Three-factor approach for modulating the content of phenols and taste attributes of Oblica cv. (*Olea europaea* L.) virgin olive oil

Trofaktorijalni pristup modulaciji sadržaja fenola i svojstava okusa djevičanskog maslinovog ulja kultivara Oblica (*Olea europaea* L.)

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INTRODUCTION

Phenols are responsible for virgin olive oil (VOO) taste characteristics and significantly determine VOO oxidative stability and shelf life, as well as its nutritive value. The content of phenols in VOO depends on many agronomic and technological factors and olive fruit ripening degree and paste malaxation duration and temperature are among the most important. The aim of this study was to investigate the main effects and interactions of these three key factors on the content of phenols and related taste sensory characteristics of Oblica cv. (*Olea europaea* L.) VOO.

MATERIALS AND METHODS

Olives were picked at three ripening degrees as green (RD1), semi-ripe (RD2) and ripe (RD3), and processed by malaxation at 22 / 30 °C, for 30 / 60 min. The obtained oils were subjected to HPLC-DAD analysis of phenols and sensory analysis by the official IOC method.

RESULTS of three-way analysis of variance (ANOVA) for the concentrations of phenols in Oblica cv. virgin olive oils, with ripening degree, malaxation temperature and malaxation duration as factors ($p < 0.05$).

Factor		Phenols (mg/kg)																			
		TYR	HYT	HAC	VAN	VAC	PCM	LUT	API	PIN	ACP	SCG	EAg I	3,4-D	Oag I	*p*-HP	O+Lag	Oag II	Lag III	Oag III	TOS	TOP
RD	RD1	10.96^{a}	13.87^{a}	0.23^{c}	0.24^{a}	0.62^{a}	11.22^{a}	3.48^{b}	0.26^{b}	1.19	10.89^{a}	0.03^{b}	0.03^{b}	168.55^{c}	124.95^{a}	70.79^{c}	156.18^{a}	196.19^{a}	7.49	10.83	735.04^{a}	788.01^{a}
	RD2	7.82^{b}	12.80^{a}	0.38^{b}	0.12^{b}	0.16^{b}	5.36^{b}	5.33^{a}	0.47^{a}	1.22	8.05^{b}	0.03^{b}	0.04^{a}	302.85^{a}	61.91^{b}	163.15^{b}	59.16^{b}	129.11^{b}	6.09	7.56	729.91^{a}	771.32^{a}
	RD3	10.26^{a}	7.02^{b}	0.66^{a}	0.10^{c}	0.04^{c}	2.64^{c}	2.78^{c}	0.23^{b}	1.14	3.13^{c}	0.04^{a}	0.04^{a}	239.40^{b}	4.98^{c}	220.07^{a}	14.81^{c}	12.02^{c}	7.94	10.12	509.43^{b}	537.43^{b}
	F-value	12.78	21.09	41.99	373.73	2041.52	830.13	130.26	195.51	1.88	807.71	5.15	15.75	20.05	799.70	151.23	2486.99	365.26	0.51	1.24	24.37	27.48
MT (°C)	22	9.72	10.72	0.42	0.14^{b}	0.30^{a}	7.12^{a}	4.10^{a}	0.35^{a}	1.22	7.33	0.03	0.04	175.41^{b}	63.43	117.40^{b}	78.76^{a}	105.79^{b}	5.15^{b}	6.79^{b}	552.77^{b}	594.18^{b}
	30	9.64	11.55	0.43	0.18^{a}	0.24^{b}	5.69^{b}	3.63^{b}	0.29^{b}	1.15	7.39	0.04	0.04	298.46^{a}	64.47	185.28^{a}	74.69^{b}	119.10^{a}	9.19^{a}	12.22^{a}	763.48^{a}	803.66^{a}
	F-value	0.02	0.83	0.12	85.95	53.05	67.02	12.34	23.94	3.85	0.14	0.07	0.25	50.44	0.18	92.08	5.86	5.59	6.72	9.19	48.91	46.08
MD (min)	30	9.52	11.13	0.49^{a}	0.14^{b}	0.28	6.53	4.04^{a}	0.33^{a}	1.17	7.50	0.03	0.04	228.09	70.47^{a}	140.20^{b}	82.33^{a}	117.29	6.67	8.79	653.19	695.04
	60	9.84	11.13	0.36^{b}	0.17^{a}	0.27	6.28	3.70^{b}	0.31^{b}	1.19	7.21	0.04	0.04	245.78	57.43^{b}	162.48^{a}	71.10^{b}	107.60	7.67	10.22	662.34	702.80
	F-value	0.36	0.00	11.05	27.11	3.13	1.99	6.64	5.50	0.40	3.40	0.07	1.00	1.04	28.33	9.92	44.99	2.97	0.42	0.64	0.08	0.06
Interactions	RD x MT	n.s.	*	n.s.	*	*	*	*	*	*	*	n.s.	n.s.	*	n.s.	*	*	n.s.	n.s.	*	*
	RD x MD	*	*	*	*	*	*	*	n.s.	n.s.	*	n.s.	n.s.	n.s.	*	n.s.	*	*	n.s.	n.s.	*	*
	MT x MD	n.s.	*	n.s.	n.s.	n.s.	n.s.	*	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.	*	n.s.	*	n.s.	n.s.	n.s.	n.s.	n.s.
	RD x MT x MD	n.s.	n.s.	n.s.	n.s.	*	n.s.	n.s.	n.s.	n.s.	*	n.s.	n.s.	n.s.	*	*	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.

* - interaction significant; n.s. - interaction not significant.

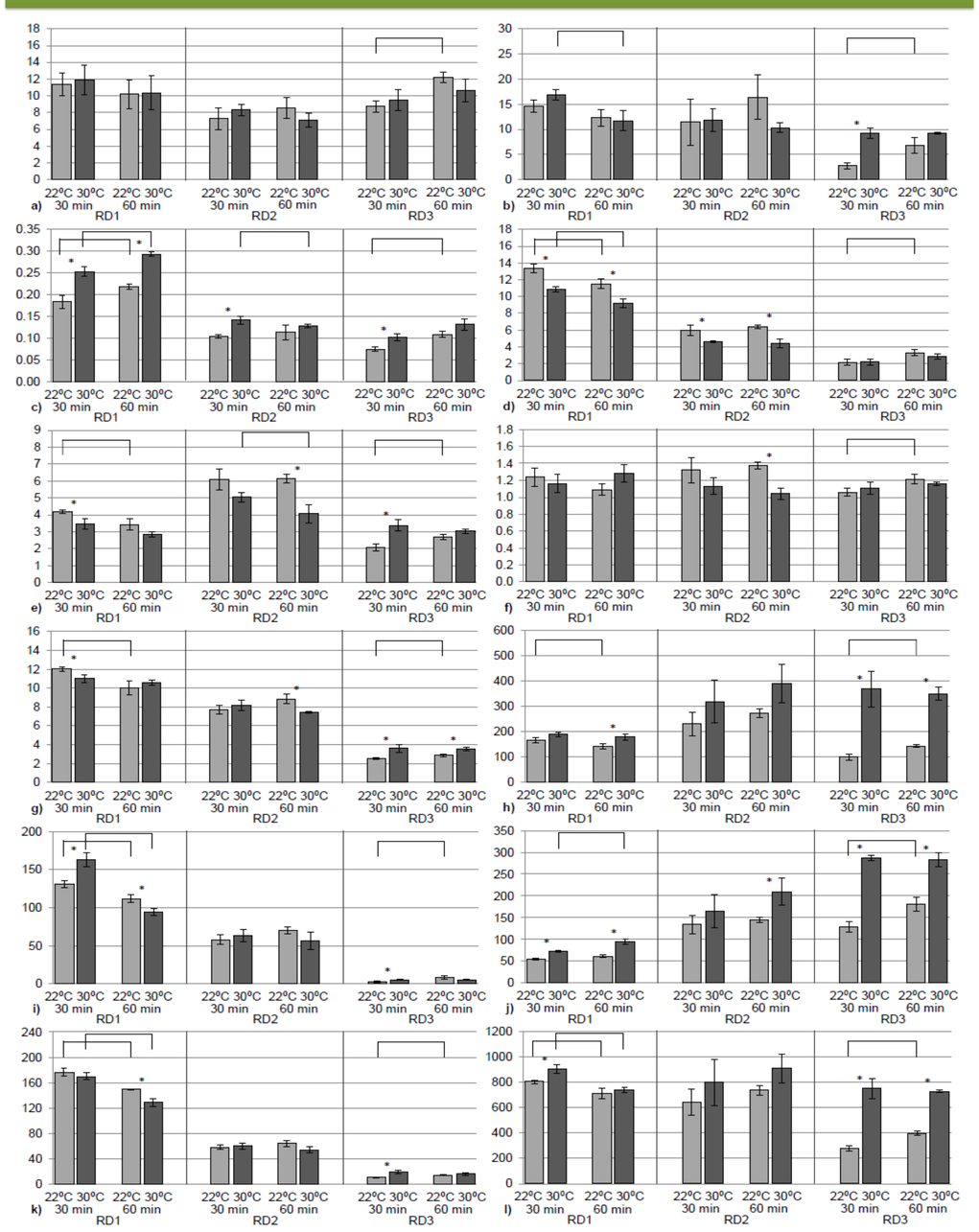
Identification of factors: RD - ripening degree (RD1: *green* fruitiness, for RD2: *green* and *ripe* fruitiness, for RD3: *ripe* fruitiness); MT - malaxation temperature; MD - malaxation duration. Identification of phenols, simple phenols: **a) TYR – tyrosol; b) HYT – hydroxytyrosol; HAC - hydroxytyrosol acetate; c) VAN - vanillin;** phenolic acids: **VAC - vanillic acid; d) PCM - p-coumaric acid;** flavonoids: **e) LUT - luteolin; API - apigenin;** lignans: **f) PIN - pinoresinol; g) ACP - acetoxypinoresinol;** secoiridoids: **SCG - secologanoside; EAg I - elenolic acid glucoside (isomer I); h) 3,4-D - 3,4-DHPEA-EDA; i) Oag I - oleuropein aglycone (isomer I); j) p-HP - p-HPEA-EDA; k) O+Lag - oleuropein + ligstroside aglycones; Oag II - oleuropein aglycone (isomer II); Lag III - ligstroside aglycone (isomer III); Oag III - oleuropein aglycone (isomer III); TOS - total secoiridoids; l) TOP - total phenols.**

RESULTS of three-way analysis of variance (ANOVA) for the intensities and scores of sensory attributes of Oblica cv. virgin olive oils, with ripening degree, malaxation temperature and malaxation duration as factors.

Factor		Sensory attributes and scores																		
		fruity[#]	*grass*	*apple*	*tomato*	*almond*	*herbs*	*chicory*	*rancid*	*bitter*	*pung*	*sweet*	*astring*	*complex*	*harmon*	*persist*	*typ odo*	*typ taste*	*typ over*	*overall*
RD	RD1	5.75^{a}	3.44^{a}	1.57^{b}	2.41	1.50^{a}	3.17^{a}	2.22^{a}	0.00^{b}	5.70^{a}	6.13^{a}	1.86^{b}	1.08^{b}	8.34^{a}	8.09^{a}	7.91^{a}	8.16^{a}	8.00^{a}	8.09^{a}	8.22^{a}
	RD2	5.20^{b}	3.13^{a}	2.26^{a}	2.49	0.71^{b}	2.51^{b}	1.44^{b}	0.00^{b}	5.17^{b}	5.84^{b}	2.03^{b}	1.49^{a}	8.13^{a}	8.19^{a}	8.00^{a}	7.81^{b}	8.13^{a}	7.97^{a}	8.02^{b}
	RD3	4.48^{c}	1.54^{b}	1.28^{b}	2.58	0.24^{c}	1.86^{c}	0.00^{c}	0.55^{a}	4.18^{c}	4.56^{c}	2.54^{a}	0.57^{c}	6.00^{b}	7.06^{b}	5.91^{b}	5.53^{c}	7.34^{b}	6.19^{b}	7.02^{c}
	F-value	75.79	67.73	15.53	0.71	34.70	49.02	67.07	218.58	62.47	101.70	9.26	29.60	223.87	103.44	174.88	248.70	38.25	277.85	637.13
MT (°C)	22	5.03^{b}	2.89^{a}	1.76	2.31^{b}	1.00^{a}	2.48	1.12	0.37	4.63^{b}	5.12^{b}	2.45^{a}	0.73^{b}	7.27^{b}	7.54^{b}	6.71^{b}	6.71^{b}	7.71^{b}	7.10^{b}	7.51^{b}
	30	5.47^{a}	2.51^{b}	1.64	2.68^{a}	0.64^{b}	2.55	1.32	0.00	5.40^{a}	5.90^{a}	1.83^{b}	1.36^{a}	7.71^{a}	8.02^{a}	7.83^{a}	7.63^{a}	7.94^{a}	7.72^{a}	7.99^{a}
	F-value	23.58	7.22	0.66	10.05	8.22	0.43	1.66	218.58	47.37	68.19	21.15	40.43	19.17	45.72	118.67	77.00	8.56	71.59	264.50
MD (min)	30	5.31	2.75	1.89^{a}	2.53	0.86	2.45	1.20	0.13^{b}	5.05	5.60	2.11	0.90	7.58	7.71	7.40^{a}	7.06	7.88	7.42	7.79
	60	5.19	2.66	1.51^{b}	2.45	0.77	2.57	1.24	0.23^{a}	4.98	5.42	2.18	1.19	7.40	7.85	7.15^{b}	7.27	7.77	7.42	7.71
	F-value	1.80	0.34	6.51	0.42	0.54	1.08	0.08	16.26	0.35	3.87	0.28	8.31	3.52	3.23	5.86	3.93	1.77	0.00	3.00
Interactions	RD x MT	*	*	n.s.	n.s.	n.s.	*	*	*	*	*	n.s.	n.s.	*	*	*	*	*	*	*
	RD x MD	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.	*	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.
	MT x MD	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.	*	n.s.	n.s.	n.s.	n.s.	n.s.	*	n.s.	*	n.s.	n.s.	n.s.
	RD x MT x MD	*	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.	*	n.s.	*	n.s.	n.s.	*	*	*	n.s.	n.s.	*	n.s.

Identification of sensory attributes and scores, odour attributes: ***green*** **- green grass and leaves**; taste attributes: ***pung*** **- pungent**; ***astring*** **- astringency**; ***complex*** **- complexity**; ***harmon*** **- harmony**; ***persist*** **- persistency**; ***typ odo*** **- typicity of odour**; ***typ taste*** **- typicity of taste**; ***typ over*** **- overall typicity**; ***overall*** **- overall quality.**

FIGURE 1. Simple effects of the two factors, malaxation temperature and malaxation duration on phenols concentration (mg/kg) in Oblica cv. virgin olive.



*** - statistically significant differences (n=3, *t*-test, *p*<0.05) between malaxation temperatures at each duration level, and forks designate statistically significant differences between malaxation durations at each temperature level.**

CONCLUSIONS

When considering the main effects of the three investigated factors, ripening degree exhibited the highest influence on Oblica virgin olive oil chemical and sensory characteristics. Phenols were generally found to decrease during ripening, however an unusual evolution pattern with increasing concentrations was observed for 3,4-DHPEA-EDA and *p*-HPEA-EDA. Generally, higher malaxation temperature induced an increase in particular important phenols.

The effects of the investigated factors were not univocal, but significantly interacted. Among many complex and specific interactions, the foremost were those observed between ripening degree and malaxation temperature. The effect of temperature was most pronounced in the processing of ripe olives, especially for 3,4-DHPEA-EDA and *p*-HPEA-EDA. The observed complexity of the interactions resulted from the synergistic and contrasting effects of all the parameters involved, which were different for each treatment.

Desirable taste sensory attributes correlated positively with particular main phenols. A decrease of bitterness and pungency, and an increase in sweetness was observed along ripening. Bitterness and pungency were enhanced by the higher malaxation temperature.

This study reported, for the first time, the results of the complex interactive effects of the three investigated factors. It showed how by manipulating these production parameters a wide range of phenol contents combinations from the same fruits can be obtained. This is of particular interest for olives with deficient or excessive phenol potentials, which has to be specifically processed to achieve a satisfactory balance.

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