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Influence of Harvest Date and Grape Variety on Sensory Attributes and Aroma Compounds in Experimental Icewines of Ukraine

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Abstract: Icewine is a sweet dessert wine whose sensory and chemical attributes are caused by technology peculiarities including the special climatic conditions and acceptable grape varieties. This study aimed to evaluate the sensory characteristics and aromatic compounds in experimental icewines produced from the grape varieties Rkatsiteli, Telti kuruk, Marselan, and Moldova that had been picked in the Odessa region at temperatures of at least minus 7 °C during the 2015 (H1) and 2016 (H2) harvests. Sensory attributes were determined by trained experts, and descriptors for icewines were chosen by consensus. A total of 33 aromatic compounds including 12 that exceeded the threshold were identified using chromatographic analysis. Rkatsiteli icewine obtained from H1 was characterized by the highest concentration of geraniol, 1-octanol, and 2-phenyl acetate, influencing the pronounced citrus and sweet fruit aromas in sensory profiles. The highest concentrations of ethyl hexanoate and ethyl octanoate associated with aromas of dried fruits were detected in Moldova and Marselan icewines made from H2. No 1-hexanol and benzyl alcohol were found in Rkatsiteli and Telti kuruk icewines. Only Moldova and Marselan icewines had γ-nonalactone and benzaldehyde, respectively. Sensory parameters and the quantity of aromatic compounds of Ukrainian experimental icewines depended on harvest date and grape varieties.

Keywords: icewine; grape variety; harvest; aroma; sensory attributes

1. Introduction

Icewine is a sweet dessert wine obtained from frozen grapes. The most important indicators of the possibility of icewine production by the natural freezing of grapes are the presence of suitable low temperatures and cold-resistant cultivars. In Canada, winemakers are allowed to harvest grapes with a required sugar concentration of a minimum of 32 °Brix at a temperature below or equal to minus 8 °C [1], according to rules of International Organisation of Vine and Wine (OIV)—at a temperature below or equal to minus 7 °C with a grape sugar content minimum of 25 Brix [2]. Rkatsiteli, Telti kuruk, Marselan, and Moldova grown in the south of Ukraine were chosen in this work due to their ampelographic characteristics acceptable for icewine grapes, on the one hand, and for the purpose of studying their winter hardiness in the climatic conditions of the area, on the other hand.

Rkatsiteli, due to its genetic characteristics, has a thick white skin that meets the requirements of grape varieties for icewine [3]. In addition to the late maturation and thick dark skin, the uniqueness of the Marselan variety lies in the limited study of its parameters, which impacts its research in icewine technology [4]. Marselan originated from the cross-breeding of Cabernet Sauvignon and Grenache in France [5]. Another dark-skinned grape variety, Moldova, is resistant to many diseases of grapes, which is also one of the conditions for leaving the vine before the first frosts [6]. Telti kuruk is a late ripening and an autochthonous white grape variety of Ukraine [6]. It should be noted that Riesling and Vidal are the most commonly used cultivars in icewine production in Canada [7].

The influence of temperature on the composition and quality of grapes and wines has been studied by numerous researchers [8–11]. It is important to note that no significant climatic changes can affect grape mechanical properties, which absolutely impacts wine technology, and hence the quality of the wine [10,11]. The icewine technology of the natural freezing of grapes depends on the frost resistance of the grape variety and the presence of minus temperatures, which should be in the range from minus 7 to minus 12 °C [2,4,5,12]. Agroclimatic factors determine the possibility of frozen grape harvesting, namely, the absence of frost or the grape unsuitability to withstand a long season before the first frosts pose the greatest risk [13]. Therefore, the natural method of freezing grapes is difficult with respect to accurately predicting the weather, as well as harvesting grapes before sunrise, which affects the complexity of the technology. The study of the peculiarities of the Northern Black Sea Coast, including the Odessa region, as a region with the necessary resources to implement the comprehensive development of winemaking and viticulture, is the aim of the investigations of Ukrainian scientists [14,15]. However, the meteorological conditions of the Northern Black Sea Coast of Ukraine, as a wine-growing and viticulture territory, have not been sufficiently studied to ensure the development of icewine production.

The main groups of aromatic compounds of icewine are higher and terpene alcohols, esters, acetates and acetals, aromatic aldehydes and ketones, lactones, and volatile phenolic acids [16–18]. Many factors can impact the wine quality including the grape variety, yeasts, winemaking techniques, harvest date, and chemical compositions [16–23]. Ethyl benzoate, ethyl octanoate, ethyl hexanoate, isoamyl acetate, and 2-phenyl acetate create honey and fruit aromas, the intensity of which is influenced by the method of freezing grapes [17]. Thus, depending on the grape variety and the date of collection in the frozen state, the mass concentrations of esters can vary in significant ranges: 1.3–1105 μg/L [20–22]. Most researchers identified compounds responsible for the aroma of dried fruits and honey in icewines [20–23]. Analysis of the chromatographic investigations of volatile aromatic substances shows that the concentrations of isoamyl acetate, hexyl acetate, ethyl 2-methylbutyrate, ethyl 3-methylbutyrate, as well as linalool and cis-rose oxide, differ significantly in wines obtained from the use of different strains of yeast [2,21,23].

The study of the organoleptic characteristics of icewines is of particular interest. Research on the sensory properties of wines made by different methods of freezing grapes shows a difference in the intensity of citrus and floral aromas [20–22], and the perceptible aromas of dried fruits in icewines indicate the maturation of grapes on the vine [18]. Intense aromas of apricot, honey, and pineapple create the aromatic profile of Canadian ice wines [24,25], and descriptors of tropical fruits, caramels, and roses are typical for icewines from China [18–20]. In addition, floral, chemical, pungent, and ripe fruit aromas were found in Gewürztraminer icewines from Croatia [17]. Thus, the physicochemical and organoleptic properties of icewines can differ significantly depending on the grape variety, harvest dates, growing area, method of freezing grapes, and yeast strain.

The aim of the current work was to determine the sensory attributes and aromatic compounds of experimental icewines produced under climate conditions in the south of Ukraine depending on the harvest date and grape variety.

2. Materials and Methods

2.1. Grapes

The experimental icewines made from Rkatsiteli, Telti kuruk, Marselan, and Moldova grapes were harvested twice from vineyards of the "Industrial-Trading Company Shabo" Ltd. in Odessa region: 30 December of 2015 (H1) and 4 January of 2016 (H2). Rkatsiteli, Telti kuruk, Marselan, and Moldova grown in the south of Ukraine were chosen in this work due to their ampelographic characteristics acceptable for icewine grapes, on the one hand, and for the purpose of studying their winter hardiness under the climatic conditions of the area, on the other hand. The characteristics of the grape varieties used are shown in Table 1. The grapes were picked when the air temperature was lower than minus 7 °C according to the definition of the vitivinicultural products code sheet of OIV [1]: minus 7.5 °C for

H1 and minus 12 °C for H2. These dates were chosen to obtain the minimum required level of sugar of at least 25 °Brix and to determine the contributions to the diversity of icewine composition including the quantity of the aromatic compounds. In addition, the temperatures were the lowest and the highest ones during the icewine crop growth period. Grapes were pressed by cultivar using a pneumatic press with periodic action of the closed type, i.e., BucherPlus (Bucher, Niederweningen, Switzerland).

Table 1. Description of grape variety, maturation, and species of *Vitis*.

Grape Variety	Maturation Period, Days [6]	The Origin of Grapes and a Brief Ampelographic Description
Rkatsiteli	Late maturing, 155–170	100%—*V. vinifera*. Technical white grape variety. The cluster is small, long, conical. The berry is round or oval. The skin is thin, very strong. The flesh is juicy. The variety is moderately resistant to mildew, weakly affected by gray rot, resistance to powdery mildew is low. Frost resistance is moderate.
Telti kuruk	Late maturing, 155–160	100%—*V. vinifera*. Technical white grape variety. Bunch of medium size, elongated-cylindrical-conical. Berry of medium size or small, slightly oval, yellowish-green, with a thick waxy coating. The skin is thin, strong. The pulp is juicy. Frost resistance is under study.
Marselan	Late maturing, 170–190	100%—*V. vinifera*. Technical red grape variety. Crossing scheme: Cabernet Sauvignon and Grenache. The cluster is small. The berry is round, small. The skin is thick, very strong. The pulp is juicy. The variety is resistant to fungal diseases. Frost resistance is under study.
Moldova	Late maturing, 155–180	56.19%—*V. vinifera*, 3.13%—*V. labrusca*, 29%—*V. rupestris*, 6.25%—*V. berlandieri*, 5.28%—*V. lincecumii*. Table red grapes. Hybrid, crossbreeding scheme: Guzal kara x Save Villar 12-375. The cluster is large, medium-dense. The berry is large, oval. The skin is thick, the pulp is juicy. The variety is resistant to mildew and gray rot. Frost resistance is high.

2.2. Wines

All samples of icewines were produced consistently with the proposed technology described by Lutskova V. [26]. The effective fining before fermentation musts was achieved using Microcol bentonite alpha (Laffort Co., Bordeaux, France) with concentration of 1 g/L and Polylact (Laffort Co., Bordeaux, France). The addition of Assotan (Esseco SRL, San Martino, Italy) was for antioxidant process. All samples of musts were formerly heated up to 18–20 °C and then they were inoculated with *Saccharomyces cerevisiae* VIN 2000 hybrid (Anchor, Cape Town, South Africa) at rate of 5 g/dal. Before fermentation, the following procedures were conducted: the rehydration of yeast to the intended concentration of 5 g/dal; then, after 15 min of rehydration, an equal volume of yeast and must previously heated to 28 °C was mixed and left for 1 h. In this starter, an equal volume of sweet must was added and left at a temperature of 25 °C, stirring every 45 min. The yeast starter was added as follows: on the 1st day of fermentation, after two days of fermentation, and after one week for acclimatization and accumulation of yeast biomass. In order to reduce the fermentation time and increase the rate of process, a complete fermentation activator and the yeast nutrient Maxaferm (DSM Food Specialties B.V, Delft, The Netherlands) at a concentration of 2 g/dal and Booster Blanc (Lallemand, Montréal, QC, Canada) at a concentration of 3 g/dal were simultaneously added to musts after two days of fermentation. The aforementioned nutrients were diluted in water, 1:10, and added once. With the achievement in wine materials of an ethyl alcohol content of 9–10% vol. on the 8th day, fermentation was stopped by cooling the wine material at a temperature of minus 5 °C. The complex system of wine stabilization for icewines, including the use of Erbsloh PVPP (Erbsloh, Geisenheim, Germany) based on Polyvinylpolypyrrolidone, Neoclar AF (Enogroup, Kishinev, Moldova), composed of gelatin, bentonite, and charcoal with subsequent cold treatment at minus 4–6 °C for 4–6 days was utilized. To ensure correct preservation of icewines, 75 mg/L of sulphur dioxide (Laffort, Bordeaux, France) was

added. To achieve bottling-ready icewines, they were processed in accordance with the "Technological instructions for the processing of wine materials in the wine industry" TI U 37471967-11.02.11 [27]. Then, the icewines were stored for no less than 20–25 days and bottled.

2.3. Chemical Methods

Chemical analysis was carried out according to the prevailing laws of winemaking of Ukraine and international documentation regarding icewine production. The sugar content of musts was measured by a Digital Hand-Held "Pocket" Refractometer PAL1 (Atago Co., Ltd., Fukaya-shi, Japan) and then converted from Brix into g/L using a table giving the sugar content of musts and concentrated musts in grams per liter, recommended by OIV [28]. The pH of musts and icewines was determined by a pH-meter S220 (Mettler-Toledo International Inc., Columbus, OH, USA). The concentrations of titrated acid (TA) and volatile acidity (VA) were determined in accordance with standard methods [29].

Determination of aromatic compounds in the experimental icewines was conducted according to a method developed by specialists of the "Industrial-trading company Shabo" Ltd. using a gas chromatograph, Agilent Technology 7890 A (Agilent Technologies, Inc., Santa Clara, CA, USA). The main characteristics were a silica capillary column VF-WAXms 60 m, the carrier gas was helium at a rate of 3 mL/min, column diameter—0.33 mm, the temperature of the evaporator and the detector was 245 °C, the temperature of the thermostat ranged from 450 to 245 °C with a rate of 40/min, sample volume—1 mcl. Method principle: 0.05 mL of internal standard solution pentanol-1 (23.54 mg/mL) was added to 10 mL of wine in a vial with a sealed stopper and actively mixed for 1–2 min. Then, 0.2 mL of chloroform was introduced and the contents mixed for 2 min until emulsions formed. The vial was centrifuged for 5 min at 3000 rpm. Then, the resulting chloroform extract (at the bottom of the vial) was transferred into a chromatographic vial with a glass micro-insert of 0.25 mL. The extract was analyzed by a chromatograph mass spectrometry detector 5973. The concentrations of compounds were calculated as the ratio of the peak areas of volatile substances relative to the internal standard peaks without correction factors. The aromatic components were identified by comparison of retention times of the substances obtained in the chromatogram and the standard library of mass spectra (Nisto2) relative to pure standards. The aforementioned method was performed on all icewine samples in triplicate. Chemical standards were purchased from Sigma–Aldrich (Oakville, ON, Canada), Aldrich (Oakville, ON, Canada), Bedoukian (Danbury, CT, USA), and Acros Organics (Geel, Belgium) (Table 2).

Table 2. Chemical standards and quantitative and qualitative ions for analyzed icewine samples.

Cas Number	Compound	Supplier	Quantification Ion (*m/z*)	Qualitative Ions (*m/z*)	Odor Characteristics
111-27-3	1-Hexanol	Sigma-Aldrich	56	69, 84	Leaf, grassy, resin, medicinal
78-83-1	Isobutanol	Aldrich	43	42, 41, 74	Fruity, floral
71-23-8	1-Propanol	Sigma-Aldrich	31	59, 42	Sweet, fruit
3391-86-4	1-Octen-3-ol	Acros Organics	57	72, 85, 99	Mushroom
98-55-5	α-Terpineol	Aldrich	59	75, 63	Lilac, citrus, lime
100-51-6	Benzyl alcohol	Aldrich	79	85, 93	flower, fruit
543-49-7	2-heptanol	Aldrich	58	73, 87	lemon, citrus
111-87-5	1-octanol	Sigma-Aldrich	56	41, 69, 84	rose, citrus
60-12-8	Phenylethyl Alcohol	Aldrich	91	122, 65, 51	Burnt, rose, oily, honey
106-24-1	Geraniol	Aldrich	90	41, 66	Citrus
106-22-9	Citronellol	Aldrich	55	41, 67, 156	Rose, citrus

Table 2. *Cont.*

Cas Number	Compound	Supplier	Quantification Ion (*m*/*z*)	Qualitative Ions (*m*/*z*)	Odor Characteristics
78-70-6	Linalool	Sigma-Aldrich	71	93, 121, 154	Lemon, flower
123-92-2	Isoamyl acetate	Aldrich	43	70, 55, 87	Pear
123-66-0	Ethyl hexanoate	Aldrich	88	99, 60, 144	Fruity, apple
7452-79-1	Ethyl 2-methyl butyrate	Aldrich	57	102, 74, 130	Blackberry, pineapple
105-54-4	Ethyl butyrate	Aldrich	71	43, 88, 116	Blackberries, tropical fruits
108-64-5	Ethyl 3-methyl butyrate	Aldrich	88	41, 70, 130	Berry, cherry
106-32-1	Ethyl octanoate	Aldrich	88	101, 127, 172	pomegranate, pineapple, peach
103-45-7	2-Phenethyl acetate	Aldrich	104	43, 91, 78	Ripe fruit, floral
110-38-3	Ethyl decanoate	Aldrich	88	101, 73, 155	Sweet, herbal
93-89-0	Ethyl benzoate	Aldrich	105	122, 77, 172	Chamomile, flower, celery, fruit
142-62-1	Hexyl acetate	Aldrich	60	56, 84, 69	Apples, pears, cherries
4455-13-4	Ethyl acetate	Aldrich	30	44, 68	Pear, fruit
98-01-1	Furfural	Aldrich	96	67, 42, 51	Bread, pastries
100-52-7	Benzaldehyde	Bedoukian	46	85, 93, 98	Almonds
121-33-5	Vanillin aldehyde	Bedoukian	45	73, 79, 85	Vanilla
97-53-0	Eugenol	Aldrich	43	78, 96	Spicy cloves, spices, oak
142-62-1	Hexanoic acid	Aldrich	60	45, 68	Fruit, herb
103-82-2	Phenylacetic acid	Aldrich	60	56, 46	Honey
124-07-2	Octanoic acid	Aldrich	60	78, 81	Oil
104-61-0	γ-nonalactone	Aldrich	85	41, 114, 156	Oil, honey, coconut
2628-17-3	4-vinylphenol	Aldrich	150	54, 97, 110	Oak
7786-61-0	2-methoxy-4-vinylphenol	Aldrich	150	54, 97	Spices, cloves

2.4. Sensory Analysis

The panel of 10 experts took part in the sensory analysis of the icewines. All judges had had theoretical and practical training in similar research and were certified in wine tasting. In our work, the objective of the sensory analysis was to characterize the aroma and taste of icewines made in Ukraine.

The sensory analysis of the experimental icewines consisted of sessions that were performed at the end of February 2016. Sessions were divided into two main parts: 10 training sessions (1 h each) and direct evaluation of the organoleptic qualities of the proposed wines. Firstly, the training part involved questions of the main icewine sensory analyses worldwide. During the next training, experts were presented with a list of 95 wine terms obtained from the literature [30] in order to determine the prospective lexicon for icewine aroma and taste. In addition, the international standard ISO 5492:2008 «Sensory analysis—Vocabulary» was used. As a result, 12 terms describing aromatic sensations and five taste perceptions were chosen by panel consensus and then used in tasting sheets. The next stage included the general methods related to expert training in conformance with ISO 8586:2012 «Sensory analysis—General guidelines for the selection, training, and monitoring of selected assessors and expert sensory assessors». For taste/tactile training, water solutions with different concentrations of sugar (sweetness), tartaric acid (sourness), caffeine (bitterness), and aluminum potassium sulfate (astringency) were produced according to

the method published by Jackson R. S. [31]. Different aromatic standards were produced by a chemical method that had been prepared prior to training [31]. In addition, an electronic wine nose «Le Nez Du Vin» was applied. In addition, various natural products including fruit and spices were used as taste and aroma standards. During the next training, three Riesling icewines from wineries Lion Gri, Cricova Zevs, Kvint (Moldova) and one from Shabo (Ukraine), Blaufränkisch, and Welschriesling icewines from Chateau Topolcianky (Slovakia) were evaluated for establishing the icewine type.

The experts determined the aroma and taste of icewine samples made from H1 and H2 in duplicate in the last session. All experts had been asked not to eat foods with strong intense aromas or flavors the day before. The samples of icewines were cooled until 10 °C and assigned three-digit codes before the tasting. Approximately 50 mL of a randomly selected sample was poured in a clean wine glass, and the tasting sheets were shown to each expert. Sensory attributes were quantified using a nine-point intensity scale (ISO 4121:2003 «Sensory analysis—Guidelines for the use of quantitative response scales») anchored from «1—low to 9—high». In the case of sensing another aroma or taste that had been not defined on the sheet, experts were allowed to add appreciate descriptors. Panelists evaluated each wine for 10 min with a 2-min break between samples. Water was provided for rinsing between icewines. After the evaluation of icewines, experts discussed and compared the obtained scores.

Testing and training sessions were conducted in a laboratory of sensory analysis in ONAFT equipped with individual booths, designed according to ISO 8589 (1988). The usual universal tasting glasses in accordance with ISO 3591:1977 «Sensory analysis—Apparatus-Wine-tasting glass» were used purchased from the Ukrainian branch of the Austrian company "Riedel".

2.5. Statistical Analysis

Statistical methods included Fisher's Least Significant Difference for mean separation at $p < 0.05$. One-way Analysis of variance (ANOVA) was performed using XL STAT (Addinsoft, Paris, France). To evaluate panel performance, the degree of concordance and reliability was calculated [32]. All figures were drawn using Excel software (Microsoft Co., Redmond, WA, USA). Mean scores of significant sensory attributes ($p < 0.05$) were calculated in order to create cobweb diagrams. An independent sample *t*-test was calculated to compare the means of the sensory data of icewines.

3. Results

3.1. Physicochemical Analysis

Physicochemical attributes of icewines from frozen grapes largely depend on two processes: grape freezing and conditions of must fermentation [24,33]. The freezing of grapes contributes to increase the mass concentrations, not only of sugars in the grape berry, but also acids, phenolic, and aromatic compounds, which then affect the formation of the icewine organoleptic indicators [24]. According to the results, harvest date was an active factor in increasing the sugar content of both white and red grapes (Table 3). Marselan and Moldova musts had the same pH values depending on different harvest dates versus those of Rkatsiteli and Telti kuruk musts. A small difference in TA was found in Telti kuruk musts of both harvests. The TA of Rkatsiteli, Marselan, and Moldova musts obtained from H2 was characterized by lower values compared to H1.

The influence of harvest dates (H1, H2) on chemical compositions of the varietal experimental icewines is shown in Table 4. All icewines samples had different residual sugar and ethanol depending on the harvest date. In icewines obtained from all grape varieties harvested in H2, the concentration of sugar and ethanol increased by 1–1.3 times. It should be noted that one of the features of icewines is a prolonged fermentation process, because the yeast is not able to immediately ferment the high sugar content into ethanol, indicating hyperosmotic shock [2,16,17,24]. In addition, values of VA play roles in icewine

quality. The experimental icewines from H1 were characterized by lower values of VA compared to H2.

Table 3. Influence of harvest date (H1, H2) on the chemical compositions of grape musts.

	H1	H2	Significance [a]
Rkatsiteli musts			
Brix	29.5a	33.4b	***
pH	3.5a	3.7b	**
Titratable acidity (g/L)	8.9a	8.4b	**
Telti kuruk musts			
Brix	30.0a	35.7b	***
pH	3.4a	3.6 b	*
Titratable acidity (g/L)	9.1a	9.2a	ns
Marselan musts			
Brix	28.5a	35.2b	***
pH	3.8a	3.8a	ns
Titratable acidity (g/L)	8.6a	8.0b	***
Moldova musts			
Brix	29.2a	34.8b	***
pH	3.7a	3.7a	ns
Titratable acidity (g/L)	9.1a	8.5b	**

[a], *, **, ***, ns: significant at $p \leq 0.05$, 0.01, and 0.001, or not significant, respectively. Treatments with the same letter within a row are not significantly different; $p \leq 0.05$, least significant difference.

Table 4. Influence of harvest date (H1, H2) on the chemical compositions of the varietal experimental icewines.

	H1	H2	Significance [a]
Rkatsiteli icewines			
Brix	145.0a	155.7b	***
pH	3.6a	3.7b	*
Titratable acidity (g/L)	9.0a	8.6b	**
Ethanol (% v/v)	10.5a	11.0b	**
Volatile acidity (g/L)	0.8a	0.95b	***
Telti kuruk icewines			
Brix	158.7a	170.1b	***
pH	3.5a	3.6b	*
Titratable acidity (g/L)	9.5a	9.5a	ns
Ethanol (% v/v)	10.2a	11.6b	***
Volatile acidity (g/L)	0.85a	1.0b	***
Marselan icewines			
Brix	142.6a	164.3b	***
pH	3.8a	3.9b	*
Titratable acidity (g/L)	9.0a	8.5b	***
Ethanol (% v/v)	9.6a	10.3b	***
Volatile acidity (g/L)	0.8a	0.9b	**
Moldova icewines			
Brix	144.0a	158.2b	***
pH	3.7a	3.7a	ns
Titratable acidity (g/L)	9.5a	8.4b	***
Ethanol (% v/v)	9.7a	10.5b	***
Volatile acidity (g/L)	0.8a	0.95b	***

[a], *, **, ***, ns: significant at $p \leq 0.05$, 0.01, and 0.001, or not significant, respectively. Treatments with the same letter within a row are not significantly different; $p \leq 0.05$, least significant difference.

3.2. Sensory Analysis

Based on the results of the tasting, the concordance coefficient was determined by a statistical method to establish the degree of agreement of scores of the sensory analyses. The concordance coefficient was 0.91%, which indicates a high degree of agreement among experts.

Dried apricot, melon, pineapple, fig, lemon, honey, raisin, caramel, spicy, and oxidized aromas were chosen by experts as aroma descriptors. In addition, they decided to add plum and cherry aromas as specific aromas for Marselan and Moldova wines [4]. Figure 1 reports the average values of the aroma descriptors and tastes of the analyzed wine samples that were significant between H1 and H2 within a certain cultivar (at $p \leq 0.05$). The sensory analysis showed that aromas of honey and raisin were characterized for all wines but differed depending on the harvest date. Notable melon, pineapple, and caramel aromas were in the Rkatsiteli icewine of H2. The most perceived oxidized aromas of fig were from wine samples produced from Telti kuruk, but general sweetness was not significant. In addition, a spicy aroma was detected only in Telti kuruk icewines. The aromas of cherry and plum were determined only in icewines obtained from dark-skinned grape varieties. The aroma of dried apricot was characteristic for Rkatsiteli and Marselan icewines. Tastes such as bitterness and astringency were not perceived by the panel.
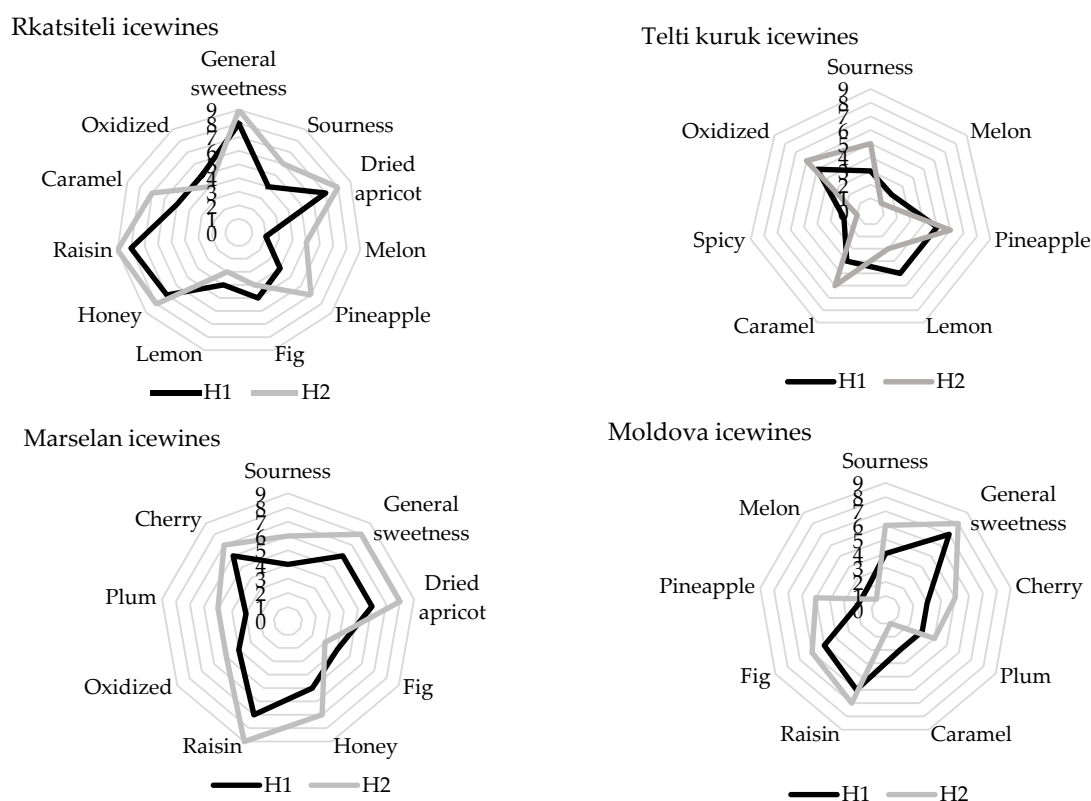

Figure 1. The intensity of sensory descriptors found in icewines made from four different varieties harvested at two different times. Sensory descriptors not shown on the plot are not significant according to a *t*-test.

3.3. Aroma Compounds

3.3.1. Odor Activity

According to chromatographic analysis, 33 aromatic compounds belonging to the following groups were found in ice wines: ethyl esters and acetates, responsible for fruit and berry aromas, higher and terpene alcohols—for floral, citrus and honey aromas, and aromatic acids and aldehydes—provide nuts and shades of tropical fruits (Tables 5 and 6). In icewines of both harvests, the concentrations of isoamyl acetate, 2-phenyl acetate, and

1-propanol responsible for honey and ripe fruit aromas were higher than the odor threshold. In Rkatsiteli icewines produced from H1, the concentration of geraniol was equal to the odor threshold characterized by citrus aromas. The highest odor activity of ethyl hexanoate and ethyl octanoate associated with aromas of dried fruits was detected in Moldova and Marselan icewines. The concentrations of ethyl 3-methyl butyrate were higher than the odor threshold in all experimental icewines. Among acid groups, only hexanoic acid, in all wine samples, had the highest odor threshold.

3.3.2. Aroma Components

Quantitative data of aromatic compounds in Rkatsiteli and Telti kuruk icewines are presented in Table 5. Wines obtained from H1 differed from ones of H2. The higher and terpene alcohols were identified as the biggest quantity aroma group among aroma volatiles. Esters and acetates accounted for 20% and 14%, respectively, of the total quantity of aromatic compounds. No 1-hexanol and benzyl alcohol were found in Rkatsiteli and Telti kuruk icewines. Telti kuruk icewine of H1 did not have ethyl benzoate and hexyl acetate. The aldehydes and acids had the same values in icewine samples.

Table 5. Quantitative data of aromatic compounds in Rkatsiteli and Telti kuruk icewines.

Compound	Rkatsiteli		Significance [a]	Telti Kuruk		Significance [a]	Threshold (µg/L)
	H1	H2		H1	H2		
Higher and terpene alcohols							
Isobutanol	1260.11a	1260.74a	ns	2120.41a	3260.3b	***	40,000 [34]
1-Propanol	129.52a	139.71b	**	150.7a	200.2b	***	120 [34]
1-Octen-3-ol	2.5a	2.5a	ns	1.5a	3.0b	*	1 [35]
α-Terpineol	30.18a	35.32b	***	80.11a	85.46b	**	250 [36]
Isoamyl alcohol	nd	560.26b	**	300.5a	570b	***	30,000 [34]
1-octanol	150.12a	120.1b	***	180.7a	125.2b	***	120 [34]
Phenylethyl Alcohol	90.45a	60.03b	***	70.85a	50.74b	***	10,000 [34]
Geraniol	6.5a	5.2b	*	6.9a	3.12b	**	7 [37]
Citronellol	nd	6.24b	*	6.56a	6.81a	ns	100 [34]
Linalool	4a	4a	ns	2a	3b	*	15 [34]
Esters							
Ethyl hexanoate	102.54a	114.25b	**	110.14a	126.2b	**	5 [34]
Ethyl 2-methyl butyrate	10.12a	10.56a	ns	10.19a	14.15b	*	18 [34]
Ethyl butyrate	50.45a	48.42b	*	51.11a	52.1b	*	20 [34]
Ethyl 3-methyl butyrate	26.21a	30.76b	*	10.23a	27.71b	**	3 [34]
Ethyl octanoate	400.29a	420.8b	**	200.19a	270.16b	***	2 [34]
Ethyl decanoate	150.5a	160.48b	**	115.62a	120.32b	**	200 [38]
Ethyl benzoate	170.52a	140.43b	***	90.17a	60.36b	***	575 [38]
Acetates							
Hexyl acetate	180.11a	180.33a	ns	nd	nd	-	670 [39]
Ethyl acetate	70.12a	80.26b	**	50.48a	70.78b	***	32,600 [40]
2-Phenethyl acetate	350.19a	150.35b	***	400.51a	170.19b	***	250 [34]
Isoamyl acetate	126.14a	107.23b	***	105.1a	106.5b	**	30 [34]
Aldehyde							
Vanillin aldehyde	nd	10.4b	*	nd	nd	-	26 [41]
Acids							
Hexanoic acid	3458a	3488b	**	4120a	4137b	**	3000 [34]
Phenylacetic acid	560a	500b	**	600a	480b	***	10,000 [34]
Octanoic acid	1110a	1112b	*	1000a	1010b	*	7000 [34]
Phenols							
4-vinylphenol	9.47	nd	*	7.5	nd	*	180 [42]
2-methoxy-4-inylphenol	3.32a	2.36b	**	1.3	nd	*	9.5 [43]
Eugenol	5.15a	3.17b	***	1.4	nd	*	6 [38]

[a], *, **, ***, ns: significant at $p \leq 0.05$, 0.01, and 0.001, or not significant, respectively; nd—not detected. Treatments with the same letter within a row are not significantly different; $p \leq 0.05$, least significant difference.

Statistical analysis showed that most of aromatic compounds with the exception of geraniol, citronellol, linalool, and ethyl benzoate found in icewines from dark grapes had differences between harvests (Table 6). All aldehydes including furfural, benzaldehyde, vanillin aldehyde and acids such as hexanoic, phenylacetic, and octanoic acids had lower concentrations in icewines of H1 compared to H2. Citronellol and linalool were not detected in Marselan icewines made from H2. Furfural was found only in Moldova and Marselan icewines. Benzaldehyde was found only in Marselan icewines of both harvests. Lactone had one representative—γ-nonalactone—found in Moldova icewines. Ethel decanoate, eugenol, 2-methoxy-4-vinylphenol, and 4-vinylphenol, belonging to phenols, were not identified Marselan icewine made from both harvests.

Table 6. Quantitative data of aromatic compounds in Moldova and Marselan icewines.

Compound	Moldova		Significance [a]	Marselan		Significance [a]	Threshold (µg/L)
	H1	H2		H1	H2		
Higher and terpene alcohols							
1-Hexanol	1200.15a	1110.52b	***	1220.45a	1110.67b	***	8000 [34]
Isobutanol	1020.24a	2010b	***	1550.17a	2010.26b	***	40,000 [34]
1-Propanol	130.19a	129.47b	*	130.8a	128.4b	*	120 [34]
1-Octen-3-ol	2.4a	3.0b	*	2.3a	5.2b	**	1 [35]
α-Terpineol	10.15a	12.27b	*	20.75a	26.43b	**	250 [36]
Benzyl alcohol	10.37a	20.96b	**	50.81a	57.18b	**	10,000 [34]
Isoamyl alcohol	200a	80b	***	100a	200b	***	30,000 [34]
1-octanol	120	nd	**	121a	120b	***	120 [34]
Phenylethyl Alcohol	10.76a	30.72b	***	10.4a	50.81b	**	10,000 [34]
Geraniol	4.47a	5.5b	**	4.13a	4.17 a	ns	7 [37]
Citronellol	1.61	nd	*	2.45	nd	*	100 [34]
Linalool	3.2a	3.24 a	ns	2.25	nd	*	15 [34]
Esters							
Ethyl hexanoate	123.11a	159.19b	***	137.75a	164.19b	***	5 [34]
Ethyl 2-methyl butyrate	11.9a	15.1b	**	25.5a	15.4b	***	18 [34]
Ethyl butyrate	60.52a	30.63b	***	45.31a	15.24b	***	20 [34]
Ethyl 3-methyl butyrate	13.7a	12.61b	**	11.28a	9.17b	***	3 [34]
Ethyl octanoate	75.13a	91.64b	***	84.14a	92.97b	***	2 [34]
Ethyl decanoate	115.52a	130.71b	***	nd	nd	-	200 [38]
Ethyl benzoate	20.1a	20.15a	ns	80.75a	25.84b	***	575 [38]
Acetates							
Hexyl acetate	210.7a	280.24b	***	180.79a	200.8b	***	670 [39]
Ethyl acetate	nd	nd	-	35.17a	38.93b	**	32,600 [40]
2-Phenethyl acetate	250.52a	245.78b	***	263.87a	219.14b	***	250 [34]
Isoamyl acetate	104.47a	100.55b	**	110.64a	105.37b	*	30 [34]
Aldehyde							
Furfural	30.35a	50.47b	***	10.15a	50.5b	***	14,100 [38]
Benzaldehyde	nd	nd	-	6.17a	10.87b	***	2000 [44]
Vanillin aldehyde	12.14a	15.15b	*	nd	6.37b	*	26 [41]
Acids							
Hexanoic acid	3205.23a	3240.51b	***	3140.62a	3170.74b	***	3000 [34]
Phenylacetic acid	640.63a	410.74b	***	510.49a	520.81b	***	10,000 [34]
Octanoic acid	650.54a	660.78b	**	580.91a	600.62b	***	7000 [34]
Lactone							
γ-nonalactone	40.14a	41.96b	*	nd	nd	-	30 [38]
Phenols							
4-vinylphenol	4.36	nd	*	nd	nd	-	180 [42]
2-methoxy-4Vinylphenol	4.54a	2.69b	**	nd	nd	-	9.5 [43]
Eugenol	1.5a	2.13b	*	nd	nd	-	6 [38]

[a], *, **, ***, ns: significant at $p \leq 0.05$, 0.01, and 0.001, or not significant, respectively; nd—not detected. Treatments with the same letter within a row are not significantly different; $p \leq 0.05$, least significant difference.

4. Discussion

4.1. Must and Wine Chemical Analysis

Analysis of the scientific literature showed that the sugar and alcohol contents of icewines differ significantly between producers and countries. Canadian icewines contain 9–12% alcohol and 170–240 g/L of sugar, while wines from Germany and other European countries have 7–10% alcohol and sugar of 150–200 g/L [34]. The physicochemical properties of the analyzed icewines showed differences in the sugar content of musts and residual sugar compared with the chemical attributes of Riesling, Vidal, and Cabernet franc icewines from Canada [21,22,24]. Such difference is especially associated with specific harvesting conditions. In addition, the peculiar change of the physicochemical properties of grapes is involved with its overripening after technological maturity. According to a study [45], in Vidal Blanc grapes from Canada during long-term ripening, the relationship between the mass concentrations of titrated acids and sugars was inversely proportional, which was also observed in our investigation. Thus, with increasing concentrations of sugars, acids decreased, which is probably due to the influence of climatic conditions on the physicochemical properties of grapes.

The fermentation of must with high sugar is known to have challenges to produce quality icewine. The main factor in choosing the yeast for icewine is the ability to accumulate a minimum content of volatile acids. For this reason, yeast strain Vin 2000, which is a hybrid, was chosen. The yeast used in the study belongs to the strain *Saccharomyces cerevisiae*, which produces acetic acid in response to the extreme osmotic stress caused by high sugar levels [24,30]. The quantity of acetic acid and glycerol and the total acidity correlate with the sugar concentration of musts [30], which was also investigated in icewines. The samples had higher values of VA compared to H1, perhaps due to the higher sugar of musts, that confirms the abovementioned statement.

4.2. Sensory Analysis

Icewines demonstrate a complex aroma formed by the quantity of aroma volatiles including esters, terpenes, norisoprenoids, alcohols, aldehydes, phenols, and lactones [33]. The sensory profiles of the experimental icewines from Ukraine consisted of typical descriptors (Figure 1) such as honey, tropical fruits, dried apricot, and raisin for white wines and red berries for icewines made from dark grapes [24,30]. All icewine samples were medium-bodied, full of sweet fruits in terms of aroma.

Rkatsiteli icewines were characterized by the pronounced aroma of dried apricot; honey and raisin contributed to the sweetness in taste. Icewines from H1 and H2 differed in the intensity of dried apricot, melon, pineapple, fig, lemon, honey, and raisin aromas ($p \leq 0.05$). Aromas of pineapple and melon in icewine obtained from H2 were more intensive. In addition, the taste of sweetness and sourness varied. The varietal aroma with floral and citrus tones is specific to Rkatsiteli table wines [46] that significantly differ from the icewine style.

Icewines produced from Telti kuruk grapes had intense aromas of lemon and caramel. In addition, a lemon aroma with the highest score was determined in Gewürztraminer icewines [47]. Telti kuruk varietal table wines are distinguished by a delicate aroma of tropical fruits, fresh notes of eucalyptus, and mint [14]. Our investigations showed that the intensity of melon, pineapple, lemon, caramel, spicy, and oxidized aromas was significantly different between icewines of different harvests ($p \leq 0.05$). In addition, oxidized aromas of these icewines were more noticeable compared to others, which may be related to relatively high values of VA, i.e., in Telti kuruk icewines—0.85 and 1.0 g/L in H1 and H2, respectively. No spicy, cherry, melon or plum was found in the icewine of H2. Aromas of raisin, honey, dried apricot, and sweetness were not different between Telti kuruk icewines. It should be mentioned Telti kuruk is a significant component of the autochthonous grape variety stock of Ukraine, as well as an important component of the genetic heritage of the domestic viticulture and wine industry.

Aroma descriptors of dried apricot and honey were characteristic of Moldova icewines but they were not significant between icewines from H1 and H2. Melon and caramel aromas were not found by experts in wine from H2. According to the statistical method, the aroma intensity of raisin, fig, pineapple, plum, and cherry was significantly different between harvests ($p \leq 0.05$). In addition, sourness and sweetness were different. There is limited scientific literature on wines made from Moldova grapes; therefore a comprehensive comparison is impossible to indicate further study of their usage and properties.

Today Marselan wines are interesting to researchers [48–50]. Aroma profiles of Marselan wines made by different wine technologies in Brazil consisted of plum, strawberry, red fruits, blackberry, cherry, tropical fruits, raisin, honey, and marmalade aromas. In addition, vegetable properties such as bell pepper, string bean, dry straw, with, broccoli, asparagus, cabbage, pepper, twig, and spicy aromatic descriptors including pepper, mint, clove, cinnamon, mushroom, nutmeg, fine herbs, vanilla, and chocolate occur in small quantities [48]. According to our observations, Marselan icewine of both harvests possessed plum, cherry, raisin, honey, and dried apricot aromas; however, in samples which had been produced from H2, these descriptors were more intense ($p \leq 0.05$). The intensity of the oxidized aroma of icewines differed. In addition, melon and pineapple aromas as well as caramel were not inherent in Marselan icewines. General sweetness and sourness of these wines were determined to be similar to Moldova grapes and differed from white-skinned cultivars.

4.3. Aroma Compounds

The volatile composition of wine is known to depend on yeast strains [21,34,50], fermentation conditions [21,47,51], and grape variety [22,52]. Analysis of scientific information highlighted icewines from the harvest of Riesling grapes have many key aromatic compounds such as: 1-octen-3-ol, ethyl benzoate, ethyl octanoate, β-ionone, ethyl isobutyrate, ethyl 3-methylbutyrate, 1-hexanol, 1-octanol, cis-rose oxide, nerol oxide, ethyl phenylacetate, γ-nonalactone, and β-damascenone [22,30]. Isoamyl alcohol, 2-methyl-1-butanol, and 2-phenylethanol were found in Cabernet franc icewines [21].

4.4. Higher and Terpene Alcohols

The alcohol content differed both qualitatively and quantitatively among the icewines. The total concentration of the alcohols was from 3260 µg/L (isobutanol of Telti kuruk icewine of H2) to 1 µg/L (citronellol of Moldova icewine of H1). Some alcohols had higher concentrations including 1-octen-3-ol, 1-propanol, α-terpineol, and benzyl alcohol; however, the latter was not detected in white icewines as well as 1-hexanol. The concentrations of such alcohols as 1-propanol, 1-octen-3-ol, 1-octanol, and geraniol exceeded the odor threshold and were identified below 300 mg/L, which contributed to the aromas of the wine [52]. Isoamyl alcohol had a lower concentration range in the analyzed wines (80–560 mg/L) compared to Canadian icewines (36,235–90,860 mg/L); similar to linalool (average values of Ukrainian experimental icewines were 2–4 mg/L and in Canadian ones—64.6–75.9 mg/L). Linalool is included in monoterpenes that are important factors, forming the aroma of white wines made from Muscat varieties (Muscat of Alexandria, Muscat de Fronttignan) and Gewürztraminer and Riesling [53]. It should be noted that higher alcohols, including isoamyl and isobutyl alcohols, are synthesized from amino acids. Moreover, aromatic amino acids, including phenylalanine and tyrosine, produce aromatic alcohols (contributing to the following aromas: honey, spice, rose, lilac [54]), such as phenylethyl alcohol whose contents in the investigated icewines were lower than the odor threshold.

4.5. Esters

Ethyl hexanoate, ethyl octanoate, and ethyl decanoate had lower concentrations in all analyzed icewines produced from H2 contrary to those of H1. Taking into account that esters are usually considered products of yeast metabolism, we can suppose that the

fermentation of grapes en picked in H2 was problematic for yeast due to a higher sugar content. The ester ethyl decanoate was not identified in Marselan icewines of both harvests as well as in table wines in a previous study [55].

There were three compounds above 100 µg/L among acetates. The contents of 2-phenethyl acetate and isoamyl acetate were higher than the odor threshold and lower for H2 in all treatments. The concentrations of ethyl acetate were higher in wines produced from overripened grapes [56], which was also observed in our study on icewines of H2.

4.6. Aldehydes, Acids, Lactones, and Phenols

The composition of aldehydes, acids, lactones, and phenols differed both qualitatively and quantitatively among the icewines. The contents of aldehydes, lactones, and phenols were at extremely low values compared to other aromatic compounds. The concentrations of furfural ranged from 10 to 50 µg/L in wines from red cultivars [57], while benzaldehyde, characterized by an almond aroma, was identified in Marselan icewines of both harvests. γ-nonalactone as well as vanillin aldehyde were present only in Moldova icewines. The compound γ-nonalactone with aromas of oil-flower, honey, and coconut is one of the main odor impact components in sweet Fiano wine [58]. In addition, some scientists have considered that the mass concentration of γ-nonalactone relates to notes of prunes, as the aroma of aging red wines, while other researchers reported a correlation between γ- and δ-lactones and the aroma of botrytized wines from Sauternes, Barsac, Lupiak [59], and Hungary (Tokay Asu) [60].

Octanoic, phenylacetic, and hexanoic acids were detected and quantified in all wine samples. The contents of acids in icewines made from H2 were higher than those from H1 but only concentrations of hexanoic acid were above 3000 µg/L. Fatty acids are produced in the lipid metabolism of yeast [61].

Phenols including 4-vinylphenol, 2-methoxy-4-vinylphenol, and eugenol tended to have the highest levels from H1 in Rkatsiteli and Moldova icewines of both harvests and Telti kuruk from H1. The compound eugenol is responsible for clove and oak aromas, described in wines made using oak chips [62]. Undesirable parts of grapes such as grape stalks may occur in fermented musts.

In addition, no concentrations of cis-rose oxide, nerol oxide, and β-damascenone were identified in the current work that had been suggested as the important odorants in icewines from Riesling and Vidal [22,63]. It is worth pointing out that contents of β-damascenone were also investigated in beer [64] and aged Riesling wines [65,66]. On this basis, the aromatic compounds of experimental icewines produced under the climate conditions in the south of Ukraine have their own quantitative and qualitative compositions that depend on the grape varieties used.

5. Conclusions

This study demonstrates that sensory descriptors and aromatic compounds of icewines produced from Rkatsiteli, Telti kuruk, Moldova, and Marselan grapes grown in the Odessa region differed depending on the harvest date. The experimental wines were characterized by aromas of dried apricot, raisin, honey, and oxidized aromas. Marselan and Moldova icewines were evaluated as more intense in cherry and plum aromas, whereas pineapple and melon aromas were more intense in Rkatsiteli and Telti kuruk icewines. Icewines produced from different grape cultivars from different harvests varied in volatile aromatic compounds. Some compounds had higher concentrations in wines of H2, especially 1-octen-3-ol, 1-propanol, α-terpineol and benzyl alcohol, hexanoic acid, ethyl hexanoate, ethyl acetate, ethyl octanoate, and octanoic acid. Icewines from white grape cultivars did not have 1-hexanol and benzyl alcohol that were found in dark-skinned grape varieties. Marselan icewines were the only ones containing benzaldehyde, but γ-nonalactone occurred only in Moldova wines. Further work could focus on investigations on the influence of different yeast strains on the aromatic compounds in icewines made from Rkatsiteli, Telti kuruk, Moldova, and Marselan grapes.

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