High-Resolution Mass Spectrometry Characterization of Essential Oil and Extractives from Spruce Buds

Omolara Mofikoya, Marko Mäkinen, and Janne Jänis

University of Eastern Finland, Department of Chemistry, P.O. Box 111, FI-80101 Joensuu, Finland

UNIVERSITY OF EASTERN FINLAND

Overview

- **Purpose:** Chemical fingerprinting of essential oil and solvent extracts from spruce buds by using high-resolution mass spectrometry.
- **Methods:** Production of essential oil and solvent extracts from the buds by hydrodistillation and solvent extraction and their analysis using ESI/APPI FT-ICR MS.
- **Results:** Compounds detected from the essential oil and solvent extracts included different monoterpene, sesquiterpene and diterpene hydrocarbons as well as acids, esters and alcohols. ESI and APPI provided complementary information on the rich chemistry of samples

Introduction

- Spruce is a coniferous evergreen tree that belongs to the *Pinaceae* family. Bruce buds (spring growths or tops) appear in the early spring.
- Spruce buds contain a variety of chemicals which possess considerable antioxidant and antimicrobial activities. The main chemical constituents include terpenes alcohols, phenols, esters, acids and their derivatives.
- In this study, we obtained essential oil and solvent extracts from spruce buds and characterized their chemical composition by using ultrahigh-resolution ESI/APPI FT-ICR MS.

Methods

- **Hydrodistillation**
  - ❖Hydrodistillation with Clevenger apparatus (200 g needles per 400 mL of water; 3 h of distillation time).
- **Solvent extraction**
  - ❖Solvent extraction with continuous Soxhlet extractor (Buchi system B-811); 40 g of spruce buds, 2.5 h of extraction time; solvents: (hexane/methanol/water).
- **Mass spectrometry**
  - ❖Direct-infusion ESI/APPI FT-ICR MS with 12-T Bruker solarix XR instrument

Results

- ESI/APPI FT-ICR MS analysis of the essential oil and extracts revealed their complex chemical nature.
  - ❖The spectral fingerprints were unique for each sample **(Figure 1)**.
  - ❖The most abundant compounds were resin acids and phenolic compounds for polar and non-polar solvents, respectively.
  - ❖Other compounds detected were different acids, esters and terpenoids.
  - ❖Some identified compounds are presented in **Figure 2**.

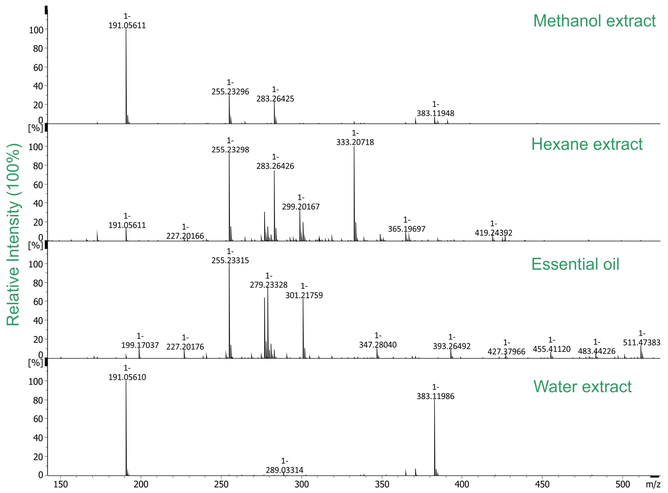

Figure 1. 12-T (-)ESI FT-ICR mass spectra of spruce sprout extracts.

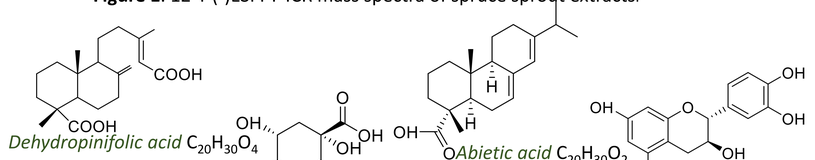

Figure 2 . Structures of some selected compounds observed in samples.

- Positive-ion APPI FT-ICR MS analysis mostly targeted nonpolar species (terpene hydrocarbons) (**Figure 3**).
- APPI provides complementary data to that of ESI.

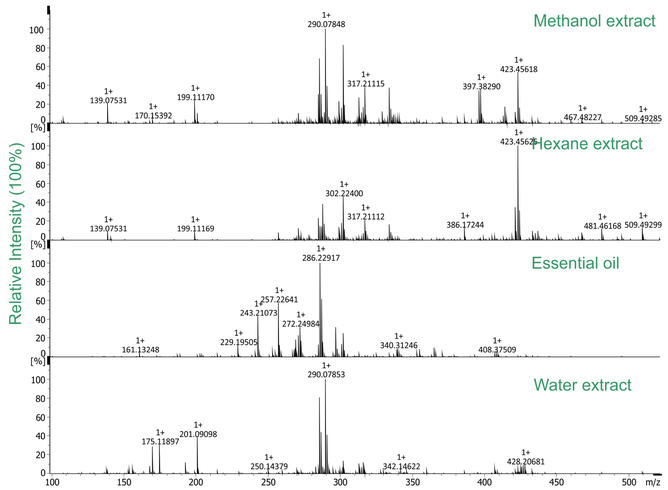

Figure 3. 12-T (+) APPI FT-ICR mass spectra of spruce sprout extracts.

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