

# **Theses of doctoral dissertation**

DOI: 10.54598/000040

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BUDAPEST  
2020**



**SZENT ISTVÁN UNIVERSITY  
FACULTY OF FOOD SCIENCES  
DOCTORAL SCHOOL OF FOOD SCIENCE**

**Effect of red wine maturation in wooden barrels  
on polyphenol composition and organoleptic  
quality**

Doctoral (PhD) dissertation

**VAS-GULD ZSUZSANNA  
BUDAPEST  
2020.**

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**Discipline:**

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Approval signature of the doctoral school and the supervisors:

The candidate fulfilled all the conditions prescribed in the Doctoral Regulations of Szent István University, the remarks and suggestions made during the workshop discussion were taken into account during the revision of the dissertation, therefore the dissertation can be submitted for defense.

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Approval of the head of the school

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Supervisor's approval

## INTRODUCTION

The topicality of my chosen topic is justified by the fact that nowadays the acquisition of professional wine tasting and wine knowledge is becoming more and more popular among students, businessmen and groups of friends. Conscious wine consumers are looking for red wine varieties on the market that have an outstanding concentration of polyphenols with antioxidant properties, as these wines have a positive physiological effect. One of the aims of my research is the comparative analysis of red wines made under factory conditions during different wooden barrel maturations, as well as the observation and quantification of the changes in the polyphenol composition as a function of sensory evaluations.

The topicality of the dissertation is supported by the fact that the objective approach of sensory evaluation methods is extremely important, both from the supply side and the demand. The need for my research is confirmed by the fact that, studying the current literature, there are few comparative analyzes available in terms of the 100-point OIV grading system and quantitative descriptive analysis (QDA). A further aim of my doctoral dissertation is to use the results of the comparison in the sensory evaluation of wines and to develop sensory methods for winemakers, wine judges, and students in viticultural engineering training for objective judgments. My aim is also to formulate a proposal for practicing winemakers regarding the use of wooden barrels, examining the relationship between the type of wooden barrel and the change in polyphenol concentration.

## OBJECTIVE

The aim of my doctoral dissertation is to investigate the polyphenol composition of different red wines during two-year wooden barrel maturation, in which I selected the components to represent the range of polyphenols that can be added to the wines from grapes or the applied technology or wooden barrel maturation.

- My goal was to examine each of the polyphenol components - *catechin and leucoanthocyanin; color tone and polymerization* - and the change in anthocyanin (*Dolphinidine-3-monoglycoside, Cyanidin-3-monoglycoside, Petunidin-3-monoglycoside, Peonidine-3-monoglycoside, Malvidin-3-monoglycoside*) concentration as a function of maturation time.
- Another goal of my research is to chemically monitor the polyphenol composition maturation phases of wine under operating conditions, to show the changes in the concentration focused on *trans-piceid and trans-resveratrol* during the two-year wooden barrel maturation.
- My further goal was to determine the presence and amount of *volatile phenols* that may develop as a function of maturation in wooden barrels.

From a scientific point of view, my aim is to compare and evaluate the methods developed for sensory evaluation, based on the results of which,

- development of a QDA reference system that performs multi-level comparisons between commercially available wine samples, ie reference samples and experimental factory barrels,
- comparison of the two sensory analysis methods (QDA and OIV system).

My goal is to determine the significance of the use of wooden barrels in winemaking, from both a technological and sensory approach.

- My goal is to be able to make a recommendation to practicing winemakers about the use of wooden barrels, in terms of how the type of wooden barrel and barrel maturation influence the change in the polyphenol composition of the wine.

## MATERIAL AND METHOD

In the Szekszárd wine region of the Pannonian Wine Region, I examined three character wine types: Kadarka, Kékfrankos and Cabernet franc. I present the harvest dates and the method of making the wines on the basis of the information summarized in the table below, which includes the date of harvest, alcoholic fermentation and other data of maturation in wooden barrels.

Wine type	<b>Kadarka</b>	<b>Kékfrankos</b>	<b>Cabernet franc</b>
<b>Harvest date</b>	mid-October	end of October	beginning of October
<b>Wine auxiliaries</b>	UVAFERM BDX (Danstar Ferment AG, Zug, Switzerland)	UVAFERM BDX (Danstar Ferment AG, Zug, Switzerland)	UVAFERM BDX (Danstar Ferment AG, Zug, Switzerland)
<b>Duration of alcoholic fermentation</b>	14 days	21 days	14 days
<b>Temperature of alcoholic fermentation</b>	25-28 °C	18-24 °C	25-28 °C
<b>Aces barrel type</b>	first filled Zemplén and Mecsek oaks	five-year-old, quarter- filled Zemplén oak	five-year-old, quarter- filled Zemplén oak
<b>Barrique barrel type</b>	first filled Zemplén and Mecsek oaks	third-filled Zemplén oak	third-filled Zemplén oak

Regarding instrumental analytical studies, I performed routine analytical, spectrophotometric, and chromatographic studies.

- The spectrophotometric examinations were performed with a MOM Spektromom 195 instrument in the research laboratory of Szent István University, Department of Oenology.
- Qualitative and quantitative determination of resveratrols and anthocyanin monomers was performed by high performance liquid chromatography (HP Series 1050) also in the research laboratory of the Department of Oenology of Szent István University.
- Sampling was performed in April 2014 and September 2014, and in April 2015 and September 2015 from both wooden barrels and the control tank. The sample preparation and analysis required for the analysis of volatile phenols, similarly to the anthocyanin test

methods, was performed by high-performance liquid chromatography in isocratic mode, from both barrique and ace barrel and counter-samples.

- Sensory examinations were designed with the elements of good sensory practice (GSP) in mind. The evaluators performed the evaluation based on two sensory evaluation methods, the 100-point OIV evaluation method, which is a fully accepted evaluation method in Hungary for determining the quality of wines, and the quantitative descriptive analysis (QDA), which is an internationally accepted standard method for characterizing foods. During the quantitative descriptive analysis (QDA) and the sensory comparative analysis of the 100-point OIV evaluation method, I compared the barrels of Kadarka, Kékfrankos and Cabernet franc produced under operating conditions with the commercially available Kadarka, Kékfrankos and Cabernet franc red wines. based on specific sensory properties.
- Informative sensory evaluation of volatile phenols was performed in a 10-point evaluation system, in which 1 point was the worst / negative and 10 points the best / positive score.

The results were evaluated by the following statistical methods:

- The performance of the trained reviewers was evaluated with Panel Check Software. PanelCheck provides all the information needed to evaluate repeatability, the distinctiveness of reviewers, and also the panel's agreement.
- The methods of analysis of variance (ANOVA) and factorial analysis of variance, STATISTICA 13 (TIBCO Software Inc., Palo Alto, CA, USA) were used in the statistical analysis because I compared the samples based on different properties - sensory properties or polyphenol concentration values in the present research.
- For principal component analysis (PCA) I used XL-Stat software (ADDINSOFT 2019) (Addinsoft, Long Island, NY, USA) for principal component analysis of reviewers and various sensory properties.
- I used XL-Stat software (Addinsoft, Long Island, NY, USA) (ADDINSOFT 2019) to run multiple factor analysis (MFA), as the aim was to analyze several different data sets together.
- For the classification estimation of different wine samples (commercial and factory barrel samples), I used the PLS-DA (partial least squares discriminant analysis) method to compare commercial wines and factory barrel samples separately using the 100-point grading method and based on QDA.

## NEW SCIENTIFIC RESULTS

1.) **I found** that *catechin and leucoanthocyanin* concentrations decreased during maturation due to polymerization. The decrease in catechin concentration was due to the polymerization of catechin monomers, while the decrease in leucoanthocyanin concentration was due to polymerization and oxidation processes. My further finding is that hue and polymerization showed a negative correlation, which is explained by the fact that during longer maturation, the degree of polymerization increases while hue decreases or remains unchanged.

2.) **I determined** and compared the content of three different varieties but of red wine trans-resveratrol from the same wine region. Using factor analysis of variance (ANOVA), I showed that the concentration of the three types of trans-resveratrol was not significantly affected by the use of different barrels (aces, barrique, steel tank) during the maturation period.

3.) Based on my results, **I showed that** the decrease in anthocyanin concentration over time can be considered statistically significant for different cultivars ( $\alpha = 0.05$ ). The most significant decrease compared to the initial values was observed in the Kékfrankos variety, at the concentrations of the following five compounds (*Dolphinidine-3-monoglycoside*, *Cyanidin-3-monoglycoside*, *Petunidin-3-monoglycoside*, *Peonidine-3-monoglycoside*, *Malvidin-3-monoglycoside*). This trend may have been a consequence of long wooden barrel maturation and polymerization.

4.) In the case of Kékfrankos, in addition to the anthocyanin content, the concentration of trans-resveratrol was also significantly higher compared to the Kadarka and Cabernet franc cultivars, taking into account the entire maturation period. According to my results, I can state that the grape variety primarily determines the concentration of trans-resveratrol, and then the applied oenological technology and wine treatment methods also appear as an influencing factor, which is also supported by the results of previous research. The harvest date, end of October, and the oenological process used, the duration of fermentation on the skins is 21 days, and a fermentation temperature of 18-24 ° C greatly contributes to the result. Resveratrol may also be released from the piceid during fermentation and maturation. At the second sampling time, the transpiceid concentrations determined in my Kékfrankos samples were higher, present in twice the amount as in the other two examined wine varieties.



5.) From my research results, **I also found** that after a one-and-a-half-year wooden barrel aging period, the concentration of 2-phenylethyl alcohol was outstanding in Kékfrankos wines and in my Kadarka barrel samples, as the concentration almost doubled from the values measured six months earlier, which exceeded the values in the literature (10-75mg / l). This increase in concentration can be explained by the fact that during the given wine-making technology, their concentration increased significantly due to the enzymatic processes and hydrolysis caused by the applied starter culture.

6.) **I created** a list of organoleptic evaluation properties consisting of 27 traits and their associated references for the evaluation of red wines, which is suitable for determining the sensory fingerprint of red wines, comparing different samples, and quantifying similarities and differences.

7.) **I have identified** that quantitative descriptive analysis (QDA) makes a more accurate comparison than the OIV rating system, although the need for resources, time and energy is higher. QDA is able to describe samples in more detail, listing positive and negative properties at the same time.

8.) **I have developed** a multivariate statistical method based on multiple factor analysis to compare the performance of wine evaluation methods, during which the joint analysis of several data sets with different structures can be performed by multi-factor analysis (MFA).

9.) **I developed** a methodology for the objective monitoring and evaluation of sensory performance in the examination of Hungarian wines, and I showed that the monitoring of individual and panel performance is best supported by the PanelCheck sensory target software. In the course of my work, I have demonstrated that by following the statistical methods of Workflow offered by the software, reviewers in need of further practice can be properly identified. Panel performance analysis would be the most appropriate method to best rule out subjectivity by analyzing the following diagrams:

1. Mixed model (ANOVA) diagram
2. Tucker joint points chart
3. Tucker correlation chart

4. Manhattan chart

5. F value chart

6. MSE plot diagram

However, subjectivity is not diminished by the method but by the training. The more qualified a reviewer is, the better the result. This requires a scrutiny, and the methods listed for checking are excellent and proven to be effective. If the judges are inexperienced, no matter what method we work with, the result will be unreliable.

10.) Based on the statistical analysis (ANOVA), **I found** that both the wine variety and the measurement time result in a significant change in the examined variables together. The oak barrels made of Hungarian sessile oak (*Quercus Petraea*) used for the aging of the wines are also suitable for the long, 24-month wooden barrel aging, which is also supported by previous research. It can be clearly stated that in the case of wines aged in wooden barrels, the phenolic components are strongly influenced by the grape variety, the type of wooden barrel and the maturation time.

## CONCLUSIONS AND RECOMMENDATIONS

In addition to the technology of grape processing and winemaking, paying special attention to the time of maturation in wooden barrels and the type of wooden barrel, I examined three different wine varieties, Kékfrankos, Kadarka and Cabernet franc. During maturation, the anthocyanin concentration showed a decrease, which occurred during the long wooden barrel maturation due to the polymerization of polyphenols. Of the three different wines examined, Kékfrankos showed the highest initial anthocyanin and trans-resveratrol concentrations. Based on my statistical results, the type and size of the wooden barrel did not significantly influence the change in polyphenol concentrations, but the grape variety, the selection of the harvest date - in addition to the examination of the technological maturity of the grapes - and the winemaking technology played a greater role.

Although I did not take vintage factors into account in my research, my results suggest that grape variety and harvest date, grape ripening status, also primarily determine trans-resveratrol and anthocyanin concentrations. During my doctoral work, I used and compared two sensory methods, the 100-point grading method and the quantitative descriptive analysis (QDA) method, in the evaluation of three red wine varieties. References for quantitative descriptive analysis were determined in 27 sensory properties. My results showed that the QDA method is more sensitive to differences between samples, so it has a higher discriminative ability. Factor analysis of variance, principal component analysis, and partial least squares discriminant analysis clearly highlighted this difference for all wines. As a suggestion, in the future the QDA method, with the development of a special wine description and reference set, may be suitable to replace and supplement the general OIV method. It is important to note that although there are some descriptive kits in the literature with reference materials, none of them have been defined as standards. Sensory evaluation was performed by humans, so it must be taken into account that the human factor always causes a higher deviation than instrumental measurements. To address the discrepancy, sensory exercises should be performed where evaluators' weaknesses can be identified and appropriate, personalized practices used to improve their sensory abilities. My results highlight the importance of performing panel tests during wine judgments, which is also statistically recommended.

In my research, I compared wines - Kadarka, Kékfrankos and Cabernet franc - produced under factory conditions - untreated, barrel batches - with commercially purchased bottled wines, Kadarka, Kékfrankos and Cabernet franc. The use of reference samples reduced the difference between the reviewers so I got more reliable data. Sensory tests based on international standards (ISO) implemented with IT support effectively aid in the design, implementation, and analysis of judgments. However, reviewers' performance monitoring systems in many cases focus on only a few areas, thus not allowing complex performance evaluation of panels (XL-StatSensory, ProfiSense). Currently, PanelCheck supports the most versatile monitoring of individual and panel performance. In my research, I have demonstrated that by following the statistical methods of Workflow, areas for improvement can be properly identified. By continuously monitoring the sensory evaluators, the basis of quality management systems can be effectively implemented, with the help of quick preventive or corrective measures taken to increase the performance of the panel, forming an integral part of the quality management system of sensory laboratories.

## PUBLICATIONS

### Impact factor journal articles:

1. **Guld Zsuzsanna**, Nyitrai Sárdy Diána, Gere Attila, Rácz Anita:  
Comparison of sensory evaluation techniques for Hungarian wines  
JOURNAL OF CHEMOMETRICS 34: 4 Paper: e3219 (2020)  
**Impact faktor: 1.633**
2. **Guld Zsuzsanna**, Rácz Anita, Tima Helga, Kállay Miklós, Nyitrai Sárdy Diána:  
Effects of aging in oak barrels on the trans-resveratrol and anthocyanin concentration of red wines from Hungary  
ACTA ALIMENTARIA: AN INTERNATIONAL JOURNAL OF FOOD SCIENCE  
48: 2 (2019) **Impact Factor: 0.384**
3. Tima Helga, **Guld Zsuzsanna**, Mohácsi-Farkas Csilla, Kiskó Gabriella, Rácz Anita:  
Deoxynivalenol, zearalenone and T-2 in grain based swine feed in Hungary  
FOOD ADDITIVES AND CONTAMINANTS PART B - SURVEILLANCE 9: 4 pp.  
275-280., 6 p. (2016) DOI WoS **Impact Factor: 1.33**

### NO IF journal article, in a foreign language:

1. Máté Andrea, Oroszi Viktor György, Slezák-Bartos Zsuzsanna, **Guld Zsuzsanna**  
Wine consumption and wine purchasing habits of young consumers in Hungary  
TRANSYLVANIAN JOURNAL OF TOURISM AND TERRITORIAL  
DEVELOPMENT 2: 1 pp. 30-46., 17 p. (2017).

### NO IF journal article, in Hungarian:

1. **Guld Zsuzsanna**, Tima Helga, Nyitrai Sárdy Diána, Kállay Miklós:  
Changes in polyphenol composition of red wines during different storage  
OENOLOGICAL BOOKLETS 26: 4 pp. 7-10., 4 p. (2015)
2. **Guld Zsuzsanna**:  
What does the year 2014 hide for the winemaker and winemaker?  
OENOLOGICAL BOOKLETS 21: 3 p. 16 (2014)
3. **Guld Zsuzsanna**:  
What are the health benefits of polyphenols?  
PRIMARY PRODUCER: FARMERS 'SHEET: 4 pp. 118-119., 2 p. (2014)

### Book, book excerpt:

1. Angler Kinga, **Guld Zsuzsanna**, Krizl Edit, Máté Andrea, Slezák-Bartos Zsuzsanna: Food Product Development - Whole Wheat Bread with Red Wine and Red Onion In: Gonda, Tibor (szerk.) Current issues of tourism and rural development in the Carpathian Basin: Studies on tourism and rural development Szekszárd, Magyarország: PTE, Faculty of Cultural Studies, Teacher Training and Rural Development, (2016) pp. 5-19., 15 p.
2. **Guld Zsuzsanna**: Viticulture and oenology trend research In: N., Horváth Béla (szerk.) The future of one of the leading sectors of Tolna county: viticulture - wine trend research Pécs, Hungary: University of Pécs, (2015) pp. 71-87., 17 p.
3. Oroszi Viktor, Gonda Tibor, **Guld Zsuzsanna**, Máté Andrea: New trends in wine tourism - Good wine tourism practices in the Pannonian Wine Region In: N., Horváth Béla (szerk.) The future of one of the leading sectors of Tolna county: viticulture - wine trend research Pécs, Hungary: University of Pécs, (2015) pp. 15-42., 28 p.

### Conference publications

#### In Hungarian, complete:

1. **Guld Zsuzsanna**, Nyitrainé Sárdy Diána, Kállay Miklós: Changes in the polyphenol content of red wines based on the technology of winemaking and maturation in wooden barrels In: Szabó, István; Bohonyi, Noémi; Haffner, Tamás; Horváth, Orsolya; Márhoffer, Nikolett; Molnár, Emese; Pál, Eszter; Schaub, Anita; Varga, Zoltán (szerk.) IV. Interdisciplinary Doctoral Student Conference 2015: 4th Interdisciplinary Doctoral Conference 2015 Pécs, Hungary: University of Pécs Doctoral Student Municipality, (2015) pp. 495-500., 6 p.

#### Summary in Hungarian:

1. **Guld Zsuzsanna**, Kállay Miklós, Nyitrainé Sárdy Diána: Changes in volatile phenols in red wines during different wooden barrel maturation methods In: Ács, Kamilla; Bencze, Noémi; Bódog, Ferenc; Haffner, Tamás; Hegyi, Dávid; Horváth, Orsolya Melinda; Hüber, Gabriella Margit; Kovács, Áron; Kis Kelemen, Bence; Lajkó, Adrienn; Schilli, Gabriella Krisztina; Szendi, Anna; Szilágyi, Tamás Gábor; Varga, Zoltán (szerk.) Book of abstracts –V. Interdisciplinary Doctoral Student Conference Pécs, Hungary: University of Pécs Doctoral Student Municipality, (2016) p. 136.
2. Kovács Vilmos, Slezák-Bartos Zsuzsanna, Krizl Edit, Angler Kinga, **Guld Zsuzsanna** Quality assurance in the food industry in Hungary and Serbia through concrete examples in the field of winemaking and brandy making In: Pintér, R; Csapó, J (szerk.) VIII. National Tourism Conference: "Environmental safety-tourism " Book of abstracts Pécs, Hungary Institute of Geography, University of Pécs, (2016) p. 19.