Characterization and Comparison of Basil Before and After Drying Using GC, GCxGC, and TOFMS

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INTRODUCTION

GC and MS are well-established techniques for the characterization of food, beverage, and flavor samples. Extending the analytical separation to two dimensions with comprehensive two-dimensional gas chromatography (GCxGC) enhances the peak capacity, allows for exploring more complex samples, and allows for determining more individual analytes within complex samples. TOFMS adds to these benefits and often leads to the identification for these isolated analytes, supporting non-target analyses where analytes of interest and importance can be determined through data evaluation. When coupled together, the benefits are often greater than the sum of the individual techniques.

Software tools that compare sets of GCxGC samples facilitate these analyses and can effectively reveal useful information from the rich data. In this work, we evaluate the aroma characteristics for basil before and after drying. This type of characterization can be useful for quality control, product development, and batch comparisons, as well as understanding the sensory profile changes associated with curing.

METHOD

Cured basil was prepared from a fresh basil sample by heating at 60 °C until the moisture level was between 5 and 10 %. Moisture levels were determined with the TGM800 moisture analyzer (LECO, St. Joseph, MI, USA), as shown in Figure 1. Fresh and cured samples were then analyzed by GC and GCxGC-TOFMS on a LECO Pegasus® BT 4D, as described in Table 1 and shown in Figure 1. Sample amounts were adjusted based on moisture content to analyze approximately 11-12 mg of plant material (100 mg of fresh and 13 mg of cured). An alkane standard was analyzed with the same methods for retention index (RI) determinations.

Table 1. Instrument Conditions

AS	**LECO L-PAL3 Autosampler**
Injection	HS-SPME, 2 min incubate and 5 min extract at 40 °C with triphase fiber
GCxGC	**LECO GCxGC QuadJet™ Thermal Modulator**
Inlet	250 °C, split 20:1
Carrier Gas	He @ 1.40 mL/min, corrected constant flow
Columns	Column 1: Rxi-5ms, 30 m x 0.25 mm i.d. x 0.25 µm coating (Restek) Column 2: Rxi-17Sil MS, 0.6 m x 0.25 mm i.d. x 0.25 µm coating (Restek)
Temperature Program	2 min 40 °C, ramp 10 °C/min to 250 °C, hold 2 min Secondary Oven: + 10 °C
Modulation	1.5 s with temperature maintained +15 °C relative to 2nd oven
Transfer Line	250 °C
MS	**LECO Pegasus BT**
Ion Source Temp	250 °C
Mass range	35-500 m/z
Acquisition Rate	10 spectra/s (GC) and 100 spectra/s (GCxGC)

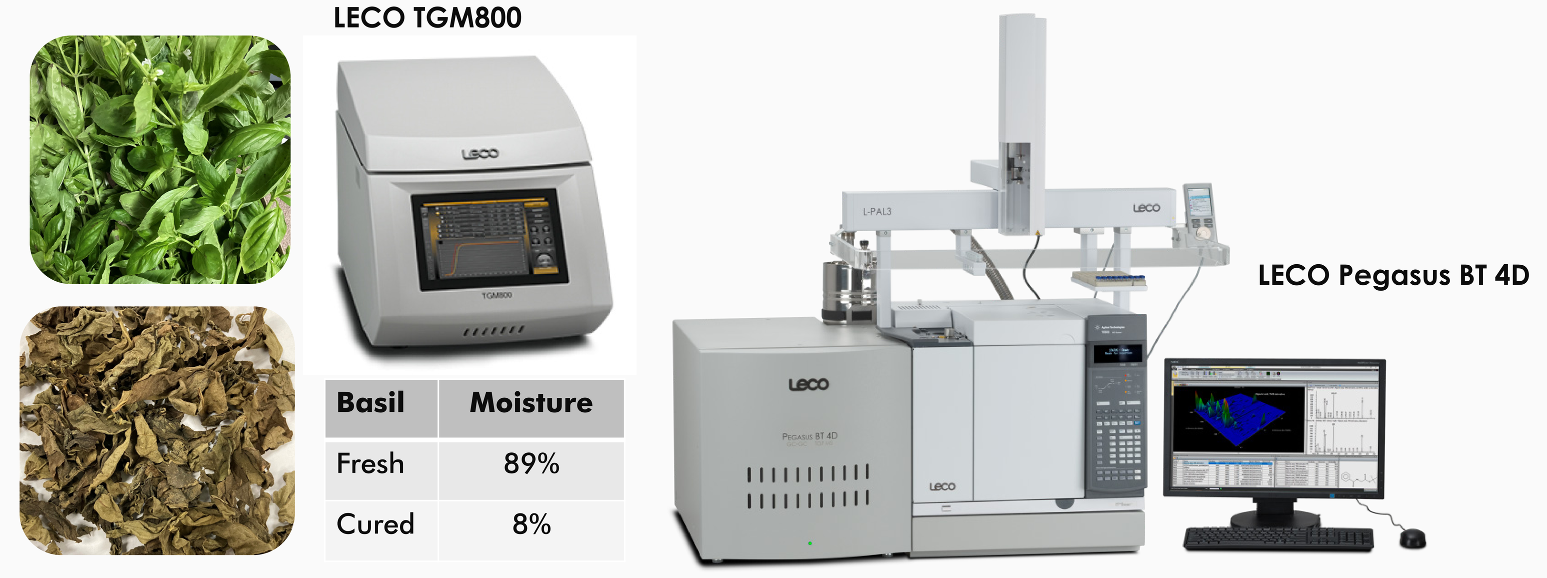

Figure 1. Fresh and cured basil were analyzed. The moisture level in the samples was measured with LECO's TGM800 and aroma profiling was done with LECO's Pegasus BT 4D.

Fresh and Cured Basil

Representative GC and GCxGC chromatograms for fresh and cured basil are shown in Figure 2. Some differences between the samples are visually apparent while others are obscured in the TIC. GCxGC improves the peak capacity and chromatographically separates more analytes to help uncover the hidden differences.

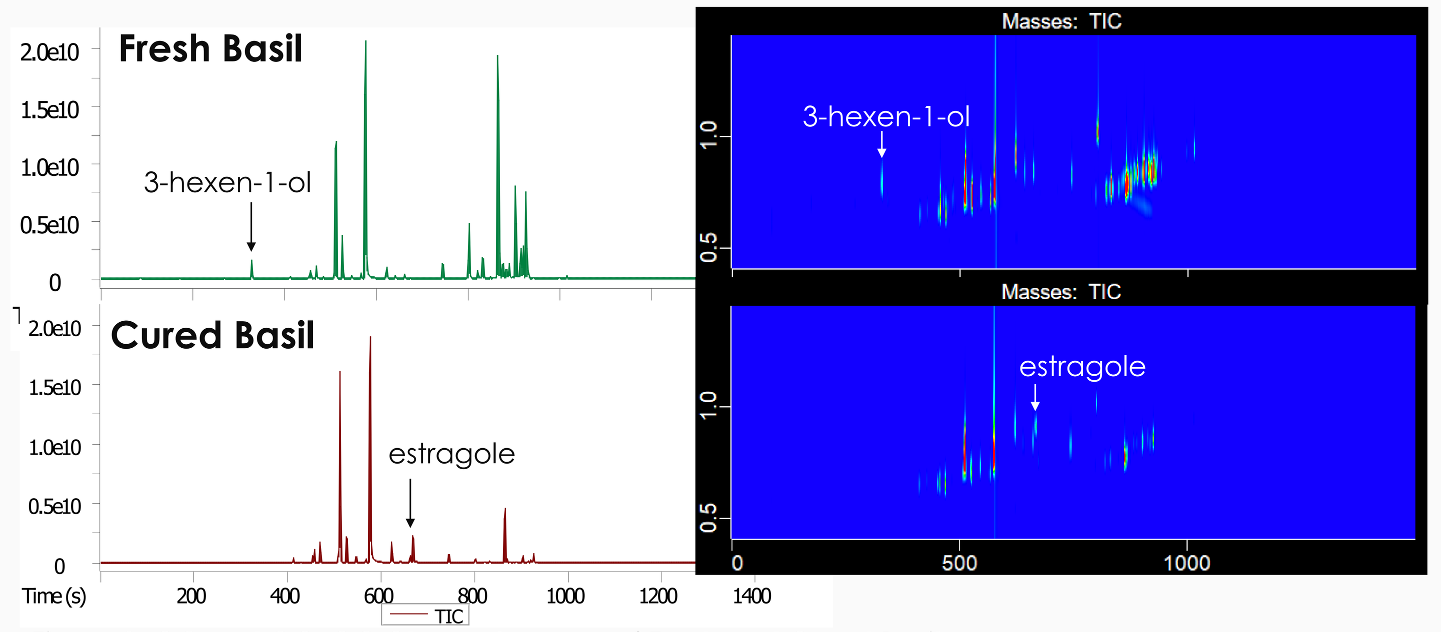

Figure 2. GC and GCxGC Chromatograms of Fresh and Cured Basil.

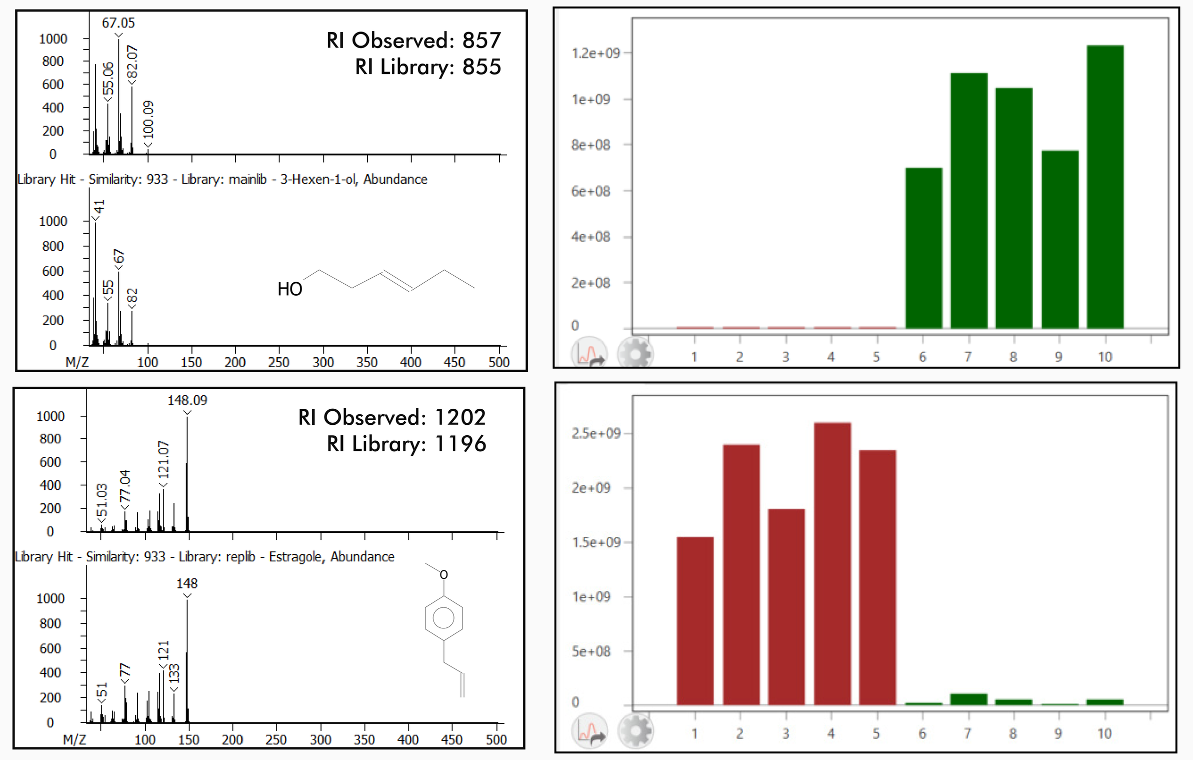

Figure 3. Representative analytes, 3-hexen-1-ol and estragole, differ between fresh and cured basil. These differences were visually apparent in both the GC and GCxGC data as highlighted in Figure 2. Both were readily found with the data analysis tools and are indicated with asterisks in Figure 5.

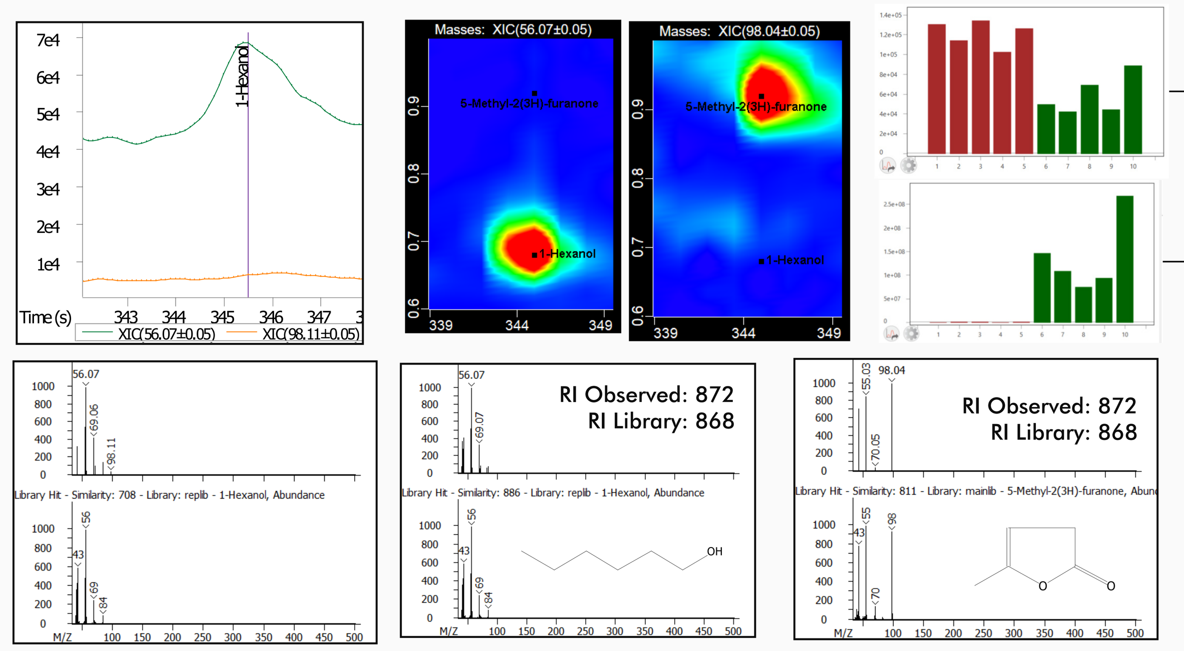

Figure 4. Other analyte differences were obscured in the 1D GC data. Two analytes that coelute in the GC data were chromatographically resolved with GCxGC, allowing for improved spectral similarity scores and revealing additional differences between the fresh and cured basil. These were also readily found with the data analysis tools, as indicated in Figure 5.

ChromaTOF® Tile to Find Features of Interest

Additional differences between the fresh and cured basil samples were explored with *ChromaTOF* Tile software. With *ChromaTOF* Tile, chromatographic windows that distinguish the sample groups are identified and the corresponding features can be determined. Some representative analytes that differ between the samples are shown in Figure 5.

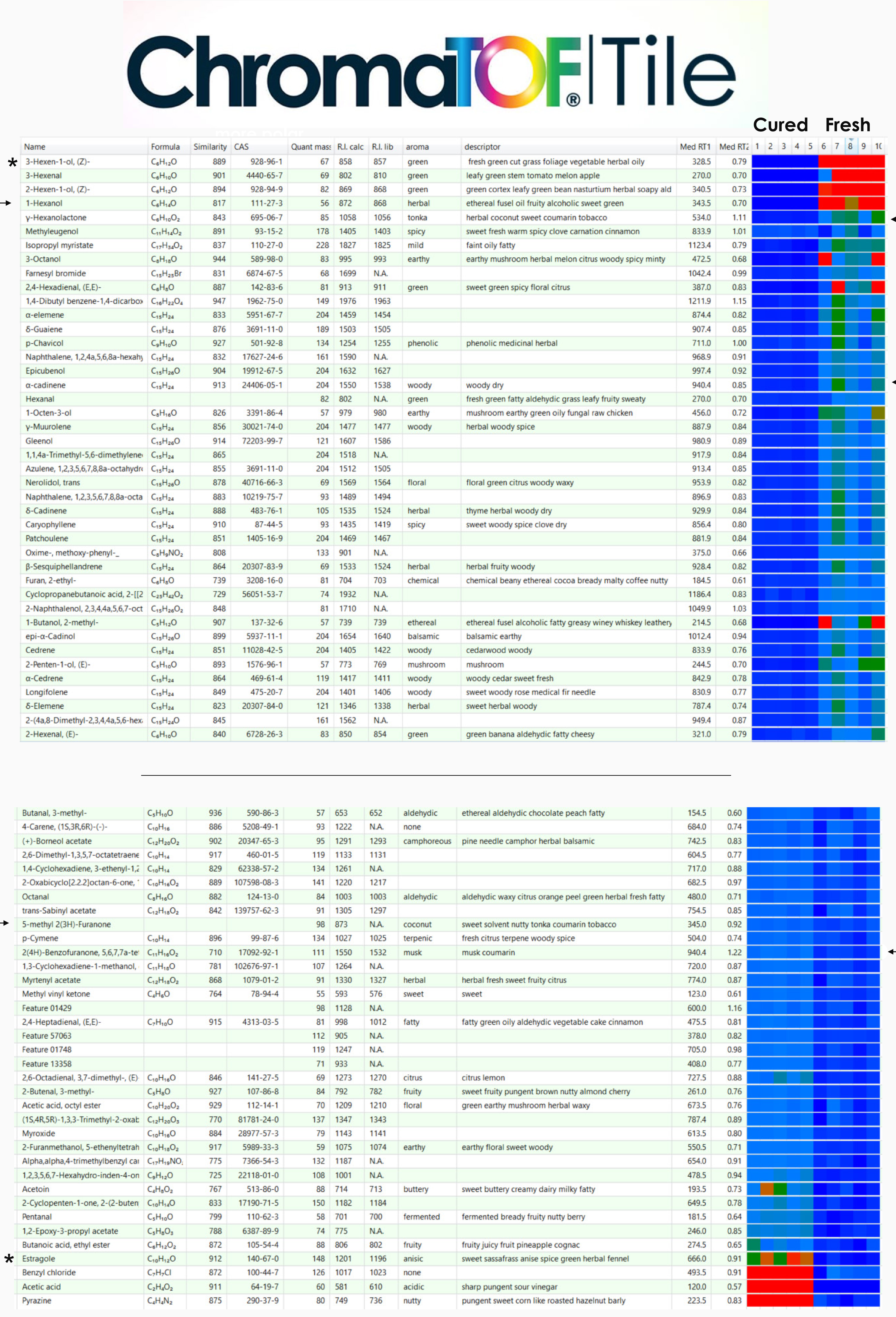

Figure 5. Representative examples of features that differ between the fresh and cured basil are shown. These were located and compiled with ChromaTOF Tile. Tentative identifications are listed.

Improved Peak Capacity with GCxGC

GCxGC was crucial for the determination of many of the analytes that differed between the fresh and cured basil. Some key analyte differences were coeluting in the 1D data, as shown in Figures 4, 6, and 7. GCxGC chromatographically separated these coelutions and then *ChromaTOF* Tile helped to locate them within the complex data.

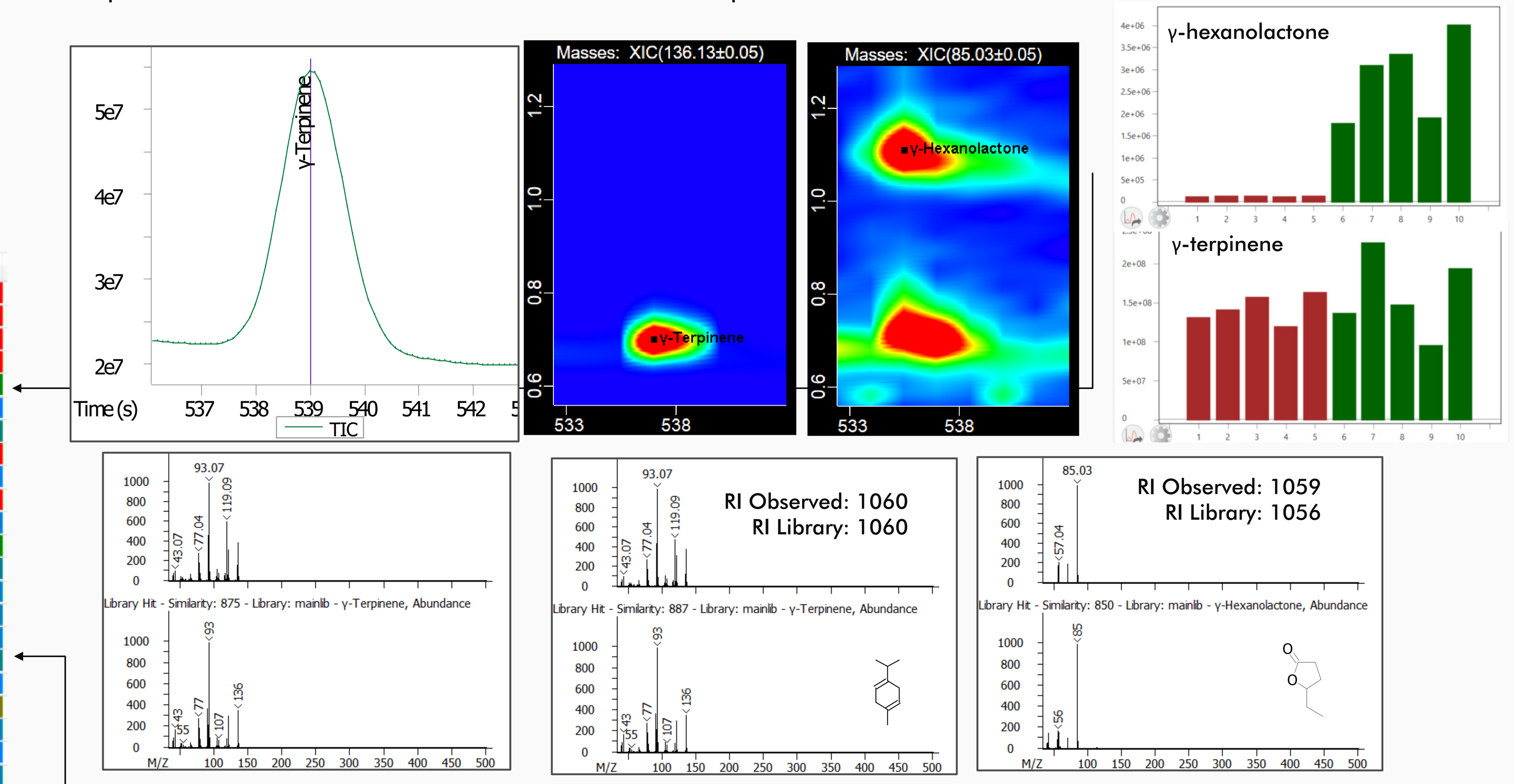

Figure 6. A lactone, observed at higher levels in the fresh basil, was hidden by a terpene in the 1D GC data. The coelution was resolved in the second dimension of the GCxGC data allowing the additional analyte to be identified and highlighted with ChromaTOF Tile.

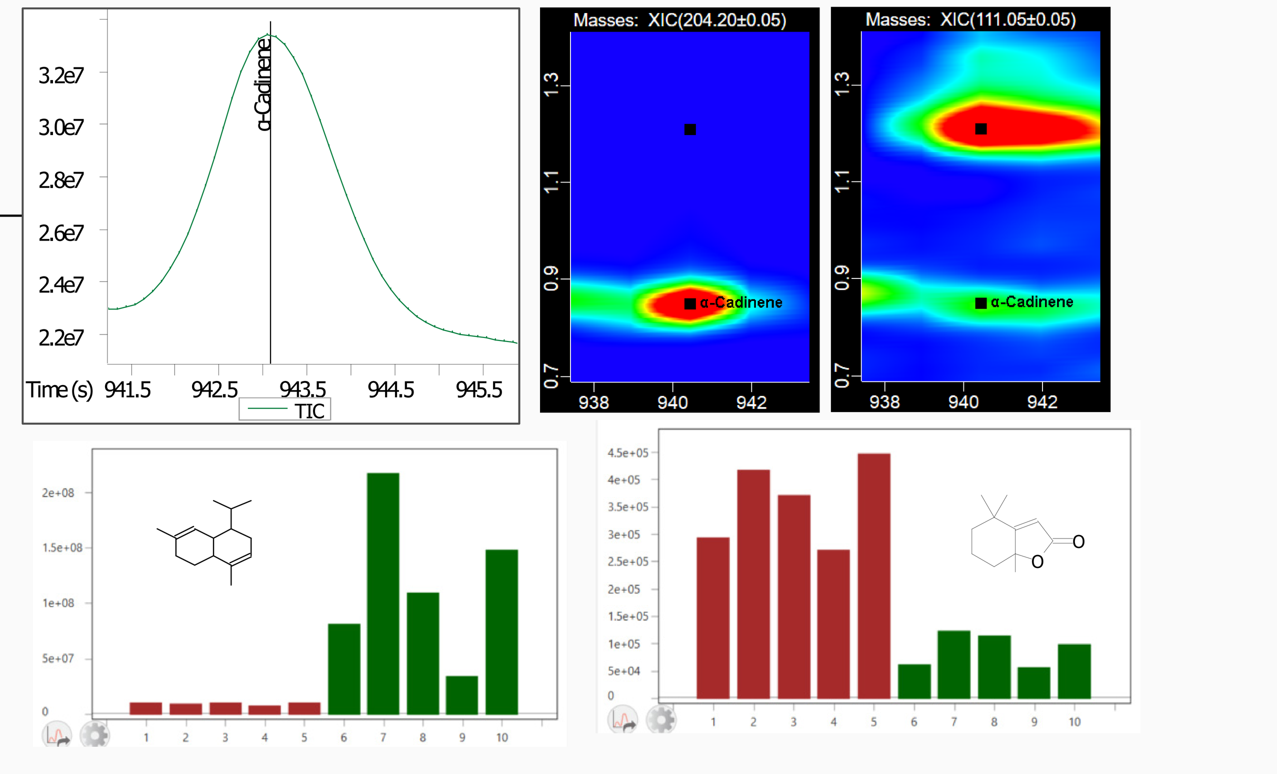

Figure 7. Two analytes that were observed to distinguish the fresh and cured basil were coeluting in the 1D GC data. The coelution was resolved in the second dimension of the GCxGC data and the difference was highlighted with ChromaTOF Tile.

CONCLUSIONS

In this work, LECO's TGM800 was used to determine the moisture level of fresh and cured basil samples and LECO's *Pegasus* BT4D GCxGC-TOFMS with *ChromaTOF* Tile were used to highlight differences between the chemicals which contribute to the sensory profiles of fresh and cured basil. This provided a better understanding of the aroma characteristics of these samples. Many differences were observed between the samples and the analytical tools used in this work were crucial for their determination.