



Seasonal analysis of the volatile constituents present in fresh (recent) and solid (aged) Oil-resin formed in *Protium heptaphyllum*

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Protium heptaphyllum (Burseraceae) produce an aromatic Oil-resin rich in volatile and non-volatile terpenes. The Oil-resin has pasty or soft consistent when its exudation is recent, and is rich in monoterpenes. Over time the Oil-resin becomes solid, and loses significant amounts of volatile compounds. In a previous study from our group by comparing the essential oil of fresh and dried oleoresin it was observed that the fresh one was rich in terpinolene and the dried one in *p*-cymene, suggesting that this last could be a degradation product. In the present work, we carry out a comparative study of seasonal samples of dried, fresh and intermediate oleoresin of *P. heptaphyllum* to see how the natural aging process affects the volatile constituents. Oil-resins were collected seasonally in São João da Barra, State of Rio de Janeiro, during the years 2014 and 2015. The samples were characterized by their texture in: Softened Oil-resin – a recent exudate; Semisolid Oil-resin – an intermediate exudate; and Solid Oil-resin – an old exudate. The oilresins were hydrodistilled with a Clevenger apparatus for 4 hours. The essential oils (EOs) obtained were analyzed by GC/FID and GC/MS, using Agilent system 5973N, with a capillary column HP-5 (30 m x 0.25 mm X 0.25 µm). The temperature was programmed from 60 to 240°C (3°C/min). The identification was made by comparison of the mass spectra (Wiley database) and retention indices calculated from the injection a series of n-alkanes. The EOs obtained from recent exudate (11.3-24.9% v/p) were mainly constituted of terpinolene (49.8-62.2%) and presented low content of *p*-cymene (4.6-13.5%) and *p*-cymen-8-ol (2.7-7.4%), whereas the EOs obtained from old exudate (2.7-4.5% v/p) had a decrease of terpinolene (21.4-26.3%) and an increase of *p*-cymene (16.1-18.8%) and *p*-cymen-8-ol (16.5-26.4%). The EOs of intermediate exudate (10.1-14.2% v/p) presented a reduction of terpinolene content (44.5-47.6%) and an increase in *p*-cymen-8-ol (7.5-9.2%) when compared to the EOs from recent exudate. With natural aging process the yield of EOs can be reduced up to 10 times, varying from 24.9% to 2.7%. The volatile composition of the Oil-resin also change during the natural aging. The increase of *p*-cymene and *p*-cymen-8-ol could be influenced by abiotic factors (light, temperature, oxygenation, humidity) that induce the conversion of the majority non-aromatic monoterpene (terpinolene) in these aromatic monoterpenes.

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