



**HAL**  
open science

## French cider characterization by sensory, technological and chemical evaluations

Jean-Michel Le Quéré, François Husson, Catherine M.G.C. Renard, Jo  
Primault

► **To cite this version:**

Jean-Michel Le Quéré, François Husson, Catherine M.G.C. Renard, Jo Primault. French cider characterization by sensory, technological and chemical evaluations. *LWT - Food Science and Technology*, 2006, 39 (9), pp.1033-1044. 10.1016/j.lwt.2006.02.018 . hal-00467413

**HAL Id: hal-00467413**

**<https://hal.science/hal-00467413>**

Submitted on 3 Oct 2023

**HAL** is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.

# French cider characterization by sensory, technological and chemical evaluations

Jean-Michel Le Quéré<sup>a,\*</sup>, François Husson<sup>b</sup>, Catherine M.G.C. Renard<sup>a</sup>, Jo Primault<sup>c</sup>

<sup>a</sup>Unité de Recherches Cidricoles—Biotransformation des Fruits et Légumes, INRA, BP 35327, 35653 Le Rheu, France

<sup>b</sup>Unité de mathématiques appliquées, Agrocampus Rennes, France

<sup>c</sup>CTPC, Station cidricoles, La Rangée Chesnel, 61500 Sées, France

A set of 90 ciders among them 50 “brut” (near dry), 30 “demi-sec” (half dry) and 10 “doux” (sweet) were collected and analysed to get a data base of about 180 variables including processing conditions, sensory descriptors and physicochemical variables. This data set was treated by a multiple factor analysis (MFA) using the sensory and analytical data as active variables whereas the “processing conditions” data were introduced as illustrative variables. Two MFAs were carried out separating the less sweet (“bruts”) from the sweeter (“demi secs” and “doux”) ciders. For both sets, the MFAs showed a strong polarization of the ciders characteristics: fruity/flowery flavours are associated with cooked flavours and opposed to the descriptors “*animal*” (animal), “*sous-bois*” (underwood), “*fond de cuve*” (vat dregs) themselves associated with bitterness and astringency and also “*pomme/cidre*” (cider/apple) flavours. The industrial ciders located on axis 1 near the fruity and cooked odours whereas the ciders of small scale producers were close to the second group of flavours and tastes.

*Keywords:* Alcoholic beverages; Fermented products; Analytical data; Food composition; Multivariate analysis

## 1. Introduction

The differentiation labels that were created these last 10 years show that the cider makers want to segment their market. Because a commercial differentiation is more effective if it is supported by a perceptible difference, the first step for creating such a differentiation may be to estimate what differences can be perceived among the existing ciders both by sensorial and physicochemical analysis. The second step is to find the methods to produce reliably these different products.

From a theoretical point of view, sensorial perceptions are due to the physicochemical composition of ciders. A lot of work in beverages deals with the sensorial effect of the composition especially in wine and beer, some of its conclusions may be extrapolated to ciders.

Fruity flavours are mainly due to hexyl acetate, ethyl caprylate and isoamyl acetate while 2-phenylethyl acetate is both fruity and flowery (Rapp & Mandery, 1986). Fruity

aroma does not mean “coming from the fruit”: most of these compounds are synthesized during fermentation (Williams & Tucknott, 1977), especially by some secondary yeasts that can produce high amount of acetate esters (Inoue et al., 1997; Rojas, Gil, Pinaga, & Manzanares, 2003). Most of the higher alcohols have perception thresholds higher than their concentrations but they are linked with fermentation conditions and can be regarded as indicators. Some compounds like phenylethanol are sensory active having a rose-like flowery aroma. The vinyl and ethyl phenols, when present at high concentrations, are usually considered as off-flavours of alcoholic beverages especially ethyl phenols. They occur during or after fermentation and are produced by bio-transformation of hydroxycinnamic acids (Chatonnet, Dubourdieu, Boidron, & Lavigne, 1993; Chatonnet, Viala, & Dubourdieu, 1997). A particular yeast, *Brettanomyces*, seems to be the main responsible for the production of ethyl phenols via vinyl phenol (Dias, Pereira-da-Silva, Tavares, Malfeito-Ferreira, & Loureiro, 2003).

In cider bitterness and astringency are due to the polyphenols especially procyanidins which are polymers

\*Corresponding author.

E-mail address: Jean-Michel.Lequere@rennes.inra.fr (J.-M. Le Quéré).

of catechins (Lea & Arnold, 1978; Noble, 2002). The degree of polymerization (DP) is the main factor that influence the ratio between the two sensations (Lea & Arnold, 1978; Lea, 1990): small procyanidins (up to DPn 4) are rather bitter and higher (DPn 5–9) are rather astringent but both sensations are usually associated. There is often an interaction (Vidal et al., 2004) with other constituents of the beverage: alcohol and polysaccharides reduce astringency while pH can increase it without changing the bitterness (Kallithraka, Bakker, & Clifford, 1997). Sugars are also known to reduce acidity (Leguerinel, Cleret, Bourgeois, & Mafart, 1987) and bitterness.

To understand and reliably reproduce these effects of composition on sensorial characteristics their causes must be identified. The composition may, in particular, be linked to fruits production and/or processing conditions.

Some of the process conditions clearly can modify the composition of cider. Juice extraction usually begins by pressing and, for some ciders, this first step is completed by water extraction in a reverse-current extractor which will dilute the juice to some extent. French cider may also be partly made by fermenting a dilution of juice concentrate (authorized up to 50% of the total sugar involved) which might bring compounds and/or flavours usually generated by heat treatments.

Juice and cider can also be clarified and one of the clarification treatment consist in adding gelatine (“Collage” in French). This treatment can be used either on the juice before fermentation or on cider before bottling. By removing selectively high DP procyanidins this treatment modifies both the total tannin content and the profile of the residual tannins, and thus may change the composition and the taste (Lea, 1990; Maury, Sarni-Manchado, Lefebvre, Cheynier, & Moutounet, 2001). But the most common clarifying method in French cider making is the keeving process which use endogenous pectin as a clarifying agent: after demethylation (Baron & Drilleau, 1982) of the pectin by an enzyme—the pectinemethylesterase—calcium is added to induce a formation of a calcium pectate gel that includes all particles of the cloudy juice. This gel is then separated by a “natural” flotation due to CO<sub>2</sub> bubbles of the beginning fermentation. This process produces a pectin-free clarified juice (Lea & Drilleau, 2003) with a reduced nitrogen amount and results in slower fermentation and better stabilization.

French cider fermentation is usually conducted without yeast pitching and at temperatures from 5 to 20 °C depending on the season and the equipment of the cider maker. These low temperatures may have a significant effect on the metabolism of yeast strains spontaneously present, and later on the growth of lactic acid bacteria and *Brettanomyces* species. At the beginning of the fermentation most of the small cider makers and farmers proceed to one or more biomass reductions (performed by filtration or centrifugation and usually followed by a new yeast growth) to deplete the cider in nutrients, slow the fermentation and keep residual sugars. When such a nutrient depletion is

achieved, the growth of micro-organisms is very limited and the ciders can be commercialized without pasteurization. But ciders can also be prepared by blending dry ciders with juice, concentrated juice or partly fermented cider. This kind of cider must then be pasteurized. Cider are commercialized as sparkling beverages and two methods are used for carbonating: Farmers usually bottle a cider that contains enough yeasts to produce up to 5 g/l CO<sub>2</sub> in the bottle whereas most of the other cider makers dissolve exogenous carbonic gas in the cider before bottling. Oxygen can be introduced during bottling (unpublished results) and can then oxidize some compounds and modify the flavour. This can be at least partially reversed by oxygen consumption by yeasts when present in bottle (unpasteurized cider) or by addition of antioxidant (SO<sub>2</sub> and ascorbic acid).

With so many different options possible, cider can be expected to be quite variable even when starting from the same apples. The relations between processing method, composition and sensorial perception are not easy to establish but the three kinds of data must be observed together to characterize the diversity of ciders. Such a data set may be also useful to know the actual composition of French ciders and then to observe the correlation between the different data as a first approach to a better causality understanding.

This data set was analysed by a multiple factor analysis (MFA) (Escofier & Pages, 1998) to extract the main global information and to determine the main associations that can be observed between the different groups of variables (sensorial, physicochemical and process conditions).

## 2. Material and method

### 2.1. Sample collection

To reach the fixed objectives the chosen ciders had to be as diverse as possible to describe the French cider diversity rather than to be representative of the production volumes. We asked a panel of 100 cider producers the processing conditions for 291 ciders. This was submitted to cluster analysis to constitute groups of ciders that could be considered as homogeneous for their process conditions. Ciders were chosen (Table 1) within those groups but without proportionality: as the smaller groups were also sampled these groups are over represented.

Table 1  
Sampled ciders

Production scale	Kind of cider			Total
	Brut	Demi sec	Doux	
A (50–500 hl)	21	16	3	40
B (500–10,000 hl)	8	12	1	21
C (> 80,000 hl)	21	2	6	29
Total	50	30	10	90

## 2.2. Processing conditions

They were collected by asking the producer to fill a form in which we had identified the process step that are known to modify the sensory perception or the physicochemical composition or which are indicators of a particular process method even if their effect is not known. The production scale (three classes: Farmer, Small cider maker, Industrial) was determined afterward on the basis of the volume of produced cider. Table 2 summarizes the variables that were asked and the possible answers.

## 2.3. Sensory analysis

In this study the sensory analysis excluded hedonic and visual appreciation. The 14 panellists were used to taste different foodstuffs and had been trained for ciders and cider apple juice evaluation (according to NF ISO 8586-2). Before this study, 10 specific training sessions were performed by tasting commercial ciders in order to generate sensory descriptors (listed in Table 3) and to get the best agreement between the assessors for these descriptors. The terms given in Table 3 are translations of these descriptors, some of them being difficult to translate: we use the term “*underwood*” for the French “*sous-bois*” to describe the humid, mouldy smell of the soil of a forest covered with decaying leaves. The same descriptors were used both for odour and flavour. Tests were performed in individual booths under red light; ciders were served at 10–12 °C in dark glasses. The assessors were asked to score the selected sensory descriptors before tasting (odour descriptors) and after tasting (flavour descriptors), by means of a linear scale from 1 to 10. Not detected attributes were scored as 0.

The ciders were kept at –5 °C from their sampling to their tasting. Designs to balance the effect of order of presentation and first-order carry-over effects were used throughout the sessions (Mc Fie, Bratchell, Greenhoff, & Valis, 1989). This allows avoiding confusion between session, assessor, product and presentation rank effects. The panel evaluated each sample in duplicate. Five ciders were tasted in each session; the experiment was performed in 36 sessions during 6 months.

## 2.4. Physicochemical data

Table 4 indicate the physicochemical variables which were used in the statistical treatment.

The different polyphenolic compounds are determined by HPLC analysis after thiolysis according to Guyot, Marnet, Sanoner, and Drilleau (2001). The degree of polymerization of flavan-3ols is determined by the ratio between extension units and terminal units.

Volatile compounds were analysed using GC with flame ionization detection. They are extracted by a method derived from (Hubert, Brunerie, Le Quéré, & Drilleau, 1990) using “Chemelut” column (Grosseron) instead of

“Extrelut” column (Merck) for solvent extraction. Injections are made in split mode ( $\frac{1}{20}$ ) on a capillary column of 50 m, 0.25 i.d. coated with a 0.20 µm film of polyethyleneglycol (CP-WAX 57-CB—Chrompack Varian). Oven temperature program: 35 °C (5 min); 35→55 °C (3°/min); 55 °C (3 min); 55→105 °C (4°/min); 105→120 (2°/min); 120→200 (7°/min); 200 (12 min); 200→225 (1.5°/min). Helium is used as carrier gas at 2 ml/min.

Methanol and ethanol are determined by GC after distillation according to OIV (1994).

Sugars (glucose, fructose and sucrose), glycerol and sorbitol, L-malic, L-lactic, D-lactic and citric acid are determined by enzymatic assays of Boehringer Mannheim/R-Biopharm (R-Biopharm AG Landwehrstr. 54 D-64293 Darmstadt) according to producer specifications.

The colour parameters—Hue, Value and Chroma—are measured by a Minolta spectrophotometer CM 3600d (Minolta Co. Ltd, 3-13, 2-Chome, Azuchi-Machi, Chuo-Ku, Osaka 541, Japan) on centrifuged (7500g 15 min) and filtrated (Analytapore Labosi—cellulose ester membrane—0.45 µm) cider. As some ciders darken when treated, the parameters are measured on both oxidized (aerated for 1 h) and reduced cider (by adding 0.4 g/l of sodium dithionite before measure). Browning capacity of the ciders is evaluated by the geometrical distance between the colour parameters of oxidized and reduced ciders. Turbidity is measured by WTW Turb-550 IR according to producer specifications.

Total residual nitrogen is analysed by Hach method (Hach, Brayton, & Kopelove, 1985) for digestion and Berthelot/salicylate method for ammonia determination.

Biochemical analysis usually determined on wine and cider are determined according to (Afnor, 1989): volatile acidity by distillation and titration, total acidity by titration, alcohol by distillation and density measurement. Density of ciders and distillate was measured by a PAAR densimeter DMA 58 (Anton-Paar-Str. 20 A-8054 Graz, Austria) according to producer specifications.

Sulfates are determined gravimetrically according to NF V76-109 and total bisulfites by a sequential analyser using 5, 5'-dithio-bis-(2-nitrobenzoic acid) known as Ellman's reagent.

Minerals were determined by atomic absorption. Among them only Ca and Cu were used in statistical treatment. Samples were mineralized and atomized in the flame of the atomic absorption spectrometer (Varian SctrAA 220 FS) with modifiers for Ca. Concentrations were derived from the measures at the adapted wave length using external standards calibration curve.

## 2.5. Statistical analysis

The final data base is made of three groups of variables: The first group contains the sensory descriptors (among them 21 flavour, 21 odour, 5 taste descriptors). The second group is constituted by most of physicochemical variables (53 of the volatile compounds and 34 of the other

Table 2

Answers of cider makers for the processing conditions of the sampled ciders

Classes	Variables	Code
<b>Row material (RM)</b>		
<i>Main kind of varieties</i>		
	Local varieties	RM-3.11
	Mixed varieties	RM-3.12
	Widespread varieties	RM-3.13
<i>Use of dessert apple</i>		
	With dessert apple	RM-3.21
	Without dessert apple	RM-3.22
<i>Kind of orchards</i>		
	Small trees	RM-3.31
	Mixed production	RM-3.32
	High trees	RM-3.33
<i>Harvest method</i>		
	Manually harvested	RM-3.41
	Mechanically harvested	RM-3.42
	Mixed harvest	RM-3.43
<i>Fruits production area</i>		
	“Basse Normandie”	RM-3.51
	“Bretagne”	RM-3.52
	“Haute Normandie”	RM-3.53
	Non specific region	RM-3.54
<i>Micro-region</i>		
	Open list	RM-3.61
	...	To
	...	RM-3.91
<b>Juice préparation (JP)</b>		
<i>Pulp maceration</i>		
	With pulp maceration	JP-4.11
	Without pulp maceration	JP-4.12
<i>Pressing</i>		
	Traditional press	JP-4.21
	Bucher press	JP-4.22
	Belt press	JP-4.23
	Pneumatic press	JP-4.24
<i>Water extraction of pomace</i>		
	Water extraction	JP-4.31
	No water extraction	JP-4.32
<i>Use of sulfites in juices</i>		
	No sulfites in juices	JP-4.41
	Less than 75 mg SO <sub>2</sub> /L in juices	JP-4.42
	More than 75 mg SO <sub>2</sub> /L in juices	JP-4.43
<i>Use of keeving</i>		
	Keeving	JP-4.51
	With or without keeving	JP-4.52
	No keeving	JP-4.53
<i>Clarification with gelatine</i>		
	Juice clarified by gelatine	JP-4.61
	Juice not clarified by gelatine	JP-4.62
<i>Other Juices clarifications</i>		
	Centrifugation	JP-4.71
	Centrifugation and filtration	JP-4.72
	Cooling and centrifugation	JP-4.73
	No other clarification	JP-4.74
<i>Use of concentrated juice</i>		
	With concentrated juice	JP-4.81
	Without concentrated juice	JP-4.82
<b>Fermentation (Fm)</b>		
<i>Biomass reduction</i>		
	Biom. Reduction by filtration	Fm-5.11
	Biom. Reduction by centrifugation	Fm-5.12
	No biom. Reduction	Fm-5.13
	No answer for biom. Reduction	Fm-5.14
<i>Fermentation temperature control</i>		
	Temperature controlled	Fm-5.21

Table 2 (continued)

Classes	Variables	Code
	Temperature not controlled	Fm-5.22
<i>Use of sulfites in cider</i>		
	No sulfites in cider	Fm-5.31
	Less than 75 mg SO <sub>2</sub> /L in cider	Fm-5.32
	More than 75 mg SO <sub>2</sub> /L in cider	Fm-5.33
	No answer for sulfites in cider	Fm-5.34
<b>Sampling (SP)</b>		
<i>Date of sampling</i>		
	Sampled in June	SP-2.11
	Sampled in September	SP-2.12
<b>Cider preparation and finning (CP)</b>		
<i>Blending of ciders</i>		
	Ciders blending	CP-6.41
	No ciders blending	CP-6.42
	No answer for of ciders blending	CP-6.43
<i>Sweetening cider by concentrated juice</i>		
	Cider sweetened by concentrated juice	CP-6.51
	Cider not sweetened by concentrated juice	CP-6.52
<i>Gelatine clarification of ciders</i>		
	Cider clarified by gelatine	CP-6.61
	Cider not clarified by gelatine	CP-6.62
<i>Filtration before bottling</i>		
	Fine filtration of ciders	CP-6.71
	Coarse filtration of ciders	CP-6.72
	No ciders filtration	CP-6.73
	No answer for ciders filtration	CP-6.74
<i>Kind of bottle</i>		
	Glass bottled	CP-6.81
	PET bottled	CP-6.82
<i>Pasteurisation</i>		
	Not pasteurised	CP-6.91
	Pasteurisation in bottle	CP-6.92
<b>Cider categories (CC)</b>		
<i>Production scale</i>		
	Farmer (50–500 hl)	CC-1.11
	Small cider maker (500–10,000 hl)	CC-1.12
	Industrial (> 80,000 hl)	CC-1.13
<i>Kind of cider</i>		
	“Brut” (near dry)	CC-1.21
	“Demi sec” (half dry)	CC-1.22
	“Doux” (sweet)	CC-1.23
<i>Assertions</i>		
	“Bio” (organic farming)	CC-1.31
	“Label Rouge” (quality label)	CC-1.32
	No assertions	CC-1.33
<i>Appellations</i>		
	“AOC” (certified origine)	CC-1.41
	“IGP Bretagne” (from Brittany)	CC-1.42
	“IGP Normandie” (from Normandie)	CC-1.43
	No appellations	CC-1.44
<b>Carbonation (CB)</b>		
<i>Kind of carbonation</i>		
	Carbonation by yeast in bottle	CB-7.11
	Carbonation by yeast in tank	CB-7.12
	Carbonation by exogenous CO <sub>2</sub>	CB-7.13
<i>Use of dry yeast</i>		
	Dry yeast added for carbonation	CB-7.21
	No dry yeast added for carbonation	CB-7.22
	No answer for yeast adding	CB-7.23
	No matter for yeast addition	CB-7.24
<i>Carbonation temperature control</i>		
	Carbonation temperature controlled	CB-7.31
	No carbonation temperature control	CB-7.32
	No answer for temperature control	CB-7.33
	No matter for carbonation	CB-7.34

Table 3  
Sensory descriptors used to characterize the ciders

Class	Variables	Code
Flowery/fruity flavours (Fff)		
	Flowery A	Fff-1a
	Flowery O	Fff-1o
	Fruity A	Fff-2a
	Fruity O	Fff-2o
	Exotic fruits A	Fff-3a
	Exotic fruits O	Fff-3o
	Cider apple A	Fff-4a
	Cider apple O	Fff-4o
	Apple fruit A	Fff-5a
	Apple fruit O	Fff-5o
	Vanilla A	Fff-6a
	Vanilla O	Fff-6o
Heavy flavours (Hvf)		
	Animal A	Hvf-1a
	Animal O	Hvf-1o
	Woody A	Hvf-2a
	Woody O	Hvf-2o
	Vat dregs A	Hvf-3a
	Vat dregs O	Hvf-3o
	Medicinal/chemical A	Hvf-4a
	Medicinal/chemical O	Hvf-4o
	Underwood A	Hvf-5a
	Underwood O	Hvf-5o
Tastes (Tst)		
	Acid T	Tst-1
	Bitter T	Tst-2
	Astringent T	Tst-3
	Sweet T	Tst-4
	Texture T	Tst-5
Heated flavours (Hf)		
	Caramel A	Hf-1a
	Caramel O	Hf-1o
	Cooked apple A	Hf-2a
	Cooked apple O	Hf-2o
Vegetal flavours (Vf)		
	Straw/Hay A	Vf-1a
	Straw/Hay O	Vf-1o
	Vegetal A	Vf-2a
	Vegetal O	Vf-2o
Fermented flavours (Ff)		
	Butter A	Ff-1a
	Butter O	Ff-1o
	Mushroom A	Ff-2a
	Mushroom O	Ff-2o
	Yeasty A	Ff-3a
	Yeasty O	Ff-3o
Vinegar/solvent flavours (Vsf)		
	Calvados/brandy A	Vsf-1a
	Calvados/brandy O	Vsf-1o
	Solvent A	Vsf-2a
	Solvent O	Vsf-2o
	Vinegar A	Vsf-3a
	Vinegar O	Vsf-3o

biochemical compounds). Some variables are calculated variables (total sugars, the advancement of malolactic fermentation which is the percentage of malic acid

transformed in lactic acid) and some variables that were initially included in the data base were eliminated because of their low variability (especially mineral compounds and minor phenols). The third group is constituted by the “processing conditions” (31 qualitative variables that include raw material, juice preparation, fermentation, cider preparation and finning, carbonation, cider category and date of sampling).

In order to simultaneously analyse sensory and physicochemical variables, we apply MFA on the data set which merges sensory and physicochemical variables. MFA allows to balance the role of the two sets of variables in a global analysis and gives visualization tools and specific indices of the grouping structure. Moreover, since MFA balances the data sets, it allows to simultaneously study quantitative and qualitative data sets: each quantitative data set is studied as PCA and each qualitative data set is studied as multiple correspondence analysis. A more complete description can be found in several papers devoted to MFA (Escofier & Pages, 1998; Pagès & Husson, 2001, 2005).

The first two groups (the sensory and the physicochemical) form the active variables of the MFA and the third group (the “processing conditions”), is introduced in MFA as illustrative data.

### 3. Results and discussion

Two MFA were performed on two separate sets of ciders: set A contains the ciders called “bruts” (less than 28 g/l of residual sugar) and set B gathers the two others categories “demi-sec” (between 28 and 42 g/l of residual sugar) and “doux” (less than 3% vol. alcohol and more than 35 g/l of residual sugar).

For the set A of 50 ciders “bruts”, factors 1 and 2 represent, respectively, 23.3% and 9.5% of the inertia. For the set B of 40 “demi-secs” and “doux” factors 1 and 2 represent, respectively, 25.4% and 11.9% of the inertia. These inertia can be regarded as high with such a number of variables. For both sets the next dimensions represent less than 6% and will not be used to describe our data.

Each variable was given a code (Tables 2–4) which is used on MFA figures. For the variables the more correlated with the axis 1 and 2 (distance from centre of the figure greater than 0.5) the complete names are written, except for volatile compounds (Fig. 2), too numerous to give the complete name. As it is difficult to read all the variables on the same graph, the active variables were divided in different categories to examine them sequentially. The maps result from the MFAs carried out with all the data; the only difference between the figures is which data are shown. Sensory data will be examined first on one figure (Fig. 1) and physicochemical variables are projected on two figures (Figs. 2 and 3). For the same reasons the illustrative data are projected on three figures (Figs. 4–6).

Table 4  
Physicochemical data analysed on the sets of ciders

Class	Variables	Code
Acetate esters (Ae)		
	Amyle acetate	Ae-1
	2-Phenylethyle acetate	Ae-2
	<i>cis</i> -3-Hexenyle acetate	Ae-3
	Hexyle acetate	Ae-4
	Isoamyl acetate	Ae-5
Fatty acid esters (Fae)		
	Ethyl 2-hydroxycaproate	Fae-1
	Ethyl 2-methylbutyrate	Fae-2
	Ethyl 3-hydroxybutyrate	Fae-3
	Ethyl butyrate	Fae-4
	Ethyl caprate	Fae-5
	Isoamyle caprate	Fae-6
	Isobutyle caprate	Fae-7
	Ethyl caproate	Fae-8
	Ethyl caprylate	Fae-9
	Isoamyle caprylate	Fae-10
	Ethyl isobutyrate	Fae-11
	Ethyl isovalerate	Fae-12
	Ethyl laurate	Fae-13
	Isoamyle laurate	Fae-14
	Ethyl myristate	Fae-15
	Ethyl oleate	Fae-16
	Ethyl palmitate	Fae-17
	Ethyl propionate	Fae-18
	Ethyl stearate	Fae-19
Other esters (Oe)		
	Ethyl formiate	Oe-1
	Ethyl furoate	Oe-2
	Ethyl lactate	Oe-3
Other odourous volatile compounds (Ooc)		
	Acetoïne	Ooc-1
	Diacetyl	Ooc-2
	Methionol	Ooc-3
	Phenylethanol	Ooc-4
Other volatile compounds (Ovc)		
	1-Butanol	Ovc-1
	1-Octen-3-ol	Ovc-2
	1-Octen-3-one	Ovc-3
	2,3-Butanediol	Ovc-4
	2-Methyl butanol-1	Ovc-5
	Isobutyrique Acide	Ovc-6
	Allylique alcohol	Ovc-7
	Amylique alcohol	Ovc-8
	Benzylque alcohol	Ovc-9
	Isoamylique alcohol	Ovc-10
	Total high alcohols	Ovc-11
	Butanol-2	Ovc-12
	Butyrolactone	Ovc-13
	<i>cis</i> -3-Hexenol	Ovc-14
	Diethyl succinate	Ovc-15
	Hexanol	Ovc-16
	Isobutanol	Ovc-17
	Methanol	Ovc-18
	Propanol	Ovc-19
Identified alteration indicator (Iai)		
	4-Ethylgâïacol	Iai-1
	4-Vinylgâïacol	Iai-2
	4-Vinylphenol	Iai-3
	Volatile acidity	Iai-4

Table 4 (continued)

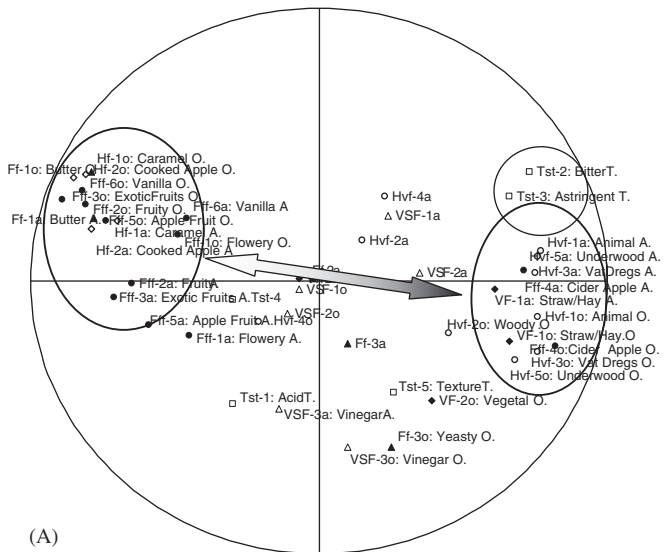
Class	Variables	Code
	D-lactate	Iai-5
	Ethanal	Iai-6
	<i>p</i> -Ethylphenol	Iai-7
Other biochemical variables (Obv)		
	Copper	Obv-1
	Dihydrochalcones	Obv-2
	Flavonols	Obv-3
Taste effect variables (Tev)		
	Acidity	Tev-1
	Measured alcohol	Tev-2
	DP-Flavan-3-ols	Tev-3
	Glycerin	Tev-4
	Initial L-malate	Tev-5
	Density	Tev-6
	PH	Tev-7
	Procyanidins	Tev-8
	Sorbitol	Tev-9
	Malolactic advancement	Tev-10
	Total sugars	Tev-11
Technological choice indicators (Tci)		
	Total alcohol	Tci-1
	Total nitrogen	Tci-2
	Calcium	Tci-3
	Disulphite	Tci-4
	Furfural	Tci-5
	Sulphates	Tci-7
Visual effect variable (Vev)		
	Caffeic acid	Vev-1
	Caffeoylquinic acid	Vev-2
	Ferulic acide	Vev-3
	<i>p</i> -Coumaric acid	Vev-4
	<i>p</i> -Coumaroylquinique acid	Vev-5
	Browning capacity	Vev-6
	Catechines	Vev-7
	Value	Vev-8
	Chroma	Vev-9
	Hue	Vev-10

### 3.1. Sensory descriptors

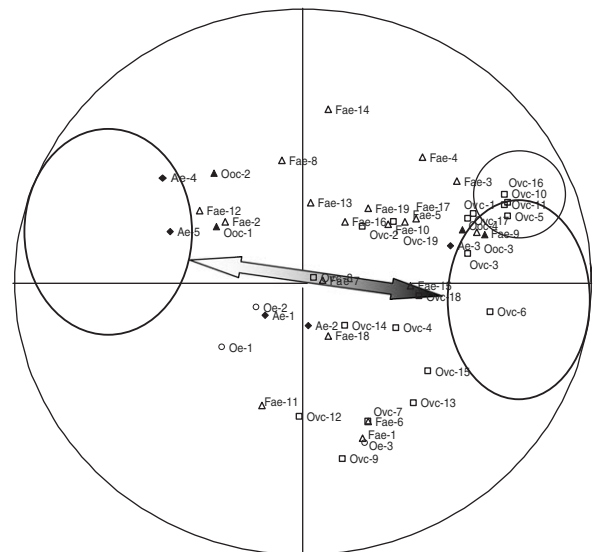
Sensory descriptors were classified into seven categories: the two main categories are fruity/floweryflavours (Fff) and a group of descriptors we called heavy flavours (Hvf) according to (Chatonnet et al., 1993). Two other groups can be easily formed by the taste/mouthfeel descriptors (Tst) and the “heated” flavours (Hf). Although the other flavours are more difficult to classify they were arranged in three categories: Vegetal (Vf), Fermented (Ff) and Vinegar/solvent flavours (Vsf).

Fig. 1(A) and (B) presents sensory data. It can be noted first that the paired descriptors (similar descriptors for odour and flavour) usually show coherent positions on axis 1 but often with an offset on axis 2.

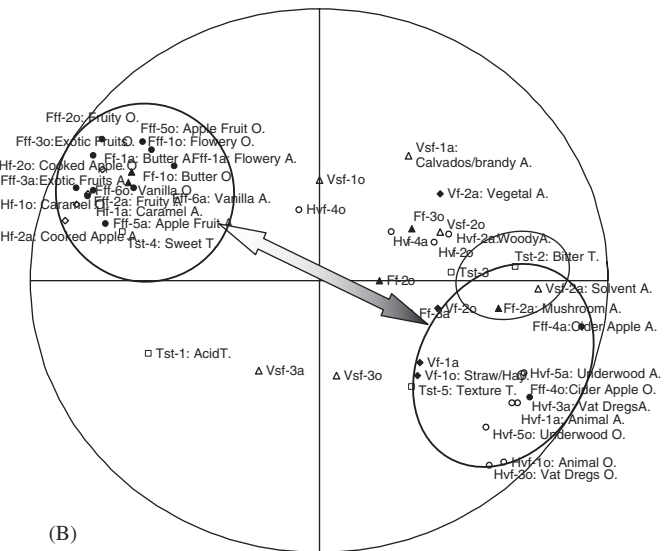
There is a strong polarization of sensory characteristics for both sets of ciders: on the left of the graphs (negatively correlated to factor 1) we find the fruity and flowery flavours such as “fruity”, “apple/fruit”, “exotic fruits”,



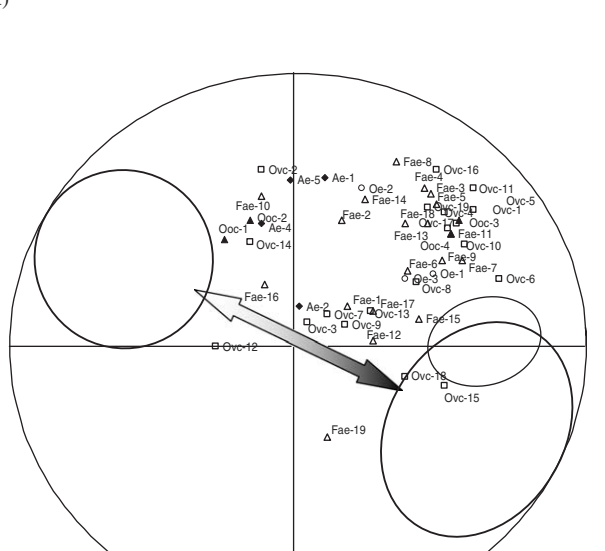
(A)



(A)



(B)



(B)

Fig. 1. Projection of sensorial data for A and B set of ciders on the two first factors. The ellipses are manually drawn and show the positions of the main groups of sensory characteristics (□ Tastes (Tst)—○ Heavy flavours (Hvf)—● Flowery/fruity flavours (Fff)—◇ Heated flavours (Hf)—◆ Vegetal flavours (Vf)—▲ Fermented flavours (Ff)—△ Vinegar/solvent flavours (Vsf)).

Fig. 2. Projection of volatile compounds on the two first factors for sets A and B (◆ Acetate esters (Ae)—△ Fatty acid esters (Fae)—○ Other esters (Oe)—▲ Other odorous volatile compounds (Ooc)—□ Other volatile compounds (Ovc)).

“vanilla”, “flowery”. However these fruity flavours are associated with some other terms like “cooked apple”, “caramel” that reminds us that ciders can be heated and also with “butter”, a descriptor that is common in fermented beverages. These first groups of flavours are opposed to “animal”, “vat dregs”, “underwood”, “woody” that can be called “heavy flavours”. However, this group of variables, positively correlated with factor 1, is also associated with “cider apple” that can be considered as a specific fruity flavour of cider. The two sets of ciders show very similar results concerning axis 1. However the flavour opposition is more involved in the definition of axis 2 for set B.

Bitter and astringent tastes present also a positive correlation with factor 1. These tastes are situated near the heavy flavour indicating that the probability to find these flavours is higher in bitter ciders.

### 3.2. Physicochemical data

Fig. 2(A) and (B) shows the projection of the volatile compounds. Among them, the esters are often considered as responsible of the fruity flavour and might explain the observed sensorial polarization. Our data are not exactly what could be expected: some of the esters known for their fruit-like flavour, such as isoamyl acetate, hexyl acetate and



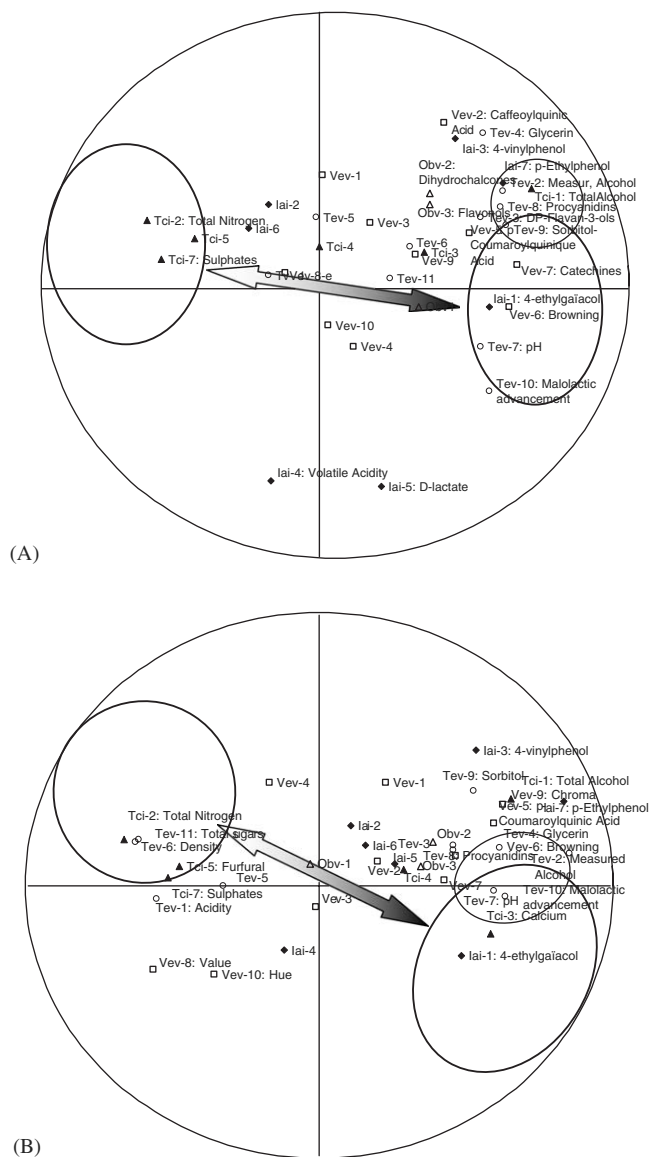


Fig. 3. Projection of different physicochemical compounds on the two first factors for sets A and B (◆ Identified alteration indicator (Iai)—△ Other biochemical variables (Obv)—○ Taste effect variables (Tev)—▲ Technological choice indicators (Tci)—□ Visual effect variable (Vev)).

ethyl isovalerate, (Fig. 2A) present a slight negative correlation with factor 1 that places them on the left of the graph with the fruity flavours. However, most of the esters are not on the left part of axis 1 and the circle that reminds of the fruity descriptors remains empty especially in Fig. 2B: on the contrary, a lot of the esters and of the other volatile compounds such as phenylethanol known for its flowery flavour are positively correlated with factor 1. They also can present a positive or negative correlation with factor 2 that place them on top or at the bottom of the graph. Among volatile compounds one of the sulfur compounds, methionol (Ooc in Fig. 2), is also positively correlated with factor 1.

Some other sensory variables can also be associated with their related analytical data: heavy flavours are close to

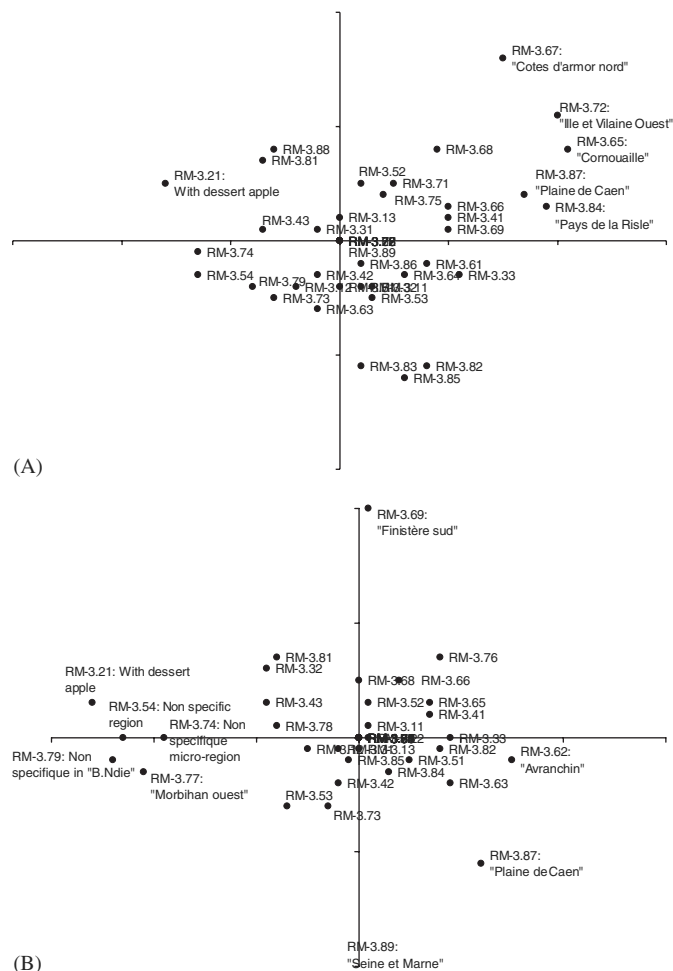


Fig. 4. Projection of apple production conditions as illustrative variables on the two first factors for sets A and B (● Raw material (RM)).

volatile phenolic compounds (Fig. 3) especially 4-ethylgalaicol. Another volatile phenolic compound, *p*-ethylphenol, is also positively correlated with factor 1 but also with factor 2. It can be remarked that methionol is situated near the volatile phenols. This could be linked to a confusion between its flavour and those of volatile phenols, so that it might contribute to “heavy” flavours.

For bitter and astringent tastes there is also a logical association with polyphenols especially with procyanidins and their degree of polymerization (DP): procyanidins of apple are known to be both bitter and astringent (Lea, 1990). Most of the phenolic compounds are correlated with axis 1 and are strongly correlated together. This correlation between all the phenolic compounds is usually observed in apple juice composition, i.e. apple juices which are high in polyphenols are high in all polyphenols with only minor variations of the proportions of the various classes. This probably explains why volatile phenols/heavy odour are associated with procyanidols/bitter-astringent: hydroxycinnamic acids, the substrates of volatile phenols synthesis are also statistically higher in bitter ciders.

Some technological consequences (Fig. 3) can also be correlated with factor 1: residual nitrogen and furfural are

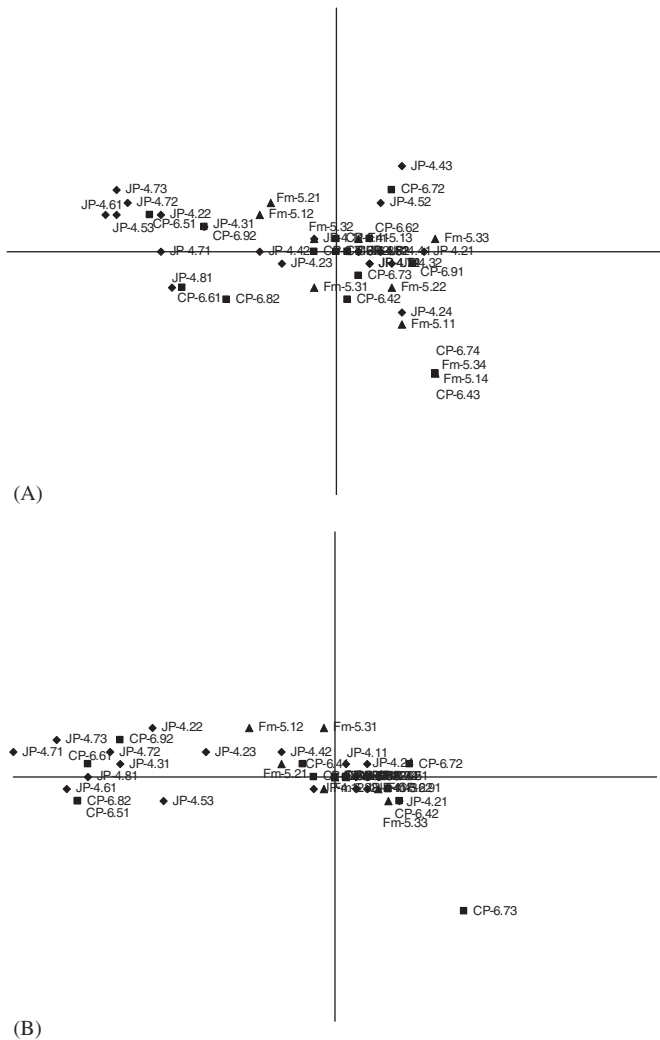


Fig. 5. Projection of juice preparation, fermentation and cider preparation/finning conditions as illustrative variables on the two first factors for sets A and B. Signification of the codes are listed in Table 2 (◆ Juice preparation (JP)—▲ Fermentation (Fm)—■ Cider preparation and finning (CP)).

situated on the left side of the graph close to the sensory descriptors “cooked apple” and “caramel”. This can be explained by the method used to obtain stability. French ciders contain enough residual sugars for bacteria or yeast growth after bottling. The most definitive solution (used for most of industrial ciders) to prevent these damages is to pasteurize the bottled cider between 50 and 60 °C and the second method (mainly used at small scale), consists in exhausting the nutrients, especially nitrogen, by several cycles of yeast growth. The presence of residual nitrogen thus indicates that the first solution was necessarily adopted, and it is therefore logical to find furfural, which is generated during heat treatments from sugars, near residual nitrogen. The sensory descriptors “cooked apple” and “caramel” might be a sensory result of this treatment.

Sulfates are also present in the same place and can be explained by an oxidation of sulfites during cider

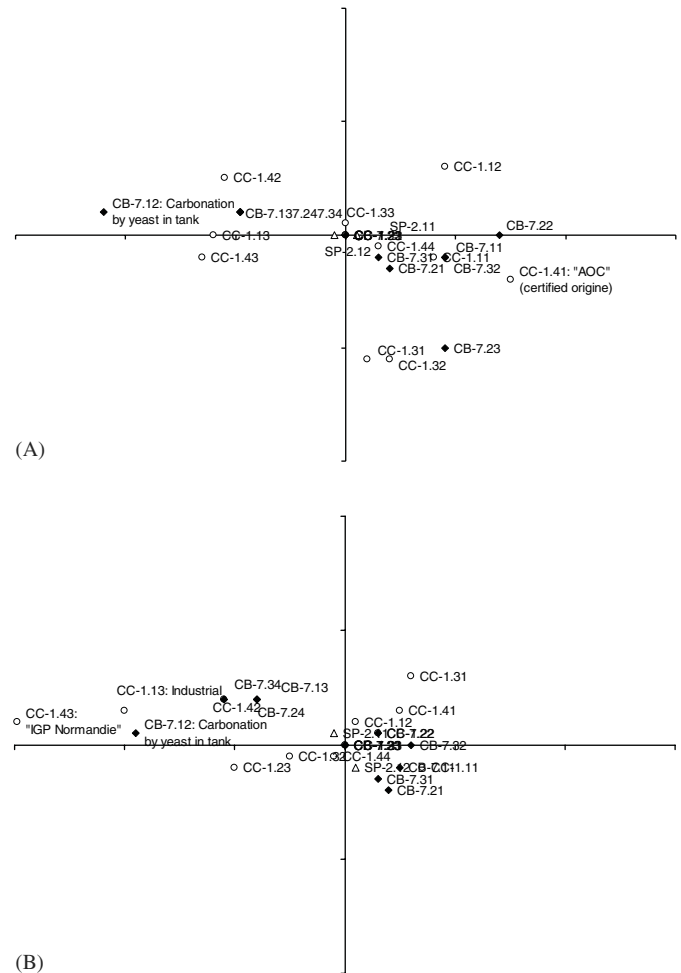


Fig. 6. Projection of cider categories, cider sampling and carbonation conditions as illustrative variables on the two first factors for sets A and B (△ Sampling (SP)—○ Cider categories (CC)—◆ Carbonation (CB)).

processing. This reaction will take place when cider is left for a long period without fermentation or after bottling.

On the other side of the graph the variable “total alcohol”, often used by French cider makers, is correlated with factor 1. This variable is calculated as the addition of analysed alcohol and the potential alcohol resulting from calculated conversion of residual sugars (17g/l for 1% vol.). The position of this calculated variable on the right part indicates that this side concerns ciders that are made from juices richest in sugars, i.e. not diluted juice.

### 3.3. Processing conditions

The processing conditions are projected on the first two axes (Figs. 4–6) as illustrative variables.

The fruit production conditions (Fig. 4) include the type of apples, the type of orchard and harvest but most of the terms that appear on the graph concern the production area—region and micro-region—because answer to this question was an open list. On the left of the graph

the answers “with dessert apple”, “non-specific region”, “non-specific micro-region” are the most negatively correlated with factor 1. These answers show that the production area cannot be defined with such a precision for the left side ciders, which is a characteristic associated with industrial ciders. On the right side there are a lot of micro regions. Such a precise origin (and manual harvesting methods) can only be at small scales.

Among the methods of juice preparation and cider fermentation and fining (Fig. 5) located on the left of axis 1 we can remark the clarifications by gelatine treatment of juices and ciders, and the use of concentrated juice both in juice and in cider. These technological choices are usually specific of industrial scale.

The terms related to carbonation and ciders categories are less correlated with the two axis: the term “carbonation in tank” is used by industrial and small cider makers but only three ciders were concerned. For the commercial differentiation terms, terms such as “IGP Normandie” (Fig. 6B) that is only used at industrial scale was situated on the left side for set B, while the French origin certification “AOC” which is only used by farmers appeared (Fig. 6A) on the right side for set A only.

As these projections of illustrative variables strongly suggested that the sensory and physicochemical classification may be linked to processing scale, we examined the occurrence of the main processing conditions in the different scales of cider. We can see (Table 5) that the smaller production scales are usually characterized by a negative answer for most of possible techniques, carbonation being the main difference between farmers and small cider makers. The other technical operations are mainly used at industrial scale. In this study it is thus impossible to distinguish the role of each of these “industrial” operations on the polarization shown by FMA.

### 3.4. Ciders positions

The projection of the ciders on the graph defined by the two first dimensions (Fig. 7) confirms the above observations: the left side concern industrial ciders and the right the ciders produced by small cider makers and farmers. The ellipses are only drawn to indicate each cider maker family without any statistical signification. The center of the graph presents an overlapping of the three scale of ciders makers. Because the processing methods are not homogeneously used by the different families of cider makers, it is not possible to draw any hypothesis from this work concerning the effect of these different methods. The link between processing scale and the sensorial and analytical classification is the only conclusion that can be drawn. As the contribution of sensorial and physicochemical data are balanced for the first dimension in MFA, we can say that the industrial ciders present both a different sensorial perception and a different composition from the two types of small scale ciders; these two last categories remaining undifferentiated. As this observation was done

Table 5  
Main technological operations in cider process that might differentiate ciders, and their representation in the sets of collected ciders

Water extraction	Main technological operations					Kind of cider makers			
	Use of concentrated juices at fermentation step	Gelatin treatment of juice	Gelatin treatment of cider	Use of concentrated juices in cider blending	Pasteurisation	Carbonation	Farmer	Small cider maker	Industrial
Yes	Yes	Yes	Yes	No	Yes	Exogenous CO <sub>2</sub>			1
Yes	Yes	Yes	No	No	Yes	Exogenous CO <sub>2</sub>			1
Yes	Yes	No	No	No	Yes	Exogenous CO <sub>2</sub>			3
Yes	Yes	No	No	No	Yes	Exogenous CO <sub>2</sub>			3
Yes	No	Yes	Yes	Yes	Yes	Exogenous CO <sub>2</sub>			2
Yes	No	Yes	Yes	No	Yes	Exogenous CO <sub>2</sub>			1
Yes	No	No	Yes	No	Yes	Exogenous CO <sub>2</sub>			1
Yes	No	No	No	No	Yes	Exogenous CO <sub>2</sub>		1	5
Yes	No	No	No	No	No	Exogenous CO <sub>2</sub>			1
No	Yes	Yes	No	No	Yes	Exogenous CO <sub>2</sub>			1
No	No	Yes	No	No	Yes	Fermentation in tank			2
No	No	No	No	No	Yes	Exogenous CO <sub>2</sub>			2
No	No	No	No	No	Yes	Exogenous CO <sub>2</sub>			4
No	No	No	No	No	No	Exogenous CO <sub>2</sub>		10	
No	No	No	No	No	No	Fermentation in tank		1	
No	No	No	No	No	No	Fermentation in bottle	40	9	2

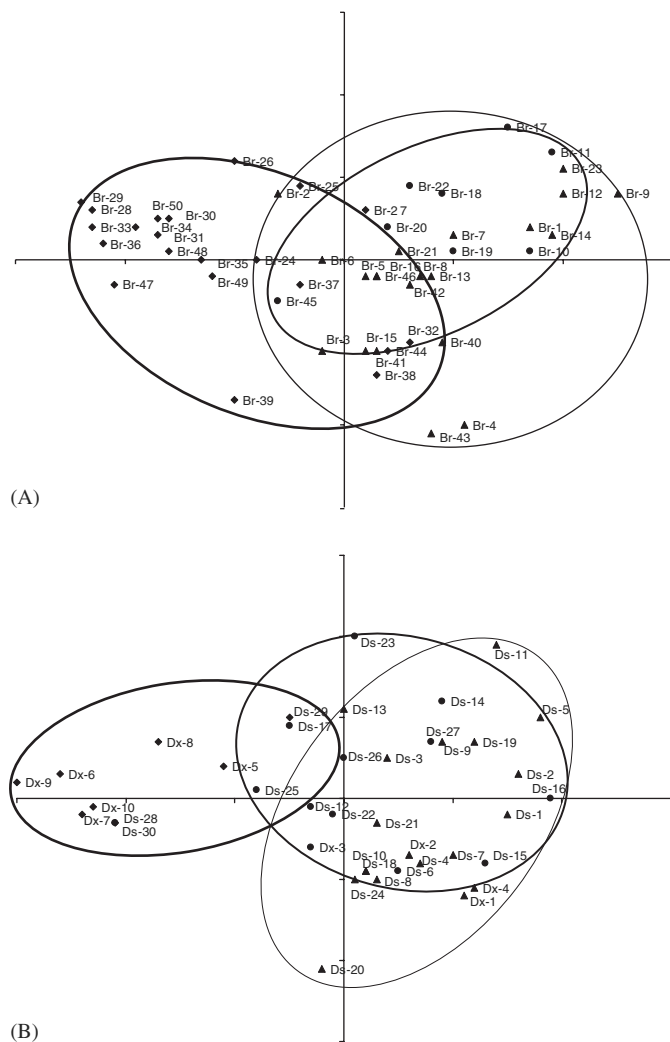


Fig. 7. Projection of the ciders on the two first factors for sets A and B (● Small cider makers—◆ Industry—▲ Farmers).

on both sets of ciders (A “bruts” and B “sweeter”) this result seems to be robust.

### 3.5. Global discussion

The industrial scale ciders could be differentiated from smaller scales ciders both on a sensory and an analytical basis with however a continuous gradation between these two extremes. The first dimension which highlight this differentiation explains enough of the total inertia to be highly significant with such a number of variables.

This differentiation of the ciders can be explained first by a strong polarization of sensory perceptions: two groups of flavours, the “fruity/flowery” flavours and the “heavy flavours”, respectively, present a negative and a positive correlation with factor 1. To this polarization can be added bitter taste, astringent mouthfeel and also a specific fruity flavour “cider apple”, which are associated with the “heavy flavours”. On the other hand some flavours such as “caramel” and “cooked apple” which are usually

attributed to heat treatments, are associated with the “fruity/flowery” flavours.

Among the analytical data some compounds can explain the sensory polarization: procyanidins, which are known to be responsible for bitterness and astringency, and volatile phenols which are considered responsible of some of the “heavy flavours” are actually close to the correspondent descriptors. The presence of furfural can also be related to heat treatments and then to the cooked flavour while high residual nitrogen indicates the necessity of this treatment in the concerned ciders.

The relation between esters and fruity flavour is less clear: isoamyl acetate, hexyl acetate and ethyl isovalerate are slightly (negatively) correlated with factor 1 close to fruity flavour but, factor 1 is also positively correlated with a lot of esters and only one fruity descriptor, “cider apple” is close to these volatile compounds. This suggest that the fruity flavours may be disguised by the presence of “heavy flavours” when these flavours are high enough and that the perception of fruity flavours in the left side ciders is explained more by the absence of “heavy flavours” than by the presence of related compounds.

The specific variable “total alcohol”, often used by cider makers and which gives an indication of the initial sugar concentration of the juice, is also positively correlated with factor 1 indicating that the small scale ciders are not or less diluted than the industrial ciders. The relation between low “total alcohol” and fruity flavours (or between heavy flavours and high “total alcohol”) may appear surprising. In fact, as the technological methods are not independent of the production scale, this higher initial density of the juice cannot be separated from the first differentiation we discuss about: industrial ciders characterized by fruity, flowery, and cooked flavour are also the more diluted because partially made from water-extracted juice whereas small scale ciders, characterized by heavy flavour bitterness and astringency usually are pure juice.

## 4. Conclusions

All these observations show the complexity of the differentiation associated with axis 1: on the industrial side more diluted ciders that are perceived by the panel as fruity but also as heated products. On the other side more “concentrated” ciders that are also more bitter and astringent but with “heavy flavours” that are often considered as off flavours in the other beverages. A possible interaction between these “heavy flavours” and fruity/flowery might be examined to better understand the signification of this polarization.

This work cannot explain the relations between the different variables but it can be used to formulate some hypotheses from their positions. The characteristics which were perceived as different by the assessors were notably the heavy and fruity flavours and also bitterness and astringency. The main observation is that the methods chosen by industry give ciders that are different from those

made by the methods chosen by the smaller scale cider makers for these characteristics. We were limited by the non-independence of the processing conditions. Experimental studies will have to be performed to identify within this succession of choices that constitute each technological path which points efficiently modify these characteristics. However, some of the processing conditions used at industrial scale can be identified to be examined first: finning with gelatine at the two steps where it can be used because of the reduction of phenols contents, heat treatments in relation with nutrients depletion, and fermentation temperature because of the control of microbial alterations.

## Acknowledgements

This work was carried out by INRA and CTPC with support from ONIVINS and Région Bretagne.

Thanks are due to S. Tisso-Le Prêtre, M. D’Haussy, J.P. Simon, Y. Gilles, A. Lepage and industrial units quality managers for collecting data and sample.

Thanks are also due S. Sibiril and H. Guichard from ADRIA Normandie for their implication in sensorial and volatile compounds analysis.

## References

- Afnor (1989). Contrôle de la qualité des produits alimentaires—Méthodes d’analyse officielles, AFNOR Tour Europe Cedex 7, Paris.
- Baron, A., & Drilleau, J.-F. (1982). Utilisation de la pectinestérase dans les industries cidricoles. In P. Dupuy (Eds.), *Utilisation des enzymes en Technologie Alimentaire* (pp. 471–476). Tech. et Doc. Lavoisier ed., Paris.
- Chatonnet, P., Dubourdieu, D., Boidron, J. N., & Lavigne, V. (1993). Synthesis of volatile phenols by *Saccharomyces cerevisiae* in wines. *Journal of the Science of Food and Agriculture*, *62*, 191–202.
- Chatonnet, P., Viala, C., & Dubourdieu, D. (1997). Influence of polyphenolic components of red wines on the microbial synthesis of volatile phenols. *American Journal of Enology and Viticulture*, *48*, 443–448.
- Dias, L., Pereira-da-Silva, S., Tavares, M., Malfeito-Ferreira, M., & Loureiro, V. (2003). Factors affecting the production of 4-ethylphenol by the yeast *Dekkera bruxellensis* in enological conditions. *Food Microbiology*, *20*, 377–384.
- Escofier, B., & Pages, J. (1998). *Analyses factorielles simples et multiples: objectifs, méthodes et interprétation* (3rd ed.). Paris: Dunod.
- Guyot, S., Marnet, N., Sanoner, P., & Drilleau, J. F. (2001). Direct thiolysis on crude apple materials for high-performance liquid chromatography characterization and quantification of polyphenols in cider apple tissues and juices. In L. Packer (Ed.), *Methods in Enzymology—Flavonoïds and other polyphenols*, Vol. 335 (pp. 57–70). New York: Academic Press.
- Hach, C. C., Brayton, S. V., & Kopelove, A. B. (1985). A powerful Kjeldahl nitrogen method using peroxymonosulfuric acid. *Journal of Agricultural and Food Chemistry*, *33*, 1117–1123.
- Hubert, C., Brunerie, P., Le Quéré, J. M., & Drilleau, J. F. (1990). Les composés volatils du cidre: Extraction rapide et dosage. *Sciences des Aliments*, *10*, 603–618.
- Inoue, Y., Trevanichi, S., Fukuda, K., Izawa, S., Wakai, Y., & Kimura, A. (1997). Roles of esterase and alcohol acetyltransferase on production of isoamyl acetate in *Hansenula mrakii*. *Journal of Agricultural and Food Chemistry*, *45*, 644–649.
- Kallithraka, S., Bakker, J., & Clifford, M. N. (1997). Red wine and model wine astringency as affected by malic and lactic acid. *Journal of Food Science*, *62*, 416–420.
- Lea, A. G. H. (1990). Bitterness and astringency: The procyanidins of fermented apple ciders. In L. R. Russell (Ed.), *Bitterness in foods and beverages* (pp. 123–143). Amsterdam, Oxford, New York, Tokyo: Elsevier.
- Lea, A. G., & Arnold, G. M. (1978). The phenolics of ciders: Bitterness and astringency. *Journal of the Science of Food and Agriculture*, *29*, 478–483.
- Lea, A. G. H., & Drilleau, J. F. (2003). Cidermaking. In A.G. Lea, & J.R. Piggott (Eds.), *Fermented beverage production* (2nd ed., pp. 59–84). Dordrecht/New York: Kluwer Academic/Plenum Publishers.
- Legerinel, J. J., Cleret, C. M., Bourgeois, P., & Mafart, I. (1987). Essai d’évaluation des caractéristiques organoleptiques des cidres par analyses instrumentales. *Sciences des Aliments*, *7*, 223–239.
- Maury, C., Sarni-Manchado, P., Lefebvre, S., Cheynier, V., & Moutouret, M. (2001). Influence of fining with different molecular weight gelatins on proanthocyanidin composition and perception of wines. *American Journal of Enology and Viticulture*, *52*, 140–145.
- Mc Fie, H. J., Bratchell, N., Greenhoff, X., & Valis, L. V. (1989). Designs to balance the effect of presentation and first-order carry-over effects in hall tests. *Journal of Sensory Studies*, *4*, 129–148.
- Noble, A. C. (2002). Astringency and bitterness of flavonoid phenols. *ACS Symposium Series/American Chemical Society*, *825*, 192–201.
- OIV (1994). Recueil des méthodes internationales d’analyse des boissons spiritueuses, des alcools et de la fraction aromatique des boissons, OIV (Office International de la Vigne et du vin)—18 Rue d’Aguessseau, Paris.
- Pagès, J., & Husson, F. (2001). Inter-laboratory comparison of sensory profiles: Methodology and results. *Food Quality and Preference*, *297*–309.
- Pagès, J., & Husson, F. (2005). Multiple factor analysis with confidence ellipses: A methodology to study the relationships between sensory and instrumental data. *Journal of Chemometrics*, *19*(3), 138–144.
- Rapp, A., & Mandery, H. (1986). Wine aroma. *Experientia*, *42*, 873–884.
- Rojas, V., Gil, J. V., Pinaga, F., & Manzanares, P. (2003). Acetate ester formation in wine by mixed cultures in laboratory fermentations. *International Journal of Food Microbiology*, *86*, 181–188.
- Vidal, S., Courcoux, P., Francis, L., Kwiatkowski, M., Gawel, R., Williams, P., et al. (2004). Use of an experimental design approach for evaluation of key wine components on mouth-feel perception. *Food Quality and Preference*, *15*, 209–217.
- Williams, A. A., & Tucknott, O. G. (1977). The volatile aroma components of fermented ciders: Minor neutral components from the fermentation of sweet Coppin apple juice. *Journal of the Science of Food and Agriculture*, *29*, 381–397.