TRANSACTIONS, ILL. STATE ACAD. SCI.
Vol. 68, No. 1 (1975)

COMPOSITION OF MONOTERPENES IN LOBLOLLY PINE AT DIFFERENT STEM HEIGHTS

A. R. Gilmore
Department of Forestry, University of Illinois
at Urbana-Champaign

Abstract. Four of the five monoterpenes found in the oleoresin of loblolly pine varied with sampling position on the stem. Data suggest that intermolecular rearrangement of alpha-pinene and beta-pinene might occur.

Differences in the composition of oleoresin from various positions in the stem have been reported for some coniferous species but not for others. For example, in Monterey and Bishop pines, Bligh and McDonald (1964) found little variation in xylem oleoresin from one stem position to another. In studying four species of *Abies*, Zavarin (1968) found monoterpenes to be quite consistent at different stem levels. Smith (1964), working with ponderosa pine, found little difference in oleoresin composition from different annual rings. Conversely, Roberts (1970) reported that oleoresin components varied with height of sampling point above the ground and that differences in oleoresin composition also occurred among annual rings of the same tree.

The variations in the composition of monoterpenes at different stem heights in loblolly pine *(Pinus taeda L.)* are reported in this note.

METHODS

Loblolly pines used in this study were planted in extreme southern Illinois in 1946 using seedlings from an unknown seed source. At the beginning of the study, the sample trees averaged 20 cm in diameter and 15 m in height. All trees were naturally pruned about two-thirds of the total height. Plots averaged 325 trees per hectare with a basal area of 39 square meters per hectare.

Oleoresin samples were collected from two trees on each of six replicate plots on 11 dates, starting May 1, 1968, and at intervals of two weeks thereafter until September 24, 1968. The oleoresin was collected at 0.3, 3.4, 6.4, and 9.4 m above ground level by making V-notch cuts that extended through the stem bark to the cambium. The oleoresin oozing from each cut was placed in a small vial and the samples were frozen shortly after collection.

The oleoresin samples were dissolved in pentane and the analyses were performed using a Hewlett-Packard, Model 5750 Gas Chromatograph

equipped with dual hydrogen-air ionization detectors and disc integrator. A stainless steel column packed with 20% Carbowax 20M liquid phase on 60-80 mesh, acid-washed Chromosorb W solid support was used in detecting the monoterpenes. Each monoterpene was identified by comparing its' relative retention time with that of a known monoterpene that had been analyzed under the same conditions as the sample. The % composition of each monoterpene was calculated from mechanical integrator traces of area under each peak as related to the total area of all monoterpene peaks.

RESULTS AND DISCUSSION

Five terpenes found in the pine oleoresin in the order in which they eluted from the gas chromatograph column were alpha-pinene, camphene, beta-pinene, myrcene, and limonene. Alpha-pinene was consistently in greater concentration, followed by beta-pinene, camphene, myrcene, and limonene were in low concentrations, usually less than five % (Table 1).

Table 1. Average terpene concentrations of loblolly pine samples at different stem heights.

Stem height (meter)	Terpene concentration				
	Alpha pinene[a]	Camphene[a]	Beta pinene[a]	Myrcene	Limonene[a]
	percent				
0.3	86.6	2.0	4.4	5.0	2.1
3.4	79.5	1.8	10.9	5.2	2.5
6.4	75.1	1.7	15.6	5.0	2.5
9.4	72.0	1.7	19.0	5.0	2.3

[a]Differences between height significant at 5% level.

Alpha-pinene and camphene decreased with increasing stem height, while beta-pinene increased with increasing stem height and limonene increased in the lower part of the stem before reaching a plateau near the crown (Table 1).

Zavarin (1968) reported that practically no seasonal change in terpene composition was observed in four *Abies* species. If his findings hold true for loblolly pine, which has been indicated in work by the author, this means that oleoresin obtained from each vertical sampling position in the present study represents oleoresin produced by and stored within differently aged tissue. It then follows that results obtained in the present study agree with those obtained by Roberts (1970) for slash pine and Poltavcenko *et al.* (1968) for Scotch pine in that alpha-pinene increases with increasing tissue age. Also, beta-pinene decreased with age in this study, as it did for slash pine, and limonene decreased with age, as it did for Scotch pine.

Of particular interest is the relationship between alpha-pinene and beta-pinene concentrations; a variation in alpha-pinene concentration is met with a corresponding complementary variation in beta-pinene concentration. For example, alpha-pinene decreased by 14.6% from the lowest to the highest position on the stem. Alike but positive change of 14.6% occurred for beta-pinene at these stem positions. Similar results were obtained in slash pine by Roberts (1970). The close similarity in molecular structure of the two terpenes, suggest that intermolecular rearrangement of alpha-pinene and beta-pinene might occur. This hypothesis is supported by Schweers (1968), who obtained radioactive beta-pinene from macerated pine material to which labeled alpha-pinene had been added.

This study demonstrates as did Rockwell (1973) that terpene concentrations in loblolly pine tissue are not constant, but vary significantly with vertical sampling positions. This factor should be considered when selecting sampling sites for terpene analysis.

ACKNOWLEDGMENTS

A portion of this research was supported by funds from the Illinois Agricultural Experiment Station, McIntire-Stennis Project 55-362.

LITERATURE CITED

Blight, M. M., and I. R. C. McDonald. 1964. Sample reproducibility in pinus essential oil studies. New Zealand J. Sci. 7:212-220.

Poltavcenko, Ju. A., *et al.* 1968. Changes in the distribution of monoterpenes in *Pinus sylvestris*. Nauc. Dokl. Vyss. Skolg (Biol. Nauki), Moskva. 10:71-76. For. Abs. 30:No. 3506.

Roberts, D. R. 1970. Within-tree variation of monoterpene hydrocarbon composition of slash pine oleoresin. Phytochem. 9:809-815.

Rockwood, D. L. 1973. Variation in the monoterpene composition of two oleoresin systems of loblolly pine. For. Sci. 19:147-153.

Schweers, W. 1968. The metabolic conversion of alpha-pinene in *Pinus ponderosa*. Tetrahedron Lett., Oxf. 42:4425-4428.

Smith, R. A. 1964. Perennial constancy of the monoterpene synthesis in the wood oleoresin of *Pinus ponderosa*. Nature 202:107-108.

Zavarin, E. 1968. Chemotaxonomy of the genus *Abies*-II. Within tree variation of the terpenes in cortical oleoresin. Phytochem. 7:99-107.