SEASONAL CHANGES IN THE VOLATILE PROFILE OF SCOTS PINE (PINUS SYLVESTRIS) NEEDLE EXTRACTS: HYDRODISTILLATION VS. SUPERCRITICAL CO_2 EXTRACTION

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The efficient utilization of by-products from forest thinning and logging operations is a fundamental objective in sustainable forestry. While residual biomass is increasingly used as biofuel, conifer needles—comprising about 20% of a tree's organic matter—are frequently classified as forestry waste. The former, however, is particularly rich in bioactive volatile phytochemicals, including terpenes and terpenoids, whose synthesis fluctuates throughout the year in response to environmental stressors and plant growth cycles. Given that Scots pine (Pinus sylvestris) is one of the most commercially significant forest-forming tree species in Northern Europe, this study explores the optimized use of its foliage with respect to seasonality.

This research investigates the seasonal changes in volatile compounds extracted from pine needles collected from the same trees near Vilnius over six sampling periods between February and December 2024. Various extraction techniques were employed to obtain volatile constituents. Essential oils (EOs) were extracted using a 3-hour hydrodistillation process with a Clevenger-type apparatus, and the resulting water extracts were subsequently freeze-dried. To obtain lipophilic extracts, a novel supercritical fluid extraction (SFE) method utilizing carbon dioxide (40 °C, 15 MPa) was applied, with extraction efficiency assessed through kinetic curve analysis. The chemical composition of volatile compounds in the lipophilic extracts was analyzed using comprehensive two-dimensional gas chromatography coupled with time-of-flight mass spectrometry (GC×GC-TOF/MS) and headspace solid-phase microextraction (HS-SPME). Additionally, gas chromatography with flame ionization detection (GC-FID) was used to characterize the composition of essential oils.

The highest yield of essential oils was recorded in February (0.5%), whereas the maximum yield of water-soluble lyophilized extracts was observed in April (1.4 g/100 ml hydrolat). Similarly, lipophilic fractions exhibited the highest extraction efficiency in February (4.2%). The composition of essential oils was dominated by α-pinene, with its highest concentration occurring in December (27.40%) and the lowest in August (19.43%). In contrast, the composition of lipophilic fractions displayed greater variability - while α-pinene was the primary volatile compound in February (19.92%) and August (14.42%), bornyl acetate was predominant in April (11.77%) and June (10.06%), whereas δ-carene (11.56%) and β-caryophyllene (9.33%) were the most abundant volatiles in October and December, respectively. The concentration of δ-cadinene fluctuated throughout the year. Notably, a peak in secondary plant metabolites in June correlated with the pine pollination period. As one of the highest yields and distributions of the main active volatile components of essential oils and lipophilic fractions were identified in February, this research suggests collecting pine needles in late winter.