

## CHEMICAL and SENSORY PROPERTIES of VINEGAR FROM DIMRIT GRAPE by SUBMERGED and SURFACE METHOD

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### Abstract

In this study, it was investigated some chemical and sensory properties of vinegar obtained from Dimrit grape by submerged and surface method. Also it was discussed convenience of Dimrit grape grown in Nevşehir-Ürgüp region for vinegar production. Vinegars were compared as regards general compositions, contents of aroma components (acetaldehyde, methanol, 2-methyl-1-butanol, 3-methyl-1-butanol, 1-propanol, 2-methyl-1-propanol, ethyl acetate, methyl acetate, 2,3-butanediol, 2-phenylethanol) and sensorial characteristics by statistical analyses. Results indicated that some properties of vinegars were different as regards their general compositions, contents of aroma components and sensorial characteristics. It was found that acidity and contents of aroma components of vinegars produced by slow method were higher and their sensorial characteristics were better. Amounts of volatile acidity changed between 36.35-54.94 g/L, whereas amounts of aroma compounds changed between 0.08-7.16 mg/L. Furthermore, ethyl acetate (4.26 -6.84 mg/L), 2,3 butandiol (3.36-5.0 mg/L) and 2-phenyl ethanol (2.38-3.42 mg/L) was determined as dominant aroma compounds in our vinegar samples. Through this study, Dimrit grape variety was evaluated for vinegar production and it was determined that vinegars from Dimrit grape were good quality.

**Key Words:** Vinegar, chemical composition, sensory properties

## DERİN KÜLTÜR YÖNTEMİ ve YAVAŞ YÖNTEM İLE DİMİRİT ÜZÜMÜNDEN ÜRETİLEN SİRKENİN KİMYASAL ve DUYUSAL ÖZELLİKLERİ

### Özet

Bu çalışmada Dimrit üzümünden derin kültür yöntemi ve yavaş yöntem ile elde edilen sirkelerin bazı kimyasal ve duyuşsal özellikleri araştırılmıştır. Aynı zamanda Nevşehir-Ürgüp bölgesinde yetiştirilen Dimrit üzümünün sirke üretimi için elverişliliği tartışılmıştır. Sirkeler genel bileşim, aroma maddeleri içeriği (asetaldehit, metanol, 2-metil-1-bütanol, 1-propanol, 2-metil-1-propanol, etil asetat, metil asetat, 2,3-bütandiol, 2-feniletanol) ve duyuşsal özellikleri bakımından istatistiksel olarak karşılaştırılmıştır. Yavaş yöntemle üretilen sirkelerin asitlik ve aroma maddeleri içeriğinin daha yüksek ve duyuşsal özelliklerinin daha iyi olduğu bulunmuştur. Uçucu asit miktarı 36.35-54.94 g/L arasında değişirken, aroma maddeleri miktarı 0.08-7.16 mg/L arasında değişmiştir. Ayrıca etil asetat (4.26-6.84 mg/L), 2,3 bütandiol (3.36-5.0 mg/L) ve 2-fenil etanol (2.38-3.42 mg/L) sirke örneklerimizdeki baskın aroma maddeleri olarak belirlenmiştir. Bu çalışma sayesinde, Dimrit üzüm çeşidi, sirke üretimi için değerlendirilmiş ve Dimrit üzümünden elde edilen sirkelerin iyi kalitede olduğu belirlenmiştir.

**Anahtar kelimeler:** Sirke, kimyasal bileşim, duyuşsal özellikler

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## INTRODUCTION

Vinegar is produced by a two stage process; being the first one the conversion of fermentable sugars to ethanol by yeasts, mostly *Saccharomyces* species, and the second the oxidation of ethanol by bacteria, mostly *Acetobacter* species. In general, two different methods is used for vinegar production. These methods are surface or traditional (slow) and submerged (quick) methods (1, 2). Vinegars produced by slow traditional surface method have better sensory quality; whereas vinegar production by submerged method is faster (about the speed of transformation of ethanol into acetic acid) and cheaper. However, vinegar production by submerged method is commercially preferred because of some advantages (high yields, more economic and faster) for producers (3-5).

Quality of vinegar is especially depending on production method and grape variety (6-9). Vinegar has distinctive flavor and aroma. Acetic acid which has a pungent flavor is responsible for the basic sensorial characteristic of vinegar. In addition to this, organic acids, volatile compounds and other fermentation products also play a role on its organoleptic properties (2).

Dimrit grape are grown in Central Anatolia, especially Nevşehir-Ürgüp region 'Dimrit' group grapes comprise. All 'Dimrit' grapes are consumed as traditional grape products (e.g. grape molasses and dried sweets made of boiled down grape juice), raisin or table grapes and the rest are used for wine production. Moreover, a significant amount of 'Dimrit' grapes is used for the production of traditional alcoholic beverage called 'raki'. Dimrit grape for wine production is not preferred for the reason that wine from Dimrit grape can easily oxidize. So, it was offered for vinegar production (10).

Methods of vinegar production affect quality of vinegar depending on fermentation duration. Especially, the final aroma profiles of vinegars are formed by aroma compounds from the substrate and fermentation (11). Aroma compounds of vinegar from slow methods are higher than submerged method. So, quality of vinegar from slow method is better than submerged method. The most important quality criterion is content of

acetic acid. Amount of total acid in wine vinegar should be at least 4 g/100 mL according to Turkish standard. pH of vinegar generally extends to 3.5 from 2.0. The other important quality criterion of vinegar is aroma components. Aroma components in vinegar are used on distinction of several vinegars. Raw material is directly effective on aroma components of vinegar. In addition to this, aroma of vinegar depends on methods of production and storage. Producers have to choose the best raw material besides the best production methods because quality products are presented to consumers (12-14).

In our country, research about wine making from Dimrit grape is quite few. Also, there isn't any research about vinegar production from Dimrit grape (15, 16). In addition to Dimrit grape, researches about other grape variety are quite scarce (17-19).

In this study, we examined the changes in some chemical characteristics of vinegars obtained from Dimrit grape (chemical composition, major aroma compounds and sensorial characteristics) during acetification in submerged and surface cultures.

## MATERIALS and METHODS

### Samples

In this study, wine produced from Dimrit grapes grown widely in Nevşehir was used as substrates. For vinegar production by traditional surface acetification, barrels were used (3 L capacity). Vinegars used as a starter (pH: 2.80, volatile acidity: 45.12 g/L, ethanol %V/V: <1) were provided from Pilot Plant in Department of Food Engineering, University of Çukurova. The mixture of substrate wine plus vinegar had a ratio of 4:1. Acetifications lasted 37 days for substrate A1 and A3, and 47 days for substrate A2 mainly depending on room temperature (Table 1). Wines were subjected to traditional surface acetification. We also used substrate B1, B2, B3 to perform submerged acetifications in a laboratory fermentor (3 L capacity). Samples (n = 6) were also taken at the beginning and at the end of the process. The acetification lasted 18 days for B1, B2 and B3 (Table 1).

Table 1. Characteristics and codes of wine substrates and vinegar samples

Acetification method	Duration	Sampling point
Surface culture	37 days	A1
	47 days	A2
	37 days	A3
Submerged culture (Laboratory fermentor)	17 days	B1
	17 days	B2
	17 days	B3

### Conditions of laboratory fermentor for submerged method

A laboratory scale fermentor (New Brunswick BioFlo 110) was used to produce wine vinegar by a submerged culture. This was equipped with: a cylindrical concave bottom glass culture vessel with a capacity of 3 L. Optimum conditions for the efficient production of vinegar samples in this study were adjusted: an air flow of 0.25 L/min. (0.25vvm), a temperature of 30 °C, a stirring speed of 200 rpm, a working volume of 2.4 L, a loading proportion of 1:4 (vinegar:wine), which results in discontinuous cycles with an average duration of 17 days (20).

### Reagents and chemicals

4-nonanol was used as internal standard (IS). All reagents and chemicals were purchased from Merck (Darmstadt, Germany). Water was obtained from a Milli-Q purification system (Millipore, USA).

### Standard Chemical Analysis

Chemical analysis of vinegar samples was as follows: total acidity, dry extract, pH, total phenols, total residual sugar, density, ethanol, acetic acid, ash, ash alkalinity, total SO<sub>2</sub>, free SO<sub>2</sub> (21, 22, 23).

### Analysis of major aroma compounds

Analyses of major aroma compounds (acetaldehyde, methanol, 2-methyl-1-butanol, 3-methyl-1-butanol, 1-propanol, 2-methyl-1-propanol, ethyl acetate, methyl acetate, 2,3-butanediol, 2-phenylethanol) were performed in duplicate using a gas chromatograph (Shimadzu GC-14B, Japan) equipped with a split/splitless injector and a flame ionization detector and a 60 m x 0.25 mm i.d. x 0.4 µm Chrompack CP-WAX-57CB capillary column. Oven temperature programme was as follows: 35 °C for 5 min, then raised by 4 °C/min up to 150 °C and by 5 °C/min from 150 °C to 180 °C and held at 180 °C for 20 min. The carrier gas was He at 1 mL/min. Injection: 1 µL in split mode.

Split ratio was 1:60. The FID temperature was 215 °C and injection temperature was 200 °C. Samples containing the internal standard (4-nonanol) were injected directly into the column. Standard solutions containing all compounds were prepared and analyzed in duplicate. Relative response factors (RRF) were calculated from peak areas for each compound (4, 24).

### Sensory Analysis

A panel of 5 assessors for vinegar sensory analysis was constituted, trained and validated. Descriptive tests were performed. Thirty milliliters of vinegar samples were randomly presented in dark glasses in each session. Vinegar sensory profile was built with previously established descriptors: general impression, aromatic richness, aromatic intensity, pungent sensation, wine character, ethyl acetate odor. A 9 cm unstructured scale was used in which each assessor marked the intensity for every attribute. All tests were made by duplicate (13, 25-27).

### Statistical analysis

Results of vinegar analysis were evaluated according to Student T- test and SPSS (28).

## RESULTS AND DISCUSSION

### Composition of vinegar

We examined the differences in composition of vinegar samples obtained by surface and submerged acetification of the same wine substrate. Wine substrate has % 11.30 (V/V) alcohol, 3.04 g/L residual sugar, 3.38 pH. Fermentation was followed by measuring the specific gravity. Table 1 shows characteristics and codes of wine and vinegar samples. Also general composition of vinegar obtained from Dimrit grape with different production methods is given in Table 2. The vinegar composition was affected by the production method. As can be seen in Table 2, amounts of ethanol, ash, ash alkalinity, total

phenols, total SO<sub>2</sub> and free SO<sub>2</sub> were unaffected by production methods, whereas results of analysis such as density, volatile acidity, dry extract, pH, total residual sugar changed according to production method. Similar results were reported in the literature (4, 6, 12, 17-19, 27, 29-32).

vinegar can transfer to liquid portion (2, 33). It was thought that these particles supported the marked increase in dry extract of our vinegar samples from surface method.

In conclusion, it was thought that the analytical parameters selected about vinegar composition

Table 2. General composition of vinegar

Analysis	A1	A2	A3	B1	B2	B3	T
Density (g/cm <sup>3</sup> ) (20 °C)	1.0135	1.0126	1.0131	1.0113	1.0115	1.0110	*
Ethanol % (V/V)	<1	<1	<1	<1	<1	<1	ns
Volatile acidity (g/L) <sup>c</sup>	49.96	54.94	51.35	36.35	40.89	39.32	**
Dry extract (g/L)	12.51	12.17	12.60	11.06	11.79	10.83	*
Ash (g/L)	1.74	1.71	1.71	1.70	1.79	1.78	ns
Ash alkalinity (meq/L)	27	21	23.5	24.5	28.5	26.5	ns
pH	2.71	2.68	2.71	2.85	2.84	2.85	*
Total phenols (mg/L) <sup>b</sup>	494.18	433.31	499.90	451.95	423.90	424.90	ns
Total Residual Sugar (g/L)	2.69	2.83	2.72	1.85	1.56	1.33	**
Total SO <sub>2</sub> (mg/L)	174.4	164.8	166.4	164.8	166.4	167.65	ns
Free SO <sub>2</sub> (mg/L)	12.8	12.8	11.2	12.8	12.8	12.8	ns

<sup>b</sup>: as gallic acid; <sup>c</sup>: acetic acid. Significance at which means differ as shown by analysis of variance. \*: significance at  $P < 0.05$ , \*\*:  $P < 0.01$ , ns: not significant.

In our study, it was seen that volatile acidity levels of the vinegar samples were generally correlated with their pH values. Amounts of volatile acidity and value of pH in vinegar samples respectively changed between 36.35-54.94 g/L and 2.68-2.85. As can be seen from our results, volatile acidity of vinegars from surface method was higher than the other. Furthermore, according to Turkish standard, pH of our vinegar samples is optimum.

As can be seen in Table 2, amounts of dry extract of vinegar samples from surface methods were higher than submerged method. In some previous studies, it was reported that in surface method, a nontoxic slime that is known as the mother of vinegar comprise yeast and acetic acid bacteria on the surface. Also some particle in mother of

were suitable descriptors to differentiate vinegar samples according to the raw material and production method.

### Major aroma compounds of vinegars from Dimrit grape

Table 3 shows major aroma compounds of vinegars from Dimrit grape. As can be seen in Table 3, use of different vinegar production methods influenced aroma composition.

It was found that out of acetaldehyde and 2-phenyl ethanol, amounts of aroma compounds in vinegars from surface method were more than submerged method. Also, as can be seen in Table 3, it was determined that amounts of ethyl acetate (4.26 -7.16 mg/L), 2,3 butandiol (3.36-5.0

Table 3. Mean concentrations of major aroma compounds in vinegars (mg/L)

Compound	A1	A2	A3	B1	B2	B3	Significance
Acetaldehyde	0.46	0.52	0.38	0.54	0.40	0.52	ns
Methyl acetate	0.14	0.10	0.08	0.10	0.10	0.08	ns
Ethyl acetate	6.84	7.16	5.74	5.78	4.26	4.68	*
Methyl alcohol	0.32	0.24	0.28	0.24	0.32	0.24	ns
1-propanol	0.22	0.26	0.22	0.06	0.08	0.12	**
2-methyl-1-propanol	0.38	0.5	0.3	0.22	0.22	0.26	*
2-methyl-1-butanol	0.22	0.36	0.26	0.04	0.10	0.06	**
3-methyl-1-butanol	0.50	0.28	0.48	0.12	0.14	0.06	*
2,3-butandiol	5.00	4.66	3.60	3.46	3.36	3.50	ns
2-phenylethanol	2.48	2.38	2.44	3.20	3.42	3.38	**

Significance at which means differ as shown by analysis of variance. \*: significance at  $P < 0.05$ , \*\*:  $P < 0.01$ , ns: not significant.

mg/L) and 2-phenyl ethanol (2.38-3.42 mg/L) in vinegars from both of submerged and surface method were higher than the other aroma compounds. Especially, ethyl acetate was detected in all samples with highest amounts due to their generation during alcoholic fermentation in agreement with previous study (9). Also, it was found that 2-phenylethanol from the yeast amino acid metabolism had considerable concentrations in agreement with previous study (11). As regards to other aroma compounds, our results are in agreement with some studies in literature (13, 26, 34, 35).

As statistical between methods, it was found that amounts of ethyl acetate, 2-methyl-1-propanol and 3-methyl-1-butanol were significantly important ( $P<0.05$ ) and amounts of 1-propanol, 2-methyl-1-butanol and 2-phenylethanol were significantly important ( $P<0.01$ ). Also it was reported that other compounds out of above were not significant.

These findings show that surface method is better in terms of aroma formation than submerged method, whereas it is slower than submerged method. Therefore, both of methods have advantage and disadvantage. For vinegar production, both of methods can be chosen according to our aim.

As a result of this study, it was obtained high

quality vinegar from Dimrit grape. Thanks to this study, Dimrit grape from inconvenient grape varieties for wine production in our country will be evaluated for vinegar production. Thus it will be contribute to economy of country.

### Sensory Analysis

The sensory profiles of the vinegars were built using the marks given for each attribute by the panel. Figure 1 shows the spider chart for each vinegar samples. As can be seen, it was determined that vinegars in terms of aromatic intensity and ethyl acetate odor were significantly different ( $P<0.05$ ), whereas there weren't differences in vinegars in terms of general impression, aromatic richness, pungent sensation, wine character. As a result, vinegar production methods (submerged and surface methods) affected some sensorial characteristics such as aromatic intensity and ethyl acetate odor. In addition to this, higher scores were obtained for sensorial characteristics in surface method. Because aromatic and sensorial quality of vinegar improved, as fermentation time increased (11). Nevertheless as statistical out of aromatic intensity and ethyl acetate odor, there weren't any differences in terms of other aromatic properties.

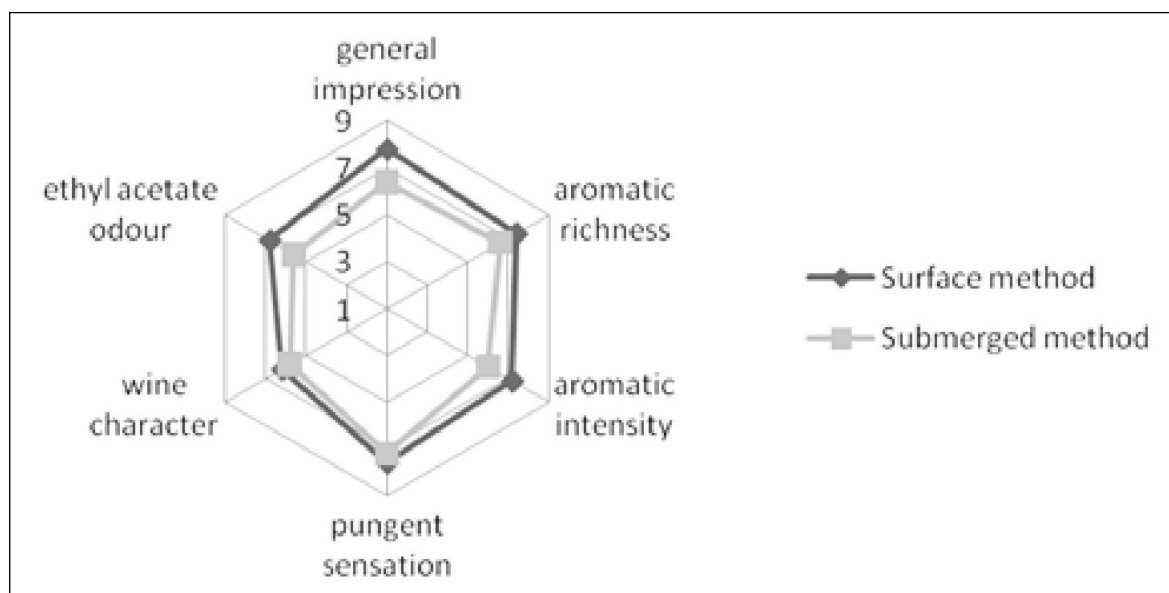


Figure 1. Sensory profile of vinegars

## CONCLUSION

In this study, vinegar was produced by both of submerged and surface method. As a result of this study, it was found that acidification conditions influenced sensorial characteristics, chemical and aroma composition of wine vinegars. Statistical analysis supported the influence of production method on quality characteristics of vinegars. It was determined that especially amounts of aroma components in vinegar from slow methods were higher and this high aroma content made positive effects on quality.

In conclusion, it was found that acidity and content of aroma components in vinegars from slow methods were better, so it was offered this method for vinegar production from Dimrit grape. Thus, Dimrit grape from inconvenient grape varieties in our country was be evaluated for vinegar production instead of wine production.

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