# Stability of fatty acid composition, phenolic and volatile compounds in varietal extra virgin olive oils during storage and heating

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DOCTORAL DISSERTATION

Zagreb, 2023

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DOCTORAL DISSERTATION

Supervisor:
Karolina Brkić Bubola, Ph.D.

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Sveučilište u Zagrebu

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Dora Klisović

# STABILNOST FENOLNIH I HLAPLJIVIH TVARI TE SASTAVA MASNIH KISELINA SORTNIH EKSTRA DJEVIČANSKIH MASLINOVIH ULJA TIJEKOM SKLADIŠTENJA I ZAGRIJAVANJA

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# STABILITY OF FATTY ACID COMPOSITION, PHENOLIC AND VOLATILE COMPOUNDS IN VARIETAL EXTRA VIRGIN OLIVE OILS DURING STORAGE AND HEATING

**Dora Klisović, MSc. med.chem.**

**Thesis performed at** the Institute of Agriculture and Tourism, Poreč
**Supervisor:** Karolina Brkić Bubola, Ph.D., Scientific Advisor

**Short abstract**
This study aimed to investigate the influence of real storage conditions and daily consumption, heating, and presence of food during storage (dried tomatoes, cheese) and heating (vegetables) of monovarietal EVOOs on its fatty acid composition, phenolic and volatile compounds, antioxidant activity, sensory properties, and quality. The results indicated that under consumption conditions used in this study (gradual headspace increasing, darkness, room temperature), EVOO's composition of fatty acid, phenolic and volatile compounds remained preserved within one month. The presence of food during storage and heating significantly decreased the quality and the concentration of total identified phenolic and volatile compounds of the used EVOO. The use of monovarietal oils indicated varietal specificity under different conditions of its use, related to the composition of fatty acids, phenolic and volatile compounds.

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# STABILNOST FENOLNIH I HLAPLJIVIH TVARI TE SASTAVA MASNIH KISELINA SORTNIH EKSTRA DJEVIČANSKIH MASLINOVIH ULJA TIJEKOM SKLADIŠTENJA I ZAGRIJAVANJA

**Dora Klisović, mag. med. chem.**

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**Mentor:** dr. sc. Karolina Brkić Bubola, znanstvena savjetnica

**Kratki sažetak disertacije**
Cilj ovog istraživanja bio je ispitati utjecaj realnih uvjeta upotrebe i svakodnevnog korištenja, zagrijavanja i prisutnosti hrane tijekom skladištenja (sušena rajčica, sir) i zagrijavanja (povrće) sortnih EDMU na sastav masnih kiselina, fenola i hlapljivih spojeva, antioksidacijsku aktivnost, senzorska svojstva i kvalitetu ulja. Dobiveni rezultati pokazali su da je sastav masnih kiselina, fenolnih i hlapljivih spojeva EDMU u uvjetima svakodnevnog korištenja (postepeno povećavanje nadprostora boce, bez svjetlosti, sobna temperatura) ostao nepromijenjen unutar mjesec dana skladištenja. Prisutnost hrane tijekom skladištenja i zagrijavanja značajno je utjecala na smanjenje kvalitete te koncentracije ukupnih identificiranih fenolnih i hlapljivih spojeva korištenog EDMU. Primjenom sortnih ulja utvrđena je sortna specifičnost u različitim uvjetima uporabe vezana uz početni sastav masnih kiselina, fenolnih i hlapljivih spojeva.

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**Ključne riječi:** antioksidacijska aktivnost, ekstra djevičansko maslinovo ulje, fenolni spojevi, hlapljivi spojevi, sastav masnih kiselina, senzorska svojstva, skladištenje, zagrijavanje

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1. izv. prof. dr. sc. Klara Kraljić
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**INFORMATION ABOUT THE SUPERVISOR**

Ph.D. Karolina Brkić Bubola is employed as a Senior Research Associate at the Institute for Agriculture and Tourism in Poreč. Since 2018, she has been elected to the scientific position of Scientific Advisor. She graduated in 2004 at the Faculty of Food Technology and Biotechnology, University of Zagreb, where she defended her doctoral thesis in 2011 and obtained the academic degree of Doctor of science in the field of biotechnical sciences, food technology. She has been employed at the Institute since 2005, where since 2005 she has been the deputy head of the Laboratory of Food Technology and Biotechnology, and since 2008 she has been the head of the accredited and official Panel for sensory analysis of virgin olive oils, recognized by the International Olive Council (IOC) and the Ministry of Agriculture of the Republic of Croatia. She participated in 12 scientific projects, mostly related to the quality and technology of olive oils and sensory properties of agro-food products. She was the principal investigator of the VIP project "Application of filtration for the purpose of improving the quality of olive oils" financed by the Ministry of Agriculture of the Republic of Croatia, and the leader of the research group of the Institute within the HORIZON 2020 project "Advanced solutions for assuring the overall authenticity and quality of olive oil - OLEUM" financed by the EU. Since 2018, she has been the head of two career development projects for young researchers founded by the Croatian Science Foundation (DOK-2018-09-2293 and DOK-2018-01-4693). To date, 1 book chapter and more than 40 scientific papers have been published, of which 31 papers are represented in the CC and SCI Expanded databases. She participated in more than 40 international conferences, at 3 of them she was a member of the organizing committee. She was cited more than 400 times with an h-index of 14. She reviewed about a hundred scientific articles for 15 reputable scientific journals. In 2008, she received an IOC scholarship for training in the sensory analysis of olive oils at the University of Jaén, Spain. In 2014, she was the winner of the "Knight of Croatian Olive Oil" charter, an award for scientific and professional work in the field of olive oil production. She is a member of professional societies: European Federation of the Science and Technology of Lipids, and Croatian Society of Food Technologists, Biotechnologists and Nutritionists. She is a member of the international judging panels of the world olive oil competitions (NYIOOC, New York, USA; EVOOLEUM, Cordoba, Spain). Since 2014, she has been a member of the working group of experts for the analysis of olive oils in the European Commission, and since 2018, a member of the IOC working group for the sensory analysis of olive oil (representative of Croatia). Since 2021, she has been a member of the Scientific field committee for biotechnical sciences of the Agency for science and higher education.

**STABILITY OF FATTY ACID COMPOSITION, PHENOLIC AND VOLATILE COMPOUNDS IN VARIETAL EXTRA VIRGIN OLIVE OILS DURING STORAGE AND HEATING**

The usage of extra virgin olive oil (EVOO) in food preparation implies its frequent contact with air, exposition to high temperatures, and interaction with other food ingredients. The influence of real conditions of EVOO usage in households has been insufficiently investigated. Therefore, this study aimed to investigate the influence of real storage conditions and daily consumption, heating, and presence of food during storage (dried tomatoes, cheese) and heating (vegetables) of monovarietal EVOOs on its fatty acid composition, phenolic and volatile compounds, antioxidant activity, sensory properties, and quality. The results indicated that under consumption conditions used in this study (gradual headspace increasing, darkness, room temperature), EVOO's composition of fatty acid, phenolic and volatile compounds remained preserved within one month. The presence of food during storage and heating significantly decreased the quality and the concentration of total identified phenolic and volatile compounds of the used EVOO. This investigation unrevealed the important role of the initial composition of the EVOO (phenolic compounds), but also of the food (moisture content) immersed in EVOO, as parameters that influence the degradation rate of EVOO during simultaneous storage and heating. The use of monovarietal oils indicated varietal specificity under different conditions of its use, related to the composition of fatty acids, phenolic and volatile compounds.

**Keywords:** *antioxidant activity, extra virgin olive oil, fatty acid composition, heating, phenolic compounds, quality; storage, sensory attributes, volatile compounds*

**STABILNOST FENOLNIH I HLAPLJIVIH TVARI TE SASTAVA MASNIH KISELINA SORTNIH EKSTRA DJEVIČANSKIH MASLINOVIH ULJA TIJEKOM SKLADIŠTENJA I ZAGRIJAVANJA**

Prilikom upotrebe ekstra djevičanskog maslinovog ulja (EDMU) u pripremi jela dolazi do njegovog učestalog kontakta sa zrakom, izloženosti visokim temperaturama i interakcijama sa sastojcima druge hrane. Utjecaj realnih uvjeta upotrebe EDMU u domaćinstvima nedovoljno je istražen. Prema tome, cilj ovog istraživanja bio je ispitati utjecaj realnih uvjeta upotrebe i svakodnevnog korištenja, zagrijavanja i prisutnosti hrane tijekom skladištenja (sušena rajčica, sir) i zagrijavanja (povrće) sortnih EDMU na sastav masnih kiselina, fenola i hlapljivih spojeva, antioksidacijsku aktivnost, senzorska svojstva i kvalitetu ulja. Dobiveni rezultati pokazali su da je sastav masnih kiselina, fenolnih i hlapljivih spojeva EDMU u uvjetima svakodnevnog korištenja (postepeno povećavanje nadprostora boce, bez svjetlosti, sobna temperatura) ostao nepromijenjen unutar mjesec dana skladištenja. Prisutnost hrane tijekom skladištenja i zagrijavanja značajno je utjecala na smanjenje kvalitete te koncentracije ukupnih identificiranih fenolnih i hlapljivih spojeva korištenog EDMU. Ovo istraživanje ukazalo je na važnu ulogu početnog kemijskog sastava EDMU (fenolni spojevi), ali i sastava korištene hrane (udio vode), kao parametara koji utječu na brzinu degradacije EDMU prilikom zajedničkog skladištenja i zagrijavanja. Primjenom sortnih ulja utvrđena je sortna specifičnost u različitim uvjetima uporabe vezana uz početni sastav masnih kiselina, fenolnih i hlapljivih spojeva.

**Ključne riječi:** *antioksidacijska aktivnost, ekstra djevičansko maslinovo ulje, fenolni spojevi, hlapljivi spojevi, kvaliteta, sastav masnih kiselina, senzorska svojstva, skladištenje, zagrijavanje*

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## LIST OF ABBREVIATIONS

ANOVA – Analysis of variance

B – Buža

DPPH – 2,2-diphenyl-1-picrylhydrazyl

EEC – European Commission Regulation

EFSA – European Food Safety Authority

EVOO – Extra virgin olive oil

FFA – Free fatty acid

GC-FID – Gas chromatography–flame ionization detection

GC-MS – Gas chromatography–mass spectrometry

HPLC – High performance liquid chromatography

IB – Istarska bjelica

IOC – International Olive Council

L – Leccino

MUFA – Monounsaturated fatty acids

VOO – Virgin olive oil

PCA – Principal component analysis

PUFA – Polyunsaturated fatty acids

PV – Peroxide value

RAF – Refined olive oil

RSA – Radical-scavenging activity

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*Other parts of the thesis will be available after the publication of all the research data.*

*(Ostali dijelovi doktorata bit će dostupni nakon objave svih podataka)*

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# Autobiography

Dora Klisović, MSc, was born in Rijeka on November 11th, 1994. After finishing grammar school, she graduated from the Department of Biotechnology at the University of Rijeka in 2018, where she gained an MSc in Medicinal Chemistry. Since 2019 she is employed at the Institute of Agriculture and Tourism in Poreč as a research assistant, hired under the Young researcher career program - Training of new doctoral students by the Croatian Science Foundation and under the OLEUM project – *Advanced solutions for assuring the overall authenticity and quality of olive oil* financed from the European Union's Horizon 2020. She is finishing her Postgraduate University Doctoral Study at the Faculty of Food Technology and Biotechnology at the University of Zagreb, in which she has enrolled in 2019. Her working skills are primarily related to analytical methods for determining oil quality, which include work on sophisticated devices (liquid and gas chromatography) for determining the composition of fatty acids, phenolic and volatile compounds, and sensory analysis of olive oils. During her employment, she spent two months at the University of Barcelona, Spain, as part of an Erasmus + scholarship for a professional stay, where she gained new knowledge on the gas chromatography analysis of virgin oils. She presented the results of her research at numerous international conferences, five of which were results produced from the doctoral research presented in the form of posters or oral presentations. To date, her research work has resulted in eight scientific papers indexed in Web of Science/Current Contents Connect in the field of food technology, while four of them were a product of the doctoral research where she was the main or corresponding author.

## LIST OF AUTHORS PUBLICATIONS

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