Stability of pure Australian Tea Tree Oil

Introduction

The Australian Tea Tree Industry Association (ATTIA Ltd) is an Australian-based, not-for-profit organization formed in 1986 as the peak body to promote and represent the interests of the Australian tea tree industry. From the grower/producers to the manufacturers of off-the-shelf products for public use, ATTIA supports and promotes the responsible use of pure Australian tea tree oil (TTO).

ATTIA's aim is to develop a stable, cohesive, environmentally friendly, and internationally competitive TTO industry producing quality assured pure Australian TTO that meets or exceeds international standards. ATTIA promotes the safe effective use of pure Australian TTO for a wide range of applications.

ATTIA Ltd is the national association representing participants in the tea tree oil industry. Our members include growers, processors, exporters and product manufacturers. We have an international associate membership of importers and manufacturers. Since 1982, we have promoted the production of pure Australian TTO as a long term sustainable crop using an Australian native plant. We are active in maintaining an Australian and International Standard for tea tree oil, and promote an industry-wide Code of Practice. Our membership accounts for around 95% of the Australian TTO crop.

This document provides a synopsis of the research conducted on the stability of pure Australian TTO and expresses ATTIA's opinion on the optimal storage times (use-by or best-before) for pure Australian TTO in both bulk and glass containers most commonly used for the transport, storage and sale of the product.

Summary

Much discussion has occurred over many years on the stability and therefore the 'use by' or 'best before' date for pure Australian TTO. A body of research data is available on the subject. The answer to an often posed question on the 'use-by' date of tea tree oil depends entirely on how it is distilled, stored, handled and transported.

Tea tree oil, in the presence of light, air (oxygen) and elevated temperature degrades over time and the levels of α-terpinene and γ-terpinene decline over time while the p-cymene and peroxide levels increase [b]

> **ATTIA recommends that the use-by (best before) date for pure Australian tea tree oil sold in commercially available small (up to100 ml) dark glass bottles stored at an ambient temperature not exceeding 25 °C be set at 6 months from when first opened or 24 months (2 years) in unopened bottles.**

For bulk storage ATTIA's Code of Practice [d] requires producers to use only stainless steel storage and transport vessels and to store the oil in cool, dark conditions with minimum exposure to air. When stored correctly in full containers pure Australian TTO can retain its quality for periods of up to 10 years [a] although this is not recommended.

> **ATTIA recommends that the use-by (best before) date for pure Australian tea tree oil sold in correctly filled, purged (Nitrogen or Argon) and tightly sealed stainless steel drums stored at an ambient temperature not exceeding 25 °C be set at 3 years from the filling date.**

If there is any doubt about the quality of pure Australian TTO a sample should be tested according to international standard ISO 4730 (2004) or the identical Australian standard AS 2782-2009 ("Oil of Melaleuca, Terpinen-4-ol type"). The levels of terpinen-4-ol, p-cymene, α-terpinene, γ-terpinene and terpinolene can then be used to determine potential levels of oxidation. If there is still doubt then the peroxide value of the oil should also be determined using appropriate analytical standards.

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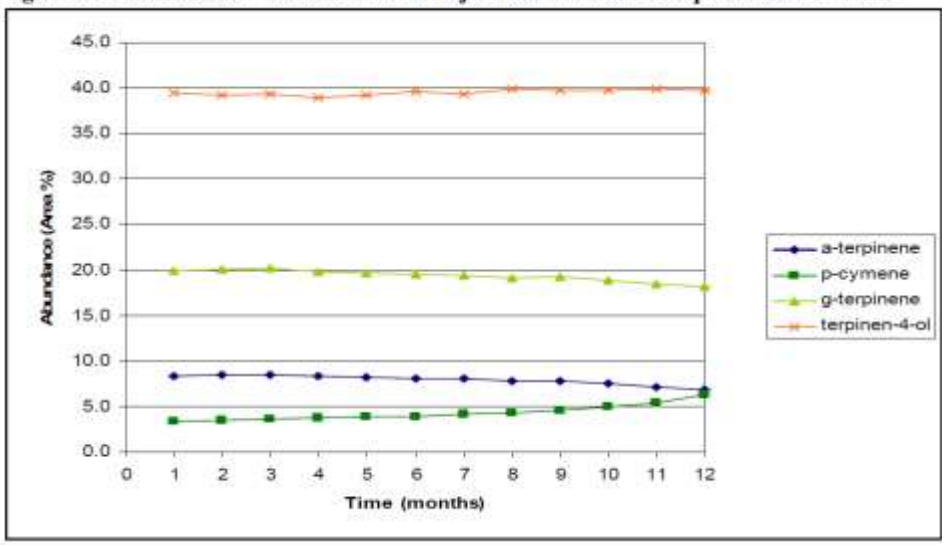
Amber Glass bottles stored in a cool (less than 25 °C), dark environment

In 2007 a joint RIRDC[1]/ATTIA commissioned study asked researchers to investigate the stability of pure Australian tea tree oil. The objective of this study was to determine the changes in tea tree oil composition and peroxide value over 12 months under simulated in-use conditions using 100 ml round amber glass bottles stored in controlled conditions at 25 °C.

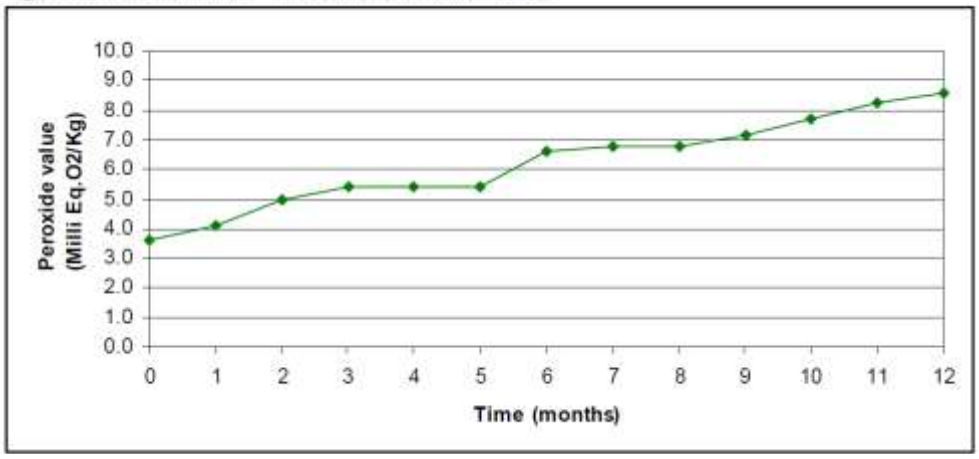
The results indicate that duplicate samples of the two tea tree oil batches tested remained relatively stable over the test period. No significant change was observed in the level of terpinen-4-ol. In both batches after 6 months there is a noticeable downward trend in α-terpinene and γ-terpinene with a similar upward trend in p-cymene. See Figure 1 below.

Figure 1. Batch 60175 – Abundance of Major Tea Tree Oil Components over Time



The peroxide level in both batches rose gradually over the test period. See Figure 3 below.

Figure 3. Batch 60175 – Peroxide level over Time



The researchers concluded that pure Australian tea tree oil monitored over 12 months at 25°C using a standard in-use protocol confirmed that the oils used in this study were stable and safe for use over that period. On the basis of these results ATTIA Ltd recommends that a use-by (best before) date for pure Australian tea tree oil sold in dark glass bottles be set at 6 months from when first opened or 24 months (2 years) in unopened bottles.

[1] Rural Industries Research and Development Corporation

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Bulk stainless steel or other non-reactive containers

For bulk storage ATTIA's Code of Practice [d] requires producers to use only stainless steel or other non-reactive storage and transport vessels and to store the oil in cool, dark conditions with minimum exposure to air. Producers are also encouraged to sparge the headspace of any storage containers with Nitrogen or Argon to exclude air. Incorrect storage and handling procedures can cause oil quality to deteriorate rapidly through oxidation. Quality may alter to the point where it is not saleable or poses a contamination risk to other oil it may be batched with. It is particularly important to manage air exposure in partially filled containers, preferably by sparging with an inert gas (Entwistle: 2005).

Southwell [b] reported that laboratories were surveyed for tea tree oil and product analyses where both peroxide value and p-cymene proportion were determined. Peroxide values for 139 tea tree oils were found. For 77 of these the p-cymene proportion had also been determined. Records also showed aged oils stable for from 3-10 years if stored correctly. (Southwell: 2006)

Brophy et al [a] reported that the variability of the composition of aged oils suggests that oil will retain original quality for 10 years or more if stored under cool, dark, and dry conditions. The extrinsic factors that accelerate oxidation are undergoing further investigation, which has already shown that, in the presence of air and sunlight for 7 months, p-cymene concentration can increase 2-fold while α-terpinene, γ-terpinene and terpinolene concentrations are halved.

Brophy et al went on to say: *In an attempt to determine whether this oxidation was brought about by aging or by extrinsic factors such as moisture, light, or oxygen, several samples of different ages and storage conditions were analysed and compositions compared. Deterioration rates were variable, with occasional samples oxidizing rapidly and failing to meet the requirements of the ISO and AS Standards. After periods as brief as 21 months, some samples contain 20-40% p-cymene and are almost devoid of α-terpinene, γ-terpinene, and terpinolene, all of which oxidize to p-cymene.*

Some of the data extracted from Table 3 in the paper from Brophy et al: 1986 is shown in Table 1 below which demonstrates the change in levels of p-cymene, α-terpinene, γ-terpinene, and terpinolene over time depending on the relative deterioration rate.

On the basis of these findings ATTIA recommends that the use-by (best before) date for pure Australian tea tree oil sold in correctly filled, purged (Nitrogen or Argon) and tightly sealed stainless steel drums stored at an ambient temperature not exceeding 25 °C be set at 3 years from the filling date.

Sample No	1	2	3	4	5
Age (Years)	10	10	5	2	1
Relative deterioration rate	Slow	Rapid	Rapid	Rapid	Moderate
Component %					
terpinen-4-ol	41.6%	31.5%	45.9%	23.8%	37.3%
p-cymene	4.3%	32.0%	21.7%	35.3%	8.0%
α-terpinene	5.8%	0.0%	N/A	0.1%	6.6%
γ-terpinene	15.0%	trace	trace	trace	17.6%
terpinolene	2.7%	trace	trace	trace	3.1%

Table 1 Some data extracted from Brophy et al: 1986 demonstrating change in component % in differing environments

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References

a. Brophy JJ, Davies NW, Southwell IA, Stiff IA, Williams LR, 1989 *Gas chromatographic quality control for oil of Melaleuca Terpinen-4-ol type (Australia Tea Tree).* Journal of Agricultural & Food Chemistry 37: 1330-5.
b. Southwell IA, 2006, p-Cymene and Peroxides, indicators of oxidation in tea tree oil, RIRDC Publication No 06/112.
c. Southwell IA, 2007, *Tea Tree Oil Stability and Evaporation Rate*, RIRDC Publication No 06/112 addendum.
d. Entwistle P, 2005, *Australian Tea Tree Industry Code of Practice*, available from url: http://www.attia.org.au/teatree_about_quality.php

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