Impact of a commercially available ion-exchange resin used in red wines to reduce toxic compounds: effect on pH, oxidation–reduction potential, color, and sensory attributes

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Abstract This research was performed to evaluate the impact of a novel ion-exchange treatment on the organoleptic, pH, color (hue and intensity), and oxidation–reduction potential (ORP) properties of commercial red wines. The ion-exchange treatment used in this study is a commercially available device containing a double cation–anion exchange resin. The device is applied, directly by the consumer, to finished bottled wines with the purpose of mitigating the effects of potentially noxious compounds such as biogenic amines and sulfites. Sensory evaluation tests included discriminative, affective, and Quantitative Descriptive Analysis sensory tests were employed to evaluate the impact on the perceived taste of treated wines. The pH, ORP and color parameters (hue and intensity) of wines were measured to assess any physicochemical alterations that may help to explain the results of the sensory evaluation. While no significant changes in color hue or intensity were found, the results showed a reduction in pH (0.07–0.12) and an increase in ORP (2.667–6.666 mV). However, the changes in pH and ORP did not result in a detectable change in wine taste for the sensory panelists. These findings have important implications for the wine industry, where many consumers select wines for their characteristic taste qualities. Thus, any treatment to

potentially remove noxious compounds should not impact the characteristic organoleptic properties of a chosen wine.

Keywords Sensory evaluation · Ion-exchange · Wine sensory · Biogenic amines

Introduction

Wine is one of the most popular and commonly consumed alcoholic beverages among global consumers (Bonn and Cho 2017). The international wine trade has experienced substantial growth over the past few decades. In particular, there has been an increase in wine consumption in Northern Europe and North America, along with a growth of exports of new world wines (Mariani et al. 2012). United States has experienced a consistent increase in annual wine production from 2010 to 2017 with an average annual growth of approximately 4% (TTB 2017). Despite the popularity and enjoyment of wine, hypersensitivity towards certain compounds in wine can cause adverse reactions in susceptible consumers. Symptoms that are commonly associated with these compounds include flushing, itching, headaches, diarrhea, urticaria, and asthma (Maintz and Novak 2007; Vally et al. 2007).

Many of the compounds long considered responsible for these adverse noxious reactions are secondary metabolites produced during fermentation in wine processing (Smit et al. 2008). Biogenic amines (BA), especially histamine, are considered to be the main groups of compounds responsible for some of these adverse reactions (Maintz and Novak 2007). Similar adverse reactions may also arise from hypersensitivity to compounds added to wine for shelf-life stability such as sulfites, benzoates, and sorbates (Smit et al. 2008; Vally et al. 2007). Red wines are often

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considered to be the main culprit for these adverse reactions, in fact, they are known to contain higher levels of amines than white wines (Romero et al. 2002; Linneberg et al. 2008). To address the hypersensitivity of these compounds a product emerged in the marketplace in 2015 that aims to alleviate some of these potentially adverse reactions. This device applies ion-exchange technology in the form of chelating agents with mixed cation and anion exchange resins with the aim of reducing BAs and sulfites. The handheld device allows the consumer to treat the wine directly prior to consumption. The manufacturer of the device reported an 85.5–86.2% reduction in BAs, a 30% reduction in free sulfites, and a 40% reduction in total sulfites content in red wine (Meadows and Ketelson 2017). A summary of these findings is presented in Table 1.

While the application of ion-exchange technology by consumers is novel, it is among the most widely used wine acidification and tartaric stabilization treatments in commercial wine processing (Lasanta and Gómez 2012). This technology has been employed in wine processing since the middle of the last century (Benítez et al. 2002a). This process is based on the use of polymerized insoluble granular resins that can exchange positive or negative ions fixed on functional groups. The ions are exchanged with other ions of the same polarity within the wines while the exchanger itself remains physically unaltered (Lasanta et al. 2013). The three ion-exchange resin configurations for wine acidification and tartrate stabilization are: (1) cation exchange resin in the H⁺ form; (2) anion exchange resin in the OH[−] form; and (3) mixed treatment (double cation–anion) in which potassium and tartrate are exchanged for H⁺ and OH[−] (Mira et al. 2006). Cation exchange resins in an acid cycle are the most commonly used resin type, and in fact, are the only resin type authorized for use in wines by the European Union (2009). Rectified concentrated grape musts are frequently treated with double anion–cation exchange resins before they are added to wine must during the fermentation process to increase potential alcohol content in the final wine (Palacios et al. 2001). In addition to ionized material, the resins also consist of chelating agents that adsorb metallic ions such as magnesium, iron, copper, potassium, calcium, and sodium in the wine (Benítez et al. 2002a; Ibeas et al. 2015).

Previous studies investigated the impact of ion-exchange treatment on the organoleptic characteristics of wines. However, the findings of these studies are somewhat conflicting. Cation exchange treatment has been found to induce negative impact for taste characteristics in some studies (Benítez et al. 2002a; Ibeas et al. 2015). Conversely, several authors have reported marked improvements in visual color, taste intensity, and overall perceived quality in treated wines (Walker et al. 2004; Lasanta et al. 2013). On the other hand, some authors have reported that ion-exchange treatment did not alter the organoleptic characteristics of treated wines (Benítez et al. 2002b; Mira et al. 2006). While a reduction in BAs and sulfites content in red wine has been reported (Meadows and Ketelson 2017), the impact of the device on the organoleptic qualities of wines has not been reported. Furthermore, the novel ion-exchange treatment used in this study is a commercially available device. Wine taste is one of the most influential intrinsic attributes and a key determinant of wine enjoyment and selection (Olsen et al. 2007; Lockshin and Corsi 2012). Therefore, it is of paramount importance to evaluate its impact on the taste of wine. Thus, the aim of this current study is to investigate the impact of an ion-exchange device, intended to reduce the concentration of noxious wine constituents, on the taste characteristics of selected red wines. In order to support the sensory findings, additional chemical measurements were conducted which include hue, color intensity, Oxidation–Reduction Potential (ORP), and pH.

Materials and methods

Ion-exchange treatment

A double anion–cation exchange resin treatment was selected for this study. This device is comprised of a mixture of both cation and anion exchange resins formed by a chloride mineral with chelating functional properties which is further described by Meadows and Ketelson (2017). The exact known types of resin, their proportions, and their properties are undisclosed by the manufacturers and are protected by US patent laws. With this

Table 1 Summary of reported findings for free sulfite, total sulfite, and biogenic amine concentrations (ppm) in untreated and treated Cabernet Sauvignon wines using the mixed bead ion-exchange treatment. [Adapted from Meadows and Ketelson (2017)]

Noxious constituents	Untreated (ppm)	Treated (ppm)	Change (%)
Free sulfite	10.00	7.00	30.0
Total sulfite	40.00	24.00	0.0
Histamine	4.63	0.64	86.2
Putrescine	22.48	3.01	86.6
Cadaverine	0.69	0.10	85.5
Tyramine	1.16	0.42	63.8

arrangement, the device is purported to be capable of binding anions or cations from the wine matrix. The application apparatus consisted of a satchel porous membrane contained in a handheld cartridge with a spoon-shaped structure, made of Polyethylene. The satchel contains approximately 3 g of ion-exchange resin. The porous walls of the satchel allow for the exchange of ions between the wine sample and the resins while at the same time preventing the resin materials mixing in the wines. The device is intended to be used directly by the end consumer prior to consumption (Meadows and Ketelson 2017).

Treatment of wine samples

To assess the repeatability of the wine treatment three commercial wines were selected for testing. Red wine was chosen as it is frequently reported as containing compounds that result in adverse reactions for some hypersensitive consumers (Linneberg et al. 2008). The particular wine brands were selected as representatives of the Cabernet Sauvignon wine variety after consultation with wine experts. The Cabernet Sauvignon variety of red wine was the most commonly purchased red wine in the US retail market in 2016 (Wine Institute, 2016). Each wine corresponded to a specific retail price segment based on the typical retail price paid per 750 ml bottle intended for home consumption (Lesschaeve et al. 2012; Thach and Olsen 2015). The price segments were low (< \$6), moderate (\$7–\$12), and high (\$15–\$20). For this work, wines are coded as Wine 1 for the low-price segment, Wine 2 for the moderate price segment, and Wine 3 for the high price segment. Wine samples were stored at room temperature (20–22 °C) for 24 h before treatment. The treatment of wines with the ion-exchange device (The Wand™, Pure-wine Inc., TX, U.S) was conducted according to the manufacturer's guidelines; one ion-exchange device was placed in 150 ml of wine for 3 min while stirring gently.

Sensory evaluation testing

Sensory evaluation testing was carried out in a wine sensory laboratory under guidelines and conditions according to ISO 8589:2010 (ISO 2010). Panelists worked in a single booth under defined conditions of 22 °C and white light. Sensory panelists were students and staff recruited at Florida International University, Miami FL. Panelist demographic information was gathered on (1) gender, (2) age, and (3) red wine consumption frequency (Table 2). The subjects were asked to refrain from eating and drinking (except room temperature water) for 1 h prior to testing. Thirty (30) ml of each sample was served between 20 and 22 °C in 50 ml clear plastic sample cups labeled with 3-digit random numbers, as recommended in ASTM E1879

Table 2 Frequency and percentage of subjects for affective sensory and discriminative tests by gender, age and drinking frequency (n = 134)

	n (%)
<i>Gender</i>	
Male	65 (48.5)
Female	69 (51.5)
Total	134 (100.0)
<i>Age group</i>	
21–24	42 (31.3)
25–34	45 (33.6)
35–44	33 (24.6)
45–54	5 (3.7)
55–64	5 (3.7)
65+	4 (3.0)
Total	134 (100.0)
<i>Wine consumption frequency</i>	
More than once a week	33 (24.6)
Once a week	27 (20.1)
Every other week	48 (35.8)
Once a month	23 (17.2)
Rarely	3 (2.2)
Total	134 (100.0)

(ASTM 2010). The panelists were required to cleanse their palates with water and crackers in between samples. Data was collected on electronic tablets using Compusense Cloud software (Compusense Inc., Guelph, Ontario, Canada). Institutional Review Board (IRB) approval was obtained before conducting the sensory evaluation using human subjects, approval protocol number IRB-16-0203.

Discrimination test

A triangle test was conducted to ascertain if a difference in taste could be detected by untrained panelists between treated and untreated samples. Testing was conducted in duplicate over two separate testing days. In order to confirm the results and avoid any bias in the data set a different set of subjects and blinded codes were used in each test. A total of 64 panelists (n = 64) participated in duplicated testing; 34 panelists (n = 34) for the initial test, and 30 panelists (n = 30) for the duplicated test. The triangle test was conducted according to the guidelines set out in ISO 4120:2004 (ISO 2004a, b). The panelists were presented with 3 samples and were advised that 2 of the samples were identical and 1 was different. Panelists were asked to identify the different (odd) sample from each triad. Samples were presented in the following order ABB, BAA,

ABB, BBA, ABA and BAB, where A is a treated wine sample, and B is the untreated wine sample.

Affective testing

A consumer hedonic test was conducted to assess the panelists overall liking of the treated and untreated wines. Seventy untrained panelists ($n = 70$) participated in the study. Subjects were instructed to evaluate their liking of the taste of the samples on a 9-point hedonic scale), where 9 = ‘extremely like’ and 1 = ‘extremely dislike’. Samples were presented in a monadic sequential randomized order to the panel. A paired-preference test directly followed the consumer hedonic tests. The panelists were presented with fresh samples and were asked to rank the samples in order of preference, from most preferred to least preferred. For these tests, panelists received 2 samples in simultaneous presentation, 50% of the samples were presented in the order A–B, the other 50% were presented in the order B–A.

Descriptive testing

Quantitative Descriptive Analysis (QDA) was performed to ascertain if a panel of trained subjects could detect differences in the taste profile of the treated and untreated wines. The QDA panel comprised of 6 people ($n = 6$), panelists were 4 females, 2 males, aged 26–32. Based on a screening test, panelists were selected from graduate students. All panelists had previously completed a variety of wine tasting classes and had significant experience in wine descriptive analysis. The panelists participated in 5 one-hour training sessions over a period of 2 weeks, where they practiced proper techniques for assessing wine, participated in taste identification, taste threshold, and line scale training and testing according to ISO8586: 2012 (ISO 2012). The panelists’ ability to differentiate between different tastes was assessed with blind coded and randomized taste reference samples. For evaluation of wine samples, the panelists were presented with 30 mL of each sample together with a glass of spring water, a spit cup for expectoration, a paper napkin, and palate cleansers (plain cracker). Sensory testing was conducted in triplicate, and sampling followed a monadic sequential randomized order. Panelists were asked to assess the intensity of taste attributes, namely; sweetness, bitterness, saltiness, sourness, and umami. The intensity of each attribute was rated using a 9-point scale (15 cm linear scale), where 9 = ‘high’ intensity, 5 = ‘medium intensity’, and 1 = ‘low intensity’. Subjects were supplied with low, medium and high concentration reference samples for each taste attribute for each taste attribute: sweet (sucrose), sour (citric acid solution), bitter (caffeine solution), salt (sodium chloride solution), and umami (monosodium glutamate).

pH and oxidation–reduction potential

The pH of wine samples (100 mL) was measured using a Mettler Toledo (FiveEasy™ FE20) benchtop pH meter following standard methods of analysis (AOAC 2016). The meter was first calibrated with two buffer solutions of pH 4 and 7. The electrode was rinsed with deionized water between measurements. Similarly, the Oxidation–Reduction Potential (ORP), mV, was determined separately by the same pH meter. All measurements were conducted in triplicate at room temperature (25 °C).

Color measurement

Wine color intensity was measured in terms of absorbance of incident light using a UV–Vis spectrophotometer (Molecular Devices Inc.) within the visible spectrum (420–620 nm) according to the Beer–Lambert law (Atkins and de Paula 2006). Prior to measurements, the spectrophotometer was zeroed using deionized (DI) water as a blank. Wine samples were diluted using deionized water by a factor of 3, and absorbance measurements were made with standard 1 cm cuvettes. An additional parameter, hue, was determined using a ratio of absorbance at 420 and 520 nm wavelengths. The color intensity is given by the sum of absorbance measurements in the violet, green and red areas of the visible spectrum:

$$\text{Color intensity} = A_{420} + A_{520} + A_{620}$$

where A_{λ} denotes the absorbance at wavelength λ .

Data analysis

Data from the discrimination test (triangle test) and the preference test were analyzed using Pearson’s Chi squared test and cross-tabulation test. Whereas, consumer liking data and QDA data were analyzed using independent t -tests to find differences among mean scores for treated and untreated samples. Data from the physicochemical analysis (ORP, pH, hue, and color intensity) were analyzed using independent t test. Differences were considered significant at the 5% significant level ($p \leq 0.05$). Statistical analysis was done through SPSS v. 20 (2016).

Results and discussion

Oxidation–reduction potential (ORP) of treated and untreated wines

The results for ORP, pH, hue, and color intensity for treated and untreated wine samples are shown in Table 3. A significant ($p \leq 0.05$) increase in the ORP for all treated

Table 3 Results of pH, oxidation–reduction potential (ORP), hue, and color intensity analysis for treated and untreated Cabernet Sauvignon wines

Treatment	Wine Type	pH*	ORP (mV)*	Hue ^{NS}	Color Intensity (A_λ) ^{NS}
Untreated	Wine 1	3.57 ± 0.00	207.333 ± 0.471	2.163 ± 0.047	1.976 ± 0.158
	Wine 2	3.79 ± 0.01	194.000 ± 0.816	2.086 ± 0.054	2.876 ± 0.170
	Wine 3	3.67 ± 0.01	199.667 ± 0.471	2.276 ± 0.035	2.440 ± 0.190
Treated	Wine 1	3.47 ± 0.01	211.667 ± 0.943	2.295 ± 0.005	2.043 ± 0.073
	Wine 2	3.72 ± 0.02	196.667 ± 0.943	2.044 ± 0.023	3.203 ± 0.105
	Wine 3	3.55 ± 0.02	206.333 ± 1.700	2.200 ± 0.006	2.881 ± 0.143

Hue is dimensionless

NS, indicates no significant difference ($p > 0.05$) between treated and untreated samples within the same wine type for the analytical measurement; A_λ absorbance units; ORP, oxidation–reduction potential

*Indicates significant difference ($p \leq 0.05$) between treated and untreated samples within the same wine type for the analytical measurement

wine samples was observed. The highest increase of ORP was found in Wine 3; this increase was 6.666 mV. The increase in ORP for Wine 1 and Wine 2 was 4.334 and 2.667 mV, respectively. These findings may be attributed to an adjustment in the isoelectric point in wines without the addition of an acid due to treatment. Furthermore, the results confirm the exchange between the ions in device resin and the ions present in the wines (Meadows and Ketelson 2017; Mira et al. 2006).

pH of treated and untreated wines

A significant ($p \leq 0.05$) decrease in pH values was found for all treated wines (Table 3). The change in pH values for treated wines ranged from 0.07 to 0.12 which corresponds to a 1.69–3.27% change. This reduction in pH conforms with the findings obtained by other authors. However, the magnitude of reduction in pH was substantially less in the current study compared to the findings reported in other studies. Lasanta et al. (2013) and Ibeas et al. (2015) treated red wines with cation exchange resins, they reported initial reductions in pH of 32.4 and 30.7%.

In contrast, Mira et al. (2006) compared the performance of cation exchange resins with that of double cation–anion exchange resins reporting considerable differences in pH reduction between resin types. They found a reduction in pH of 45% for the cation exchange resins. While, they found that the use of double cationic–anionic exchange resins resulted in a considerably lower reduction in pH of only 30%. Their findings suggest that the strong acidic component of cation change resin can have a greater impact on wine pH than a double cation–anion exchange resin. Cation exchange resin is known to provide a significant adjustment of the acid–base balance of wines and an overall improvement of tartaric stability (Lasanta et al. 2013). Therefore, in the current study, the relatively marginal reduction of pH in treated wines may be explained by the use of a relatively weaker double cation–anion

exchange resin compared to the more conventional cation exchange resin (Mira et al. 2006).

Hue and color of treated and untreated wines

The results for hue and color intensity analysis for all treated and untreated wines are presented in Table 3. No significant ($p > 0.05$) changes in both hue and color intensity were observed. These findings are contrary to observations of other authors where an improvement in hue and color intensity have been reported (Benítez et al. 2002a, b; Walker et al. 2002; Mira et al. 2006; Lasanta et al. 2013). A decrease in hue with an increase in color intensity is typically associated with the retention of phenolic compounds by exchange resins and a reduction in pH in wines (Lasanta et al. 2013; Mira et al. 2006). Thus, the differences between the findings in this study and other studies is explained by a relatively marginal reduction in pH. Furthermore, a lack of retention of wine color pigments such as anthocyanin in the surface of the resin may also elucidate the results for hue and color (Mira et al. 2006; Ibeas et al. 2015).

Sensory analysis

Discrimination test

A table presenting the findings for sensory evaluation testing has been provided as supplementary material. Chi squared analysis of the discrimination sensory data revealed that the panelist could not significantly ($p > 0.05$) distinguish between treated and untreated wine samples. More specifically, the results for Wine 1 showed that only 12 respondents out of 34 correctly identified the odd sample. Comparative results were found for Wines 2 and 3, where 15 and 10 respondents correctly identified the odd sample, respectively. Considering this test was an alternative forced choice test (AFC) with a probability of 1-in-3 of

selecting the correct answer by chance, these results indicated that the panelists could not discriminate between the taste of treated and untreated wine samples.

Preference test

For the paired-preference sensory test, the Chi squared analysis indicated that no significant ($p > 0.05$) preference for treated or untreated wine existed. Furthermore, for Wine 1, 38 panelists preferred the treated sample, while in comparison 32 panelists preferred the untreated sample. Similarly, 38 panelists preferred the treated sample for Wine 2, with 30 panelists preferring the untreated sample. A comparable trend was found for Wine 3, 41 panelists preferred the treated sample compared to 29 panelists for the untreated sample.

Consumer test

Similar findings were found for the consumer hedonic tests, no significant ($p > 0.05$) differences between the mean overall liking of wine taste for all treated and untreated wine samples was found. Mean liking score for Wine 1 were 6.40 ± 1.65 for the treated sample and 6.16 ± 1.65 for the untreated sample. While for Wine 2, mean scores were 5.94 ± 1.93 for treated samples and 5.76 ± 2.05 for untreated samples. The mean liking score for Wine 3 were 5.93 ± 2.27 for the treated sample and 5.69 ± 2.08 for the untreated sample. Thus, the results from the two affective sensory tests (hedonic and paired-preference) indicated that treated wines were found to be equally acceptable as untreated wines.

Descriptive test

Analysis of variance (ANOVA) was carried out on the QDA data. These results showed that no product $\times$ replication and product assessor interactions were significant ($p \leq 0.05$). This indicates that the training program was adequate to reach high reliability of the panel, as each product was not evaluated differently in different replications or by different assessors. The mean scoring of each sensory attributed for treated and untreated wine samples are depicted in Figs. 1, 2, and 3. The trained panel did not detect any significant ($p > 0.05$) differences in taste attributes between treated and untreated wines. These findings are particularly interesting considering a reduction in pH for all treated wines was found. It appears that this reduction was not sufficient to produce detectable differences in the acidic taste.

While these findings do conform with results reported by Lasanta (2009), Mira et al. (2006), Palacios et al. (2001) and Walker et al. (2004), some other authors reported

Fig. 1 Comparative descriptive sensory analysis results between treated and untreated Wine 1 samples rated on a 9-point scale (15 cm linear scale) for taste attributes ($n = 6$). Means analyzed by independent two-tailed t -test, differences considered significant at 5% significant level ($p \leq 0.05$)

Fig. 2 Comparative descriptive sensory analysis results between treated and untreated Wine 2 samples rated on a 9-point scale (15 cm linear scale) for taste attributes ($n = 6$). Means analyzed by independent two-tailed t -test, differences considered significant at 5% significant level ($p \leq 0.05$)

alterations in the organoleptic characteristics of treated wines. For instance, Ibeas et al. (2015) observed lower punctuation in taste equilibrium, intensity, and persistence in wines treated with a cation exchange resin. On the other hand, Lasanta et al. (2013) observed that wines treated with a cation exchange resin resulted in significant improvements in taste intensity, visual aspect, and overall quality.

Fig. 3 Comparative descriptive sensory analysis results between treated and untreated Wine 3 samples rated on a 9-point scale (15 cm linear scale) for taste attributes ($n = 6$). Means analyzed by independent two-tailed t -test, differences considered significant at 5% significant level ($p \leq 0.05$)

Implications of the study

The differences in the findings compared to some studies may be explained by the intended application of the ion-exchange device in the current study, and therefore the choice of resin. Strong cation exchange resins are typically used in the industrial processing of wine for tartaric stabilization. These resins are known to produce extreme reductions in wine pH during processing and in many cases this reduction can be excessive (Lasanta and Gómez 2012). Subsequently, this requires the mixing of treated wines with untreated wines to produce a more palatable final product (Mourgues 1993). In contrast, the practical objective of the device tested was not to inhibit tartrate crystal growth in wine must during processing, but rather to reduce the concentration of SO_2 and BA in finished wines. Hence, a weaker double anion–cation exchange resin was used that produced minimal changes in pH.

The results of the current study have practical implication for the food and beverage industry as a whole. For wine consumers, the expectation of wine taste can be formed from previous consumption experiences or from the descriptive wine label itself (Lesschaeve et al. 2012). In this sense, a consumer concentrates on the expected characteristic taste qualities of a wine before consumption (Wansink et al. 2007). Any alterations of these qualities during consumption may lead to a negative hedonic response. Thus, it is important that the taste characteristics of wine are not altered or masked through treatments such as ion-exchange. While consumers with hypersensitivities to noxious compounds in wines may wish to offset their potential negative effects, they may not be willing to alter

the taste profile of the chosen wine. This might be especially true if the characteristic taste of the wine was a determining factor in the wine selection process.

Conclusion

The present study indicated that there were no significant changes in hue and color intensity and sensory characteristics as a result of the ion-exchange treatment. However, reductions in pH and corresponding increase in ORP of treated wines was observed. The reduction in pH proved to be marginal in comparison to observations of other authors where a stronger cation exchange resins were used. The results from the sensory evaluation testing suggest that the ion-exchange device did not alter the pH of the wines to a level that was detectable by the panelists. Thus, the findings in the current study provide important implications for the practical application of the ion-exchange treatment, where any alterations in wine taste may render the wine unrecognizable. Since most consumers purchase wine for hedonic purposes, retaining the hedonic value of the wine and meeting consumer expectations are of utmost importance. Further study should investigate the efficacy of the ion-exchange device to reduce the concentration of potentially noxious compounds in wines, and its impact on other organoleptic attributes of wines in addition to taste.

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