



# Prediction of sensory characteristics of cider according to their biochemical composition: Use of a central composite design and external validation by cider professionals

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## ABSTRACT

A large experimental design containing 72 cider-like model solutions was developed in order to evaluate the impact of apple procyanidins, fructose, acid content and ethanol on four sensory attributes: bitterness, astringency, sweetness and sourness. Based on cider composition, this work investigated the linear and quadratic effects of these four quantitative factors and the interactions between them. The results underline the role of procyanidin content (procyanidin profile and concentration) in cider, which highly impacts bitterness and astringency but also affects sweetness and sourness. Using an external validation based on a sensory evaluation by cider professionals, one predictive model for each sensory attribute is proposed.

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## 1. Introduction

Cider is a slightly alcoholic beverage resulting from the fermentation of apple juice. Originally processed in several countries in Europe (France, Ireland, Great Britain, Poland, Latvia and Spain), it is now also produced in Argentina, Australia, and South Africa. The main chemical constituents are water, sugar (principally fructose), organic acids, ethanol and polyphenols (Lea & Drilleau, 2003). Mastering the quality and the regularity of production needs a better understanding of the way cider components, notably procyanidins, interact to construct the final flavor of cider (MAIPROCI, 2011). Although the aromatic dimension should not be forgotten, the cider mouthfeel perception results from interactions between these constituents and their effect on sweetness, sourness, bitterness and astringency.

In cider research, very few works have studied the link between chemical composition and mouthfeel perception. Only Lea and

Arnold (1978), Symoneaux, Baron, Marnet, Bauduin, & Chollet (2014) and Symoneaux, Chollet, Bauduin, Le Quéré, and Baron (2014) have evaluated cider with a focus on the effects of procyanidins on these characteristics, working with model solutions. They identified that small procyanidins, notably tetramers and pentamers, were more bitter. They also noticed that the higher the average degree of polymerization (aDP) was, the more astringent the solutions were. Symoneaux, Baron, et al. (2014) also studied the role of procyanidins in sweetness and sourness, demonstrating that only the concentration of procyanidins (not their aDP) impacts these two characteristics, but in a cider-like model solution with a low acid and sugar content. The effect of the average degree of polymerization (aDP) and its interactions with fructose, malic acid and ethanol content in a range of commercial ciders was the main focus of the work done by Symoneaux, Chollet, et al. (2014). They confirmed the positive impact of aDP on astringency and the role of apple procyanidin pentamers in enhancing bitterness in cider without any significant interactions between the polymerization degree and the other constituents. Nevertheless, these two works were carried out using a half fractional design due to the amount of purified procyanidins available. Thus, although the way

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procyanidins (concentration and aDP) impacted mouthfeel characteristics could be identified, it was not possible to access all interactions satisfactorily, notably those between other constituents (fructose, ethanol and acid), and to generate generic models adapted for predicting the mouthfeel perception of ciders.

In wine, research on the effects of chemical compounds on organoleptic characteristics is extensive (Arnold, Noble, & Singleton, 1980; Brossaud, Cheynier, & Noble, 2001; Fontoin, Saucier, Teissedre, & Glories, 2008; Robichaud & Noble, 1990; Vidal et al., 2004). However, the composition of wine is quite different from cider with more alcohol, different acid characteristics and different types of procyanidins. For these reasons, it is not possible to generalize results from wine to cider.

Moreover, the influences of constituents are most often studied independently and no study has focused on the effects of the interaction of cider components in mixture formulations through experimental designs and modeling.

Besides, in experimental designs, models are generally validated internally by the calculation of  $R^2$  or by evaluating the lack of fit using a variance analysis approach integrating the replicate central points (Droesbeke, Saporta, & Fine, 1997; Myers, Montgomery, & Anderson-Cook, 2009). Nevertheless, in sensory science, the results could be panel-dependent. In an inter-laboratory comparison, Pagès and Husson (2001) indicated that contradictory conclusions could occasionally be observed between several panels performing descriptive tasks. This point relates to the reproducibility of results discussed in the guidelines for monitoring and assessing the overall performance of a quantitative descriptive panel (ISO, 2012). A good way to assess reproducibility is to ask a second panel to evaluate products coming from the same experimental domain and to check if the predicted values are in accordance with the observed data from the second panel.

The aim of this study was to build predictive models for cider-like model solutions integrating the effects of fructose, acid, ethanol and polyphenol content on bitterness, astringency, sweetness and sourness using a central composite design including interactions and quadratic effects. This design enabled the results from the two previous works done with a half fractional design (Symoneaux, Baron, et al., 2014; Symoneaux, Chollet, et al., 2014) to be challenged and the study of the main effects and interactions not investigated previously with the range of concentrations encountered in French commercial ciders to be completed. The predictive models were validated externally using a different panel composed of cider makers tasting another set of experimental products. The use of professionals for this validation should allow a better generalization of the conclusions.

## 2. Materials and methods

### 2.1. Procyanidin extracts

In order to evaluate the impact of procyanidin profile on mouthfeel perception, two fractions of procyanidins were obtained using the following procedure. Five hundred liters of a very bitter and astringent cider containing a high concentration of procyanidins was used for this purpose. Fraction A was directly extracted from 200 L of this cider and Fraction B was extracted from 300 L of this cider previously treated to reduce the high DP procyanidin concentration of the original cider: for this treatment the 300 L cider sample was left for 2 h at 0 °C in contact with apple mash (previously washed with water) using a method derived from previous works (Le Bourvellec, Le Quere, & Renard, 2007).

Then, the polyphenols were extracted from these two samples by adsorption on a food grade column (Amberlite FPX66/Rohm and Haas). After rinsing, the polyphenols were eluted by ethanol 96% vol

and the resulting fractions were then concentrated to remove ethanol and freeze-dried for storage. These extracts were diluted to prepare two mother solutions adjusted to the same procyanidin content. An analysis of the DP profile was done using a method described by Kelm, Johnson, Robbins, Hammerstone, and Schmitz (2006) on a diol stationary phase columns and an UV detector. This method gives a good evaluation of the proportion of each DP but not of the average DP nor the total procyanidins content because of the bad resolution on the higher DP. Total procyanidins and aDP were then quantified following the method described by Guyot, Marnet, Laraba, Sanoner, and Drilleau (1998). As expected the procyanidin profiles of the two fractions obtained were different (Table 1) and the aDP of Fraction A coming from the original cider was significantly higher than that of B due to a higher proportion of procyanidins with a polymerization degree greater than 4.

### 2.2. Experimental design and model solutions

An experimental design with 5 factors (procyanidin fraction, procyanidin concentration, fructose, malic acid (and pH) and ethanol content) was built. The first factor had 2 levels corresponding to the two fractions obtained, A (coded 1) and B (coded –1). The effect of the four other quantitative factors, with five levels each (–2, –1, 0, 1, 2), was arranged following a central composite design. It resulted in seventy-two samples including twelve replicates of the central point for each fraction as recommended by Cliquet, Durier, and Kobilinski (1993) in order to evaluate the direct effects of the five factors and the interactions between them and the quadratic effects of the quantitative ones. These replicates enabled the isovariant by rotation and the orthogonal properties of the design to be conserved.

The concentration ranges were chosen to include the compositions of French commercial ciders (database studied in Le Quéré, Husson, Renard, & Primault, 2006) and were previously studied by Symoneaux, Chollet, et al. (2014): polyphenols (0, 375, 750, 1125, 1500 mg/L), fructose (0, 20, 40, 60, 80 g/L), malic acid (2.43, 3.3, 4.17, 5.04, 5.91 g/L) being linked to pH by adjusting respectively pH to 4.17, 3.94, 3.71, 3.48 and 3.25, and ethanol (1.2, 2.7, 4.2, 5, 7.2% vol.).

For the external validation by the second panel, thirty model solutions were prepared with a 50/50% blend of fractions A and B. They were selected to cover the main variations in bitterness, sweetness and astringency. This validation was performed simultaneously with a training course about mouthfeel modulation in cider.

**Table 1**

Procyanidin content of the two polyphenol fractions after dilution in order to have the same procyanidin content. Procyanidin content and aDP was calculated from the method described by Guyot et al. (1998). The concentration of each DP was calculated using the proportion of each DP obtained by the method developed in Kelm et al. (2006). SD: pooled standard deviation calculated according to Box, Hunter, and Hunter (1978).

Mother solution used				
	DP	Fraction A	Fraction B	SD
Concentration of each DP (mg/L)	2	7.59	9.25	0.04
	3	5.80	6.24	0.03
	4	5.01	4.86	0.04
	5	4.02	3.82	0.04
	6	2.90	2.55	0.02
	7	2.16	1.71	0.05
	8	1.41	0.89	0.03
	9	0.80	0.50	0.04
	10	0.31	0.18	0.02
Procyanidin content (mg/L)		30.00	30.00	
aDP		3.22	2.99	

DP: degree of polymerization; aDP: average degree of polymerization.

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