dried to less than 1% moisture and then compounded with plastic and additives in a 32-mm compounding twin-screw extruder (D-TEX extruder, Davis Standard, Pawcatuck, Connecticut). The screw design and setup are shown in Figure 2. The raw materials were fed into the main feed throat using a gravimetric feed system. The plastic was melted and the various components were blended into the polymer in the mixing zones. Atmospheric

and vacuum vents were used to remove any remaining water vapor or other volatiles. Screw rotation and feed rates were chosen to ensure adequate blending yet keep the melt temperature below about 200°C (392°F) to avoid thermally degrading the wood flour. The melt was forced through a strand die. The strands were then cooled and cut into pellets, which were then dried and used as a feedstock for either profile extrusion or injection molding. Formulations compounded in this manner are summarized in Table 1, blends 1–3 (injection-molded composites) and Table 2 (extruded composites).

However, for the accelerated-weathering investigation, a different method for preparing compounds was used (Table 1, blends 4 and 5). Both the manufactured saltcedar wood flour and the purchased pine wood flour were screened to a mesh size of –40/+80. The wood flour was dried before being combined with 5 MFI HDPE at 50% by weight wood flour. Small batches were compounded using a 1-L thermokinetic mixer (K-mixer, Synergistics, Inc., St. Remi de Napierville, Quebec) in 150-g batch sizes for the saltcedar/HDPE composites and 120-g batch sizes for the pine/HDPE composites. The thermokinetic mixer was run at 5,500 rpm. An infrared sensor monitored the composite melt temperature, and the discharge temperature was set at 196°C (385°F). The molten composite was pressed into a patty, cooled at room temperature, and granulated.

Dry, compounded pellets of the formulations in Table 1

Table 1—Formulations of injection-molded composites, which included no additives such as lubricants, coupling agents, UV stabilizers, or pigments[a]

Blend #	Plastic content (%)	Plastic type	Wood species	Wood content (%)	Compounding method
1	60	PP [b]	Pine	40	Extrusion compounding
2	60	PP	Saltcedar	40	Extrusion compounding
3	60	PP	Juniper	40	Extrusion compounding
4	50	HDPE [c]	Pine	50	Thermokinetic mixing
5	50	HDPE	Saltcedar	50	Thermokinetic mixing

[a] Particle-size fraction comprised of particles that pass through 40-mesh screen but not 80-mesh screen.
[b] Polypropylene.
[c] High-density polyethylene.

Table 2—Formulations of extruded composites wit 50% wood content (percentages by weight)

Blend #	HDPE [a] content (%)	Wood species	Particle-size fraction (mesh)	Lubricant level (%)	Coupling agent (%)	UV package (%)
6	44	Pine	As received [b]	6	0	0
7	41	Pine	As received	6	3	0
8	41	Pine	As received	6	0	3
9	38	Pine	As received	6	3	3
10	42	Juniper	–40 [c]	8	0	0
11	39	Juniper	–40	8	3	0
12	39	Juniper	–40	8	0	3
13	36	Juniper	–40	8	3	3
14	42	Saltcedar	–40[c]	8	0	0
15	39	Saltcedar	–40	8	3	0
16	39	Saltcedar	–40	8	0	3
17	36	Saltcedar	–40	8	3	3

[a] High-density polyethylene.
[b] Commercial wood flour supplied as a –40 mesh with some fines removed.
[c] Wood flour produced at the Forest Products Laboratory and screened to –40 mesh (particle-size fraction comprised of particles that pass through 40-mesh screen.). No fines removed.

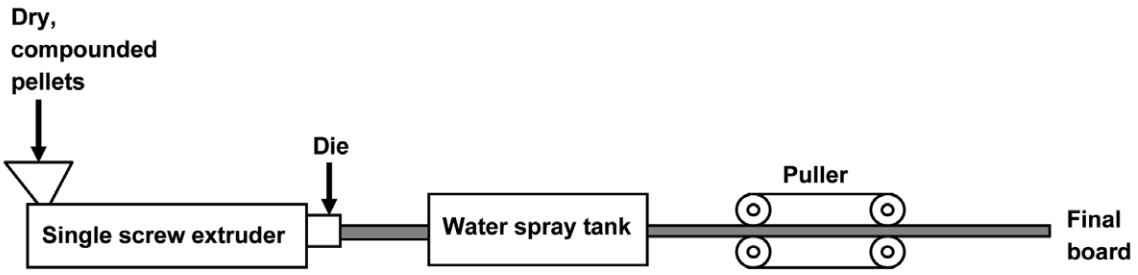

Figure 3—Single screw profile extrusion line.

were injection-molded into specimens using a 33-ton reciprocating-screw injection molder (Cincinnati Milacron, Batavia, Ohio) to test for the following properties: flexural as per ASTM D 790 (ASTM 2005b), tensile (Type I, ASTM D 638 (ASTM 2005a)), and Izod impact (ASTM D 256 (ASTM 2006)) . For the injection-molded PP composites, the barrel temperatures ranged from 188 to 193°C (370° to 380°F), and a mold temperature of 93°C (200°F) was necessary to obtain a good surface on the highly filled compounds. The injection speed was approximately 1.9 cm/s (0.75 in/s) and the cycle time was approximately 50 s. For the injection-molded HDPE composites for the accelerated weathering investigation, the barrel temperatures ranged from 182°C to 191°C (360°F to 377°F) and the mold temperature was 99°C (210°F).

Dry, compounded pellets of the formulations in Table 2 were formed into 12.7- by 1.3-cm (5- by ½-in.) radius-edged boards using FPL's profiling line (Figs. 3 and 4) at a rate of about 64 m/h (210 ft/h). The pellets were melted in the extruder and pumped through the die to the desired dimensions. A Model 6-39 belt puller (The Conair Group, Pittsburgh, Pennsylvania) was used to pull the boards through a Model MSBHI-16-9 water spray tank (The Conair Group, Pittsburgh, Pennsylvania), which cooled the boards. The boards were then cut to 1.8-m (6-ft) lengths by a Model MST-4 traveling cut-off saw (The Conair Group, Pittsburgh, Pennsylvania).

Rheology

The MFI values of various formulations were measured according to ASTM D1238 (ASTM 2005d).Viscosity was more completely characterized by extrusion capillary rheometry using a Plasticorder 19-mm single-screw extruder with capillary dies (C.W. Brabender Instruments, South Hackensack, New Jersey) following ASTM D 5422 protocols (ASTM 2005e). Straight lines were not found on the Bagley plots. Therefore, apparent viscosities and shear rates are reported from runs using a 3-mm-diameter and 12.3-mm-long capillary over shear rates of approximately 50 to 500 s^{-1}. Melt temperature in the die was maintained at 180°C (356°F).

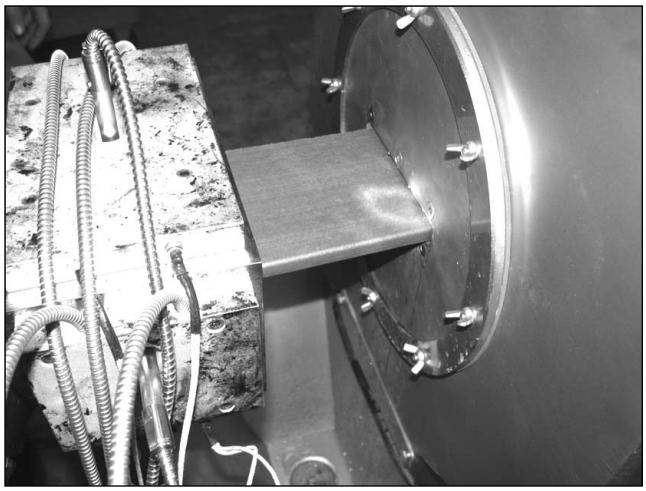

Figure 4—Board exiting the extruder die (left) and entering cooling tank (right).

Mechanical Properties of Composites

Flexural, tensile, and Izod impact tests were performed on the injection-molded composites according to ASTM D 790 (ASTM 2005b), D 638 (ASTM 2005a), and D 256 (ASTM 2006). Specimens were tested dry. At least five replicates were tested.

The extruded boards were tested for flexural modulus of elasticity (MOE) and modulus of rupture (MOR) according to ASTM D 6109 (ASTM 2004b). The profile of the boards tested was as extruded, and they were cut into 46-cm-(18-in.) long samples. The boards were tested in the flat position with a support span of 20 cm (8 in.) and loaded at two points with an outer fiber strain rate of 0.01. The supports on the under side of the board were on pivots that moved with the board as it flexed. The average of five replicates is reported.

Composite Color

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 three-dimensional color space measuring the lightness of the sample (L*) and color coordinates (a* and b*). L* ranges between 0 (black) and 100 (white). An increase in L* means the sample is lightening. L* was measured for five replicate samples.

Weathering Composites

Accelerated Weathering

The injection-molded composites for the weathering investigation (blends 4 and 5, Table 1) were placed into a xenon-arc type weathering apparatus and weathered for 3,000 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 2004a). The xenon-arc lamp was fitted with borosilicate inner and outer filters, resulting in a spectral irradiance distribution similar to solar radiation. The samples were removed after 1,000, 2,000, and 3,000 h of weathering. During weathering, irradiance between 300 and 400 nm was measured. The removal of the samples corresponded with a radiant exposure of 41, 83, and 125 kW-h/m^2, respectively. The composites were monitored for changes in flexural properties and L* values.

Natural Weathering

Extruded samples were cut to a length of 45.72 cm (18 in.) and mounted on 90° racks facing south in Madison, Wisconsin. No backing was used on the racks. The composites were monitored periodically for changes in L*. At the time of this report, they have been weathered for 9 months. They will be weathered for a total period of 2 and 5 years, after which they will be removed and tested for flexural MOR and MOE. L* will continue to be monitored during this time period.

Results and Discussion

Wood Flour Particle Dimensions and Composition

Wood flour particle size and aspect ratio (length-to-diameter) distributions were determined by image analysis and are summarized in Figures 5 and 6. The particle-size distributions of the different species were similar. Though saltcedar had more small particles with aspect ratios from 1.5 to 2.5, their overall distributions were similar. Average aspect ratios of 4.0, 3.2, and 4.4 were found for pine, saltcedar, and juniper, respectively.

Chemical analyses were performed on the wood to determine acid-insoluble lignin content as well as the 5- and 6-carbon sugars remaining after hydrolysis of the carbohydrate fraction (cellulose and hemicelluloses, Fig. 7). Differences in composition were found. For example, juniper had much more lignin than did the other two fillers, and saltcedar had more than double the xylan content of the other two. These compositional differences can play important roles in the performance of these species as wood fillers. For example, hemicelluloses are generally considered the least thermally stable of the three major wood components cellulose, hemicellulose, and lignin (Rowell and LeVan-Green 2005), and their degradation has been shown to correlate with such properties as the initial strength loss in solid wood at

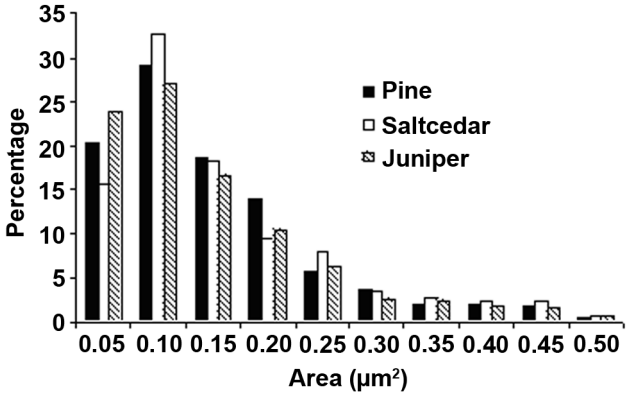

Figure 5—Particle-size distributions (–40/+80 mesh wood flour fractions) determined by image analysis of at least 500 fibers for each species.

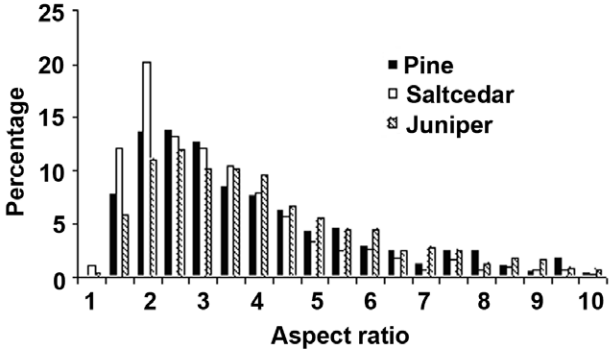

Figure 6—Distributions of aspect ratios (length-to-diameter ratios) determined by image analysis of at least 500 fibers for each species.

elevated temperatures (Winandy and Lebow 2001). Therefore, wood flour containing less thermally stable hemicelluloses may result in WPCs with reduced performance if processing temperatures are too high. The three major components are also susceptible to ultraviolet (UV) degradation. However, lignin is the predominant component that absorbs light (Hon 2000). Therefore, juniper composites may degrade faster during UV exposure than pine or saltcedar composites.

Because of the exposure of the composites to water when in service, we focused on the water-soluble extractives of the wood. The results are summarized in Table 3. The extractive content for saltcedar was at least twice that of the other wood flours. Additionally, the extract of saltcedar had more color than that of either pine or juniper flours. Since wood plastic composites are not typically protected by sealing, painting, or staining, this suggests greater potential for extractives leaching from WPCs made from saltcedar. The best protection against this would be careful formulation (for example, limiting wood content to 50%) and processing the composites to minimize moisture intrusion. Alternatively, the composites could be used in interior applications where the potential for staining would not be an issue.

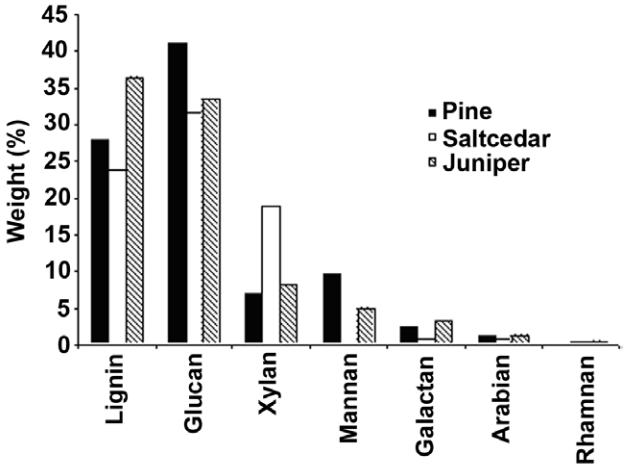

Figure 7—Sugar and acid-insoluble lignin content after hydrolysis for the wood flour species investigated.

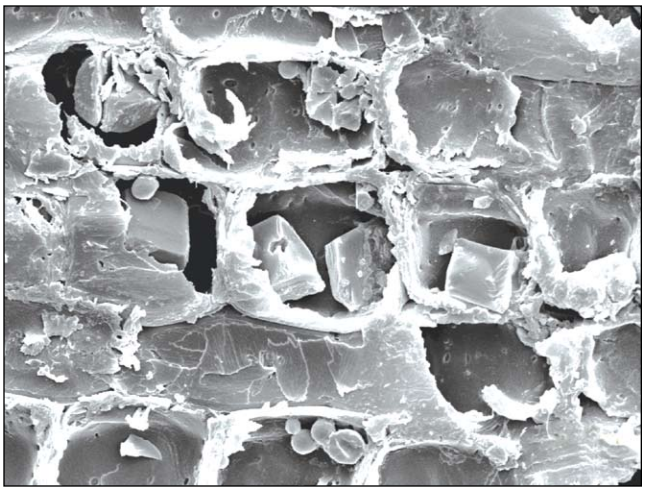

Figure 8—Scanning electron micrograph of ray cells of saltcedar containing salt crystals.

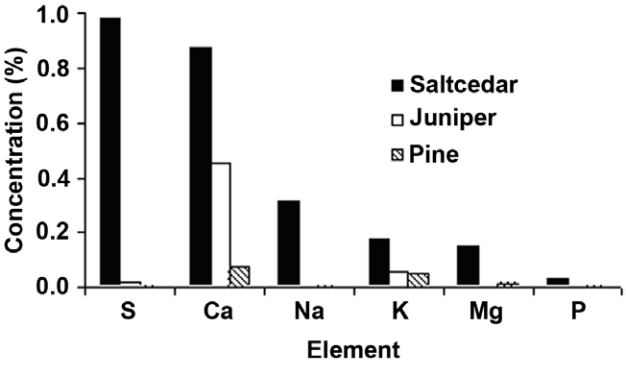

Figure 9—Mineral content determined by inductively coupled plasma (ICP) analysis of unextracted wood flour made from different wood species. Content of Fe, Zn, B, As, Cu, Pb, and Cd was less than 0.01% by weight.

Since 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. 8).

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. 9). Except for about 0.5% calcium in the juniper wood flour, very small amounts of minerals were found in the other two wood flours. The total content of the minerals investigated was 2.5%, 0.5%, and 0.2% of the total weight for the saltcedar, juniper, and pine wood flours, respectively. Since this is the mineral content only, the total salt content would be considerably higher.

Figure 10 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, were removed during the extraction, suggesting that a significant fraction of the water-soluble extractive content in Table 3 is salt.

Thermal Stability of Wood Flour

Thermal stability of wood fillers is important since WPCs are processed at temperatures as high as about 200°C (392°F). Wood's lack of thermal stability limits the number of plastics that can be processed with wood and the number of applications where WPCs can be used. Additionally, sometimes it is desirable to process even low melting-point plastics at higher temperatures to improve processing. For example, raising melt temperature can lower viscosity, decrease motor loads, and increase output during extrusion or can prevent premature solidification when injection molding thin-walled parts. Hence, processors sometimes push the processing temperatures, which can create volatiles that must be vented or cause discoloration and increased odor.

Figure 11 shows the weight loss rates for the investigated wood flours as a function of temperature as determined by TGA. All tests were run in a nitrogen atmosphere. 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°C to 325°C (Winandy and Lebow 2001). The faster degradation rate of saltcedar at low temperature suggests that it contains hemicelluloses that are less thermally stable.

To further investigate the relative thermal stability of the different species, isothermal tests were performed at temperatures more typical of WPC processing. After a drying step, the temperature was elevated to the desired temperature and held for 45 min. Figure 12 shows the weight loss curves for the different species at 93°C (200°F). Though weight losses were not large, even small ones can indicate the release

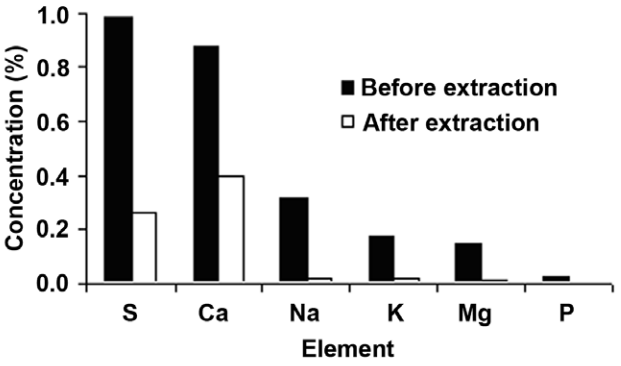

Figure 10—Mineral content determined by inductively coupled plasma (ICP) analysis of wood flour from saltcedar before and after a 4-h Soxhlet extraction with water.

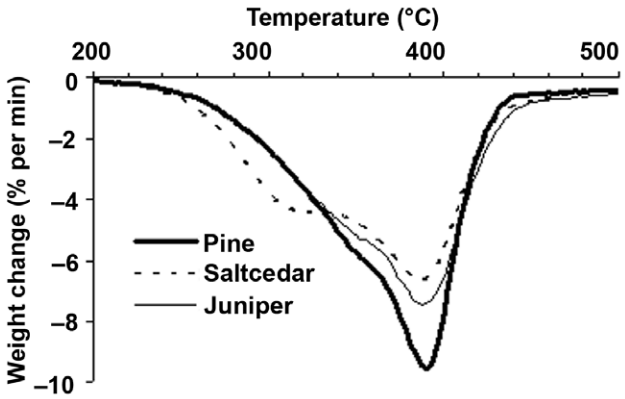

Figure 11—Weight loss rate with temperature for several species by thermogravimetric analysis (10°C/min heating rate under nitrogen).

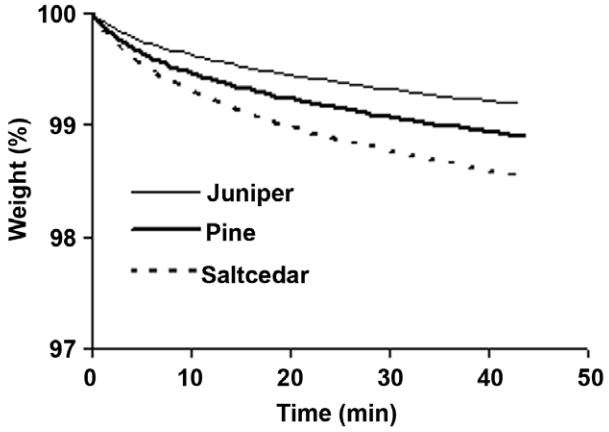

Figure 12—Weight loss of unextracted samples of several species under nitrogen at a constant temperature of 200°C (392°F).

of significant quantities of volatiles that need to be removed during processing to avoid undesirable voids in the finished product. The largest weight losses were found for saltcedar and the smallest for juniper. Since saltcedar also had the largest water-soluble extractive content and juniper the least

Table 3— Water-soluble extractive content of wood flours

	wt %	
Species	Room temperature [a] (%)	Hot water [b] (%)
Pine	3.4	5.0
Saltcedar	9.3	10.9
Juniper	2.4	4.4

[a] Water extraction at room temperature was performed according to ASTM D 1110 (ASTM 2005c).
[b] 4-h Soxhlet extraction in water.

(Table 3), at least part of the weight loss may be due to extractives. In similar tests on Soxhlet-extracted wood flours (Fig. 13), lower weight losses were found overall, and little difference was seen between the species suggesting that the weight loss differences between the species were due to extractives.

Rheology of WPC Blends

Processing equipment such as extruders and injection molders rely on the ability of molten materials to flow through barrels and dies and into cooled molds to blend constituent materials and form 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 melt-flow index is a crude but quick measure of viscosity that measures the amount of a melt forced through an orifice by a weighted piston (ASTM 2005d). Figure 14 shows the melt-flow indices for HDPE composites containing 50% wood flour of various particle sizes and species. Composites containing pine flour with mesh sizes of –80/+100 and –100 were not made because little wood flour of this particle size was present in the commercial wood flour. Composites with saltcedar of any particle size had the largest melt-flow indices (the lowest viscosity), whereas composites with pine had the lowest melt-flow indices.

Because of its potential importance in WPC processing, this species effect on viscosity was more thoroughly investigated using extrusion capillary rheometry (ASTM 2005e). 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 processing rates.

Figure 15 shows the viscosity curves for composite melts containing 50% wood flour (–40/+80 mesh). Viscosity decreased with increasing shear rate as is common for thermoplastics and their composites. Differences between the species were found over the entire shear-rate range investigated, although the greatest differences were at low shear

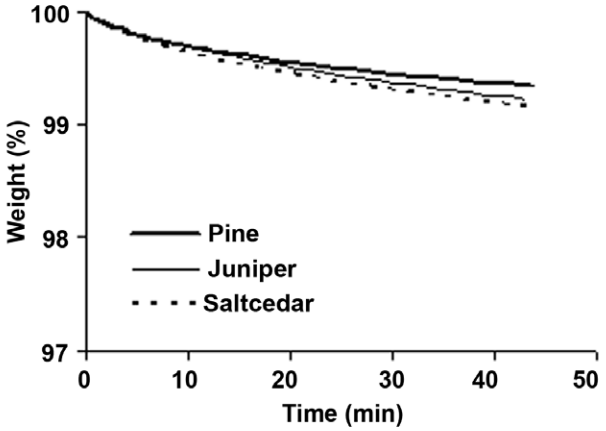

Figure 13—Weight loss of several species under nitrogen at a constant temperature of 200°C (392°F). Samples from wood flour extracted for 4 h in water using a Soxhlet extraction unit.

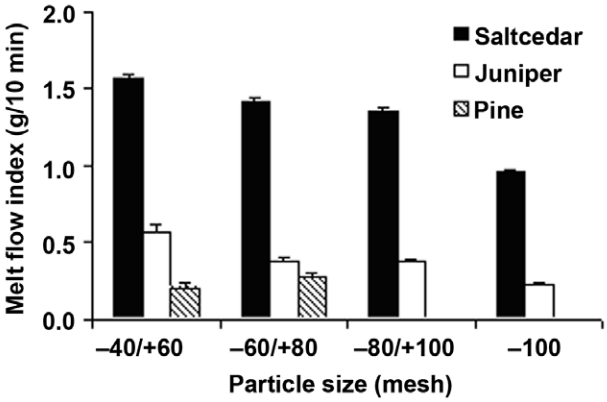

Figure 14—Melt-flow indices for HDPE composites containing 50% wood flour of various particle sizes and species according to ASTM D 5422-03 (ASTM 2005e). Composites containing pine flour with mesh sizes of –80/+100 and –100 were not made.

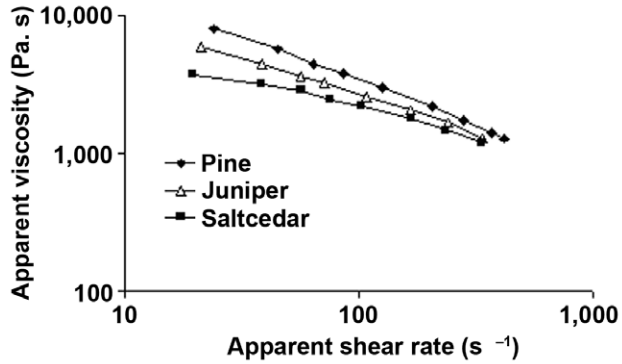

Figure 15—Viscosity curves for HDPE composites with 50% wood flour by extrusion viscosity capillary rheometry.

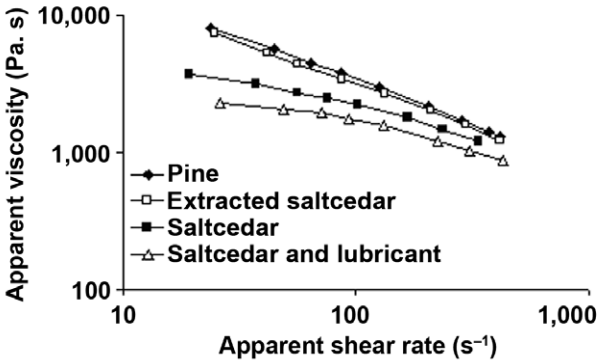

Figure 16—Viscosity curves for selected HDPE composites with 50% wood flour by extrusion viscosity capillary rheometry.

rates. 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 due to water-soluble extractives. When the extractives were removed from the saltcedar, this viscosity reduction disappears (Fig. 16).

In extruded WPCs, about 3% to 6% of lubricants and processing aids are added to reduce viscosity, increase output, and prevent tearing of the melt as it exits the extrusion die. Though the saltcedar extractives greatly reduced viscosity, they did not reduce viscosity as thoroughly as the addition of 6% of a commercial lubricant. However, this is the upper end of lubricant level used in extruded composites, and a lower level would likely result in higher viscosity. Even though extractives in saltcedar significantly reduced the viscosity of the composite melt, tearing of the melt was still a problem. Identification of an appropriate and inexpensive external lubricant to improve the surface appearance of the saltcedar blends could perhaps result in a more optimal composite formulation and might reduce the amount of lubricant needed. This could be advantageous in retaining mechanical performance (lubricants typically reduce them) and perhaps reduce cost. However, developing a new lubricant system for the saltcedar composite was beyond the scope of the current project.

Composites

Both injection-molded and extruded composites were successfully produced from all the wood flour species. Composition was carefully controlled in the injection-molded composites so that direct comparisons between the wood flour species could be made. Several practical formulational considerations such as the need for fines removal during wood flour production and the effects of additives on weatherability were explored in the extruded composites. More detailed information on the injection-molded and extruded composites is given below.

Injection-molded Composites

The compositions of the injection-molded WPCs are summarized in Table 1. In blends 1–3, an injection-molding grade of PP was used, and the wood flour content was

Table 4—Mechanical properties of injection-molded polypropylene (PP) with 40% wood filler

	Bending		Tensile		Izod impact	
Filler type	Modulus (GPa)	Strength (MPa)	Modulus (GPa)	Strength (MPa)	Notched (J/m)	Reverse notched (J/m)
Western pine	4.54 (0.03) [a]	57.4 (0.8)	4.59 (0.24)	28.2 (0.3)	27.3 (2.7)	64 (12)
Saltcedar (–40/+80 mesh) [b]	4.10 (0.12)	51.2 (0.7)	4.20 (0.07)	25.5 (0.6)	18.1 (4.6)	66 (14)
Juniper (–40/+80 mesh)	4.00 (0.02)	57.6 (0.8)	4.06 (0.05)	29.2 (0.1)	20.5 (2.3)	69 (5)

[a] Numbers in parentheses are one standard deviation.
[b] Particle-size fraction comprised of particles that pass through a 40-mesh screen but not a 80-mesh screen.

limited to 40% by weight to keep the viscosity low enough that specimens could be easily molded. No additives were used, which could potentially interact differently with the different species. Also, similar particle-size distributions were used so that direct comparisons between the species could be made.

The mechanical properties of the injection-molded composites are summarized in Table 4. Juniper composites had the lowest moduli, approximately 12% lower than that of the pine composites, but strengths were similar. Saltcedar had lower strengths and moduli than those of the pine composites, about 11% and 9%, respectively. The reversed notch-impact strengths for composites made with the invasive species were similar to the pine composites, but the notch-impact strengths were considerably lower. Generally, the mechanical properties decreased when pine flour was replaced with saltcedar or juniper flours. How much of an effect these property reductions would have on their use depends largely on the specifications of the application. For example, stiffness is very important in a deck board application but strength is less so. Where problematic, appropriate design changes (thicker boards or walls, ribbing) could be used to compensate for deficiencies.

The color of all WPCs produced using the invasive species were considerably darker than those made with the commercial pine flour. Though pigments and colorants can be added to WPCs to lighten the composites, the dark color from the invasive species will limit the color range that can be economically achieved. Alternatively, a dark color may be advantageous if such a color is desired and if the color is sufficiently stabilized.

Because it is important to prevent weathering of WPCs used in exterior building applications, weathering was investigated in more depth. During exposure, composite lightness (L*), flexural MOE, and flexural strength were monitored. The results are shown in Figures 17, 18, and 19, respectively.

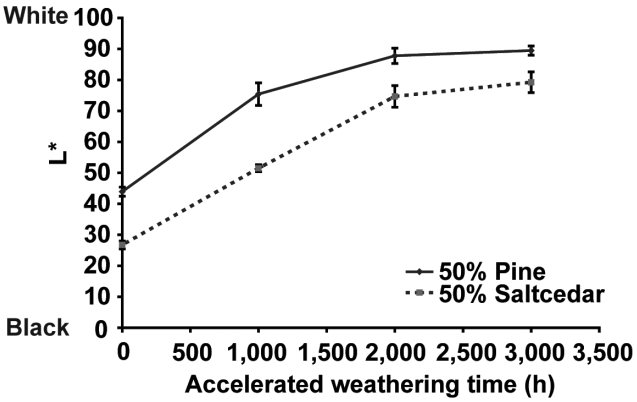

Figure 17—Composite lightness (L*) of injection-molded HDPE composites containing 50% wood flour (–40/+80 mesh) during accelerated weathering.

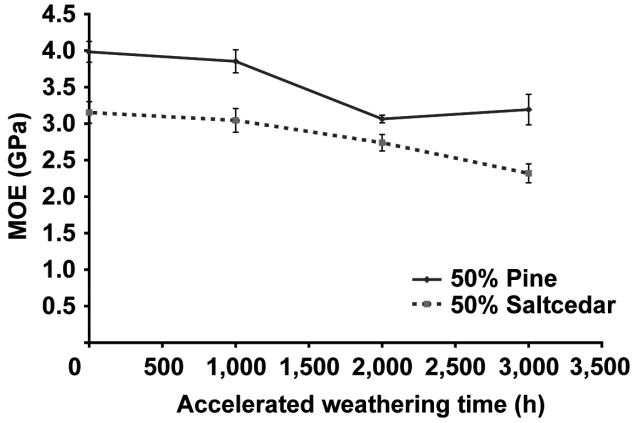

Figure 18—Flexural modulus of elasticity (MOE) for injection-molded HDPE composites containing 50% wood flour (–40/+80 mesh) during accelerated weathering.

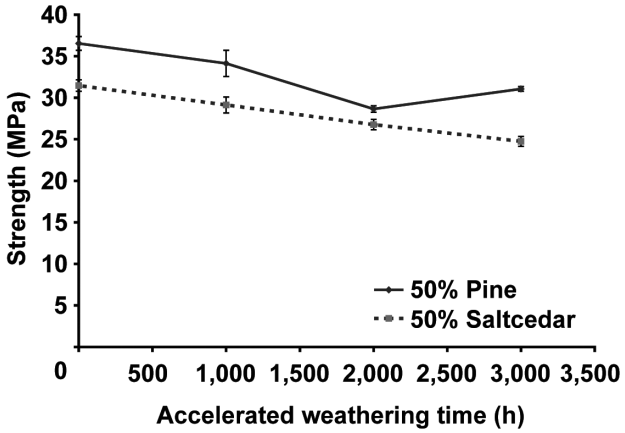

Figure 19—Flexural strength of injection-molded HDPE composites containing 50% wood flour (–40/+80 mesh) during accelerated weathering.

Figure 20—Extruded boards of HDPE containing 50% saltcedar (left), pine (center), or juniper (right) and lubricant and no coupling agent or UV package. See Table 2 for details on formulations.

Initially, the saltcedar composites were slightly darker than the pine composites (smaller value for L*). During weathering, L* increased for both composites (Fig. 17). The largest increases occurred early in the weathering period, with increases leveling off between 2,000 and 3,000 h of exposure. Overall, the saltcedar composites lightened more than the pine composites (L* increase of 52 versus 46, respectively). However, the saltcedar composites remained slightly darker than the pine composites throughout weathering.

The flexural MOE and strength of the saltcedar composites were lower than those of the pine composites before weathering (Figs. 18 and 19). During accelerated weathering, the MOE of the pine composites decreased through 2,000 h and then did not change between 2,000 and 3,000 h. The strength of the pine composites also decreased through 2,000 h but then increased slightly between 2,000 and 3,000 h. The MOE of both the pine and saltcedar composites decreased by 0.8 GPa. The strength of the pine and saltcedar composites decreased by 5 and 7 MPa, respectively. Mechanical property changes that occur in WPCs during weathering are discussed elsewhere (Stark and Matuana 2004).

Although the saltcedar composites initially were darker and had lower flexural properties, the results presented in Figures 17–19 indicate that saltcedar composites and pine composites perform similarly during weathering.

Extruded Composites

HDPE, the most common plastic used in extruded WPCs, was used in our extruded formulations. For extruded composites, a –40 mesh saltcedar and juniper wood flour was used (particles that pass through a 40-mesh screen) because large quantities of wood flour were needed, and removal of the fines (–80 mesh) significantly slowed down wood flour production and could potentially increase the cost of the wood flour in a commercial setting.

A lubricant was added to all extruded formulations to reduce viscosity, improve output, and prevent tearing of the surface of the composite as it exited the extruder die. These lubricants used in WPCs tend to migrate to the interface between the wood particles and the plastics. The larger interfacial area in the composites made from the finer wood flour (juniper and saltcedar) required that more lubricant be used (Table 2). Because wood flours with different particle sizes were used in the extruded composites, direct comparisons between species were not made. Consequently, direct species comparisons were made only with the injection-molded composites.

Several additives were also used in the extruded formulations. Besides lubricant, a coupling agent and UV stabilization package were added to some of the formulations to investigate their influence on composite performance (Table 2). As with the injection-molded composites, the color of all WPCs produced using the invasive species was considerably darker (Fig. 20) than those made with the commercial pine flour.

A scanning electron micrograph of a microtomed specimen taken from an extruded composite of HDPE containing 50% saltcedar and lubricant without any coupling agent or UV stabilizer package (formulation 9 in Table 2) is shown in Figure 21. The micrograph is a composite image of secondary electron and backscattered 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 where salt would accumulate. Figure 22a is a close up of a wood flour particle containing salt crystals. The x-ray spectra taken for one of these salt crystals is shown in Figure 22b and has similar proportions of sulfur and calcium as found in the ICP analysis (Fig. 9).

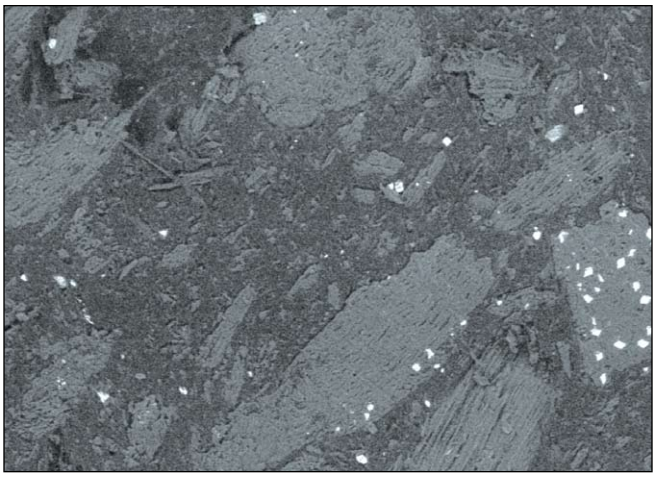

Figure 21—Scanning electron micrograph of microtomed specimen taken from an extruded composite of HDPE containing 50% saltcedar and lubricant (formula 9, Table 2). Micrograph is a composite image from 50% secondary electrons and 50% backscattered electrons; bright areas are salt crystals.

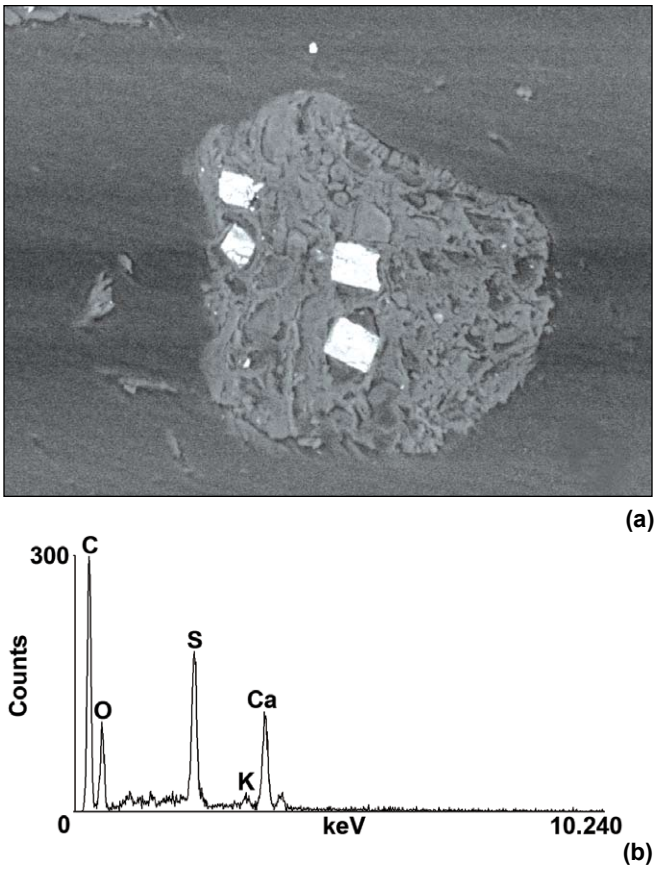

Figure 22—Scanning electron microscopy analysis of an HDPE–saltcedar wood flour composite: (a) close up of wood particle surrounded by HDPE and containing salt crystals, (b) x-ray spectra of one salt crystal showing the intensity versus energy in kiloeclectron volts (keV) for elements present in salt crystal.

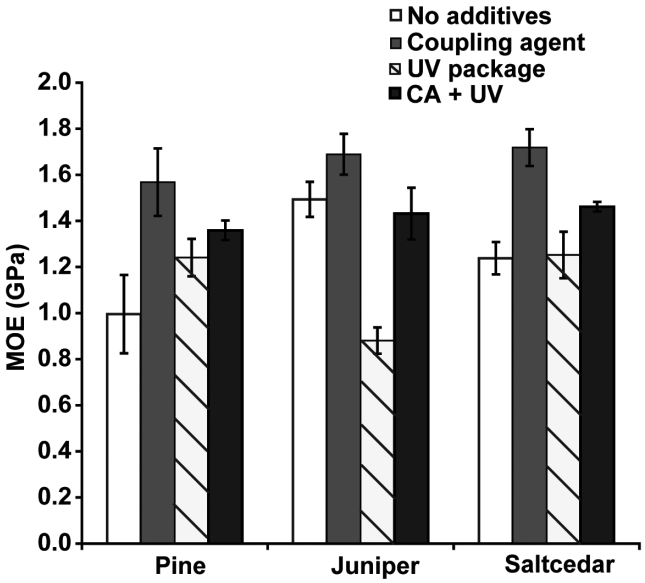

Figure 23—Flexural modulus of elasticity (MOE) of extruded HDPE composites with 50% wood flour.

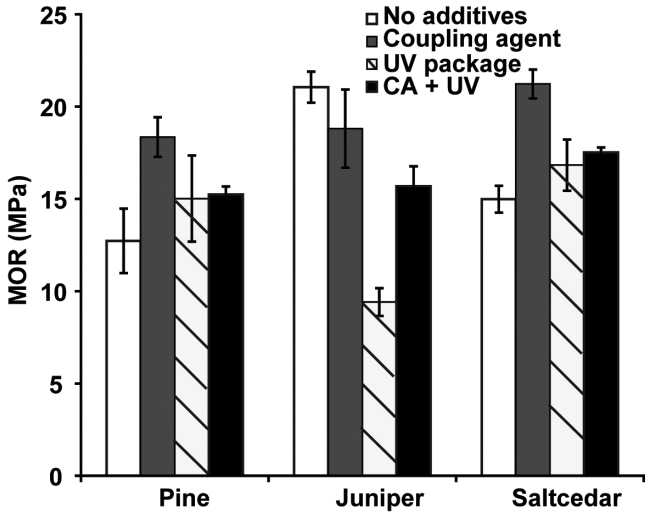

Figure 24—Flexural modulus of rupture (MOR) of extruded HDPE composites with 50% wood flour.

The flexural MOE and MOR of the extruded composites are shown in Figures 23 and 24, respectively. In general, the mechanical performance of the extruded composites is quite low. The formulations with the lowest mechanical properties—the pine composites without the UV package or coupling agent and the juniper composites with the UV package—also had the lowest densities (Fig. 25), indicating that the extruded boards were not of the same quality as the others. However, the mechanical properties of all of the blends were lower than expected, and further optimization of the manufacturing variables would be necessary if extruded composites from these invasive species were to be pursued.

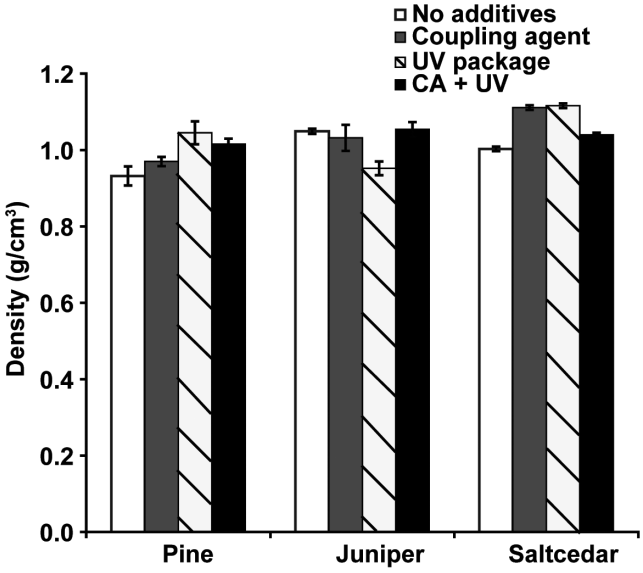

Figure 25—Density of extruded HDPE composites with 50% wood flour.

Figure 26—Extruded composite boards manufactured with pine wood flour, juniper wood flour, and saltcedar wood flour on a test rack for natural weathering in Madison, Wisconsin.

Some general trends were found regarding the influences of the additives. Adding the coupling agent to the composites increased the MOE for all composites and increased the MOR of the pine and saltcedar composites. Compared with composites with a coupling agent, the composites with the UV package performed worse, and both the MOE and MOR were lower. Adding the coupling agent with the UV package to the composites generally improved the mechanical performance over the composites with only the UV package added, but the performance still was not equivalent to the performance of the composites with only the coupling agent.

The 12 extruded composites (Table 2) are currently undergoing natural weathering in Madison, Wisconsin. They were installed on 90° racks (Fig. 26) on October 18, 2005. The L* of the composites has been monitored periodically, and the data are shown in Figure 27. Initially, the juniper and saltcedar composites were darker than the pine composites. For the lighter pine composites, the composites with the UV package, which consisted of an ultraviolet absorber and brown pigment, were darker than composites without. During natural weathering, the L* of all of the composites increased. Generally, the L* of the composites without the UV package have increased throughout the natural weathering cycle, while the L* of the composites with the UV package increased through the first 6 months of exposure, then did not change between 6 months and 9 months of exposure. The total change in L* for each composite during 9 months of exposure is reported in Table 5. It can be clearly seen that the addition of the coupling agent only did not dramatically reduce the lightening effect, but the addition of the UV package did. Additionally, the overall increase in L* was greater for the juniper and saltcedar composites compared with the overall increase for the pine composites. However, because the juniper and saltcedar composites were initially darker, the final L* of the pine composites was still higher than the final L* of the juniper and saltcedar composites.

Summary and Conclusions

Two invasive species, saltcedar (*Tamarisk ramosissima*) and Utah juniper (*Juniperus osteosperma*), were selected by the Bureau of Land Management (BLM) for investigation as fillers in wood–plastic composites (WPCs). Small saltcedar logs, previously harvested from the lower Colorado River near Yuma, Arizona, were chipped at the USDA Forest Service, Forest Products Laboratory (FPL). Chips of Utah juniper were provided by the BLM from wood harvested on BLM lands in Utah. Chips of both species were made into wood flour by hammermilling and screening. Chemical composition and thermal stability were determined and compared to a commercial pine wood flour.

Some differences were found in lignin content and the composition of the carbohydrate fraction. Saltcedar had the largest salt content of the species investigated. Saltcedar was found to contain the most water-soluble extractives, and its extract was significantly more colored than that of either pine or juniper flours. This suggests the potential for extractive leaching in WPCs used in exterior applications if care is not taken in formulating and processing.

Small weight losses were found in isothermal thermogravimetric analysis (TGA) tests at temperatures typical of wood–plastic composite (WPC) manufacture. Saltcedar lost the most weight; juniper the least. Species differences appeared to correlate with the water-soluble extractive content and their removal eliminated these differences. Volatiles resulting from thermal degradation would need to be vented during processing.

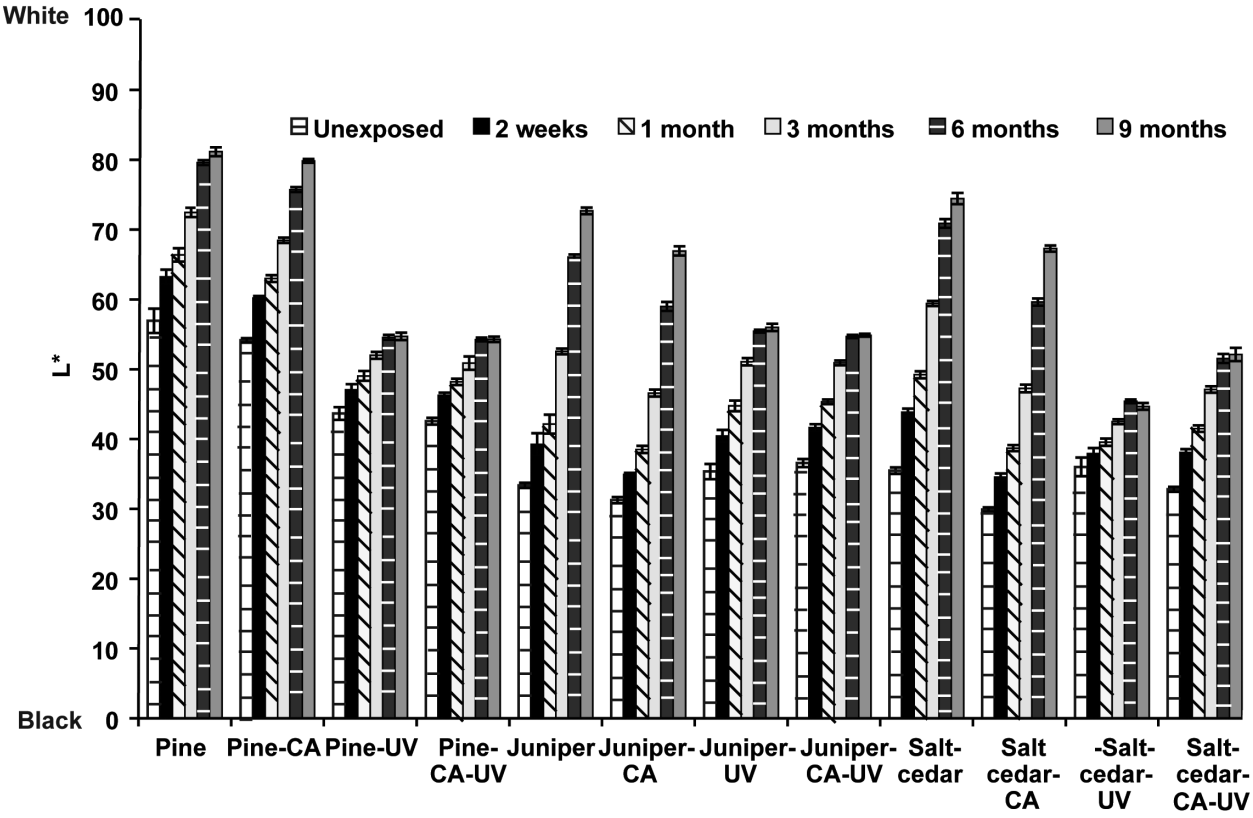

Figure 27—Composite lightness (L*) of extruded composites that have been exposed to natural weathering in Madison, Wisconsin.

Table 5—Increase in composite lightness (L*) of extruded composites with 50% wood after 9 months of natural weathering

Blend #	Wood species	Coupling agent	UV package	Change in L* [a]	
1	Pine	—	—	24.2	(1.8)
2	Pine	X	—	25.7	(0.4)
3	Pine	—	X	11.0	(1.0)
4	Pine	X	X	11.7	(0.6)
5	Juniper	—	—	39.3	(0.6)
6	Juniper	X	—	35.7	(0.8)
7	Juniper	—	X	20.7	(1.2)
8	Juniper	X	X	18.3	(0.6)
9	Saltcedar	—	—	39.0	(0.9)
10	Saltcedar	X	—	37.4	(0.5)
11	Saltcedar	—	X	8.7	(1.4)
12	Saltcedar	X	X	19.3	(1.0)

[a] Numbers in parentheses represent one standard deviation.

The wood flours were compounded with plastic and additives, and the viscosities of the composite melts containing the different species were compared. Water-soluble extractives significantly influenced the viscosity of the wood–plastic blends, especially at low shear rates. Saltcedar had the lowest viscosities, which could be advantageous in some processing scenarios. Adding a synthetic lubricant, a necessary additive when producing the extruded boards, overrode the effects of extractives on viscosity in extruded formulations. However, optimizing the synthetic lubricant package might help to take better advantage of rheological benefits of using saltcedar and possibly lead to cost savings.

Composites were successfully produced from the compounded material by profile extrusion and by injection molding. The mechanical performance, appearance, and weatherability of the composites were evaluated. In injection-molded composites, mechanical properties were generally lower for WPCs from invasive species. However, the impact of this difference would largely depend on the application and its performance targets. Additionally, design considerations could offset performance deficiencies if necessary. Wood–plastic composites made from both saltcedar and juniper were considerably darker than those made with pine, potentially limiting the color range that can be achieved and leading to greater color fade if not stabilized properly. However, the composites performed similarly to those made with a commercial pine wood flour in accelerated weathering tests.

Extruded composites could be made from each of the different species, but because of the large amount of fines in

composites containing the invasive species, more lubricant was required to make acceptable profiled boards from them. Material parameters such as particle size and additive type greatly influenced mechanical performance. Natural weathering tests on the extruded composites are ongoing.

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 from invasive species would not be problematic, and in some instances, may even be preferable. Development of formulation and evaluation of durability of these composites needs to continue so that informed decisions regarding applications can be made.

The economic feasibility of using invasive species 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. Even if more competitive markets such as deck boards were not economically feasible, less competitive or captive markets may offer opportunities for using these species in WPCs. For example, land managers could partner with manufacturers to make composite lumber from their invasive species to be used for trails and pedestrian bridges, where the combination of inherent durability with low maintenance is desirable. Some commercial entries of WPCs from other invasive species have already occurred. For example, in New Mexico, signs for state and federal agencies have been manufactured from small-diameter oneseed (*Juniperus monosperma*) juniper trees and recycled plastic milk bottles (Livingston 2006).

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