

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 time of infusion for mint-flavored green tea.



Materials & Methods

Samples

A commercial of mint green tea was used for analysis: Brightley® (store brand). This tea has been infused at different times as described in Table 1. Each level of infusion was analyzed in three replicates.

Samples	Code
Brightley® - Infusion time 1 min	B1
Brightley® - Infusion time 2 min	B2
Brightley® - Infusion time 3 min	B3
Brightley® - Infusion time 5 min	B5
Brightley® - Infusion time 10 min	B10
Brightley® - Infusion time 15 min	B15
Brightley® - Infusion time 20 min	B20

Table 1. Samples set

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, 180 µm 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 pre-screening the chemical compounds and giving sensory features by directly clicking on the chromatograms' peaks.

Smell analysis

Analytical conditions

The analytical parameters optimized for this analysis 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 showed quantitative differences.

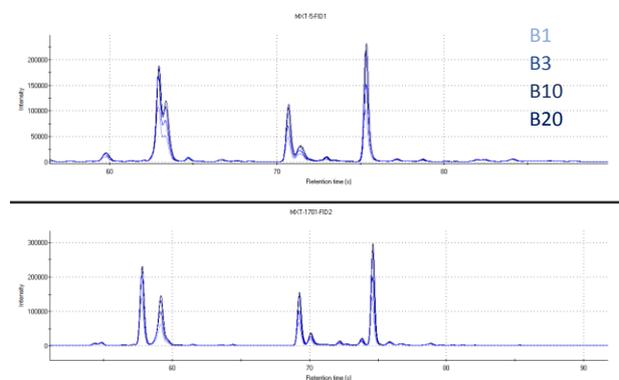


Figure 3. Heracles NEO e-nose volatile profiles of tea samples

An odor map based on Principal Component Analysis can be generated using all volatile compounds (Figure 4). There is a good discrimination between the four tea samples. There is a shift induced by infusion time, samples are distributed along PC1 axis.

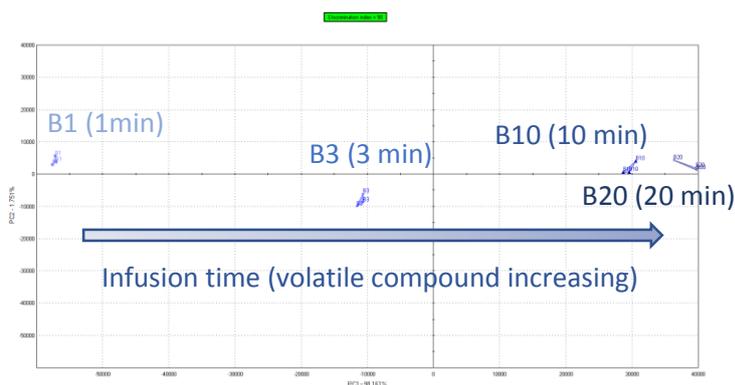


Fig.4: Odor map of 4 infusion time tea samples based on principal component analysis (PCA)

The area of the six main compounds (identified using AroChemBase) was followed with AlphaSoft (Figure 5). Long infusion times induce a higher release of compounds like menthol or carvone.

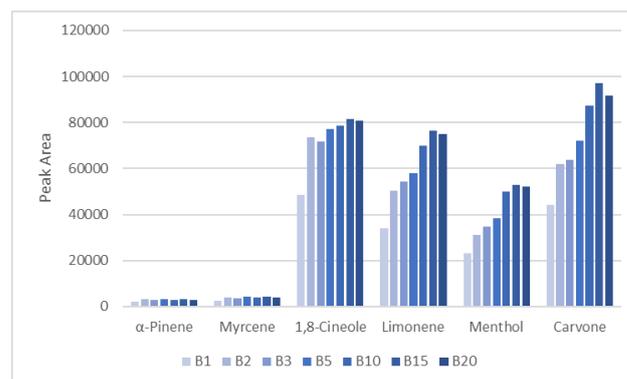


Figure 5: Peak areas for the 6 main compounds at every infusion time

Quality control

AlphaSoft has several quality control models to assess sensory features of products and ensure quality of tea is conform to the reference one. Statistical Quality Control module (SQC) compares the organoleptic distance between reference group and every other groups to give an easy-to-understand result, conform or different. This typical model can be used online for quality control. The 3-minutes infusion samples (time advised by the manufacturer) were used as reference (Fig 6). Other samples were then projected as unknown. The acceptable area is displayed in blue, if sample is projected inside it is considered conform to the reference and outside are samples considered as non-acceptable. Only sample B2 (2 minutes of incubation) was projected in the

conform area which indicates that 2 or 3 min allows a similar flavor intake.

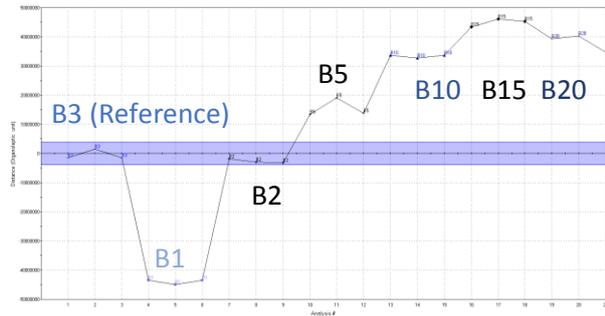


Figure 6: SQC results for tea samples

Predictable model of infusion

A PLS (Partial Least Square) model can be used to predict the infusion time of tea. On Figure 7, samples B1, B3, B10 and B20 were used to build the correlation curve. Then, samples B2, B5 and B15 were projected as unknown. Results are presented in Table 3.

Sample	Real infusion time	Projected infusion time	Standard deviation
B2	2.0	3.1	0.1
B5	5.0	5.4	0.4
B15	15.0	18.8	0.6

Table 3: Projection results of unknown samples

Conclusion

Infusion of mint-flavored green tea samples was investigated by using the HERACLES NEO e-nose.

The e-nose was able to clearly determine that steeping time has a clear impact on VOC release overtime.

SQC model was able to monitor the quality of tea infusion with important change of aroma from 1 to 5 minutes.

This study demonstrates that HERACLES NEO e-nose can be valuable tool for quality control of teas and suitable solutions for tea industries.

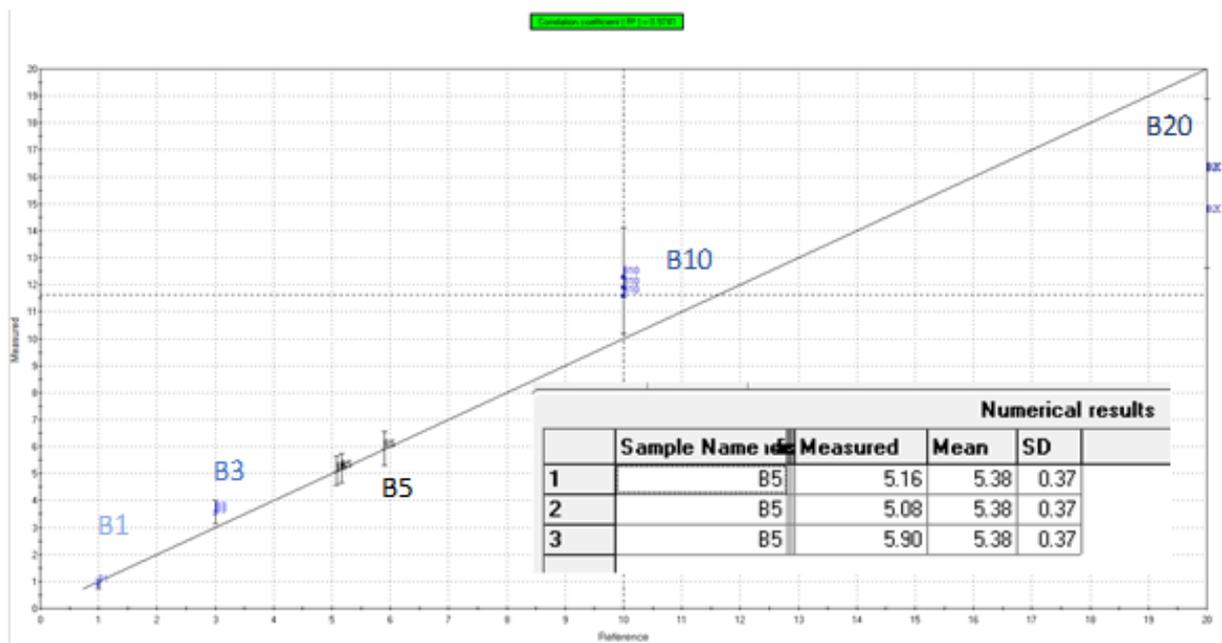


Figure 7: PLS predictable model of infusion time with B5 sample projected results