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Relationship between ethanolic extracts of yellow birch and tree characteristics

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ABSTRACT

The traditional wood industry has gone through an important crisis over recent years due to a decline in demand for certain traditional products coupled with a decreased demand due to competing developing economies, among others. In this context, the production of high value-added products and the exploration of new markets could promote the transition to a more resilient forest industry. One of the up and coming markets involves the plant-based extracts, which can be used in several industries, such as pharmaceuticals, cosmetics and nutraceuticals. In order to better understand the potential that biorefining products from yellow birch (*Betula alleghaniensis*) have to produce high value-added extracts, this study evaluated the relationship between total phenols, major triterpenes and phytosterols content in wood and bark of the studied species and selected tree characteristics. Results showed that the majority of the studied chemical compounds were related to tree health. The wood of non-vigorous trees presented the highest total phenols content among the three vigour classes, with an average of 394.9 ± 138.6 mg GAE g^{-1} of oven-dry extract, while presenting the lowest β-sitosterol content (3.0 ± 0.9 mg g^{-1}). The bark of moribund trees contained the lowest total phenols content (317.6 ± 46.8 mg GAE g^{-1}), but the highest betulin content (6.6 ± 4.2 mg g^{-1}). Variations in bark compounds were also frequently correlated with the presence of fungal fruiting bodies on the stem, being associated with lower extract values for the betulin, lupeol and lupenone. Tree vigour and the presence of sporocarps in the stem explained a significant part of the variation in the studied chemical compounds from wood and bark. A remaining unexplained variation may be attributed to stand-level conditions.

1. Introduction

Yellow birch (*Betula alleghaniensis* Britt.) is an important species of the temperate hardwood forests of North America. It has been used traditionally as a raw material for appearance wood products, such as furniture and flooring, for timber and panelling, as well as in the production of pulp and paper products (Burns and Honkala, 1990). The traditional wood industry, however, has gone through an important crisis over recent years. According to Mockler and Fairbairn (2009), the main causes for the crisis were: (i) a structural decline in demand for newsprint; (ii) a decreased demand due to competition with developing economies; (iii) a cyclical decrease in demand from the construction industry; and (iv) other aggravating factors, such as exchange rate, cost of energy, low-value commodity-based product matrix, etc. This combination of factors led to the closure of many pulp and paper mills and sawmills in Canada (Natural Resources Canada, 2015). In order to be less susceptible to cyclical downturns, the forest sector needs to diversify and innovate (FPAC, 2010).

In this changing context, the exploration of new markets, such as biorefining, could facilitate the transition to a more resilient forest industry. Considering the production of high added-value extracts as an additional use for yellow birch wood and bark becomes even more interesting when the availability of this raw material is taken into account. The annual allowable cut is 1 million m^3/year of merchantable yellow birch for the Canadian province of Quebec alone (a small fraction of its natural distribution) (Bureau du Forestier en Chef, 2014). Considering that the average sawlog volume fraction in a mature tree rarely exceeds 75%, and that the average lumber conversion rate for sawlogs is of 50%, approximately 0.6 million m^3/year of pulpwood and sawmilling wood residues is available for uses, such as pulp and paper, panelling, biomass and biorefining products (Alderman et al., 1999; Fortin et al., 2009; Hanks 1977). In addition, this timber conversion generates an estimated 97000 dmt of yellow birch bark each year in the province of Quebec (based on the allowable annual cut for this species and a conver-

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sion factor of 0.095 dmt m^{-3}) (Bureau du Forestier en Chef, 2014; CIFQ, 2014). Currently, this material is primarily used for energy production through direct burn, but high added-value uses could be explored to help diversifying the products and, consequently, strengthening the forest industry. Among those uses it is important to highlight the extraction of chemical compounds from wood and bark. The use of co-products and sawmill residues for producing high value-added extracts could improve the current value chain without competing directly with the traditional products.

According to Royer et al. (2013), several tree species, including yellow birch, have the potential for biorefining, especially for pharmaceutical, nutraceutical and cosmetic industries. Research investigating the properties of plant extracts has been done to prove their efficacy against cancer, certain viruses and diabetes (Alakurtti et al., 2006 Anhê et al., 2013; Baltina et al., 2003; Dehelean et al., 2012; Mulvihill et al., 2011; Pavlova et al., 2003; Serafim et al., 2014), efficacy reducing cholesterol levels (Best et al., 1954; Rideout et al., 2012) and as an antioxidant (Garcia-Perez et al., 2008; Royer et al., 2011). Some of these functional extracts can be found in tree species, and their quantification is crucial when aiming a large-scale production.

The wood and bark constituents of yellow birch have been the subject of several studies. Seshadri and Vedantham (1971), Lavoie and Stevanovic (2007) and Habiyaremye et al. (2002) characterized the main triterpenes found in the wood and bark from recently harvested trees, and Lavoie and Stevanovic (2006) also investigated the constituents from sawdust subjected to weathering in a sawmill yard. St-Pierre et al. (2013) quantified the constituents of ethanolic extracts of wood and bark from trees of different vigour classes. However, those studies were based on analyses from a small number of sample trees, limiting their capability of capturing the tree-to-tree variation in terms of extracts. Furthermore, none of them investigated how the external and internal tree characteristics, such as the occurrence of defects on the stem and wood decay, would be correlated with the chemical compounds.

In order to further develop the understanding of the relationship between the extract contents and individual tree characteristics, two objectives were established for this project: a) to assess the quantity of major ethanolic extracts, namely total phenols, major triterpenes and phytosterols, in yellow birch wood and bark samples; and b) to analyse the relationship between the ethanolic extracts content and selected tree characteristics. Such information on the characteristics of the raw material is an essential step for the development of a new processing pathway dedicated to the production of high added-value extracts.

2. Material and methods

2.1. Sampling

Four sampling sites were selected near Mont-Laurier, Quebec, Canada, in the sugar maple-yellow birch bioclimatic region. All four sites were located on public land and dominated by yellow birch (Table 1). Sampling was carried out in early summer, during the months of June and July.

From each stand, 12 merchantable yellow birch (*Betula alleghaniensis* Britt.) trees were sampled. Before cutting, they were classified into three vigour classes, based on the overall tree health (Boulet, 2007). This classification was put in place to prioritize harvesting, where trees that are bound to die before the end of the next cutting cycle (i.e. about 20 years) should be harvested before the most vigorous trees. Moribund trees are those expected to die before the next rotation, non-vigorous trees are those that should survive until the next intervention despite being defective and declining in health, and vigorous trees are those that present little to no defects and should be retained in the forest. In each stand four trees from each vigour class were selected. The presence of external fungal fruiting bodies (or sporocarp), observed in 19 sample trees, was also recorded.

Discs were collected from each tree, at heights of 0.3 m, 1.3 m, 2.3 m, and at every two metres up to the end of the main stem. Whenever a bifurcation occurred, the largest or most vigorous stem was chosen and sampling was resumed until discs reached 9 cm in diameter. The samples were immediately stored in plastic bags to protect from contamination, and kept in a cool shaded area. Since most volatiles compounds found in the wood and bark were not part of the groups of interest in this study, no further precautions were taken. The samples were later transported to Laval University, where they were stored in a freezer at −5 °C until further processing. Since most volatile compounds were not among the

2.2. Sample preparation

Diameters with and without bark of all discs along the stem were measured with the aid of a measuring tape. With these measurements the volume of each section was calculated using the Smalian formula (Wenger, 1984) (Eq. (1)), and the stem volume was obtained as the sum of all section in a tree.

$$V_s = \left(\frac{s_b + s_t}{2}\right) * l \quad (1)$$

where V_s is the volume of each section (m^3), s_b is the area of the large end of the section (m^2), s_t is the area of the small end of the section (m^2), and l is the section length (m). The perimeter of the wood containing decay or hollow areas was also measured to estimate the unsound wood area. If decay occurred in one disc, but not in the subsequent, a decay value of zero was attributed to the latter. The Smalian formula was used to estimate decay volume in every stem section and the total decay content in the stem (%) was then calculated.

Even though variations in extractives content occur along and across the stem (Gierlinger and Wimmer, 2004; Morais and Pereira, 2012), only the samples collected at breast height were used for the chemical analyses, simplifying the assessment of the relationship between extracts content and tree characteristics. The discs collected at breast height (1.3 m) were debarked and both wood and bark were transformed into chips. This material was then dried in an oven at 30 °C for 48 h to reduce moisture content and facilitate further processing, and ground with a Fritsch Pulverisette 19 grinder. The sawdust was sieved and the fraction retained between sieves 400 μm (40 mesh) and 250 μm (60 mesh) was kept for the analysis.

2.3. Maceration

Since total phenols are thermosensitive components and we wished to preserve their antioxidant activity as much as possible, we chose to use an extraction method that did not involve high temperatures (Jordão et al., 2012; Lim and Murtijaya 2007; Ross et al., 2011; Zhang et al., 2015). For the same reason, the sawdust was not oven-dried to 0% moisture content prior to the analysis. Instead, the moisture content of the sawdust was determined concurrently with the maceration process following the standard ASTM D4442 (ASTM, 2015). For both wood and bark, 5 g of sawdust were extracted with 50 mL of 95% ethanol, using an orbital bench-top shaker from Barnstead Lab-Line model 4633 set at 200 rpm for 24 h at room temperature. Ethanol was chosen as solvent for being able to extract both total phenols and triterpenes (Conde et al., 2013; Rizhikovs et al., 2015; St-Pierre et al., 2013). The extract was vacuum-filtered using a Whatman No. 4 qualitative filter paper mounted on a Büchner funnel. Then, the wood fibres were washed