Effect of a commercial tannin on the sensorial temporality of astringency

M. Medel-Marabolí⁎, J.L. Romero, E. Obreque-Slier, A. Contreras, A. Peña-Neira

Faculty of Agronomic Sciences, University of Chile, Santa Rosa #11315, La Pintana, Santiago, Chile

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ABSTRACT

Astringency is a tactile sensation that is generated by a reduction in lubrication in the oral cavity and is generally attributed to the interaction of proanthocyanidins or condensed tannins with salivary proteins. Several factors influence tannin-protein interactions, such as pH, alcohol, sweetness, oxygen and polyphenol content. A scarcely studied factor is the effect of the tannin content on the perception of astringency. The objective of this study was to evaluate the effect of different concentrations of commercial oenological tannin (COT) on the timing of the perception of astringency. For this model, a vinous solution enriched with three concentrations of COT was used. The samples were subjected to a storage period of three months. Additionally, a panel was trained in the perception of astringency in red wines using a method of temporal dominance of sensations (TDS). Astringency descriptors were selected, and the TDS method was used to characterize the astringency. The samples were evaluated using traditional descriptors in TDS and astringency descriptors in TDS.

In traditional TDS curves, treatments with higher concentrations of COT showed a higher and more persistent dominance index in the descriptor astringency. Moreover, the stimulus duration variable increased as the COT level increased. Likewise, temporary astringency was dominant over alcohol. For astringency TDS, at low concentrations of COT, the soft and adhesive descriptors were dominant, whereas at high COT concentrations, aggressive and drying were perceived as the dominant descriptors. An increasing concentration of tannin in the vinous solution generated an increased duration and dominance of astringency and reduced the duration of the sensation of alcohol. Finally, the type of perceived astringency was closely related to the tannin concentration.

1. Introduction

Astringency is an oral perception characterized by dryness, roughness and wrinkling of the mucosa in the oral cavity (Lee & Lawless, 1991). This tactile sensation is generated by the loss of lubrication (Smith & Noble, 1998) associated with the interaction of tannins and salivary proteins (Baconn & Rhodes, 2000). Several factors are involved in the perception of astringency, such as the ethanol concentration (Obreque-Slier, Peña-Neira, & Lopez-Solis, 2010), pH (Obreque-Slier, Peña-Neira, & López-Solís, 2012), sweetness (Ishikawa & Noble, 1995), oxygen (Waterhouse & Laurie, 2006), and tannin concentrations (Vidal et al., 2004). Regarding polyphenols, Vidal et al. (2004) found that the maximum astringency intensity was significantly correlated with the total content of phenols and catechins. On the other hand, Monteleone, Condelli, Dinnella, and Bertuccioli (2004), Gawel, Francis, and Waters (2007) and Cliff, King, and Schlosser (2007) found similar correlations between perceived astringency and the total phenolic composition of the wines studied. Gawel et al. (2007) established a relationship between different concentrations of phenols and different astringency descriptors, finding that the amounts of total phenols and total tannins are positively related to the rough and abrasive descriptors; therefore, increasing the concentration of these variables would increase the intensity of the descriptors. Cliff et al. (2007), who worked with different varieties and crops of Canadian wines, observed a strong correlation between the perceived astringency and the phenol and total tannin contents. Monteleone et al. (2004) observed that increasing the concentration of tannic acid and grape seed extract elevated the intensity of the perceived astringency. Thus, low levels of tannic acid (0.5–2 g/L) generated an increase in the intensity of the astringency perception, while at higher levels (2–3 g/L), the intensity remained stable. Fontoin, Saucier, Teissedre, and Glories (2008) worked in model vinous solutions, and found that as ethanol level and pH values increased, the astringency perception was lowered. While pH affected only astringency, ethanol contributed also to the perceived bitterness of tannin oligomers, especially at typical wine ethanol levels (11–15%). Golder and Zamora (2010) worked in Malbec wine; they used a time-intensity analysis, and found that at an increase in polyphenol concentration, the intensity of astringency also increased. Vidal et al. (2016) worked in dynamic characterization of Tannat wine astringency; they were asked to describe the astringency in a Temporal Dominance of Sensations...
(TDS) task comprising a list of 8 terms. Between two and three terms were significantly dominant to describe the astringency and enabled to discriminate samples with different astringency characteristics.

Quijada-Morín, Williams, Rivas-Gonzalvo, Doco and Escribano-Bailón (2014) observed significant correlations between the overall intensity of the astringency and the structural characteristics of the proanthocyanidins; a higher proportion of epicatechin subunits in the extension position and terminal gallocteinches increased the perception of astringency. However, the amount of epigallocatechin in both extension and terminal positions was negatively correlated with perceived astringency.

Pineau et al. (2009) studied the simultaneous evolution of several attributes over time and integrated the different perceptions into the temporal domain of sensations (TDS) method. This method has been used to describe the temporality of wine sensations, and it has proven to be an adequate methodology to identify wine quality descriptors (Meillon, Urbano, & Schlich 2009). Meillon et al. (2009) worked with TDS in wines deialcoholized in Merlot and Syrah and found that decreased alcohol content increased the perception of astringency in both varieties.

Based on the above characteristics and because astringency is a highly relevant and dynamic attribute of wine quality, the TDS method is a sensory tool that can be applied to evaluate of astringency over time. The objective of this study was to evaluate the effect of different concentrations of commercial tannin on the temporality of the perception of astringency. In addition, it is postulated that the gradual addition of tannin to a wine medium increases the dominance and duration of astringency and that the type of astringency is closely related to the tannin concentration.

2. Materials and methods

2.1. Model solution

A commercial oenological tannin (COT) brand, a grape skin tannin, Graptan S (Ferco, St. Montan, France) was used. Model solutions were prepared with 12% ethanol in water, containing 3.5 g/L of tartaric acid, with the pH adjusted to 3.2. In the characterization of Graptan S, at a concentration of 1 g of tannin/L in the model solutions, 633 mg equivalents of gallic acid/g of tannin were obtained in the analysis of total phenols by Folin-Ciocaltel, 612 mg equivalents of cyanidin per g of tannin of proanthocyanidins by the method of Singleton VL and JA Rossi (1965), and 2.08 average degree of polymerization by the method of Vivas et al. (2004) and Obreque-Slier et al. (2013b). Once the solution was prepared, the solution was fractionated into three containers to add the corresponding tannin doses (C1: 1 g/L, C2: 2 g/L, C3: 3 g/L). The bottles of these solutions were stored for 3 months at ≈ 16 °C in an underground cellar.

2.2. Evaluation procedure

The training to characterize astringency was conducted in 12 sessions (1.5-hour duration), and 4 sessions were used to evaluate the model solutions (40-minute duration). Samples are expected during training, there were 9 samples in the evaluation sessions of the model solutions, panelists absorbed 30 ml of model solutions. Samples were served in randomly coded black INAO cups and were presented according to a Williams Latin square plan to balance the order and decrease contrast effects. The sessions were conducted in insulated booths at 20 °C ± 1 °C. The operating temperature was 17 °C ± 1 °C.

2.3. Sensory panel training

Potassium alum and pectin sensory standards purchased from Sigma Corporation (Saint Louis Missouri, USA) and Cramer were used for training. In addition, Santa Rita 2012 cabernet sauvignon and various solvents were purchased from the Mitchelson Drugstore (Santiago, Chile). Fourteen people were selected for panel training, of which 13 completed the final evaluations. The members of the panel belonged to the Faculty of Agronomic Sciences of the University of Chile. During the training and evaluations, each evaluator was compensated as an incentive to participate.

The methodology of constant stimuli (Centeno, 2006) was used to determine the threshold of perception. Six solutions of potassium alum were used with concentrations between 0.1 g/L and 0.8 g/L. First, the probable threshold area was established using the alternative forced choice (AFC) method (Centeno, 2006). According to the ability of each evaluator to recognize the astringent sample, the minimum concentration for astringency perception was determined. The concentration had to be recognized with an accuracy of 100% in a series of three triangular 3-AFC tests to be considered as the detection threshold.

The characterization of astringency was based on the methodology of Gawel, Oberholster, and Francis (2000). As an introduction to the concept of astringency, a series of diverse textures was presented for manual manipulation, and they were associated with the perceived descriptors. These sensations were evaluated for some astringent foods and materials, as follows.

- Group of material: satin, suede, silk, leather, velvet, fine sandpaper, medium sandpaper, coarse sandpaper, fine burlap, medium burlap, coarse burlap, corduroy, smooth Trevira, stamped Trevira, Osnaburg, t alc, clay, sawdust, and plasticine.

- Group of food: tea, persimmon, banana peel and grape.

Through open discussion, an agreement was reached regarding the appropriate descriptors for each of the samples. The use of manual texture references for each descriptor was considered based on the astringency wheel of Gawel et al. (2000). Subsequently, in open sessions, the judges were presented with solutions, both in water and in wine, of astringent compounds, such as potassium alum and oenological tannin. The judges were asked to indicate the appropriate terms to describe the astringency of the samples using the list previously selected during the generation of descriptors. The astringency descriptors of Gawel et al. (2000) were used in the model solutions to determine the most representative descriptors of the different tannin concentrations. The panel of judges selected the descriptors; soft, adhesive, mouthfeel, aggressive and dry, which were used in the temporal domain of the astringency sensations.

2.4. Chemical and temporal sensory analysis

A Shimadzu UV-1700 Pharmaspec UV-vis spectrophotometer and an Agilent Technologies 1200 series high-performance liquid chromatograph with an Aligned model L-7455 photodiode array detector (HPLC-DAD) and an L-7200 automatic injector were used for the chemical analysis. FIZZ (Biosystemes, Couternon, France) was used for the sensorial analysis but was not used to construct the graphs and to obtain the temporal domain parameter data.

The following chemical analyses were performed at bottling (T0) and at the end of the storage period (T1) using the methodologies proposed by García-Barcelo (1990): pH, alcohol content, and total phenolics. Additionally, the total tannin content (Mercurio, Dambergs, Herderich, & Smith, 2007) and low-molecular-weight phenols were quantified by HPLC (Obreque-Slier, Peña-Neira, López-Solís, Ramírez-Escudero, & Zamora-Marín, 2009).

Percentage quantification was performed in the headspace using a Checkpoint portable gas analyser (Dansensor, Spain), and the percentage of oxygen contained in this volume of air was determined.

Characterization of the astringency in model vinous solutions was performed with different concentrations of COT (C1, C2, and C3). The panel of judges evaluated the dominance of astringency and characterized it using the previously selected descriptors. The TDS method was used with different descriptors. In traditional TDS, the sensorial wine descriptors were used according to Meillon et al. (2009).
contrast, for astringency TDS, the astringency descriptors, which had a higher frequency of occurrence in the wine model evaluations during training, were taken from the astringency wheel of Gawel et al. (2000).

The dominance index in the temporal graphs was determined by the sensory evaluation. In addition, two temporal parameters were measured:

- Appearance time (T) — the moment the judge selected as the beginning of the dominance of the descriptor (Pineau et al., 2009).
- Dominance duration (D) — the total time that the descriptor remained dominant (Pineau et al., 2009).

### 2.5. Experimental design and statistical analysis

Statistical analysis of the chemical and sensory results was conducted using InfoStat software (Córdoba, Argentina).

The chemical analysis used a split-plot (3 × 2) experimental design with the tannin concentration and time in the bottle as factors. The sensory evaluation was structured in random blocks with each panelist as a block.

There were three levels of tannin: 1 g/L (C1), 2 g/L (C2) and 3 g/L (C3), with a 16 mL head space level (Kwiatkowski, Skouroukounis, Lattey, & Waters, 2008). The time in the bottle factor had two levels: bottling time (T0) and 3 months after bottling (T1). Treatments, defined as tannin levels, were performed in triplicate. The experimental unit was a pair of bottles of a model solution: one was evaluated at T0 and the other at T1. Each bottle was a sample unit. In terms of repetition for session, two sessions in T0 and two sessions in T1 were performed for each of the measurement times.

The data were analysed using the following statistical model.

\[ Y = \mu + \tau_i + T_k + (\tau T)_{ik} + P_j + B_{ik} + e_{ijkl} \]

where
- \( Y \) response to treatment.
- \( \mu \) overall average.
- \( \tau_i \) effect of tannin, i: 1, 2, 3
- \( T_k \) effect of time, k: 0, 1
- \( P_j \) effect of plot, j: 1, 2, 3
- \( B_{ik} \) effect of the bottle, s: 1, 2
- \( e_{ijkl} \) standard error

The model was analysed as a mixed linear model using InfoStat statistical software. The Fisher test was used to test for significant differences at a significance level of 5%.

### 3. Results

#### 3.1. Astringency detection threshold

Table 1 shows that the mean detection threshold of the panel for astringency was 0.14 g/L. Of the 14 panel members, 64.3% had a lower threshold than the panel average.

#### 3.1.2. Intensity order

Table 2 shows the astringency intensity rankings. In the first three sessions, it was observed that the panel lost the ability to discriminate by initially tasting a water matrix and then a wine matrix. However, the use of a pectin in the rinse solution between samples helped to restore lubrication, decreasing the saturation of the panelists. Only the results in which the panel discriminated between the samples are presented.

#### 3.1.3. Temporal profiles of the model solutions

Fig. 1 shows the temporal profiles of 3 model solutions with different concentrations of COT. In the model solutions, increases in the dominance index of astringency and in the persistence of the dominance of astringency were observed as the tannin concentration increased. In addition, this increase in dose produced a decrease in the dominance of the remaining descriptors.

#### 3.2. Sensory evaluations

A quantitative phase was performed in which the temporal profiles were obtained, along with their time variables, using the two TDS modalities: traditional and astringency.

#### 3.2.1. Temporal profiles of traditional TDS

Fig. 2 shows the profiles evaluated at T0 and T1. Independent of the time point, there is an increase in the dominance index and a significant increase in the duration of the dominance (seconds) of the astringency as the tannin level increases (C1: 27.85 ± 6.03 bc; C2: 45.83 ± 5.78 a). In addition, a significant decrease in the duration of the alcohol descriptor (seconds) was observed with increasing tannin concentration (C1: 15.4 ± 3.43 ab; C3: 7.7 ± 3.62 c). With respect to the acidity and bitterness, although there were no significant differences, a lower balance was observed due to the significant increase in astringency.
3.2.2. TDS temporal profiles of astringency

Fig. 3 presents the profiles evaluated at T0 and T1. Different tannin concentration levels have different dominant descriptor compositions. In treatments with a low tannin concentration (C1), the dominant descriptors were mouth filling and soft. In treatments with a medium tannin concentration (C2), the dominant descriptors were mouth filling, aggressive, drying and adhesive. In the treatments with a high tannin concentration (C3) the dominant descriptors were aggressive and drying. In addition, the duration of the aggressive (C1: 18.1 ± 4.11 b, C3: 27.26 ± 3.34 a) and drying (C1: 14.07 ± 4.38 b, C3: 26.8 ± 3.81 a) descriptors increased as the tannin concentration increased. Changes in the time of appearance were also observed relative to the tannin concentration, for example, the appearance of the adhesive descriptor was delayed and the appearance of the drying descriptor was earlier at higher tannin concentrations.

3.3. Chemical analysis

3.3.1. Phenol and total tannin contents

Table 3 shows the total phenol and tannin contents of the model vinous solutions for different concentrations of a COT (C1, C2 and C3). A substantial increase in the total phenol and tannin content was observed as the COT concentration increased. Additionally, the total phenol content was higher in T1, and the total tannin content decreased significantly from T0 to T1.

3.3.2. Quantification of low-molecular-weight phenols in the model vinous solutions by HPLC-DAD

Table 4 shows the procyanidin, flavanol (only catechin and epicatechin) and benzoic acid contents of the model vinous solutions. A decrease was observed in the concentrations of the procyanidins and flavonols between the two measurement times for C2 and C3. On the other hand, as the tannin concentration increased, the concentration of both variables increased. A decrease in the benzoic acid concentration was observed between the two measurement times for C2 and C3. However, as the tannin concentration increased, the concentration of benzoic acid increased.

4. Discussion

The perception of astringency is a highly dynamic process that continuously changes during ingestion, especially after expectoration or
swallowing (Noble, 1995). Many methodologies, trials and models have been proposed to objectively evaluate astringency (Obreque-Slier, 2010). However, sensory evaluation by a trained panel is the most commonly used methodology (Gawel, Iland, & Francis, 2001). Currently, intensive research is being conducted on the evolution of organoleptic sensations brought about by wine (Meillon et al., 2010; Pineau et al., 2009). The TDS methodology was developed to incorporate time into the traditional descriptive analyses and to determine in real time the changes in the perception of sensations. Pineau et al. (2009) demonstrated that TDS can be used to simultaneously evaluate the dominance of several descriptors to describe the global context of the perception of a complex matrix. However, there is no information regarding the use of this methodology in the perception of astringency under conditions of different COT concentrations over time. This study evaluated the perception of astringency generated by a model wine solution enriched with tannins, using TDS as the central methodology to evaluate the parameters of dominance and duration.

Training sessions were performed to determine the panel’s astringency threshold using solutions of potassium alum in water. The average threshold was 0.14 g/L, which is in agreement with Vazallo (2016), who observed an average of 0.15 g/L. Regarding the order of intensities, as a whole, the panel discriminated between the concentrations of potassium alum in water in agreement with Centeno (2006), and there was high regularity in the response of the evaluators. The above result supports the discriminative ability of the panel in terms of the astringency intensity of COT in model vinous solutions. The validity of the temporal characterization training was reflected in the clear differences in the index of dominance and the persistence of dominance between different model vinous solutions.

For the traditional TDS results, treatments with higher concentrations of COT showed a higher rate of dominance and duration of astringency. This observation was closely related to the higher presence of polyphenols, specifically the concentration of condensed and hydrolysable tannins, which increased as the COT concentration increased. These compounds have been described as important precipitants of the salivary proteins, causing a loss of lubrication of the buccal cavity, consequently generating a greater sensation of dryness or astringency (Vidal et al., 2004).

An indirect relationship was observed between the duration of the alcohol descriptor and the COT concentration; at higher COT concentrations, the duration of the alcohol descriptor decreased and the duration of the astringency descriptor increased. According to Meillon et al. (2010), decreasing the alcohol concentration would increase the dominance of the astringency, which could be because alcohol interrupts the hydrogen bridge that is formed between the tannin and salivary proteins (Noble, 1990). However, some studies have reported that higher alcohol contents increase the intensity of the astringency perceived by a panel (Obreque-Slier et al., 2010). In the present study, an increase in COT content decreased the duration of the alcohol descriptor and increased the duration of astringency.

After using the traditional TDS method as a tool to simultaneously evaluate the dominance of several descriptors, including astringency, a specific TDS was performed for the perception of astringency. The descriptors were selected based on their frequency of appearance in the evaluation of samples with different COT concentrations using the wheel of astringency of Gawel et al. (2000) as a reference. According to the responses in the dominance curves of the treatments, a change in the dominant descriptors was observed as a function of the COT concentration. Thus, at the lowest concentration, the dominant descriptors were soft, mouth filling and adhesive, while at the highest concentration, the dominant descriptors were aggressive and drying. Both observations were closely related to the degree of lubrication loss in the

![Fig. 3. TDS astringency profiles.](image)

### Table 3

<table>
<thead>
<tr>
<th>Total phenols (mg L$^{-1}$ gallic acid)</th>
<th>Total tannins (mg L$^{-1}$ catechin)</th>
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<tr>
<td></td>
<td>T0</td>
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<tr>
<td></td>
<td>Mean</td>
</tr>
<tr>
<td>C1</td>
<td>394 ± 8 f</td>
</tr>
<tr>
<td>C2</td>
<td>760.4 ± 8 d</td>
</tr>
<tr>
<td>C3</td>
<td>1138 ± 8 b</td>
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</tbody>
</table>

The values are presented as the mean ± standard error. Lowercase letters indicate significant differences (Fisher test, $p < 0.05$) between treatments.
The values are presented as the mean ± standard error. Lowercase letters indicate significant differences (Fisher test, p < 0.05) between treatments.

### Table 4

<table>
<thead>
<tr>
<th>Procyanidins (mg L⁻¹ eq. catechin)</th>
<th>Flavonols</th>
<th>Benzoic acids (mg L⁻¹ eq. gallic acid)</th>
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</thead>
<tbody>
<tr>
<td></td>
<td>Mean E.E.</td>
<td>Mean E.E.</td>
</tr>
<tr>
<td>C1 5.14 ± 0.3 de</td>
<td>4.58 ± 0.3 e</td>
<td>9.76 ± 0.64 de</td>
</tr>
<tr>
<td>C2 15.86 ± 0.3 b</td>
<td>5.94 ± 0.3 d</td>
<td>26.41 ± 0.64 b</td>
</tr>
<tr>
<td>C3 21.17 ± 0.3 c</td>
<td>9.52 ± 0.3 c</td>
<td>38.27 ± 0.64 a</td>
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