PROCESS-RELATED MECHANICAL DEGRADATION OF THE WOOD COMPONENT IN HIGH-WOOD-CONTENT WOOD–PLASTIC COMPOSITES

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Abstract. Micromorphological studies of wood–plastic composites (WPC) are crucial for deeper understanding of their physical, mechanical, and durability properties. The objective of this study was to examine process-related mechanical degradation of the wood component in an extruded high-wood-content WPC. WPC with ≈70% wood content and three distinctly different ground wood components were manufactured by a conical extrusion technology, ie WPC were prepared with an unmodified, acetylated, or thermally modified wood component. Size and shape of wood components were determined before and after the extrusion process. Micromorphology of WPC samples was studied using a scanning electron microscope (SEM) and a surface preparation technique based on UV laser ablation. This micromachining technique was also applied to prepare thin specimens for micromechanical analysis using a tensile stage mounted in a SEM. Results show that extrusion processes cause a significant mechanical degradation of the wood component. Degradation was most pronounced for the thermally modified wood component, and interestingly, this resulted in a more homogenous WPC micromorphology compared with WPC with unmodified and acetylated wood components. WPC with thermally modified wood also exhibited the highest micromechanical strength.

Keywords: WPC, particle size, micromorphology, acetylated wood, thermally modified wood, mechanical degradation.

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INTRODUCTION

During recent decades, wood–plastic composites (WPC) have rapidly increased their market share as a building material (Carus and Gahle 2008). Despite the recent economic downturn, which has leveled off this growth, a continued global market advance is predicted (Anon 2011). This success may partly be attributed to the fact that WPC are a competitive alternative to tropical hardwoods and that they are considered to require less maintenance than conventional wood products.

The majority of today's WPC are manufactured using a continuous thermoforming process, ie an extrusion process in which ground wood is mixed with a thermoplastic matrix. In general, most WPC properties depend on the thermoplastic used, normally polypropylene (PP), polyethylene, or polyvinyl chloride. In extruded WPC, the thermoplastic matrix is considered to partly encapsulate the wood component and this decreases moisture transport and increases resistance to microbial attack (Klyosov 2007). If wood content is greater than ≈50%, an interconnecting network of particles or fibers may be created and the encapsulation that slows moisture transport is lost (Caulfield et al 2005). When WPC are made with a high wood content, eg 70% by weight, decreasing the rate and level of moisture uptake is important to protect the wood component from swelling and microbial attack.

There are several ways of decreasing moisture uptake in high-wood-content WPC, eg chemical modification by acetylation or thermal modification of the wood component. Both modification concepts have been known for decades, but they have not gained notable commercial success until recent years (Hill 2006). Acetylation is a single-site reaction in which wood is impregnated with acetic anhydride, which reacts with wood hydroxyl groups at elevated temperature (Tarkow et al 1946; Stamm and Tarkow 1947; Rowell 2006). In the case of thermal modification, material is heated to between 180 and 260°C, which leads to chemical changes to the macromolecular wood constituents (Stamm 1964; Hill 2006). Both of these modifications give products increased dimensional stability, increased decay resistance, and lower equilibrium moisture levels compared with unmodified wood (Rowell et al 2009).

The wood component in WPC is generally in the form of wood meal (10-80 mesh), sawdust, shavings, or fibers (Caulfield et al 2005). Advantages of using wood rather than inorganic fillers, such as talc and calcium carbonate, are mainly that it is renewable, widely available, inexpensive, and nonabrasive. Higher filler content is also possible in WPC, making them more suitable for recycling than conventionally reinforced filler-containing plastics (Jacobson et al 1995). Major challenges for WPC as a building material are that they are hygroscopic and partly biodegradable, making them a risk for unacceptable dimensional instability and degradation by microorganism. The lignin component in wood is furthermore highly susceptible to degradation by UV radiation, which also may result in an accelerated UV degradation of wood–plastic combinations. Also, an obvious limitation during WPC processing relates to the risk for thermal degradation of the wood component, which means that they must be processed at lower temperatures compared with eg glass fiber-reinforced plastics.

The wood component may also be mechanically damaged when mixed with the plastic matrix in the extruder. To ensure complete mixing, a powerful shearing action is required. In this process, the wood geometry is changed and the wood structure is damaged (Rowell 2007). Rowell et al (1997) showed that length and width of oil palm fibers were decreased during processing into composites. In a study of injection-molded WPC, Segerholm et al (2007) found that the length-to-width ratio of unmodified and acetylated particles was decreased during processing into WPC. Gacitua et al (2008) used a nanoindentation technique to evaluate damage to the wood cell walls caused by processing into WPC. Processing in this case could decrease Young's modulus of the S2 layer of the wood cell wall by 40-70%. A difference was also observed between earlywood and latewood cells, in which the latter suffered less collapse and damage.

In this study, WPC with a wood content of about 70% were prepared using acetylated and unmodified Scots pine (*Pinus sylvestris* L.) and thermally modified Norway spruce (*Picea abies* Karst.) (Segerholm et al 2005, 2009; Larsson Brelid et al 2006; Westin et al 2006). PP was used as a matrix in the composites. Coupling agent, lubricant, temperature stabilizer, and UV absorber and stabilizer were also added to improve performance and processability of composites.

The objective of this study was to examine process-related mechanical degradation of the wood component in an extruded high-wood-content WPC prepared with unmodified, acetylated, or thermally modified wood. The study involves analyses of size and shape of the wood component before and after the extrusion processes as well as examinations of overall micromorphology and micromechanical behavior of different WPC samples.

EXPERIMENTAL

Wood Material

Wood material was prepared from acetylated and unmodified boards of Scots pine sapwood and thermally modified boards of Norway spruce, about 100 kg for each sample. The wood acetylation process followed principles described by Rowell et al (1986) in a pilot plant with a microwave-heated reaction vessel (0.67 m^3). The degree of acetylation was about 20% expressed as wood acetyl content. Thermal modification was performed by Stora Enso Oyj, Helsinki, Finland, according to the ThermoWood D procedure, which has a peak temperature of 212°C (Anon 2003). From the boards, ground wood compounds were prepared by a two-step process. First, boards were cut into 190-mm-long blocks, which were fed into a disk flaker (Bezner, Ravensburg, Germany) and processed into thin veneer strands. Then veneer strands were fed into a dry grinding knife-mill (Condux, Mankato, MN) and chopped into fine particles. The procedure involving these two steps created about the same size and shape distribution in both unmodified pine sapwood and unmodified Norway spruce (Segerholm 2006). Differences in size and shape reported in this article are therefore not attributed to differences in species but to different modifications.

Wood Component Size and Shape Analysis before Extrusion

Size and shape distributions of ground wood components were characterized by sieve analysis and light microscopy (Olympus BX51, Center Valley, PA). In sieve analysis, wood components were divided into five different mesh fractions, –120 (<0.125 mm), –60 + 120 (0.125-0.25 mm), –35 + 60 (0.25-0.5 mm), –18 + 35 (0.5-1.0 mm), and +18 (>1.0 mm). Approximately 100 g of each wood component was used for sieve analysis. The largest sized fraction, not passing through the 18-mesh screen, amounted to <1% in all types of wood components and was neglected in subsequent analysis. Lengths and widths of the ground wood component were determined with light microscope image software (analySIS; Version 5.0; Olympus Soft Imaging System GmbH, Münster, Germany), and at least 100 particles of each fraction were measured. Based on these measurements, length-to-width (L/W) ratios were calculated. The term aspect ratio was not used in this case because of the irregular form of wood components.

Composite Manufacture

The thermoplastic matrix used was PP (Moplen HF 500N; LyondellBasell, Houston, TX). Unmodified and modified wood components were dried to <1% MC, mixed with PP and additives, and compounded in a counterrotating twin screw extruder at OFK Plast AB in Karlskoga, Sweden. Additives used were 2% w/w coupling agent (Licomont AR504), 2.9% w/w lubricant (Licomont ET 141; Clariant GmbH, Augsburg, Germany), 1% w/w temperature stabilizer (AmeriChem 56082 P-1, Cuyahoga Falls, OH), 0.7% w/w UV stabilizer (Cyasorb UV-3808PP5; Cytec Industries, Inc., Woodland Park, NJ), and 0.1% w/w UV absorber (Cyasorb UV-531).

Additives used were the same for all three formulations, and their influence on composite performance was not further investigated in this study. Pellets (compound) based on unmodified, acetylated, and thermally modified wood produced had about 70% wood content based on dry weight. The pellets were then fed into a conical extruder (Conex; Conenor Ltd., Tampere, Finland) and extruded into rectangular-shaped hollow profiles. Melt temperature was between 180 and 185°C, and die pressure was about 10 MPa. Approximately 40 m of the profile was extruded with each formulation. Cross-section of the hollow WPC profiles was 60 × 40 mm^2 with 8 mm thick walls (Fig 1a).

Wood Component Size and Shape Analysis after Extrusion

The PP matrix was extracted from the wood component to examine size and shape of the wood component after it was processed into a composite. Two specimens of each material, measuring 10 × 10 × 8 mm^3, were prepared from profiles, and the PP matrix was extracted in a soxtec (Foss, Höganäs, Sweden) extractor with xylene as a solvent. Specimens were subjected to boiling xylene solvent until all PP had dissolved after approximately 4 h. The resulting solution was poured through a fine mesh to retrieve the wood component. Residue was then washed with hot xylene, dried at room temperature, and finally spread on microscope slides. Lengths and widths of extracted particles were studied in a light microscope without any further fractionation. About 100 particles were measured of each sample. Length-to-width ratio was used to describe the form of the wood component, because the wood component might not only have been shortened in length along the grain, but it may also have been split transverse to the grain.

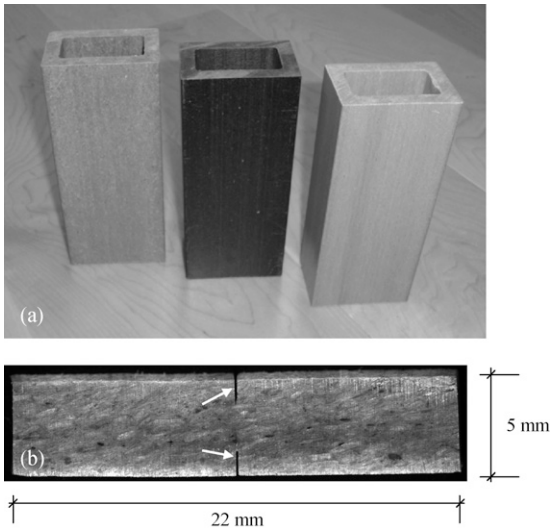

Figure 1. (a) Extruded wood–plastic composite (WPC) profiles (from left, unmodified, thermally modified, and acetylated wood component) and (b) microtensile specimen (arrows indicate UV excimer laser-ablated notches).

Micromorphological and Micromechanical Scanning Electron Microscope Analyses

From hollow WPC profiles, small samples measuring 22 × 5 × 7 mm^3 were prepared using a band saw. These samples were then further micromachined into thin veneer-like tensile test specimens by means of a UV excimer laser ablation technique. The technique for ablating wooden materials is further described by Seltman (1995) and Wålinder et al (2009). Specimens for micromechanical analysis measured 22 × 5 × 1.1 mm^3. A UV excimer laser ablated notch 1 mm deep and 0.5 mm wide was placed on each side of the specimen (Fig 1b). These notches ensured a crack propagation across the region viewed in a scanning electron microscope (SEM), Model JSM-5310LV Jeol (Tokyo, Japan). Specimens for the tensile test were prepared both in the extrusion direction and perpendicular to the extrusion direction of the original WPC.

Flat surfaces of the specimens were first examined with regard to their overall micromorphology, including size and shape, and also compression or lumen filling of the wood component. Specimens were mounted in a tensile test stage with a 50-N loadcell, and the tensile test stage was then placed inside the SEM chamber. Specimens were loaded with a stepwise increase of 5 N in the load. New micrographs were

acquired at each load level until ultimate load was reached. Ten specimens of each formulation and loading direction (perpendicular and parallel to extrusion direction) were tested. These tests do not provide mechanical data fully comparable with macroscopic mechanical data for the materials, but they provide a basis for comparing effects of size and shape of different wood components along and across the extrusion direction. It is important to remember that these SEM micrographs were captured stepwise at different loadings with intermediate relaxation and that notches acted as crack initiators when material was stressed. Results from strength tests were analyzed by statistical methods to evaluate possible statistically significant differences in mean values. The significance test was performed with analysis of variance (Tukey) using a 95% confidence limit and a 0.05 significance level.

RESULTS AND DISCUSSION

Wood Component Size and Shape Analysis before Extrusion

Figure 2 shows size distributions according to sieve analysis of the three ground wood components before extrusion. As shown, unmodified and thermally modified wood components had a similar size distribution, whereas acetylated wood had a larger amount of the –35 + 60 mesh fraction. Mesh fractions were further analyzed in a light microscope to determine length, width, and L/W ratios of the wood component (Table 1). Figure 3 visually summarizes shape and size of these different mesh fractions. Figure 3a shows a series of light microscope images of different mesh fractions for the thermally modified wood component, and as shown, all fractions contained a large portion of a splinter-shaped wood component in accordance with the comparably high average L/W ratio of 10.4 presented in Table 1. Figures 3b and c show a corresponding image series for acetylated and unmodified wood components, respectively. Approximately 70% of the ground acetylated wood consisted of the –35 + 60 mesh fraction with a high portion of a splinter-shaped wood component, also in this case in accordance with the comparably high overall average L/W ratio of 7.8 (Table 1). The unmodified wood component had the lowest average L/W ratio of examined wood components.

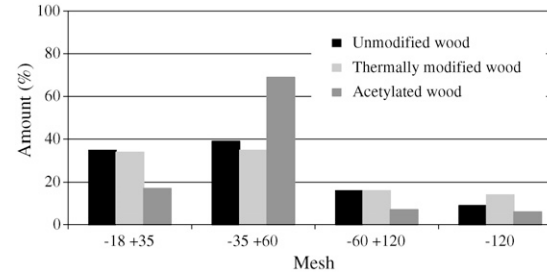

Figure 2. Size distribution of wood components before wood–plastic composite (WPC) processing.

Table 1. Average length, average width, and length-to-width (L/W) ratio of wood components before and after processing (standard deviations are shown in parentheses).

Type of wood component	Average length (μm)	Average width (μm)	L/W ratio
Unmodified pine			
Before processing	1277 (589)	302 (143)	5.5 (3.8)
After processing	533 (314)	170 (105)	3.5 (1.4)
Thermally modified spruce			
Before processing	578 (306)	80 (70)	10.4 (7.0)
After processing	125 (150)	36 (53)	3.9 (1.5)
Acetylated pine			
Before processing	874 (547)	143 (91)	7.8 (5.4)
After processing	355 (192)	67 (44)	6.6 (3.8)

These results clearly show that the thermally modified wood is split into a splinter-shaped wood component during the grinding process compared with the unmodified wood. This is probably because of the fact that the wood matrix polymers of thermally modified wood were partly degraded (Hill 2006; Rowell et al 2009), probably resulting in an altered, more brittle fracture behavior during normal machining operation. Furthermore, acetylated wood was fixed in a swollen state, and results shown here indicated that acetylated wood therefore may have been split into a more slender and splinter-shaped wood component compared with unmodified wood. In addition, the lower equilibrium

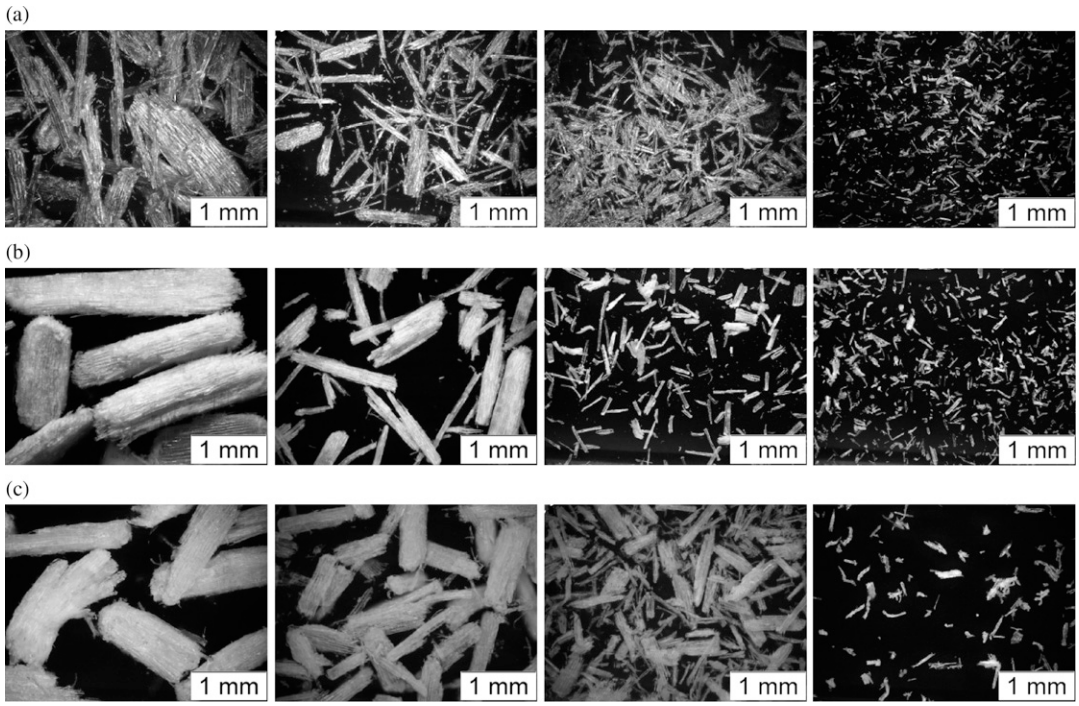

Figure 3. Shape and size of different wood components before wood–plastic composite (WPC) processing (a) thermally modified spruce, (b) acetylated pine, and (c) unmodified pine in four different mesh fractions: from left to right –18 + 35, –35 + 60, –60 + 120, and –120.

moisture content of the modified woods means that they were more brittle than unmodified wood.

Extracted Wood Component Size and Shape Analysis after Extrusion

Light microscope analysis of wood components extracted from extruded WPC profiles showed that all three types of wood components were mechanically degraded during extrusion processes. Table 1 presents average lengths and widths as well as L/W ratio of the three different wood component samples. As shown in Table 1, extrusion processes clearly significantly decreased both average length and width. For unmodified and thermally modified wood components, a significant decrease of average L/W ratio was also observed.

Table 1 also shows that the thermally modified wood component was the most mechanically degraded during processing. Interestingly, average width of thermally modified particles was equal to the diameter of a normal softwood tracheid, although the standard deviation was large because of the presence a few larger wood tissues built up of fiber bundles. Figure 4a shows a light microscope image of the extracted thermally modified wood component after processing. This image shows that a large portion of the thermally modified wood component has been split into cell wall fragments during WPC processing. This suggests that the morphology of the wood component, as expected, was severely altered during the previous thermal modification process and possibly also further degraded during extrusion processes. These thermally induced alterations possibly occur both at the micro- and molecular level because of partly degraded wood matrix polymers, ie hemicelluloses and lignin (Hill 2006). Interestingly, however, this degradation resulted in a more homogeneous WPC

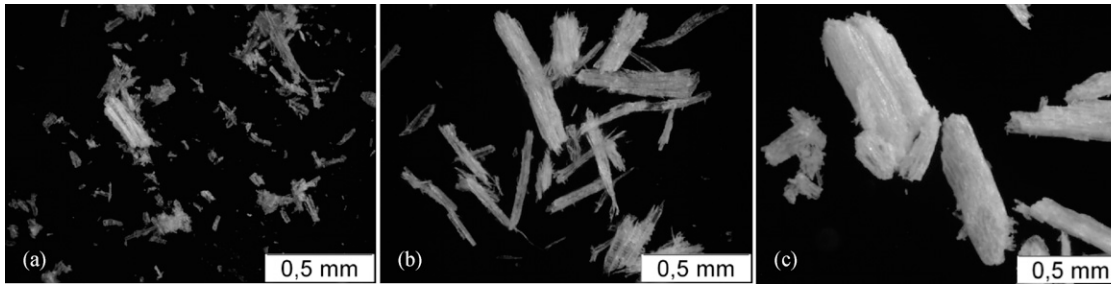

Figure 4. Wood components extracted from extruded wood–plastic composites (WPC) with (a) thermally modified spruce, (b) acetylated pine, and (c) unmodified pine.

microstructure with a distinctly decreased size and size distribution of the wood component compared with unmodified wood component WPC (Fig 5). Similarly, WPC with a thermally modified wood component were also more homogeneous than those with acetylated wood.

Figure 4b shows the corresponding extracted acetylated wood component after processing. As indicated, the acetylated wood component consisted mostly of single tracheids or larger fiber bundles with a large size distribution. L/W ratio of the acetylated wood component was fairly constant before and after extrusion processing. Figure 4c shows the extracted unmodified wood component after processing. The wood component in this case was not split into single tracheids during WPC processing, although a large size distribution could be observed.

Micromorphological and Micromechanical Scanning Electron Microscope Analyses

SEM micrographs of the UV excimer laser-ablated WPC specimens revealed that all materials contained a larger fraction of cell wall fragments compared with the shape and form of the wood components prior to processing. The highest amount of cell wall fragments were found in WPC specimens with a thermally modified wood component. It was obvious that the main micromorphological differences among samples related to wood component size. Specimens with acetylated and unmodified wood components had a higher portion of larger sized wood tissues than specimens with the thermally modified wood component. One important feature of WPC micromorphology that was clearly observed in this study was that lumens of visually intact tracheids (ie tracheids that are not compressed or collapsed) were filled with PP matrix in all specimens. It was also obvious that the wood component was predominantly oriented in the extrusion direction, but some random orientation could also be seen.

Table 2 presents micromechanical tensile strength data of WPC specimens obtained by the tensile test stage in SEM. WPC with unmodified and acetylated wood components performed similarly in both directions with no significant differences. WPC with the thermally modified wood component had a notably higher strength than the other two formulations, ie about 30% higher in the extrusion direction and about 70% higher perpendicular to the extrusion direction. These results suggest that the comparably larger wood components present in specimens will induce increased stress concentrations at the interface between the wood component and the matrix compared with smaller wood components. This would be even more devastating if these materials were stressed perpendicular to the extrusion direction (Table 2). Another possible influence could be related to the altered surface free energy of the thermally modified wood, ie a less hydrophilic characteristic (Bryne and Wålinder 2010). In this case, this could mean that the thermally modified wood could be more compatible with the hydrophobic PP matrix. The added coupling agent may, however, oppose such an effect.

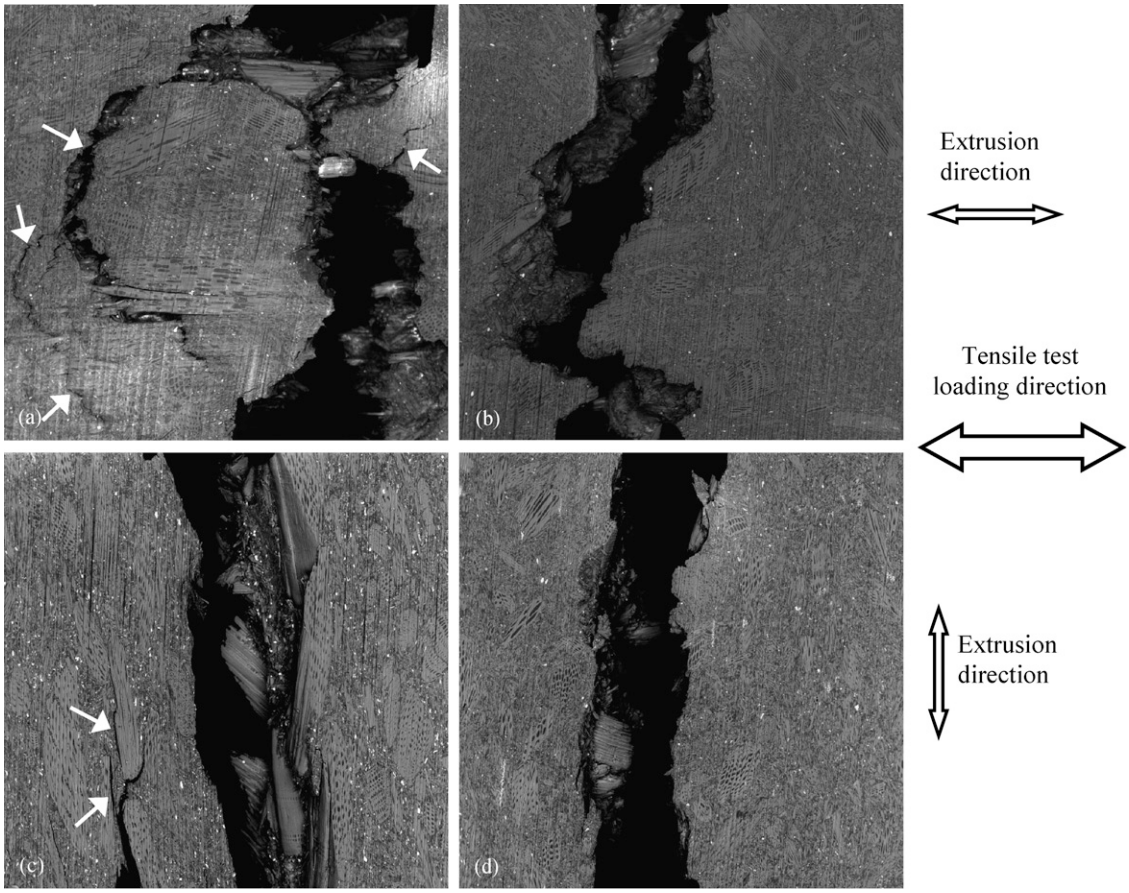

Figure 5. Scanning electron microscope (SEM) micrographs of fracture zones in tensile-tested wood–plastic composite (WPC) specimens in direction of extrusion with unmodified (a) and thermally modified (b) wood components and perpendicular to extrusion direction with unmodified (c) and thermally modified (d) wood components. White arrows indicate additional cracks in tested WPC specimens with unmodified wood.

Table 2. Micromechanical tensile strength of wood–plastic composites (WPC) in extrusion direction (∥) and perpendicular to extrusion direction (⊥).

Type of wood component in WPC	Strength∥ (MPa)	Strength⊥ (MPa)
Unmodified pine	14.3 (2.1)[A]	6.3 (1.5)[C]
Thermally modified spruce	18.0 (3.1)[B]	10.5 (2.1)[D]
Acetylated pine	14.3 (1.6)[A]	6.0 (0.8)[C]

[a] Standard deviations are shown parentheses. Statistically significant differences are represented with different letters.

Figure 5 shows micrographs of fracture zones of tensile-tested WPC specimens with unmodified and thermally modified wood components. Specimens in Figs 5a and b were tested in the direction of extrusion, and specimens in Figs 5c and d were tested perpendicular to the extrusion direction. Specimens tested along the extrusion direction showed an extended and more complicated fracture path than did specimens tested perpendicular to the extrusion direction. This difference is attributed to orientation of larger sized wood components. Specimens with a thermally modified wood component showed a less irregular fracture path than the ones with acetylated and untreated wood, presumably because of the absence of larger sized wood components within fracture zones. For specimens with unmodified and acetylated wood components, several additional cracks occurred at zones close to the fracture path (Figs 5a and c, white arrows).

CONCLUSIONS

In this study, it is shown that mechanical action during extrusion processing of a high-wood-content WPC severely damaged the wood component. This was most pronounced in the case of WPC with a thermally modified wood component, which after processing exhibited significantly shorter and more damaged wood tissues than before the extrusion process. Before processing, L/W ratio was highest for the thermally modified wood component in all analyzed size fractions followed by the acetylated wood component. After processing into composites, average length of the thermally modified wood component was significantly shorter than that of both unmodified and acetylated wood components. Composite samples prepared with acetylated wood contained the highest average wood component L/W ratio.

Based on micromechanical analysis of WPC, it was concluded that size of the wood component plays a central role in WPC fracture behavior. In this case, larger wood components in WPC have less ability to redistribute stresses in the material compared with smaller wood components. Micromorphological analysis of fracture zones showed that such larger wood components were more frequently present in WPC with unmodified and acetylated wood than in WPC with thermally modified wood.

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