
Density, heating value, and composition of pellets made from lodgepole pine (*Pinus concorta* Douglas) infested with mountain pine beetle (*Dendroctonus ponderosae* Hopkins)

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Zaini, P., Sokansanj, S., Bi, X., Lim, C.J., Mani, S., Melin, S. and Kadla, J. 2008. **Density, heating value, and composition of pellets made from lodgepole pine (*Pinus concorta* Douglas) infested with mountain pine beetle (*Dendroctonus ponderosae* Hopkins).** Canadian Biosystems Engineering/Le génie des biostèmes au Canada 50: 3.47-3.55. Mountain pine beetle (MPB) (*Dendroctonus ponderosae* Hopkins) has become a serious infestation problem in Western Canada. The infested lodgepole pine (*Pinus concorta* Douglas) becomes host to blue staining fungi and the infested trees die in 4-6 years. The dead trees gradually lose their suitability for dimension lumber and pulp chips due to excessive cracking and spoilage. Recovering the killed wood and processing it to pellets for bioenergy and other applications could partially avert the economic loss, and reduce overloading of the forest floor and potential for forest fires. In this study, heating value, density, and chemical composition of pellets made from MPB-infested Pine wood were measured and compared with those measured for pellets made from uninfested pine. Chemical analysis showed minor decrease in lignin and sugar contents of pellets made from MPB wood. The mean density of pellets manufactured from MPB (about 975 kg m⁻³) was slightly higher than the density of pellets from uninfested wood (934 kg m⁻³). This difference was not statistically significant ($\alpha = 0.05$) due to a large variation in the measured data. The high heating value (HHV) of MBP at a mean 19.20 MJ kg⁻¹ was not significantly different from 19.05 MJ kg⁻¹ measured for pellets made from uninfested pine. Biologically cultured MPB pellets did not show any staining fungi growth. The overall conclusion is that MBP infested wood can be used to produce comparable pellets to non-infested wood pellets. **Keywords:** mountain pine beetle, *Dendroctonus ponderosae*, lodgepole pine, *Pinus concorta*, pellet, density, heat value, blue stain fungi, chemical composition, spruce, fir.

Le dendroctone du pin ponderosa (DPP) (*Dendroctonus ponderosae* Hopkins), est devenu une cause d'infestation sérieuse dans l'Ouest canadien. Les pins tordus (*Pinus concorta* Douglas) infestés sont devenus les hôtes de champignons de bleuissures et les arbres infestés meurent après 4 ou 6 ans. Les arbres morts perdent graduellement leur valeur comme bois de sciage et copeaux de bois dû à un craquage excessif et à la pourriture. La récupération du bois mort et la transformation de celui-ci en cubes pour la production de bioénergie ou pour d'autres fins

pourrait permettre de limiter les pertes économiques et réduire la surcharge des sous-bois et les feux de forêt potentiels. Dans cette étude, la valeur calorifique, la densité et la composition chimique des cubes faits à partir de pins infestés par le DPP ont été mesurées et comparées à celles évaluées à partir de cubes provenant de pins non infestés. Les analyses chimiques ont montré une faible diminution du contenu en lignine et en sucre des cubes faits à partir de bois infesté par le DPP. La densité moyenne des cubes fabriqués à partir de bois DPP (environ 975 kg m⁻³) était un peu plus élevée que la densité des cubes provenant de bois non infesté (934 kg m⁻³). Cette différence n'était pas statistiquement significative ($\alpha = 0,05$) à cause de la grande variabilité des données mesurées. La valeur calorifique de 19,20 MJ kg⁻¹ du bois DPP était supérieure à celle de 19,05 MJ kg⁻¹ provenant de cubes de pin non infesté; toutefois ces valeurs n'étaient pas statistiquement différentes. Les cultures biologiques faites à partir de cubes DPP n'ont démontré aucune croissance de champignons à bleuissures. La conclusion générale de cette étude est donc que le bois infesté de DPP peut être utilisé pour produire des cubes qui sont comparables aux cubes provenant de bois non infesté. **Mots-clés:** dendroctone du pin ponderosa, *Dendroctonus ponderosae*, pin tordu, *Pinus concorta*, cube, densité, valeur calorifique, champignon de bleuissure, composition chimique, épinette, sapin..

INTRODUCTION

British Columbia is one of the major forest resources in Canada with 12 million ha of lodgepole pine species (*Pinus concorta* Douglas) located in the Interior of the province (COFI 1996). The province is currently experiencing the largest recorded mountain pine beetle (*Dendroctonus ponderosae* Hopkins) outbreak in North America. Approximately 7 million ha or roughly 450 million cubic meters of lodgepole pine have been killed to date. Projections suggest that approximately 80% of the 12 million ha will be killed by 2014.

Mountain pine beetles (MPB) bore through the bark into the phloem layer where they feed on sap and lay eggs. This causes discoloration and degradation of the internal

structure of lodgepole pine. Reductions in specific gravity, lignin content, and extractives in MPB-affected wood have been reported (Chapman 1940; Schirp et al. 2003; Woo et al. 2005), along with increasing water permeability (Woo et al. 2005). The beetles also carry blue stain fungi, which block the production of defensive resins to fight the beetle attack. The fungi destroy the phloem layer, cutting off the flow of water and nutrients to other parts of the tree. Pine needles turn a reddish color as the tree dies, and eventually a grayish color, which is a sign of a completely killed tree.

According to Uzunovic et al. (1999) fungi of the *Ascomycota* genera *Ophiostoma* and *Ceratocystis* are responsible for blue stain. Gao et al. (1994) showed that the staining fungi survive on extractives and not on cellulose, hemicelluloses or lignin. However, others have proposed the enzymes produced by fungi might also degrade hemicelluloses (Schirp et al. 2003). Ballard et al. (1983) concluded that the sap-staining fungi attack and destroy parenchyma cells. The fungi may also degrade extractives such as triglycerides, fatty acids and resin acids available in the parenchyma cells.

Lignin is one of the components responsible for the flexibility of wood (Gindl et al. 2002). According to Crawford (1981) fungi do not attack lignin and cellulose. But observed infestation indicates that lignin degradation is mostly caused by white rot fungi attack of the dead tree (Watson 2005). Hemicelluloses are preferentially removed when lignin is degraded by white rot fungi (Blanchette and Abad 1988). Dos Santos Abreu et al. (1999) studied the effect of lignin on fiber elasticity. They observed decreasing fiber elasticity with decreasing lignin content. Reduction in elasticity might cause brittleness, which has been observed in the grinding operation of MPB-infested wood (Byrne 2005).

Wood biomass can be densified to form pellets to enhance the energy-to-volume ratio of biomass in order to reduce energy and cost during storage, handling and transportation (Robinson et al. 2003; Sokhansanj et al. 2005; Mani 2005). Pellets are produced by several manufacturers throughout BC and other Canadian provinces. The pellets are used mostly for fuel, with some being used for animal bedding. Currently, Canada exports roughly 750 000 t of wood pellets to Europe as a fuel for heat and power (Swaan 2006).

Several studies have reported the properties of densified wood residues in various process and environmental conditions (Sjostrom 1993; Chin Chin and Siddiqui 2000; Li and Liu 2000; Lehtikangas 2001). The most important physical properties of wood pellets are bulk and pellet density, heating value, moisture content, and durability (Stahl et al. 2004; Obernberger and Thek 2004; Sokhansanj et al. 2005). Table 1 summarizes the important properties of wood pellets and their relevance to industrial applications.

The production of wood pellets from mill residue involves initial shredding (chipping) of the wood, drying, grinding and compacting. The wet biomass is dried in a rotary drum dryer to a moisture content of about 10% (Mani 2005). The dried biomass is ground in a hammer mill, compacted in a pellet mill and stored as bulk or in

Table 1. Pellet properties and their importance for subsequent handling and utilization.

Parameter	Importance
Moisture content	Storability, caloric value, self ignition
Heat value	Fuel utilization, plant design, economic value
Fungi spores	Health risk during fuel handling
Bulk density	Transport and storage expenditures
Particle density	Combustion properties, specific heat conductivity, rate of gasification
Durability	Physical damage during transshipment

bags. Moisture content, density, heating value, durability, hardness and color of pellets depend upon biomass species and plant part (heartwood, sapwood, or bark); particle size (chips, shavings, or saw dust); and process conditions (drying temperature, compaction pressure and temperature) (Rhen et al. 2005; Tabil and Sokhansanj 1996). Table 2 lists the measured physical and chemical properties of a number of commercially produced pellets in British Columbia. The commercial pellets are 6.5 mm in diameter with a length of almost twice the diameter. The density of a single pellet is 1.15 g/cm³ (1150 kg/m³). Moisture content of commercial pellets is at 5.2% (s.d. = 1.4%). The pellets have a low ash content of about 0.3%.

Objective

Owing to the chemical and structural changes reported with MPB attack, it is important to develop engineering data on properties of MPB-infested pine for wood pellets. The objective of this research is to compare chemical composition, density, and heat value of pellets made from mountain pine beetle infested wood and to compare these properties with those measured for pellets made from uninfested wood. This paper also presents density data for a few pellets made from spruce and pine wood chips.

Table 2. Some of the physical and chemical properties of commercial pellets measured by authors. Wood pellets were from a mixture of spruce, pine and fir grown in British Columbia.

Property	Unit	Number	Average	Std dev.
Diameter	mm	80	6.5	0.1
Length	mm	80	13.4	4.7
Unit density	g/cm ³	40	1.15	0
Number of pellets in 100 g		2	260	27
Heating value	MJ/kg	2	19.572	0.453
Moisture content	% wb	2	5.2	1.4
Ash content	% dry	2	0.3	0.1
Cellulose	% dry	2	39.5	1.5
Hemicelluloses	% dry	2	20.4	0.3
Lignin	% dry	2	28.7	0.0
Extractives	% dry	2	4.4	0.3
Carbon	% dry	2	47.1	0.1

MATERIALS AND METHODS

Wood chips of healthy (uninfested) pine (*Pinus contorta* Douglas) and MPB-infested pine (2 years since attack) and wood chips of white spruce (*Picea glauca* Voss) and Douglas fir (*Pseudotsuga menziesii* Franco) were provided by Paprican Canada. Samples were all fresh, mature sapwood. The uninfested and MPB-infested samples were harvested from the same site. The harvest site for spruce and fir was unknown. Wood chips were air dried in the laboratory prior to grinding in a laboratory Wiley mill (Thomas Scientific) using two different screen sizes (3.2 and 4.7 mm). Moisture content of the samples was measured according to ASABE Standard S358.2 (ASABE 2007a) for forages, drying 25 g of ground sample in 103°C oven for 24 h. Milled wood was further ground using 40-mesh screen size (0.04 mm) in a laboratory Wiley mill for chemical analysis.

Chemical composition

Chemical analysis of uninfested and MPB-infested pine samples was conducted following TAPPI (Technical Association of the Pulp and Paper Industry) standard methods, starting with moisture content followed by extractives removal, Klason lignin and sugar analysis. Moisture content was measured according to T 412 (TAPPI 2002a) procedure; a 2 g ground sample (passing through 40 mesh screen) was dried at $105 \pm 2^\circ\text{C}$ for 2 hours. Extractives (dry basis) were measured according to T 280 (TAPPI 2002b) procedure, and determined by weighing the residue after Soxhlet extraction with acetone for 16 hours. Acid-insoluble lignin (extractive and moisture free) was measured following T 222 (TAPPI 2002c) procedure, and determined from the weight of the residue obtained after sulfuric acid hydrolysis of the ground samples. Acid soluble lignin (extractive and moisture free) was measured following T 249 (TAPPI 2002d), UV Spectra procedure, from the absorption band at 205 nm using a Perkin Elmer spectrometer. Sugar analysis (extractive and moisture free) was conducted following T 249 (TAPPI 2002d) HPLC procedure; the sulfuric acid hydrolysate was analyzed using a HPLC model Dionex ICS2500 (Shimadzu Scientific Instruments, North America) equipped with an ED50 electrochemical detector using an anion exchange column (Dionex CarboPac PA1) with a flow rate of de-ionized water at 1 mL/min. Three replications for extractives and six replications for Klason lignin and sugar analysis were conducted.

Pelletization, density and heating value measurements

The moisture content of the ground material was adjusted prior to pelletizing. A small quantity of water was sprayed on the ground material, and the moistened material was thoroughly mixed in a container. The process of adding moisture and mixing was repeated until a pre-determined moisture content was reached. The moistened samples were kept in sealed plastic bags in the laboratory (23°C) and equilibrated for 24 hours.

Pellets were made using the single pelletizer shown in Fig. 1. A change of 0.50 to 0.55 g of conditioned sample was manually loaded into the die, and compressed into a

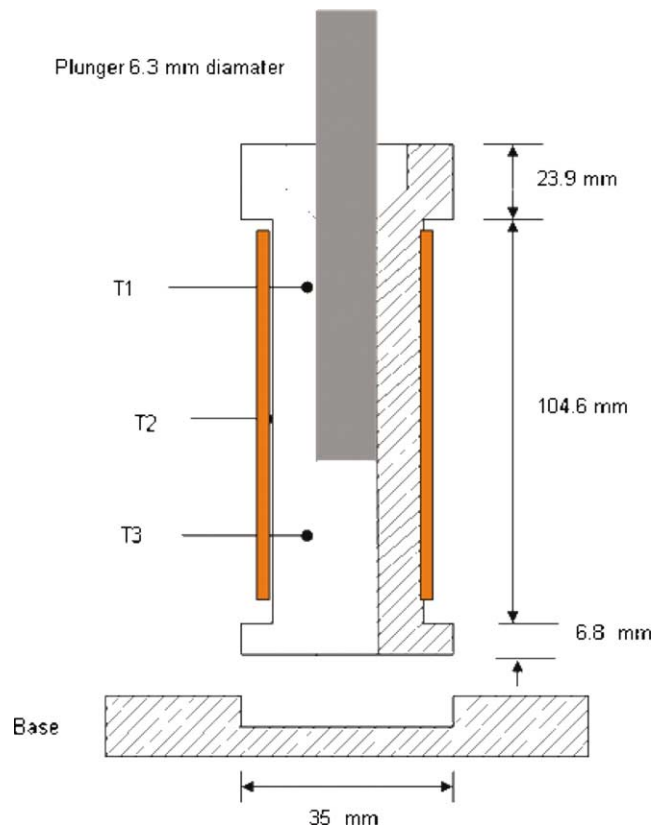


Fig. 1. Diagram of laboratory pelletizer that consists of a plunger and cylinder die with internal diameter of 6.35 mm. The die is heated with an electrical heating blanket. T1, T2, and T3 are locations of thermocouples.

6-mm diameter pellet using a moving plunger attached to an Instron machine model 1132. The die assembly was heated with a heating tape. Two thermocouples were used for detecting and controlling die temperature near the forming pellet. A third thermocouple measured the interface between the die and heater. The pressure-displacement data were acquired by a computerized data logger.

Densification took approximately 5 to 6 min. This time included time for loading the die, densifying and holding (about 1 min), relaxing (about 1 min) and removing the pellet. Pellets were cooled to the ambient temperature and kept in plastic bags for 1 day. The variables studied included size of ground biomass (3.2 and 4.7 mm), moisture content (10 and 15%), compaction pressure (80 and 127 MPa), and compaction temperature (90, 100, and 110°C). Table 3 lists the combination of these variables to produce pellets. Five pellets were made for each combination.

The length and diameter of pellets were measured using a digital caliper and were used to calculate pellet volume. Pellet mass was measured using a digital balance with 0.01 g precision. The density of a single pellet was calculated from the ratio of its mass to its volume. The high heating value (HHV) of pellets was measured according to ASTM E711-87 (2004) standard using an

Table 3. Grinder screen size, biomass moisture content, pelleting pressure and temperature combinations and resulting pellet densities made from various wood species including mountain pine beetle (MPB) infested pine.

Biomass property		Pelletizing condition				Pellet density (kg/m ³)	
Screen size(mm)	Moisture content (% wb)	Pressure (MPa)	Temp (°C)	Spruce	Fir	Uninfested pine	MPB-infested Pine
4.7	10	127	90	934.8	910.0	1001.8	1041.0
4.7	10	127	100	1017.2	925.6	972.2	1024.6
4.7	10	127	110	1058.6	944.2	1030.6	1045.8
4.7	10	80	90	843.2	767.0	812.8	957.2
4.7	10	80	100	892.0	870.2	917.4	947.4
4.7	10	80	110	859.4	828.4	924.6	946.6
4.7	15	80	90	890.6	782.8	882.0	893.2
4.7	15	80	100	897.8	786.4	857.0	893.6
4.7	15	80	110	1004.2	945.2	887.4	898.0
3.2	10	127	90	1004.2	945.2	1013.2	1041.4
3.2	10	80	100	978.6	931.0	958.6	995.0
3.2	10	80	110	982.2	929.4	974.0	1010.7
Average (<i>M</i>)				946.9	880.5	936.0	974.5
Standard deviation (<i>S</i>)				69.7	70.3	67.2	59.6
Sample size (<i>n</i>)				12	12	12	12
<i>t</i> -calculated				1.35	3.54	1.49	
<i>t</i> -critical				2.07	2.07	2.07	

oxygen bomb calorimeter, Parr model 1108 (Preiser Scientific, Louisville, KY). Microbiological tests were performed on some of the pellets made in the laboratory. Two types of media were used for culture samples: 2% malt extract agar (MEA), and 2% MEA+benomyl. Samples were cultured in Petri dishes and kept at room temperature (25°C) for 1 mo (four replicates for each media) (Gao et al. 1994).

Statistical analysis

The Student *t*-test is used to compare the resulting data from tests on MBP infested and uninfested wood and pellets. For each pair of data to be compared a *t* is calculated using the following equation (Devore 2004):

$$t = \frac{M_1 - M_2}{\left(\frac{S_1^2}{n_1} + \frac{S_2^2}{n_2}\right)^{0.5}} \quad 1$$

where M_1 and M_2 are means, S_1 and S_2 are standard deviations, and n_1 and n_2 are the number of data points for each group to be compared. A *t*-critical was calculated assuming a significance level of $\alpha = 0.05$ with a degree of freedom

$$d.f. = n_1 + n_2 - 2.$$

The null hypothesis is that pair treatments are identical. This hypothesis is rejected if $t > t$ -critical. Microsoft Excel spreadsheet is used for data organization and calculating average, standard deviation, and *t*-critical.

RESULTS AND DISCUSSION

Chemical analysis

Table 4 lists mean, standard deviation, and number of samples for the chemical composition tests for uninfested and MPB-infested pine samples. Chemical tests were not conducted on spruce and fir samples. The calculated *t* values using equation 1 and *t*-critical values using *tin*v(0.05,d.f.) for each pair of tests are listed.

Compared with those for uninfested pine, a slight decrease in mean acid insoluble lignin, xylose, galactose, and mannose was noticed; the mean percent of glucose and extractives increased. The increase in extractives in MPB-infested wood is statistically significant ($\alpha = 0.05$). This increase is in contrast to the conclusion of Ballard et al. (1983) that sap-staining fungi may degrade extractives such as triglycerides, fatty acids and resin acids available in the parenchyma cells. We offer the following explanation for the increased extractives in MPB pellets compared with uninfested pellets: (1) Non-uniform degradation due to staining fungi causes extractive variations; (2) extractives could migrate from one site to another to protect the attacked and injured parts of the tree; (3) extraction does not show the exact amount of the wood extractives because the protein content of the fungal body and the degraded functional groups of lignin interfere with measurements. Reduction in lignin and monomers of polysaccharides (arabinose and galactose) implies the probable decay of wood from cell-wall-degrading enzymes. Rot fungi that may follow the beetle infestation may also be responsible for lignin degradation.

Table 5. Statistical testing of the significance between the pellet density for two groups of pellets made from MPB-infested and uninfested pine and between four treatments.

Parameters	MPB-infested pine			MPB-uninfested pine			Statistics*		
		Mean (kg m ⁻³)	Std. dev. (kg m ⁻³)	<i>n</i>	Mean (kg m ⁻³)	Std. dev. (kg m ⁻³)	<i>n</i>	<i>t</i>	<i>t-critical</i>
Grinder	4.7 mm	961	62	9	926	68	10	1.17	2.11
screen size	3.2 mm	1016	24	3	982	28	3	1.59	2.78
Statistics**	<i>t</i>	2.21			2.08				
	<i>t-critical</i>	2.23			2.20				
Moisture	10%	1001	41	9	958	62	10	1.81	2.11
	15%	895	3	3	875	16	3	2.05	2.78
Statistics	<i>t</i>	7.65			3.78				
	<i>t-critical</i>	2.23			2.20				
Pressure	80 MPa	943	45	8	902	53	8	1.66	2.14
	127 Mpa	1038	9	4	998	26	5	3.24	2.36
Statistics	<i>t</i>	5.71			4.37				
	<i>t-critical</i>	2.23			2.20				
Temperature	90°C	983	72	4	927	97	4	0.92	2.45
	110°C	975	66	4	954	62	4	0.47	2.45
Statistics	<i>t</i>	0.16			0.46				
	<i>t-critical</i>	2.45			2.45				

*Statistical comparison between two entries in the row (MPB-infested and uninfested Pine).

**Statistical comparison between two treatments (2 grinder screen size, two moistures, two pressures, two temperatures).

that the *t* calculated values for spruce and uninfested pine are less than *t-critical*. This indicates that their densities do not differ statistically from the density of pellets made from MPB-infested pine. The density of pellets made from fir is significantly different from that of pellets from MPB-infested pine.

The standard deviation in density data for MPB pellets was lower than the density variations among uninfested pellets. A *t-test* between the two groups using equation 1 [(*t* = 1.49) < (*t-critical* = 2.7), $\alpha = 0.05$] shows that the overall density values for pellets made from MPB-infested and uninfested woods are not statistically different. The apparent increase in mean density of MPB could be attributed to lignin and hemicelluloses structures in MPB wood. Molecular flexibility of lignin is related to the type and amount of inter-unit linkages within the lignin macromolecule (Dos Santos Abreu et al. 1999). As degradation of lignin occurs by breakage of some of the inter-unit bonds in lignin, e.g. β -aryl ether linkages (Crawford 1981), this can be expected to have an impact on the molecular flexibility. Similarly, hemicelluloses, which act as interface between the lignin and cellulose, are also responsible for the strength of cell walls. Any changes in the structure of lignin and hemicelluloses might therefore affect the cell wall's flexibility, brittleness, rigidity, and spring-back after pressure removal. In a recent review, Byrne (2005) stated the mechanical properties of MPB wood exhibit increased brittleness. More rigidity and less spring-back would help the wood particles to stay more intact after densification. Spring-back is an important parameter to change the density of materials after removing the load (Kultikova 1999).

Pelletization treatments

Table 5 presents mean and standard deviation of the density data grouped into MBP-infested and uninfested, and within each of these two groups the data are rearranged in four treatments: grinder screen size, moisture content, pelletization pressure, and die temperature. Density data for spruce and fir are not included in this table. Comparing the two groups of uninfested and MBP infested for each of treatments, we note that except for high- pressure (127 MPa) treatment, the calculated statistics *t* is greater than or equal to *t-critical* ($\alpha = 0.005$). Therefore the two groups of pellets made from uninfested and MBP infested pine showed a similar response to the treatments. Within treatments in each group, pellets made from MPB-infested wood showed a statistically significant response to moisture and pressure. A slight drop in the density for MPB-uninfested pellets was observed when the die temperature was increased from 90 to 100°C (Table 3), but the density increased with increasing temperature from 100 to 110°C.

Increasing die temperature from 90 to 100°C may initiate water loss that may lead to lower pellet density. Water is a good plasticizer and bonding agent for wood adhesion (Pizzi 1994). The glass transition temperature of lignin and hemicelluloses is very much dependent upon moisture content. Therefore, increasing temperature to 110°C may lead to softening of the amorphous components, specifically lignin leading to an increased pellet density. Increasing pressure further facilitates pellet pressing and increased density. Table 3 shows that the mean density of MPB-infested pellets decreased slightly when

Table 6. Statistical summary of heating value of pellets made from mountain pine beetle (MPB) infested and uninfested pine.

Pellets made from	Property	Unit	Mean	Std. dev.	Number of tests
MPB-infested wood	Heating value	GJ/Mg	19.20	0.18	6
Uninfested wood	Heating value	GJ/Mg	19.08	0.16	6
Statistics	<i>t</i>		1.22		
	<i>t-critical</i>		2.23		

the temperature of the dies increased from 90 to 110°C. The mean density of uninfested wood also showed a slight increase, but variations in standard deviations in measured density were large to make these trends statistically significant.

Heating value

Randomly selected pellets made from wood particles having 10 and 15% moisture contents were tested for their heating values. Table 6 summarizes the results of heating values for MPB-infested and uninfested wood pellets. The results do not show any significant difference [$(t = 1.49) < (t_{critical} = 2.7), \alpha = 0.05$] between MPB-infested and uninfested wood. This confirms the applicability of MPB-infested wood as a fuel source in the form of pellets.

Microbiological tests

Fungi growth was visually observed by light microscopy after preparing cultures on the MPB-uninfested and infested wood pellets. Mold (*Penicillium*) was the only fungi was observed in each culture. No sap-stain or other type of fungi was observed. Microbiological test on both MPB uninfested and infested pellets did not show any growth of decaying fungi. Past experiments on other biological materials (Gao et al. 1994, Clausen and Yang 2007) showed that moisture content larger than 20% is required for fungi growth. This microbiological assessment is based on a few observations. This research requires further expansion to identify conditions in which microorganisms including mold may grow on pellets.

CONCLUSIONS

We compared the chemical composition, pellet density, heating value and microbiological activity of MPB-infested and uninfested lodgepole pine.

Chemical composition analysis did not show significant differences between the sugar and lignin contents of MPB-uninfested and infested pine except minor reductions in lignin and certain hemicelluloses monomers.

Pellets made from MPB-infested pine had a mean value for density larger than those made from uninfested pine. A Student t-test did not indicate any statistical difference (at $\alpha = 0.05$) between the two types of pellets.

Heating values of the pellets from MPB-infested wood were similar to those measured for pellets from uninfested wood.

A preliminary observation of mold growth did not show any further staining or other decay fungi growth for the pellets made from MPB-infested wood.

The overall conclusion is that pellets made from MPB-infested wood are similar to pellets made from uninfested wood in density, heating value, and most chemical constituents.

Density data had large variations. This indicates that the number of replicates must be increased in future experiments.

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