



Quality Characteristics of Piquette: A Potential Use of Grape Pomace

Aude A. Watrelot * D and James Hollis

Department of Food Science and Human Nutrition, Iowa State University, 536 Farm House Lane, Ames, IA 50011, USA; jhollis@iastate.edu

* Correspondence: watrelot@iastate.edu

Abstract: Grape pomace is a common waste product that can be used as compost, as animal feed or discarded. The goal of this study was to evaluate the quality and consumers' perception of a value-added grape pomace beverage, piquette, made using different red grape cultivars, yeast strains and grape pomace to water ratios. Petite Pearl and Marquette grape pomace were soaked using different pomace to water ratios in water for 2 days, prior to being pressed. Cane sugar was added to the juices prior to inoculation with three yeast strains (Cross Evolution, ICV D254, and Exotics Mosaic). The piquettes were bottled before chemical analysis and sensory evaluation by an untrained sensory panel following 8 months of storage. Piquettes made from Petite Pearl grape pomace, regardless of yeast strain, were preferred by consumers. Petite Pearl piquettes were fruity and pink, especially using D254 yeast. Piquettes made from different ratios of Petite Pearl pomace to water on a larger scale lacked nutrients at the beginning of fermentation, which led to "rotten-egg" aromas and were the least accepted by consumers.

Keywords: valorization; wine waste; sparkling grape beverage; Marquette; Petite Pearl; by-product

1. Introduction

Wine production in the U.S. accounts for 12% of global output, with more than three million liters of wine produced in 2017 (World Statistics | OIV, n.d.). About 20-30% of the fresh weight of the processed grapes is generated as grape pomace (GP). It is estimated that for every 6 L of wine produced, about 1 kg of GP is generated, and the estimated production of GP in the US was approximately 0.6 million tons in 2017 [1]. Grape pomace is obtained after juice or wine pressing, and includes 250 kg of stems, 430 kg of skins, 230 kg of seeds, and remaining pulp per 1000 kg of pomace [2]. This is considered a waste product of the grape and wine industry and is commonly used as compost, to feed animals, or dumped in landfills [3]. However, as GP is composed of skins, seeds, stems, and pulp, it is rich in fibers, accounting for about 190 to 380 g/kg of fresh matter, phenolic compounds, and organic acids. Phenolic compounds are antioxidants that help protect wines against oxidation and maintain wine quality, including maintaining color stability. In GP, the concentration of phenolic compounds ranges from 11 to 27 mg gallic acid equivalent per kg of dry white and red skin pomaces, and between 91 to 203 g/kg of dry matter in GP [4]. Therefore, the use of GP has become increasingly popular, as it is rich in health-beneficial compounds [5]. Studies have shown various uses of GP, including as a growth medium for biomass production, fortifying baked products [6,7], and chocolate formulations [8]. In addition to the development of those value-added products, the production of piquette has increased over the past few years in the US. Lora is the name of a wine-based beverage developed by Romans that was made from the addition of water to GP after pressing [9]. In French, this beverage has been called "Piquette", meaning "prickle". The process of making it involves adding water to grape GP, pressing, fermenting residual sugars, and bottling before completion of fermentation, leading to some bubbles [10]. This beverage was made



Citation: Watrelot, A.A.; Hollis, J. Quality Characteristics of Piquette: A Potential Use of Grape Pomace. *Beverages* **2024**, *10*, 64. https:// doi.org/10.3390/beverages10030064

Academic Editors: Vita Di Stefano, Vittorio Farina and Nicola Francesca

Received: 1 July 2024 Revised: 22 July 2024 Accepted: 23 July 2024 Published: 26 July 2024



Copyright: © 2024 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). rapidly during the winemaking season from the common waste, pomace and was given to vineyard and winery workers as a low alcohol fizzy drink. However, this beverage is not popular despite the quantity of pomace wasted every year and the demand from consumers for low-alcohol beverages. In addition, the legislation around the production of piquette is country-dependent. The EU prohibits the production of piquette from grape pomace if not used for distillation or for consumption in wine-producers' households (Regulation (EU) No 1308/2013, annex 8).

In winemaking, many factors impact the final quality of wines, including grape cultivars, location, environment and growing conditions, in addition to the process of winemaking. Yeasts are the key microorganisms in alcoholic fermentation, which transforms juice into wine, with *Saccharomyces cerevisiae* yeasts being the most commonly used species in winemaking. Yeasts produce ethanol, carbon dioxide, and heat during alcoholic fermentation, and are responsible for the production of glycerol and secondary aroma compounds. Higher concentrations of glycerol, up to 15 g/L, are produced by yeasts in red wines due to more oxygen exposure, higher temperature of fermentation, and higher pH than in white wines [11]. In addition, yeast strains can produce diacetyl aroma compounds and hydrogen sulfide due to a lack of nutrients, which could be managed by adding diammonium phosphate (DAP) to wines during the process [12]. However the use of DAP in red wines can lead to low red color stability [13].

In this study, piquettes were produced from different red grape cultivars, different ratios of pomace to water, and different yeast strains to evaluate the impact on quality and consumers' perception of those piquettes. This is the first study focusing on the production of piquette and on their quality parameters. The production of piquette from red grape pomaces could add value to a common waste product, make the industry more sustainable, and increase the profitability of the grape and wine industry.

2. Materials and Methods

2.1. Piquette Process

The list of conditions and sample names are provided in Table 1.

Name	Grape Variety	Yeast Strains	Pomace to Water Ratio	
PPB1		Evotion Monaio	1:5	
PPB2		Exotics mosaic	1:2.5	
PP1	 Petite Pearl	ICV D254		
PP2		Cross Evolution	-	
PP3		Exotics Mosaic	- 1.2	
M1		ICV D254	- 1.2	
M2	Marquette	Cross Evolution	-	
M3	—	Exotics Mosaic	-	

Table 1. List of sample names corresponding to grape variety, yeast strains and pomace to water ratio.

2.2. Impact of Pomace to Water Ratio

Petite Pearl pomace obtained on 7 October 2022 from The Walker Homestead Vineyard and Farm (IA) at pressing using a bladder press was placed into two macrobins. One macrobin contained 90 kg of Petite Pearl pomace (PPB1) and one macrobin contained 136 kg of Petite Pearl pomace (PPB2). In both macrobins, 378.5 L of dechlorinated water, 20 g of potassium metabisulfite, and 200 g of citric acid were added. The contents of both macrobins were mixed and covered for two days at 14 °C. After those two days, the pomace solutions were pressed and Petite Pearl juice obtained from 23 kg of Petite grapes was added to the pomace solutions to obtain a Brix of 12. The juice obtained after pressing was inoculated with Exotics Mosaic yeast strain (Scott Laboratories, Petaluma, CA, USA) at 15 °C. After two days of alcoholic fermentation, 40 g of Fermaid K and 20 g of diammonium phosphate (DAP) were added to each fermenting tank. Due to a hydrogen sulfide smell, 100 g of Fermaid K and 50 g of DAP were added again on the next day. On 15 October 2022, the piquettes (PPB1 and PPB2) were bottled in crown cap, clear glass bottles with an average Brix of 2.1.

2.3. Impact of Yeast Strains

The pomace of Marquette and Petite Pearl grape cultivars from the Iowa State University Horticulture Research Station was stored at -10 °C after pressing, on 1 September 2022 and 15 September 2022, respectively. On 9 December 2022, the GP were thawed in a cold room (4 °C overnight) prior to being placed in 8 L buckets. Two kilograms of Marquette pomace (2 kg) were placed in three 8 L buckets and 2 kg of Petite Pearl pomace were placed in three 8 L buckets. Four liters of de-chlorinated water, 2.5 g of citric acid, and 0.1 g of potassium metabisulfite were added to each bucket. The GP were soaked at 18 °C for two days. The juices were pressed using a benchtop press and 1.2 kg of sugar was added to each bucket to reach 12 Brix. In Marquette and Petite Pearl juices, the yeast assimilable nitrogen (YAN) was evaluated using an enzymatic kit (Megazyme) and was 40 mg/L and 20 mg/L, respectively. Fermaid K (0.3 g/L) was added at the same time as the yeast inoculation. ICV D254 (M1, PP1), Cross Evolution (M2, PP2), and Exotics Mosaic (M3, PP3) yeast strains (Scott Laboratories, Petaluma, CA, USA) were inoculated with Go-Ferm, following the standard provider recommendations, in one bucket of Marquette and one bucket of Petite Pearl juices. The Brix, temperature, and density were recorded during fermentation and used to estimate the time of bottling. On 18 and 19 December 2022, Marquette and Petite Pearl piquettes were bottled in four 750 mL green champagne bottles (Midwest Supplies, Roseville, MN, USA) for each condition, when the Brix was at 1.6 \pm 0.2, and closed with a crown cap. This lower degree Brix was chosen to avoid the strong effervescence observed in the other piquettes bottled at a Brix of 2.1 (in Section 2.1). The bottles were then stored at 15 °C in a wine cooler until chemical analysis and sensory evaluation.

2.4. Chemical Analysis

The Brix of the juices was analyzed using a digital refractometer (Atago® model PAL-1, Tokyo, Japan). Brix remaining at bottling and ethanol content were analyzed using a DMA (Anton Paar USA Inc., Ashland, VA, USA). The pH was measured using a digital pH meter (ThermoScientific® model Orion Star A211, Waltham, MA, USA). The titratable acidity (TA, expressed as g/L tartaric acid equivalents) was determined by titration with a pH meter to a final pH of 8.20 using 0.1 N sodium hydroxide. Wine color parameters, full visible spectrum scans (280 to 700 nm), and CIELab color coordinates were determined in 1 cm path-length UV-Visible cuvettes using a UV-Visible Spectrophotometer (Genesys 150, Thermofisher scientific, Waltham, MA, USA). CIELab coordinates including L* (lightness), a* (green/red component), and b* (blue/yellow component) were calculated using the Visionlite software version 2.0 (Thermofisher scientific, Waltham, MA, USA). The L*, a*, and b* parameters were then converted into color patches using the free color converter software (Nix Color Sensor, Nix Sensor Ltd 2024). Organic acids, residual sugars, and glycerol concentrations in piquettes after 8 months of storage were quantified by reversed-phase high-performance liquid chromatography (HPLC) (1200 series, Agilent Technologies, Santa Clara, CA, USA) with a refractive index detector (RID) (Agilent 1200 series). Briefly, 20 µL of sample was injected at a flow rate of 0.7 mL/min in an isocratic mode of 0.005 M sulfuric acid. The compounds separation was performed on a Hi-Plex H column (Agilent Hi-Plex H, 7.7×300 mm, 8 μ m, Agilent Technologies, Santa Clara, CA, USA) protected with the same material guard column (PL Hi-Plex H Guard Column 7.7 × 50 mm, Agilent Technologies), the oven temperature was 60 $^{\circ}$ C, and the RID was set at 55 $^{\circ}$ C. The concentration of each compound (tartaric acid, malic acid, lactic acid, glucose, fructose, succinic acid, and glycerol) was determined using external calibration curves of standards.

2.5. Sensory Analysis

A consumer sensory evaluation study was carried out on the 8 samples PPB1, PPB2, PP1, PP2, PP3, M1, M2, M3 after 8 months of aging. All study procedures were approved by the Iowa State University Institutional Review Board (IRB ID 23-149) and all participants provided written consent. The sensory evaluation was composed of four 1-h sessions over a period of one week. Fifteen participants were recruited through an email distributed to all faculty, staff, and students at Iowa State University. To be included in the study, participants had to be older than 21 years of age, fluent in English, and not have a diagnosed color, smell, and/or taste disorder. Participants were asked not to eat, drink, or smoke for at least one hour prior to the sensory evaluation sessions. Participants were compensated for their participation in the study with a gift card. At the beginning of the first session, demographic information was collected using a questionnaire that posed questions about gender, ethnicity, age, occupation, and the number of times per month the participant consumes sparkling wine, sparkling beer, and sparkling water. At each of the 4 sessions, 5 or 6 samples were evaluated. Participants were asked to take a sip of the product, evaluate the pleasantness of taste and mouthfeel, and expectorate the product. Participants evaluated the pleasantness of the color, appearance, and aroma of the samples using a 9-point hedonic scale (anchored with "dislike extremely" (1) and "like extremely" (9)). Appearance included the fizziness and cloudiness of the product. Participants were also asked to answer some questions about their preferences regarding the samples and how much and why they would pay for a 750 mL bottle of the sample. This questionnaire was administered using Compusense software v23.0.26998 19 April 2023 (Compusense Inc., Guelph, ON, Canada). Participants had a 2 min break between samples to cleanse their palate with water and crackers before being provided the next sample to evaluate. This process was repeated until all the samples were evaluated.

2.6. Statistical Analysis

Data were collected as analytical replicates or biological replicates as noted where appropriate. Means and standard errors were calculated for all chemical variables. Chemical and sensory data were analyzed using a one-way ANOVA followed by Tukey's HSD post-hoc test to identify differences between samples. Means and standard deviations are presented for all sensory data. Relationships between sensory data and the chemical/physical characteristics of the samples were determined using the Pearson correlation coefficient. Statistical analysis was completed on JMP Pro 17 software (SAS Institute Inc., Cary, SC, USA). Statistical significance was set at $\alpha < 0.05$.

3. Results

3.1. Chemical Parameters

The chemical parameters of piquette samples after 8 months of storage are shown in Tables 2 and 3. The pH of the samples ranged from 3.35 to 3.61, with a statistically significant main effect of sample (F(7,20) = 14.5, p < 0.0001), with a pH higher in PPB1 than the other samples (p < 0.0001). The titratable acidity of the samples ranged from 5.15 g/L tartaric acid eq. to 6.75 g/L tartaric acid eq., with a statistically significant main effect of sample (F(7,20) = 12.9, p < 0.0001). Titratable acidity was higher in PPB1 and PPB2 than the other samples (p < 0.0001). In addition, the titratable acidity was higher in PPB2 compared to PP3 (p < 0.05) and M3 (p < 0.05). There was no statistically significant main effect of sample on ethanol concentration (F(7,20) = 2.5, p = 0.08).

Samples	рН	Titratable Acidity (g/L Tartaric Acid eq.)	Ethanol Conc. (Vol %)	L*	a*	b*
M1	$3.36\pm0.00b$	$6.10\pm0.09\mathrm{bc}$	4.77 ± 0.06	69.90 ± 2.00 a	$23.90\pm0.52~\mathrm{ab}$	$14.55\pm0.21~\mathrm{c}$
M2	$3.39\pm0.01~\mathrm{b}$	$5.70\pm0.42\mathrm{bc}$	4.60 ± 0.00	$69.65\pm0.92~\mathrm{ab}$	$22.49\pm0.30~\text{abc}$	$15.47\pm0.18~\mathrm{bc}$
M3	$3.38\pm0.02b$	$5.15\pm0.71~\mathrm{c}$	4.77 ± 0.21	$74.43\pm2.36~\mathrm{a}$	$21.60\pm0.62~abc$	$14.45\pm0.75~\mathrm{c}$
PP1	$3.35\pm0.00~\text{b}$	$6.30\pm0.21\mathrm{bc}$	3.50 ± 0.00	$56.35\pm7.99~\mathrm{ab}$	$22.14 \pm 1.96~\mathrm{abc}$	$7.04\pm0.16~\mathrm{d}$
PP2	$3.38\pm0.00~b$	$5.33\pm0.95\mathrm{bc}$	4.15 ± 1.28	$43.85\pm0.07b$	$20.19\pm0.17~\mathrm{abc}$	$6.88\pm0.26~\mathrm{d}$
PP3	$3.38\pm0.01b$	$5.15\pm0.48~{\rm c}$	4.83 ± 0.15	$53.00\pm16.89~\mathrm{ab}$	$26.39\pm5.73~\mathrm{a}$	$5.84\pm0.24~d$
PPB1	3.61 ± 0.03 a	$6.75\pm0.52\mathrm{b}$	3.63 ± 0.35	59.83 ± 1.84 ab	$15.07\pm0.21\mathrm{bc}$	$16.55\pm0.67\mathrm{b}$
PPB2	$3.48\pm0.12b$	$9.08\pm0.53~\mathrm{a}$	3.95 ± 0.00	$60.10\pm2.26~\text{ab}$	$17.62\pm0.62~\mathrm{c}$	$19.60\pm0.46~\mathrm{a}$

Table 2. Chemical and CIELab parameters of piquette samples at the time of sensory evaluation (i.e., after 8 months of storage).

Means followed by the same letter within columns are not significantly different according to Tukey's HSD p > 0.05. Values are listed as mean \pm standard error of three replicates.

Table 3. Concentrations (g/L) of organic acids, residual sugars, and glycerol in piquette samples at the time of sensory evaluation.

Samples	Tartaric Acid	Fructose	Lactic Acid	Acetic Acid	Glycerol
M1	$1.55\pm0.08~\mathrm{a}$	$0.39\pm0.00~{ m bc}$	$0.06 \pm 0.01 \text{ a}$	$0.10\pm0.00~\mathrm{ab}$	$10.60 \pm 0.05 \text{ b}$
M2	1.15 ± 0.19 a	$0.45\pm0.11~\mathrm{b}$	$0.06\pm0.01~\mathrm{a}$	$0.08\pm0.01~\mathrm{ab}$	$10.77\pm0.32~\mathrm{b}$
M3	1.28 ± 0.11 a	$0.25\pm0.02~bcd$	$0.05\pm0.03~\mathrm{a}$	$0.07\pm0.00~\mathrm{b}$	$10.48\pm0.07~\mathrm{b}$
PP1	$1.07\pm0.04~\mathrm{a}$	$1.93\pm0.20~\mathrm{a}$	$0.04\pm0.00~\mathrm{a}$	$0.15\pm0.00~\mathrm{ab}$	13.24 ± 0.24 a
PP2	$1.05\pm0.10~\mathrm{a}$	$0.00\pm0.00~{ m d}$	$0.04\pm0.01~\mathrm{a}$	$0.07\pm0.00~\mathrm{ab}$	$10.63\pm0.11~\mathrm{b}$
PP3	$1.18\pm0.22~\mathrm{a}$	$0.13\pm0.11~\text{cd}$	$0.04\pm0.00~\mathrm{a}$	$0.08\pm0.00~ab$	$10.22\pm0.19b$
PPB1	$1.00\pm0.27~\mathrm{a}$	$0.23\pm0.03~\mathrm{bcd}$	$0.09\pm0.03~\mathrm{a}$	$0.13\pm0.09~\mathrm{ab}$	$8.41\pm1.10~{\rm c}$
PPB2	$1.25\pm0.30~\mathrm{a}$	$0.05\pm0.07~\mathrm{d}$	$0.09\pm0.00~\mathrm{a}$	0.21 ± 0.00 a	$7.91\pm0.30~\mathrm{c}$

Means followed by the same letter within columns are not significantly different according to Tukey's HSD p > 0.05. Values are listed as mean \pm standard error of three replicates.

The L* values of the samples ranged from 43.85 to 74.43, with a statistically significant main effect of sample (F(7,20) = 4.5, p = 0.01). L* was higher in M3 than PP2 (p < 0.0001). The a* values ranged from 15.07 to 23.90, with a statistically significant main effect of sample (F(7,20) = 6.0, p = 0.004). a* was higher in PP3 than PPB1 (p = 0.002), higher in M1 compared to PPB1 (p = 0.01), and higher in PP3 compared to PPB2 (p = 0.03). The b* values ranged from 5.84 to 19.60 with a statistically significant main effect of sample (F(7,20) = 300.1, p < 0.0001). b* was higher in PPB2 compared to all the other samples (p < 0.003), PPB1 was higher than M1, M3, PP1, PP2, and PP3 (p < 0.0001) and PPB2, PPB1, M2, M1, and M3 were higher than PP1, PP2, and PP3 (p < 0.0001).

In all the samples, no malic acid, glucose, or succinic acid was detected and therefore not shown. The tartaric acid concentrations of the samples ranged from 1.00 to 1.55 g/L (Table 3), with no statistically significant main effect of sample (F(7,20) = 1.8, p = 0.18). The concentrations of fructose ranged from 0 to 1.93 g/L, with a statistically significant main effect of sample (F(7,20) = 98.1, p < 0.0001). Fructose concentration was higher in PP1 compared to the other samples (p < 0.0001). Moreover, M2 was higher than PP3, PPB2, and PP2 (p < 0.01), and M1 was higher than PPB2 and PP2 (p < 0.01). The lactic acid concentrations ranged from 0.04 to 0.06 g/L, with a significant main effect of sample (F(7,20) = 3.2, p = 0.03). However, post-hoc analysis failed to reveal any statistically significant differences between the samples. The acetic acid concentrations ranged from 0.07 to 0.15 g/L, with a statistically significant main effect of sample (F(7,20) = 3.2, p = 0.03). However, post-hoc analysis failed to reveal any statistically significant differences between the samples. The acetic acid concentrations ranged from 0.07 to 0.15 g/L, with a statistically significant main effect of sample (F(7,20) = 3.1, p = 0.04). Acetic acid concentration was higher in PPB2 compared to M3 (p < 0.049). The glycerol values ranged from 7.91 to 13.24 g/L, with a statistically significant main effect of sample (F(7,20) = 24.0, p < 0.0001). Glycerol concentration was higher in PP1 compared to the other samples (p < 0.0001). In

addition, M2, PP2, M1, M3, and PP3 were higher in glycerol concentration compared to PPB1 and PPB2 (p < 0.01).

3.2. Sensory Evaluation

Participants for the sensory study were 5 males and 10 females, including 11 white and 2 Asian/Asian American individuals. The participants' ages ranged between 25 to 75 years old with an average of 39 years old (standard deviation = 12). Forty percent of the panelists mentioned consuming sparkling wine once a month and 40% consumed sparkling wine fewer times than once a month. Forty percent of the panelists reported consuming sparkling beer fewer than once a month. Twenty percent of the panelists reported consuming sparkling water three times a week, thirteen percent consumed sparkling water daily, and thirteen percent consumed it fewer than one time each month.

Piquette characteristics evaluated by sensory evaluation are provided in Table 4. The color of piquette samples was statistically different (F(7,119) = 5.25, p < 0.0001), with the color of M3 and PPB1 being rated statistically lower than M1 and than PP1, PP2, and PP3. There was a significant main effect of sample on appearance (F(7,119) = 11.15, p < 0.0001). The appearance of M3 piquette was rated the lowest by participants. PP1, PP2, and PP3 were all rated statistically higher than PPB1 and M3. There was a significant main effect of sample on smell (F(7,119) = 7.32, p < 0.0001). The smell of M1 and PP1 was statistically rated higher than the smell of PPB1 and PPB2. The smell of PPB2 was rated statistically the lowest. There was a significant main effect of sample on taste (F(7,119) = 3.74, p = 0.004). The taste of PP1 was rated statistically higher than the taste of PPB2. There was not a significant main effect of sample on mouthfeel (F7,1) = 1.96, p = 0.06). There was a significant main effect of sample on acceptability (F(7,119) = 6.53, p < 0.0001). Tukey's HSD test for multiple comparisons found mean differences between M1 and M3 (p = 0.02), M2 and M3 (p = 0.005), M3 and PP1 (p < 0.0001), M3 and PP3 (p = 0.005) and PP1 and PPB2 (p = 0.009). No other comparisons were statistically significant.

Table 4. Sensory characteristics of piquette samples evaluated by panelists.

Samples	Color	Appearance	Smell	Taste	Mouthfeel	Acceptability
M1	7.4 ± 1.2 a	$6.2\pm2.1~\mathrm{ab}$	7.0 ± 1.0 a	$6.2\pm1.6~\mathrm{ab}$	$6.6\pm1.6~\mathrm{ab}$	$6.3\pm1.6~\mathrm{ab}$
M2	$7.1\pm1.2~\mathrm{ab}$	$6.3\pm1.7~\mathrm{ab}$	$6.4\pm1.2~\mathrm{ab}$	$6.4\pm1.2~\mathrm{ab}$	$6.5\pm1.5~\mathrm{ab}$	$6.5\pm1.3~\mathrm{ab}$
M3	$6.5\pm1.8~\mathrm{b}$	$4.5\pm2.5~c$	$5.6\pm2.5~\mathrm{ab}$	$5.6\pm1.9~\mathrm{ab}$	$5.8\pm2.0b$	$5.1\pm1.9~\mathrm{c}$
PP1	7.4 ± 1.3 a	6.5 ± 1.9 a	6.6 ± 1.6 a	6.8 ± 1.4 a	$6.9\pm1.7~\mathrm{a}$	6.7 ± 1.6 a
PP2	7.3 ± 1.6 a	$6.3\pm1.9~\mathrm{a}$	$5.4\pm2.5~\mathrm{ab}$	$5.6\pm2.1~\mathrm{ab}$	$6.3\pm1.8~\mathrm{ab}$	$5.7\pm2.0~\mathrm{abc}$
PP3	$7.6\pm1.7~\mathrm{a}$	$7.1\pm1.9~\mathrm{a}$	$6.7\pm1.6~\mathrm{ab}$	$6.4\pm1.8~\mathrm{ab}$	$6.6\pm2.0~ab$	$6.7\pm1.9~\mathrm{ab}$
PPB1 PPB2	$6.0 \pm 1.8 \text{ b} \\ 5.8 \pm 2.0 \text{ ab}$	$\begin{array}{c} 4.4\pm1.9~\text{bc}\\ 4.8\pm2.0~\text{ab} \end{array}$	$\begin{array}{c} 4.4\pm2.7\ \mathrm{bc}\\ 3.5\pm2.2\ \mathrm{c} \end{array}$	$5.0\pm2.1~\mathrm{ab}$ $4.5\pm2.1~\mathrm{b}$	$5.8 \pm 1.9~\mathrm{ab}$ $5.4 \pm 2.0~\mathrm{ab}$	$4.8\pm2.2~\mathrm{abc}$ $4.4\pm2.2~\mathrm{bc}$

Means followed by the same letter within columns are not significantly different according to Tukey's HSD p > 0.05. Values are listed as mean \pm standard deviation of three replicates.

In order to provide a better understanding of the color of samples, the L*, a*, and b* parameters shown in Table 2 have been converted to color (Figure 1).



Figure 1. Color swatches of the piquette samples obtained after the conversion of the L*, a*, and b* parameters to color.

7 of 10

Correlations between the chemical parameters and the sensory characteristics of piquettes found that the ethanol concentration and the lightness L* parameter were not correlated with any sensory descriptors (Table 5). The color of piquettes perceived by panelists was negatively correlated with pH (p = 0.0226) and b* (p = 0.0167), and positively correlated with a* (p = 0.0081). The smell of piquettes was significantly negatively correlated with pH (p = 0.0380) and with titratable acidity (p = 0.0421), and positively correlated with the a* parameter (p = 0.0053). The taste was significantly negatively correlated with the pH (p = 0.0387) but not with the titratable acidity. The acceptability of piquettes was positively correlated with the a* color parameter (p = 0.0115).

Table 5. Pearson's correlation coefficients between chemical and sensory parameters of all piquettes. L*, a*, and b* are the CIELab color parameters.

Correlation	Color	Appearanc	e Smell	Taste	Mouthfeel	Acceptability
pH Titratable Acidity Ethanol (vol%)	$-0.779 * -0.701 \\ 0.328$	-0.679 -0.458 0.272	$-0.734 * \\ -0.724 * \\ 0.487$	-0.732 * -0.685 0.317	$-0.630 \\ -0.577 \\ 0.184$	$-0691 \\ -0.604 \\ 0.303$
L* a* b*	-0.243 0.846 ** -0.801 *	-0.400 0.787 * -0.751 *	0.146 0.867 ** -0.584	0.102 0.805 * -0.623	-0.091 0.709 * -0.630	-0.095 0.826 * -0.680

* = *p*-value < 0.05, ** = *p*-value < 0.01.

4. Discussion

Marquette is an interspecific hybrid red grape cultivar predominantly grown and used to make red wines in the US Midwest [3]. Due to its genetic background (*Vitis vinifera* crossed with *Vitis riparia* species), the grapes produced tend to be acidic, rich in anthocyanin mono- and di-glucosides in the skins and flesh, and are poor in tannins [14]. It also contains high concentrations of terpenes that are aroma compounds associated with the "aromatic" grape varieties such as Muscat, Riesling, and Gewürztraminer [15]. Little is known about wine characteristics made from Petite Pearl red grapes, as this is also an interspecific hybrid grape released in 2009. This grape cultivar tends to have a lower acidity level than Marquette, have a different optimal harvest maturity, and a different color profile [16].

The chemical characteristics of piquettes made with different cultivars, yeast strains, and ratios of pomace to water were correlated with the sensory evaluation data. The pH of these piquettes was associated with the concentration of organic acids present in the pomace after pressing and the citric acid content added before pressing. Traces of lactic acid were found in these piquettes, which likely was from the lactic acid produced by malolactic bacteria during the winemaking process and remained in the pomace after pressing. The main differences in acidity were observed in Petite Pearl piquettes made with different ratios of pomace to water. PPB2, which was made with the highest amount of pomace, contained slightly more tartaric acid, even though it was not significantly different from PPB1, but also had significantly higher titratable acidity and lower pH. However, this difference in acidity between those piquettes was not perceived by panelists, most likely due to the different acids and pH that provide different levels of sourness and overall acceptability [17]. The color parameters in grapes and wines are related to the presence of pigments. The main pigments in grape skins are anthocyanins that are monoglucoside forms in *Vitis vinifera* grape cultivars, while interspecific hybrid cultivars are rich in mono- and di-glucoside forms, which do not provide the same color intensity and stability to wines [18,19]. Marquette grapes have been shown to contain fewer monomeric anthocyanins than Petite Pearl grapes and therefore have a lower color intensity than Petite Pearl [16,20]. In piquettes, the opposite was observed, with the lowest L^* and b^* parameters observed in Petite Pearl piquettes compared to Marquette piquettes, suggesting that piquettes of Petite Pearl were less color intense and more purple. This might be explained by the extraction of most of the anthocyanins and pigments from grape skins and flesh occurring in wines throughout alcoholic fermentation, and the color parameters of the

piquettes being based on only remaining pigments from the pomace [21]. However, due to the technique of piquette processing used in this study, phenolic compounds have been extracted from pomace only by diffusion of pomace into water, and therefore a very low concentration of phenolics was found in those piquettes (below the limit of quantification). It was interesting to observe that the pH of the piquettes was significantly correlated with color and taste of piquettes, but that titratable acidity was not. pH is known to be an important chemical characteristic influencing the structure of anthocyanins, leading to a wine with a more red color at low pH compared to a more blue color at a high pH [22]. The ethanol concentration in all piquettes was low as expected, due to the low initial amount of sugar added to the pomace prior to alcoholic fermentation, and it was not significantly different in the different conditions, most likely because the yeast strains used are tolerant to low alcohol and consume the sugar at the same rate. Ethanol was not correlated with any sensory parameters, most likely due to the low concentration in those piquettes, as it has been previously shown that ethanol content of 9% and above increases the acceptance of wine and the duration of aroma compounds perception [23].

Glycerol is an important component of the wine matrix and may lead to some perceived sweetness in wines, and it can play a critical role as an osmoprotectant during fermentation [24]. The production of glycerol varies depending on the yeast strains, as observed in this study. Low glycerol-producing yeasts, e.g., ICV D254, produce between 6 to 7.5 g/L of glycerol, as per the supplier description. High glycerol-producing yeast, e.g., Cross Evolution, produce between 7.5 and 9.5 g/L of glycerol, as per the supplier description, and very high glycerol-producing yeasts, e.g., Exotics Mosaic, produce between 9 and 13 g/L of glycerol, as per the supplier description. Therefore, it was expected to observe a higher concentration of glycerol in piquettes made from the Exotics Mosaic yeast strain. However, the high concentration of glycerol was observed only in Petite Pearl PP1 and not in Marquette M1. This difference could be explained by the highest content of fructose remaining in the piquette PP1, as the production of glycerol increases when the concentration of sugars increases. Overall, the acceptability of the piquettes by panelists was highly correlated with the a* value, which was the highest in PP3 and corresponded to a more red color. It has been previously shown that the color of rosé wine is highly related to consumer preferences, as a pink salmon shade is a marker of Provence rosé but does not represent a unanimous preference [25,26].

Therefore, it seems that piquette produced from Petite Pearl pomace using Exotics Mosaic (PP3) was the most accepted by panelists. This was confirmed by the price that consumers would be willing to pay for each piquette. PP3 was the piquette for which 10% of the panelists would be willing to pay \$20 or more; 37% of panelists were willing to pay between \$15 to \$19.99 for PP1; 46% of the panelists were willing to pay between \$10 and \$14.99 for M1 and M2; and more than 50% of the panelists were willing to pay less than \$10 for the other piquettes, especially PPB1 and PPB2, which were the least appreciated, most likely due to the appearance, smell, and mouthfeel.

Author Contributions: Conceptualization, A.A.W.; methodology, A.A.W.; software, J.H.; validation, A.A.W. and J.H.; formal analysis, A.A.W. and J.H.; investigation, A.A.W.; resources, A.A.W. and J.H.; data curation, A.A.W. and J.H.; writing—original draft preparation, A.A.W.; writing—review and editing, A.A.W. and J.H.; visualization, A.A.W.; supervision, A.A.W.; project administration, A.A.W. All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Institutional Review Board Statement: The study was conducted in accordance with the Declaration of Helsinki and approved by the Institutional Review Board of Iowa State University (IRB ID 23-149) on 16 May 2023.

Informed Consent Statement: Informed consent was obtained from all subjects involved in the study.

Data Availability Statement: The datasets generated and/or analyzed during the current study are available from the corresponding author on reasonable request.

Acknowledgments: Kent Foulker from Cedar Ridge Distillery and Winery, IA is gratefully acknowledged for his generous donation of piquette and yeast strains, and for going through the process of making piquette with us for this project. We would like to thank Yiliang Cheng and Julain Sinkler of Iowa State University for assistance in chemical analysis. This work is a product of the Iowa Agriculture and Home Economics Experiment Station, Ames, Iowa, USA, project No. IOW05647, which is sponsored by State of Iowa funds.

Conflicts of Interest: The authors declare no conflicts of interest.

References

- Jin, Q.; O'Keefe, S.F.; Stewart, A.C.; Neilson, A.P.; Kim, Y.-T.; Huang, H. Techno-Economic Analysis of a Grape Pomace Biorefinery: Production of Seed Oil, Polyphenols, and Biochar. *Food Bioprod. Process.* 2021, 127, 139–151. [CrossRef]
- Beres, C.; Costa, G.N.S.; Cabezudo, I.; da Silva-James, N.K.; Teles, A.S.C.; Cruz, A.P.G.; Mellinger-Silva, C.; Tonon, R.V.; Cabral, L.M.C.; Freitas, S.P. Towards Integral Utilization of Grape Pomace from Winemaking Process: A Review. *Waste Manag.* 2017, 68, 581–594. [CrossRef]
- 3. Watrelot, A. Iowa Wine Industry and Its Educational Needs. J. Ext. 2022, 60, 13. [CrossRef]
- Bordiga, M.; Travaglia, F.; Locatelli, M. Valorisation of Grape Pomace: An Approach That Is Increasingly Reaching Its Maturity–A Review. Int. J. Food Sci. Technol. 2019, 54, 933–942. [CrossRef]
- Lutz, M.; Fuentes, E.; Ávila, F.; Alarcón, M.; Palomo, I. Roles of Phenolic Compounds in the Reduction of Risk Factors of Cardiovascular Diseases. *Molecules* 2019, 24, 366. [CrossRef]
- Nakov, G.; Brandolini, A.; Hidalgo, A.; Ivanova, N.; Stamatovska, V.; Dimov, I. Effect of Grape Pomace Powder Addition on Chemical, Nutritional and Technological Properties of Cakes. LWT 2020, 134, 109950. [CrossRef]
- Walker, R.; Tseng, A.; Cavender, G.; Ross, A.; Zhao, Y. Physicochemical, Nutritional, and Sensory Qualities of Wine Grape Pomace Fortified Baked Goods. J. Food Sci. 2014, 79, S1811–S1822. [CrossRef]
- 8. Acan, B.G.; Kilicli, M.; Bursa, K.; Toker, O.S.; Palabiyik, I.; Gulcu, M.; Yaman, M.; Gunes, R.; Konar, N. Effect of Grape Pomace Usage in Chocolate Spread Formulation on Textural, Rheological and Digestibility Properties. *LWT* **2021**, *138*, 110451. [CrossRef]
- 9. Harutyunyan, M.; Viana, R.; Granja-Soares, J.; Martins, M.; Ribeiro, H.; Malfeito-Ferreira, M. Adaptation of Ancient Techniques to Recreate 'Wines' and 'Beverages' Using Withered Grapes of Muscat of Alexandria. *Fermentation* **2022**, *8*, 85. [CrossRef]
- 10. Pickard, C. What Is Piquette? Meet Wine's Easy-Drinking, Low-Alcohol Style | Wine Enthusiast. Available online: https://www.wineenthusiast.com/culture/wine/piquette-easy-drinking-low-alcohol-wine/ (accessed on 4 May 2024).
- 11. Nieuwoudt, H.H.; Prior, B.A.; Pretorius, L.S.; Bauer, F.F. Glycerol in South African Table Wines: An Assessment of Its Relationship to Wine Quality. S. Afr. J. Enol. Vitic. 2017, 23, 22–30. [CrossRef]
- 12. Swiegers, J.; Bartowsky, E.; Henschke, P.; Pretorius, I. Yeast and Bacterial Modulation of Wine Aroma and Flavour. *Aust. J. Grape Wine Res.* 2005, *11*, 139–173. [CrossRef]
- Carrau, F.; Gaggero, C.; Aguilar, P.S. Yeast Diversity and Native Vigor for Flavor Phenotypes. *Trends Biotechnol.* 2015, 33, 148–154. [CrossRef]
- 14. Watrelot, A.A. Tannin Content in Vitis Species Red Wines Quantified Using Three Analytical Methods. *Molecules* **2021**, *26*, 4923. [CrossRef] [PubMed]
- Slegers, A.; Angers, P.; Ouellet, É.; Truchon, T.; Pedneault, K. Volatile Compounds from Grape Skin, Juice and Wine from Five Interspecific Hybrid Grape Cultivars Grown in Québec (Canada) for Wine Production. *Molecules* 2015, 20, 10980–11016. [CrossRef] [PubMed]
- 16. Scharfetter, J.; Workmaster, B.A.; Atucha, A. Preveraison Leaf Removal Changes Fruit Zone Microclimate and Phenolics in Cold Climate Interspecific Hybrid Grapes Grown under Cool Climate Conditions. *Am. J. Enol. Vitic.* **2019**, *70*, 297–307. [CrossRef]
- CoSeteng, M.Y.; McLellan, M.R.; Downing, D.L. Influence of Titratable Acidity and pH on Intensity of Sourness of Citric, Malic, Tartaric, Lactic and Acetic Acids Solutions and on the Overall Acceptability of Imitation Apple Juice. *Can. Inst. Food Sci. Technol. J.* 1989, 22, 46–51. [CrossRef]
- 18. Manns, D.C.; Lenerz, C.T.M.C.; Mansfield, A.K. Impact of Processing Parameters on the Phenolic Profile of Wines Produced from Hybrid Red Grapes Maréchal Foch, Corot noir, and Marquette. *J. Food Sci.* **2013**, *78*, C696–C702. [CrossRef]
- 19. Burtch, C.E.; Mansfield, A.K.; Manns, D.C. Reaction Kinetics of Monomeric Anthocyanin Conversion to Polymeric Pigments and Their Significance to Color in Interspecific Hybrid Wines. J. Agric. Food Chem. 2017, 65, 6379–6386. [CrossRef]
- 20. Rice, S.; Koziel, J.A.; Dharmadhikari, M.; Fennell, A. Evaluation of Tannins and Anthocyanins in Marquette, Frontenac, and St. Croix Cold-Hardy Grape Cultivars. *Fermentation* **2017**, *3*, 47. [CrossRef]
- Busse-Valverde, N.; Gómez-Plaza, E.; López-Roca, J.M.; Gil-Muñoz, R.; Bautista-Ortín, A.B. The Extraction of Anthocyanian and Proanthocyanidins from Grapes to Wine during Fermentative Maceration Is Affected by the Enological Technique. *J. Agric. Food Chem.* 2011, 59, 5450–5455. [CrossRef]
- 22. Khoo, H.E.; Azlan, A.; Tang, S.T.; Lim, S.M. Anthocyanidins and Anthocyanins: Colored Pigments as Food, Pharmaceutical Ingredients, and the Potential Health Benefits. *Food Nutr. Res.* **2017**, *61*, 1361779. [CrossRef]
- Baker, A.K.; Ross, C.F. Wine Finish in Red Wine: The Effect of Ethanol and Tannin Concentration. Food Qual. Prefer. 2014, 38, 65–74. [CrossRef]

- 24. Tilloy, V.; Ortiz-Julien, A.; Dequin, S. Reduction of Ethanol Yield and Improvement of Glycerol Formation by Adaptive Evolution of the Wine Yeast Saccharomyces Cerevisiae under Hyperosmotic Conditions. *Appl. Environ. Microbiol.* **2014**, *80*, 2623–2632. [CrossRef]
- 25. Fan, S.; Liu, C.; Li, Y.; Zhang, Y. Visual Representation of Red Wine Color: Methodology, Comparison and Applications. *Foods* **2023**, *12*, 924. [CrossRef]
- 26. Peres, S.; Giraud-Heraud, E.; Masure, A.-S.; Tempere, S. Rose Wine Market: Anything but Colour? Foods 2020, 9, 1850. [CrossRef]

Disclaimer/Publisher's Note: The statements, opinions and data contained in all publications are solely those of the individual author(s) and contributor(s) and not of MDPI and/or the editor(s). MDPI and/or the editor(s) disclaim responsibility for any injury to people or property resulting from any ideas, methods, instructions or products referred to in the content.