TORION TECHNOLOGIES INC.

Volatile and Semivolatile Compound Screening of Cocoa Raw Materials and Chocolate Products using Headspace SPME and GC-TMS

Solid phase microextraction (SPME) was used to generate volatile and semivolatile compound profiles of raw materials used in the production of chocolate products. The headspace of cocoa beans, cocoa butter, and finished chocolate were sampled using SPME and analyzed using a portable gas chromatograph-toroidal ion trap mass spectrometer (GC-TMS). This SPME-GC-TMS screening method can be used to insure better raw material and end-product quality.

Introduction

The quality of raw materials used for the production of chocolate greatly impacts the finished product quality where taste and odor can be greatly influenced by both the source, transport, and storage conditions of the raw materials. Volatile and semivolatile organic compounds (VOCs, SVOCs) found in products originating from cocoa beans have been characterized [1] and compounds indicative of quality can be identified [2]. Analysis of raw materials prior to use in the chocolate-making process can be advantageous for the manufacturer since the presence of chemical defects such as subtle molding or elevated free fatty acid content resulting from improper storage conditions can impact a cocoa bean's taste and odor characteristics. By detecting off-flavor compounds early in the supply chain and during the manufacturing process, chocolate makers can eliminate poor quality raw materials and decrease rejection rates of finished products not meeting quality standards.

Headspace SPME, when combined with portable GC-TMS, is an effective tool for the on-site analysis of VOCs and SVOCs prior to shipping and upon receipt of raw materials by the cocoa manufacturer. The ability to quickly fingerprint raw materials for the presence of mold indicator compounds at various points in the supply chain can reduce costs by allowing the procurer (shipper, buyer, broker, etc.) to rapidly evaluate the quality of a shipment before it is purchased. In addition to cocoa beans, prescreening of, raw materials and end-products can be broadly applied to numerous other foodstuffs to determine food quality and ensure food safety.

Sample Preparation and Analysis

Cocoa beans, cocoa butter, and finished chocolate product were provided by Theo Chocolate (Seattle, WA). A CUSTODION SPME syringe with a 65 µm Polydimethylsiloxane/Divinylbenzene (PDMS/DVB) fiber was used for extraction by exposing the fiber directly into the headspace above each sample for 20 sec to 20 min at elevated temperatures (45-65°C). This sampling technique improved sample off-gassing and analyte collection of VOCs and SVOCs in the sample.

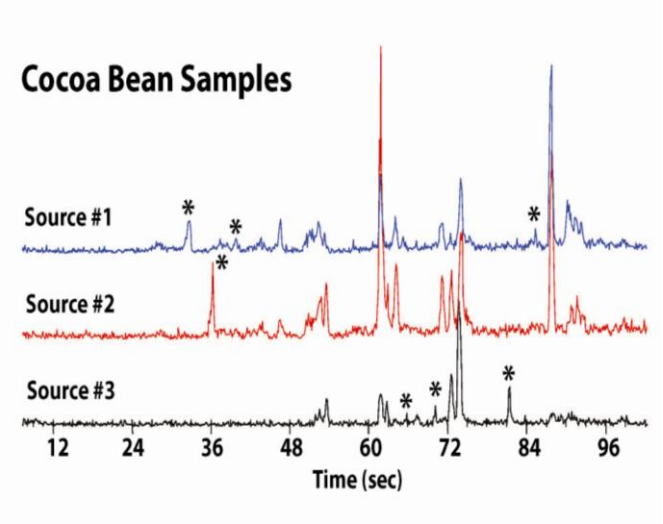

Figure 1: Chromatogram of cocoa bean headspace from three sources (PDMS/DVB SPME fiber, 20 min exposure, 65°C). Asterix (*) represents quality marker compounds

Following each sample preparation, the SPME syringe was inserted into the TRIDION™-9 GC-TMS injection port where the target analytes were desorbed into a low thermal mass injector (270°C) coupled to a capillary GC

column (MXT-5, 5 m x 0.1 mm, 0.4 µm d_f [Restek, Bellefonte, PA]). After an initial 10 second hold at 50ºC, the GC temperature was increased at 2°C/sec to 280ºC. The capillary GC is coupled to a TMS detector having a mass range of 45-500 *m/z* and a scan rate of ~10Hz.

Experimental Conditions

Sampling:	Solid phase microextraction (SPME)
SPME Phase:	Divinylbenzene/Polydimethylsiloxane (DVB/PDMS, 65 µm)
GC Inj. Temp:	270°C
GC Column:	MXT-5, 5 m x 0.1 mm, 0.4 µm d_f
GC Carrier Gas:	Helium, 0.2ml/min, constant pressure
GC Column Temp:	50-280°C at 2°C/sec
Transfer Line:	250°C
Injector Split Ratio:	20:1
Mass Analyzer:	Toroidal ion trap (TMS)
TMS Mass Range:	45-500 Da
Ionization Mode:	In-trap electron impact
Detector:	Electron multiplier
Vacuum:	Roughing and turbo molecular pumps
Resolution:	Less than unit mass to 230 amu, nominal unit mass to 500 amu

Results

Figure 1 shows the GC-TMS chromatograms for three organically cultivated, fermented, dried cocoa beans from different sources. Key components in the headspace eluted in less than 90 seconds under the GC-TMS conditions listed above. Peaks marked with an asterix(*) represent candidate VOC and SVOC markers which potentially can be used to evaluate the acceptability of a raw material shipment. By comparing VOC and SVOC compounds emitted by the sample to a representative VOC and SVOC profile from an acceptable lot, the analyst can make an initial determination of commodity quality.

Conclusions

VOCs and SVOCs from foods and commodities can be quickly analyzed at the buyer source using headspace SPME-GC-TMS. Using the CUSTODION SPME syringe and TRIDION-9 GC-TMS, both field and laboratory screening of marker compounds can indicate initial raw material quality and support rapid decision making for purchases. The ability to analyze a wide variety of organic compounds provides timely and critical information at the commodity source. The short GC-TMS analysis time allows the user to quickly analyze multiple samples on-site if required.

References

1. Frauendorfer, F., Schieberle, P., J. Agr. Food Chem. 2006, **54**, *15*, 5521–5529.
2. Humston, E.M., Zhang, Y., Brabeck, G.F., McShea, A., Synovec, R.E., J. Sep. Sci. 2009, **32**, 2289 – 2295.

Acknowledgements

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