

Introduction

Tea is a very popular drink around the world and its consumption continue to grow. With large number of varieties and additional flavors, it constitutes an interesting coffee alternative.

Producers of tea always look for new tastes or better quality of their products to increase their market place.

In this study, it is proposed to use HERACLES NEO fast gas chromatography, aroma analyzer to evaluate different brands of mint green tea.



Materials & Methods

Samples

Three commercial brands of mint green tea were used for analysis: Brightley® (store brand), Lipton® and Twinings®. These teas have been infused in water at 80°C at different times 1, 2, 3, 5, 10, 15 and 20 minutes. Each level of infusion was analyzed in three replicates. 3-minutes infusion samples have been used for benchmarking (Table 1). All samples were compared for flavor profile study.

Samples	Code
Brightley® - Infusion time 3 min	B3
Lipton® - Infusion time 3 min	L3
Twinings® - Infusion time 3 min	T3

Table 1. Samples set for benchmarking study

HERACLES NEO Smell analyzer

HERACLES NEO Electronic Nose (Fig. 1) is based on ultra fast gas chromatography. It features 2 metal columns of different polarities (non polar MXT-5 and slightly polar MXT-1701, 10m length, 180µm diameter, Restek) in parallel

and coupled to 2 Flame Ionization Detectors (FID). Two chromatograms are obtained simultaneously, allowing a sharper identification of the chemical compounds. It allows headspace or liquid injection modes.

The integrated solid adsorbent trap thermo-regulated by Peltier cooler (0-280°C) achieves an efficient pre-concentration of light volatiles and shows a great sensitivity.

With fast column heating rates (up to 480°C/min), results are delivered within seconds and the usual analysis cycle time is 8 minutes.



Fig. 1: Ultra Fast GC based HERACLES NEO Electronic Nose (Alpha MOS, France)

The electronic nose is coupled to an autosampler (PAL3 RSI, CTC Analytics) to automate sampling and injection.

The instrument is monitored by Alpha Soft software. In addition to classical chromatography functionalities, it provides chemometrics data processing tools such as sample fingerprint analysis for comparison, qualitative and quantitative models, quality control charts.

AroChemBase: Kovats Index library for chemical & sensory characterization

HERACLES NEO e-nose is additionally equipped with AroChemBase module (Alpha MOS, France) that can be used within AlphaSoft E-Nose software. It consists of a library of chemical compounds with name, formula, CAS number, molecular weight, Kovats retention Index, sensory attributes and related bibliography. It allows to pre-screen the chemical compounds and giving sensory features by directly clicking on the chromatograms' peaks.

Smell analysis

Analytical conditions

For all samples, one teabag was infused at the corresponding time in 100 mL of water at 80°C. 1mL of these samples was transferred into a vial. The analytical parameters optimized are described in Table 2.

Parameters	
Sample volume	1 mL
Vial	20 mL
Acquisition duration	110 s
Incubation temperature	40°C
Incubation time	20 min
Injection volume	5 mL

Table 2: HERACLES NEO e-nose analytical parameters

A standard mixture of n-alkanes (n-pentane to n-hexadecane) is used to calibrate the system, to allow retention time conversion into Kovats indices.

Volatile profiles

The odor profile comparison of tea samples is displayed on Fig 3. The chromatograms of the 3 brands showed quantitative and qualitative differences.

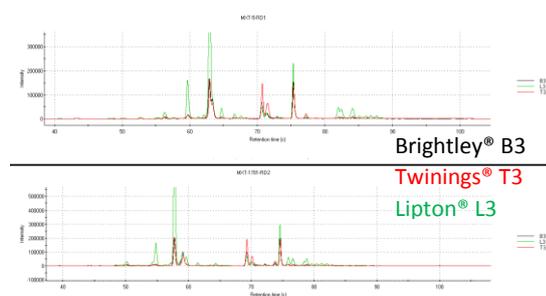


Figure 3. Heracles NEO e-nose volatile profiles of tea samples

An odor map based on Principal Component Analysis can be generated using discriminating volatile compounds (Figure 4). There is a good discrimination between the three tea samples. Brightley® and Twinings® are closer than Lipton®.

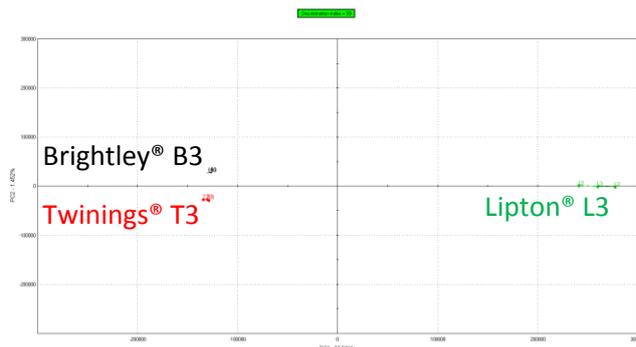


Figure 4: Odor map of 3-minute infusion tea samples based on principal component analysis (PCA)

Flavor profile comparison

AlphaSoft allows to follow the evolution of overall volatile profile of the three brands of tea overtime, thanks to shelflife algorithm. This evolution is shown as a distance compared to the first time of infusion (reference).

Infusion time representation for the three different tea samples shows different evolution (Figure 5). Lipton® and Twinings® have their most intense volatile profile around 2-3 min of infusion and decrease from the 5th minute. Brightley®, at the opposite, seems to have an aroma profile that still increase after 3 min of infusion.

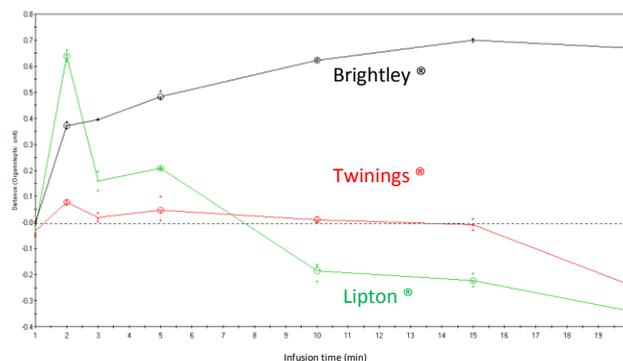


Figure 5. Infusion time representation of tea samples based on shelflife modelisation

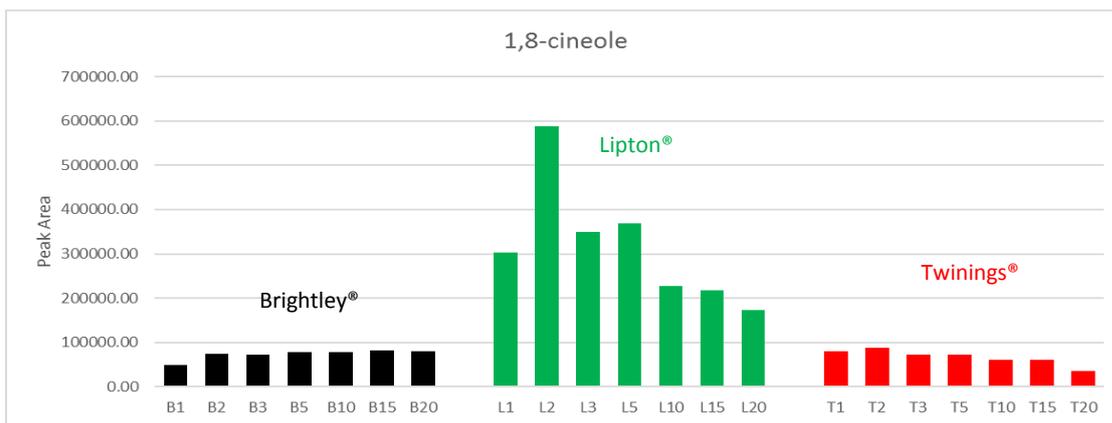


Figure 6. Evolution of 1,8-cineole overtime

AroChemBase: volatile compounds identification

The nature of most discriminative volatile compounds in tea samples was investigated using their Kovats index and the AroChemBase database.

The possible identification of compounds is presented in Table 3. Terpenes are the main discriminative compounds. AroChemBase also allows to have access to sensory descriptors with odor threshold for detection and recognition (example for Carvone in figure 7.) These compounds gave fruity and minty notes to the tea samples.

RI MXT-5 (±20)	RI MXT-1701 (±20)	Possible identification	Descriptor
952	964	α-pinene	Herbaceous, Terpenic
997	1027	Myrcene	Fruity, Geranium, Etheral
1048	1073	1,8-cineole (Eucalyptol)	Camphor, Herbaceous, Mentholic
1054	1094	Limonene	Citrus, Fruity, Minty
1182	1289	Menthol	Mentholic
1275	1411	Carvone	Basil, Bitter, Minty, Peppermint

Table 3: AroChemBase identification of the main compounds

Intensity of each molecule can be followed for each infusion time. Figure 6 shows the follow up of the one of the main compounds intensity, identified as 1,8 cineole by AroChemBase.

Conclusion

This study of commercial brands of mint flavored green tea was investigated by using the HERACLES NEO e-nose.

The e-nose was able to clearly discriminate the three different brands and to show the aroma profile evolution during infusion for each brand. The software also allows to follow intensities of molecules at different time.

AroChemBase showed that main compounds of teas are Menthol, 1,8-Cineole and Carvone with some fruity and minty notes.

This study demonstrates that HERACLES NEO e-nose can be a valuable tool for quality control of teas and suitable solutions for the development of new flavors.

	Value	Minimum	Maximum	Median	Mean	S-D	Number
DB-5	1264	1253	1276	1264	1264	12	2
DB-1701	1400	1386	1413	1400	1400	14	2

	Type	Value	Minimum	Maximum	S-D	Unit	Number
Air	Detection	0.03	0.02	0.15	0.05	mg/m3	5
Air	Recognition	0.11	0.10	0.12	1.e-2	mg/m3	1
Aqueous solu	Detection	5.e-3	5.e-3	5.e-3	-	mg/kg	1
Phthalate	Detection	8.00	0.32	2.82e+3	1.33e+3	mg/kg	2

Figure 7: Sensory attributes of Carvone