Alpha MOS

ANH32–Quality investigation on Yerba Mate tea

Introduction

Popular throughout the Middle East and South America, *Ilex paraguariensis*, commonly called yerba mate, is a flowering tree. Traditionally, its leaves are brewed into a stimulating tea, with a lot of health benefits.
Drying, milling the leaves and storage are different necessary steps to manufacture this tea. They are essential to reveal the characteristic flavors of the plant.
In this study, it is proposed to use HERACLES fast gas chromatography Electronic Nose and IRIS visual analyzer to evaluate the olfactory profile and visual aspect between good and bad qualities and to identify compounds responsible for the bad smell.

Equipment

HERACLES Electronic Nose

HERACLES Electronic Nose (Alpha MOS, France – Fig. 1) is based on ultra fast chromatography. It features 2 metal columns of different polarities (non polar RXT-5 and slightly polar RXT-1701, length = 10m, diameter = 180µm, *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 (0-260°C) achieves an efficient pre-concentration of light volatiles and shows a great sensitivity (in the pg range).
With fast column heating rates (up to 600°C/min), results are delivered within seconds and the analysis cycle time is around 5 to 9 minutes.

Fig. 1: Ultra Fast GC based HERACLES Electronic Nose

The electronic nose is coupled to an autosampler (HS 100, CTC Analytics) to automate sampling and injection.
The instrument is operated through Alpha Soft software. In addition to classical chromatography functionalities, it provides chemometrics data processing tools such as sample fingerprint analysis and comparison, qualitative and quantitative models, quality control charts.

AroChembase: Kovats Index library for chemical& sensory characterization

HERACLES e-nose was 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.

IRIS Electronic Eye

Through a CCD camera, the IRIS analyzer achieves a detailed visual assessment of both color and shape parameters of the overall products or selected portions of these products.

Fig. 2: IRIS Electronic Eye

The instrument is operated with the same AlphaSoft software platform that allows analytical parameters monitoring, data acquisition and processing.

Smell analysis

Samples & Analytical Conditions

Three samples of two yerba mate qualities were analyzed to understand the differences of each odor profiles.

Sample	Quality	Sensory quality
GB1/GB2/GB3	Optimal	Fresh, green, low bitter
BB1/BB2/BB3	Poor	Humidity, strong, more bitter

Table 1: Yerba mate samples

HERACLES Parameters	Values
Sample mass	1 ± 0.01 g
Water added	1 mL
Sample incubation	20 min at 70°C
Injected volume	5 mL
Acquisition time	110 s

Table 2: HERACLES e-nose analytical parameters

First, a standard mixture of n-alkanes (n-pentane to n-hexadecane) was analyzed, to allow retention time conversion into Kovats indices.

Chromatograms

The comparison of the volatile profiles of good and bad qualities shows significant differences (Figure 2). It seems that the good blends present higher peak intensity than the bad blends.

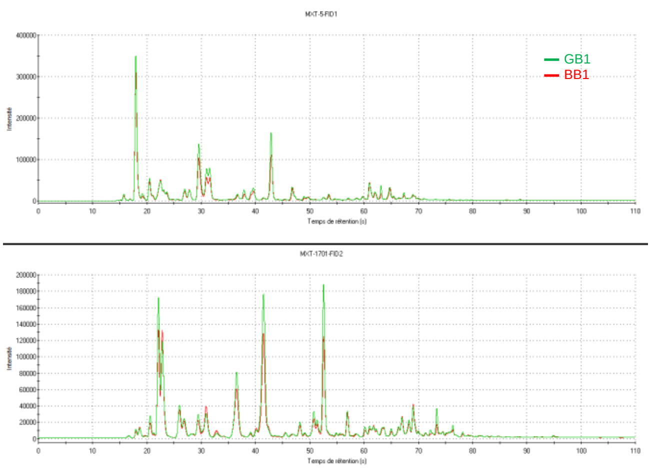

Fig 2. Heracles e-nose chromatograms of yerba mate optimal and less optimal quality samples

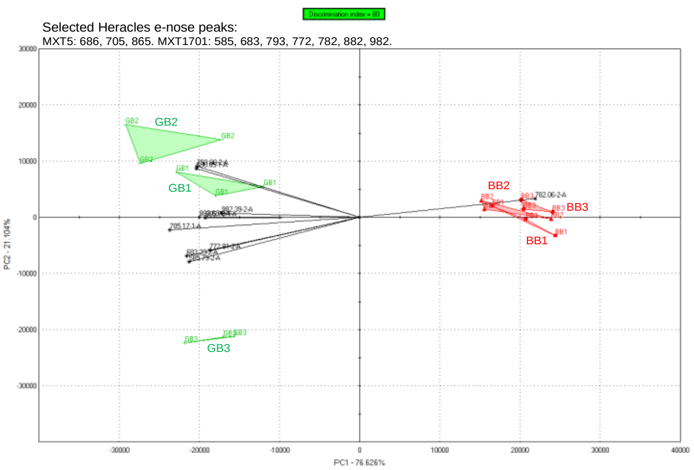

Fig 3. Yerba mate samples odor map based on principal component analysis (PCA) with projection of the selected volatile compounds of Heracles e-nose

Odor Profile Comparison

To rapidly and easily compare the odor profiles of the different samples, an odor map based on Principal Components Analysis (PCA) was set up (Figure 3).

A selection of the most discriminative molecules was made and allows identifying the compounds responsible for the differences between the various batches.

On this map, the closer the samples are, the closer their flavor profiles are. It shows a clear discrimination between the different qualities of blends and some peaks are characteristic of each quality.

Chemical Compounds Investigation

The nature of most discriminant volatile compounds involved in the smell differences was investigated using their Kovats index and the AroChemBase database (Table 3). To compare the presence of each compound in the various samples, a histogram was built (Figure 4).

The bad quality presents less volatile compounds in concentration compare to the good sample. Most of the odorous compounds correspond to aldehydes as pentanal and propanal, or esters as methyl isobutyrate. The associated descriptors are fruity, floral or almond, which could explain the fresh sensory quality of good samples.

RT MXT-5 (± 0.1s)	RT MXT-1701 (±0. 1s)	RI MXT-5 (± 20)	RI MXT-1701 (± 20)	Possible identification	Descriptor
17.94	22.13	503	**585**	**propanal**	plastic, pungent, solvent
17.94	22.84	503	601	2-propanol	alcohol, etheral
22.49	30.91	603	701	2-butanol	-
22.49	36.53	603	750	acetic acid	acidic, vinegar
26.94	29.47	656	**683**	**nd**	-
29.56	36.53	**686**	750	**methyl isobutyrate**	floral, fruity, sweet
31.53	41.47	**705**	**793**	**pentanal**	almond, green, malty
37.88	48.17	762	856	2-methylpentanal	earthy, etheral, fruity
39.61	39.09	777	**772**	**octane isomer**	alkane
39.61	40.14	777	**782**	**octane isomer**	alkane
39.61	50.75	777	**882**	**1-hexen-3-ol**	green
42.87	52.54	806	897	hexanal	acorn, fatty, fruity, herbaceous
46.75	56.91	843	946	4-methylpentanol	fruity, green, oily, yeasty fermented
48.93	60.13	**865**	**982**	**1-hexanol**	dry, floral, grassy, resinous
60.98	69.02	994	1100	3-octanol	herbaceous, mushroom, nutty
61.98	68.26	1006	1090	2-ethyl-3-methylpyrazine	balsamic, roast
63.11	73.38	1022	1169	2,4-heptadienal	fatty, nutty, orange oil
64.71	66.97	1044	1072	limonene	citrus, fruity, minty

Table 3.Volatile compounds identified in yerba mate

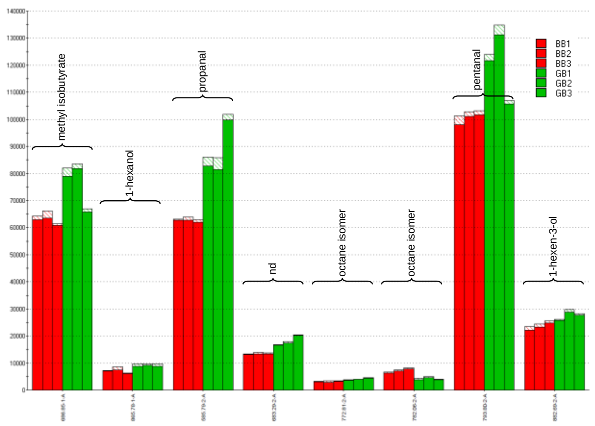

Fig 4.Histograms of the peak intensity of the most discriminant molecules from yerba mate (area of selected peaks) on Heracles e-nose

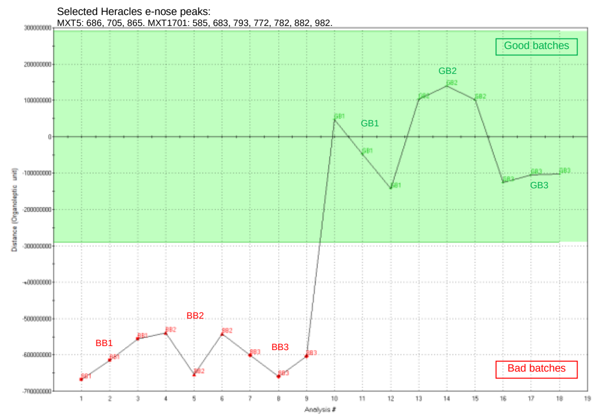

Fig 5.Quality control (SQC) of yerba mate odor based on selected peaks detected with Heracles e-nose

Quality control model

Building a quality control model helps to follow up the quality evolution during the processing steps. Based on Statistical Quality Control (SQC) algorithm, this chart was built with the three good batches as reference (Figure 5).

The sampling and number of reference batches are very important to define a good model. To be considered as robust this model should be built with more references of each batch.

The three bad batches are projected out of specification and considered as different from the reference.

Visual analysis

Samples & Analytical Conditions

For this analysis, two qualities were analyzed in duplicates (tables 4& 5).

Sample	Quality	Sensory quality
Good green color	Optimal	Fresh, green
Bad green color	Poor	Toasted

Table 4: Yerba mate samples

IRISParameters	Values
Lens	5 mm
Image size	1703x1278
Lighting	Top and bottom light
Sample tray	36x24 cm
Sample mass	68 g

Table 5: IRIS e-eye analytical parameters

Images

Four pictures of the 2 good batches and of 4 pictures of one bad batch were taken with the Iris system (Figure 6). The samples are slightly different in color but statistical analysis will help compare them to each other.

Good green color

Bad green color

Figure 6. Pictures of green yerba mate samples for color analysis

Color spectrum

The color spectrum of green yerba mate can be analyzed using AlphaSoft software. The system displays the proportion (in %) of each color (Figure 7).

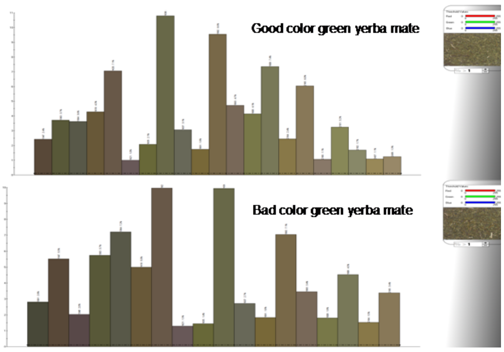

Figure 7.Color spectrum comparison for the good and bad green yerba mate batches

Statistical analysis on color

To easily evaluate the dissimilarities of color distributions in each sample, the most discriminative colors were selected and a chart of their proportions in each sample was built (Figure 8). The bad green color yerba mate presents more dark colors than the good green one.

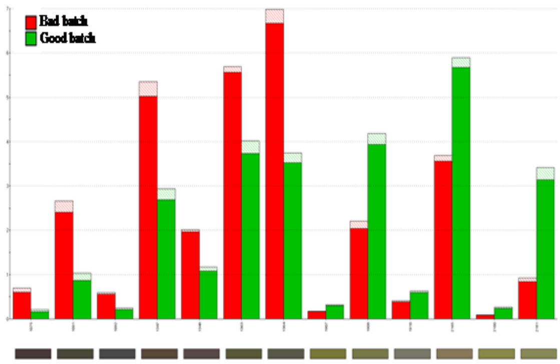

Figure 8.Comparison of colors distribution of good and bad green yerba mate batches based on global color spectrum

Quality control model

A quality control model based on color can be designed to check the quality of new batches using current quality batches as reference group (Figure 9).

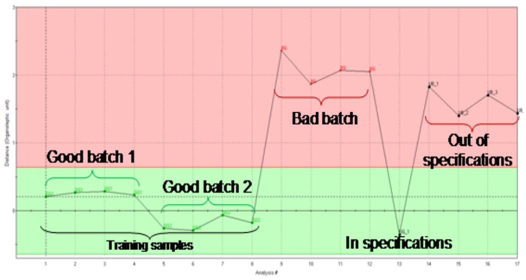

Figure 9.Statistical Quality Control (SQC) chart based on selected colors with current quality samples as reference group

Conclusion

The HERACLES system allows discriminating the various qualities of yerba mate tea based on their odor profiles.

The chromatograms showed that most of the discriminant volatile compounds involved in variation between the good and bad batches are characterized by a decrease in intensity in bad samples. Thanks to the Arochembase database, it was possible to display a list of several molecules involved in odor profile variations between yerba mate samples.

The Iris system was able to detect the different qualities of yerba mate batches. The good batches contain more green color and the bad ones darker colors.