Solid phase microextraction of *Eucalyptus ixophleba* leaves

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Solid Phase Microextraction of Eucalyptus loxophleba leaves

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Introduction

The volatile essential oils of the numerous Eucalyptus species have been widely studied for their chemical and commercial value, and also because a study of the oils can be used in supporting botanical relationships. In most cases the oils are obtained by traditional steam distillation methods, but also solvent extraction and headspace techniques have been employed.

The present study describes a study of the use of solid phase microextraction of the headspace to provide a convenient, solvent-free and clean method for the examination of the volatile components. The work examined the dried leaves of E. loxophleba ssp. lissophloia, which is of interest because of the presence of a number of anti-feeding sideroxylonls which make the plant unpalatable for koalas.

The advantage of solid phase extraction as a technique is potentially twofold. It should trap the volatile essential oil constituents and concentrate them and it should also provide a solvent free sample enabling very volatile and rapidly eluted analytes to be investigated, which might otherwise be lost under a solvent peak. However, because the sample is being partitioned into the SPME fibre coating, the most volatile constituents are those which often show the lowest recovery. In this study the SPME method was therefore compared with direct headspace injection and with solvent extraction.

Comparison of methods

All three methods extracted the same main components. The solvent extraction appeared to contain a number of extra peaks, some of which appeared to be from the solvent. Direct headspace analysis provided the lowest sample concentration and seemed in particular to omit the less volatile analytes. Solid phase microextraction of a headspace provided a cleaner sample in a higher concentration than headspace analysis and more of the less volatile components and was a clean and simple method to carry out the extraction.

All three methods showed the presence of the unusual 4-methyl-2-pentyl acetate.

Method

The dried leaves of E. loxophleba (25 mg) were placed in a sealed 2 ml sample vial and heated for 50 min at 60 °C. Either a 1 ml sample of the headspace was taken for direct analysis or the headspace was sampled with a 100 μm polydimethylsiloxane (PDMS) microextraction fibre (Supelco) for 50 min. 7 μm PDMS and polyacrylate fibres were also examined but gave lower recoveries.

For comparison a sample of the leaves was extracted with ethyl acetate (10 mg/ml) and 1 μl sample examined.

The gas chromatographic separations were carried out on a methylphenyl silicone coated capillary column (30 m x 0.32 mm with a 0.25 μm film). The temperature programme was 50 °C for 1 min, increased at 1 °C/min to 60 °C then 30 °C/min to 290 °C. The peaks were identified using similar conditions on a GC-MS instrument with a library match.

Results

This species is interesting because it is one of a small number Eucalyptus species found only in Western Australia which yield the unusual non-terpenoid ester, 4-methyl-2-pentyl acetate which is highly volatile and can be lost with the solvent on evaporation.

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