

Analytical characteristics of olive oils produced by two different extraction techniques, in the Portuguese olive variety 'Galega Vulgar'

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RESUMEN

Características analíticas de los aceites de oliva producidos por diversas técnicas de extracción con la variedad portuguesa 'Galega Vulgar'.

Se han comparado dos líneas de procesamiento de aceite, utilizando un sistema de prensas (SP) o un decantador centrífugo (DC). El procesamiento que recurre al sistema de prensas es un sistema discontinuo que, sin embargo permite obtener aceites vírgenes de gran calidad. Para este estudio se ha utilizado la variedad *Portuguesa Galega* común. Las aceitunas fueron tratadas contra el ataque de plagas, y recogidas en un punto de maduración predeterminado. Del mismo modo, se evaluó el porcentaje de aceite obtenido de la cosecha. Se han considerado los resultados analíticos teniendo en cuenta la determinación de la influencia de la extracción en el producto final. Este punto ha sido evaluado por medio de un tratamiento estadístico. Aunque se han observado diferencias significativas entre los resultados de algunos de los parámetros analizados, sólo las diferencias verificadas en el grado de acidez son susceptibles de modificar la clasificación final del aceite, situándolo en la categoría lampante.

PALABRAS CLAVE: Aceite de oliva – 'Galega Vulgar' – Técnicas de extracción – Tecnología.

SUMMARY

Analytical characteristics of olive oils produced by two different extraction techniques, in Portuguese olive variety 'Galega Vulgar'.

A metal hammer-decanter (HD) olive processing line was compared to a traditional metal hammer-press (HP) line, a discontinuous method which, when properly used, yields high-quality virgin olive oils. Galega olives (traditional Portuguese variety) were used. Olives were picked at a predetermined maturation stage and plagues and oil content were evaluated before processing. Years, extraction technology, data replicates, and years*extraction, were taken into account and compared using statistical treatment. In spite of significant differences among the results obtained, only acidity was statistically significant and sufficient for classifying the produced olive oil into a lampante category.

KEY-WORDS: Extraction technique – 'Galega Vulgar' – Olive oil – Technology.

1. INTRODUCTION

Olive trees belong to the *Olea europea L.* family but among them different cultivars with different

characteristics can be found in the world's production areas. The most important cultivars used in Portugal are *Galega Vulgar*, *Carrasquenha*, *Cordovil*, *Cobrançosa* and *Verdeal* (Gouveia, 1995; Bartolini *et al.*, 1998). Portuguese cultivars with a major interest in the olive oil industry are those responsible for the olive oils of Protected Denomination Origins (DOP) (Bartolini *et al.*, 1998; Gouveia *et al.*, 2002), where the predominant variety is Galega Vulgar, representing 80% of the olive patrimony in Portugal (Gemmas *et al.*, 2002).

In general terms, olive oil quality is related to olive ripeness, olive sanitary condition and processing but also to origin, variety and storage. (Vinha *et al.*, 2005; Torres *et al.*, 2006). Processing is, in fact, a major factor affecting olive oil quality. Pressed oil obtained under the proper processing conditions is usually of great quality. Press extraction was almost the only olive oil extraction process used for centuries. However, Olive oil processing has progressed significantly since the beginning of the seventies, when the centrifugation system appeared. Since then, many articles comparing the so-called three-phase centrifugation extraction system with the two-phase centrifugation system (Vlyssides *et al.*, 2004) have been written. When compared to the press system these processes are sometimes regarded as producing olive oils of inferior quality (Rannalli *et al.*, 2001). To verify olive oil characteristics, chemical and sensorial analyses can be used. Several studies have been carried out comparing aroma compounds, oxidative stability, phenolic compounds, color and other chemical parameters (Boskou, 1996; Rannalli *et al.*, 1997; Aparicio and Luna, 2002).

In this work olive oil produced by two different extraction technologies, decanter and pressing by hydraulic press, were evaluated, by means of routine regulated analyses. The results obtained were evaluated in order to verify if differences could be significant for placing the olive oil in different classifications. If different classified olive oils were obtained we could conclude that not only the intrinsic quality was affected but also its commercial value. Technological studies similar to this can provide important information to determine not only

the differences among the produced olive oils but also to improve technology .

2. MATERIALS AND METHODS

2.1. Sampling

Experiments were carried out by processing mechanically picked olives from the Portuguese cultivar *Galega Vulgar* under defined conditions. All olives were picked under proper, controlled sanitary conditions. Olives were picked during the harvests of 2001, 2002, 2003 and 2004. A 120 Kg sample was collected. Fruits were stored in open boxes at ambient temperature (5-15°C) with reasonable air flow and without direct light incidence. Extraction was made during the next 24h. Before extraction, leaves and dirt (soil, stones, etc) were removed by washing under cold running water.

Maturation index was determined according to Hermoso *et al.* (1991). Fat yield and humidity percentage were also determined before extraction. For these olives the percentage of *Gloeosporium olivarum* Alm. or *Dacus oleae* Rossi (olive fly) attack was determined by visual inspection. Three replicates for each extraction procedure (pressing and centrifugation) were made for each collected sample with the exception of the 2001 harvest, where only two replicates were made due to severe climatic conditions.

2.2. Extraction technology

A homogeneous 20 Kg sample was processed (each time) for each one of the technologies under study: a hammer-mill press line (Vieirinox, Portugal) and a hammer-mill integral decanter line (Oliomio, Italy) were used. No water was added to the olive paste in both systems and malaxing time, about 1 h, was equal for both methods. For the pressing system the liquid-liquid separation was made by natural process, decantation. Three replicates were made for each extraction process.

2.3. Olive Oil Analysis

Acidity was determined according to the EC regulation nº 2568/91 annex II, Spectrofotometry UV absorption was made according to the EC regulation nº 2568/91 annex IX; Peroxide value followed the EC regulation nº 2568/91 annex III; Rancidity was analyzed according to the Portuguese norm NP 4158 de 1991; Sterols, uvaol and eritrodol were in accordance with the EC regulation nº 2568/91 annex V; fatty acids with the EC regulation nº 2568/91 annex XB; waxes with the EC regulation nº 2568/91 annex IV and triglycerides with the EC regulation nº 2568/91 anexe VIII.

For Polyphenol analyses, a procedure developed at Instituto Superior de Agronomia,

based on the Folin Ciocalteu's method, was followed (internal proceeding IT065, Lab. de Estudos Técnicos, ISA, UTL. Lisboa Portugal),

Tocopherols were determined according to the method described by P. Rovellini *et al.* (1997), which uses High Performance Liquid Chromatography; connected to an ultraviolet detector (HPLC-UV).

2.4. Statistics

For the ANOVA, General Linear model analysis the software used was the Minitab version 12 for Windows (Minitab Inc., State College, USA).

3. RESULTS AND DISCUSSION

Tables 1 and 2 show the homogeneity of the samples in terms of maturation, fat content, humidity and dry matter. According to the results in Table 1 we might consider that harvesting was carried out at similar maturation stages. The same

Table 1
Results of maturation index
for 2002, 2003 e 2004

	Olive (Galega V.)
2002	4,6
2003	4,7
2004	4,5
Median	4,6
RSD	0,1
Relative Standard Deviation (%)	1,7

Table 2
Results from RMN tests for fats and humidity

	Humidity %	Fat-RMN	% of fat in dry matter
Median 2002 (n=3)	43,90	27,28	48,63
Median 2003 (n=3)	46,38	24,04	44,83
Median 2004 (n=3)	46,00	21,85	40,46
Median of Medians	45,43	24,39	44,64
SD	1,33	2,73	4,09
RSD (%)	2,94	11,20	9,16

can be stated as far as humidity percentage is concerned but not for the fat content. In fact, for fat content a standard deviation of 11.20 was obtained.

The results obtained for pest control *Gloeosporium olivarum* Alm. or *Dacus oleae* Rossi (olive fly) show a significant difference among years (Table 3). These verified differences, although acceptable, cannot be explained by climate conditions (rain) since 2004 was dryer than 2003.

On the other hand, the sanitary state observed in each of the studied years may justify the difference verified for fat content (Boskou, 1996; Barranco *et al.*, 2001), since this plague definitely affects olive oil quality and is, in Portugal, quite difficult to control if a severe attack occurs.

For olive oil results (Tables 4-8) we might consider that most data, for all studied parameters, lie in the legal intervals considered for extra virgin olive oil

Table 3
Pests control *Gloeosporium olivarum* Alm. (G) and *Dacus oleae* Rossi (Fly), for variety Galega Vulgar.

	Olive Fly in %	Olive Fly with deposition in %	Fly in %	G in %	Infested Olives (%)
2003	0,3	0,0	0,3	6,0	6,3
2004	10,0	2,0	13,0	60,0	76,1
Media	5,6	1,5	7,1	31,6	40,5
SD	6,5	1,1	7,6	40,5	50,5
RSD %	116,1	71,0	106,5	127,9	124,7

Table 4
Results for the fatty acids content during studied harvests in both extraction systems used

Year	2001	2001	2002	2002	2003	2003	2004	2004
Extraction (n=3)	2 Phases	Press	2 Phases	Press	2 Phases	Press	2 Phases	Press
C14:0 (%)	0,01	0,01	0,01	0,01	0,01	0,01	0,01	0,01
C16:0 (%)	14,85	14,18	14,55	14,33	14,19	14,70	14,25	13,80
C16:1 (%)	2,91	2,25	2,03	1,85	1,99	1,79	2,06	1,76
C17:0 (%)	0,11	0,10	0,12	0,04	0,11	0,08	0,12	0,09
C17:1 (%)	0,30	0,25	0,31	0,29	0,30	0,19	0,32	0,22
C18:0 (%)	1,55	2,05	1,84	2,26	1,79	2,08	1,90	2,08
C18:1 (%)	73,60	73,05	75,89	74,13	75,19	73,29	76,08	74,29
C18:2 (%)	5,07	6,46	3,93	5,68	5,04	6,43	3,91	6,33
C20:0 (%)	0,33	0,37	0,30	0,33	0,31	0,34	0,30	0,33
C18:3 (%)	0,83	0,83	0,69	0,74	0,72	0,71	0,64	0,67
C20:1 (%)	0,28	0,28	0,23	0,22	0,24	0,23	0,27	0,27
C22:0 (%)	0,10	0,12	0,08	0,09	0,08	0,10	0,09	0,10
C24:0 (%)	0,05	0,06	0,04	0,04	0,03	0,05	0,04	0,05
Trans C16:1 (%)	0,12	0,12	0,11	0,11	0,11	0,11	0,10	0,09
Trans C18:1 + Trans C18:2 (%)	0,01	0,03	0,02	0,02	1,16	0,02	0,01	0,02

Table 5
Results for waxes content during studied harvests in both extraction systems used

Year	Extraction (n=3)	C 40 (mg/kg)	C 42 (mg/kg)	C 44 (mg/kg)	C 46 (mg/kg)	Total Wax (mg/kg)
2001	2 Phases	28,40	39,63	34,56	25,84	128,43
2001	Press	25,67	36,38	30,22	22,01	114,28
2002	2 Phases	11,36	20,07	8,83	10,18	50,43
2002	Press	13,87	19,13	10,18	12,45	55,63
2003	2 Phases	6,94	12,65	8,36	12,91	40,85
2003	Press	23,75	32,24	29,14	35,15	120,28
2004	2 Phases	10,12	18,20	9,04	13,88	51,24
2004	Press	15,96	21,94	21,44	16,42	75,76

Table 6
Results for triacylglycerols content during studied harvests in both extraction systems used

Year	2001	2001	2002	2002	2003	2003	2004	2004
Extraction (n=3)	2 Phases	Press	2 Phases	Press	2 Phases	Press	2 Phases	Press
LLL	0,06	0,09	0,04	0,11	0,21	0,09	0,09	0,07
OLLn	0,31	0,30	0,32	0,43	0,14	0,26	0,14	0,25
PLLn	0,09	0,11	0,03	0,07	0,05	0,07	0,09	0,11
OLL	0,81	1,42	0,44	1,21	0,50	1,38	0,46	1,31
OOLn	2,19	2,09	1,24	1,50	1,64	1,61	1,72	1,46
PoOL	0,97	0,85	0,00	0,29	0,68	0,64	0,00	0,39
PLL	0,00	0,00	1,02	0,70	0,00	0,22	0,75	0,76
POLn	0,13	0,15	0,34	0,12	0,11	0,12	0,15	0,14
OOL+LnPP+PoOO	12,21	13,07	10,05	11,35	10,16	12,43	10,06	12,08
POL+StLL	6,04	6,56	4,65	5,71	4,84	6,00	5,08	6,49
PPoO+PPL	0,85	1,05	0,84	0,96	0,89	0,79	1,04	1,13
OOO	38,98	38,54	43,54	40,70	43,57	40,65	42,14	39,12
POO	28,22	26,47	28,40	26,98	27,68	26,20	28,62	27,20
PPO	4,80	4,30	4,56	4,42	4,51	4,21	4,55	4,34
PPP	0,32	0,17	0,10	0,24	0,25	0,20	0,16	0,13
StOO+StOSt	3,04	3,68	3,59	3,97	3,53	3,80	3,54	3,74
PStO	0,86	0,94	0,85	1,12	1,03	1,06	0,97	0,98
PPSt	0,12	0,20	0,30	0,12	0,20	0,23	0,44	0,29

Table 7
Results for polyphenols e tocopherols content during studied harvests in both extraction systems used

Year	Extraction (n=3)	Polyphenols	γ Tocopherols	α Tocopherols
2001	2 Phases	21,99	27,30	281,67
2001	Press	17,83	23,99	239,14
2002	2 Phases	42,77	11,36	215,77
2002	Press	64,54	14,92	244,56
2003	2 Phases	80,45	8,69	201,49
2003	Press	110,92	14,06	246,12
2004	2 Phases	64,12	19,00	260,66
2004	Press	39,64	16,26	198,94

Table 8
Results for sterols content during studied harvests in both extraction systems used

Year	2001	2001	2002	2002	2003	2003	2004	2004
Extraction (n=3)	Press	2 Phases	Press	2 Phases	Press	2 Phases	Press	2 Phases
Cholesterol	0,24	0,33	0,12	0,12	0,17	0,25	0,14	0,22
Campesterol	1,71	3,15	2,75	2,82	2,76	2,7	2,94	2,87
Stigmasterol	0,93	1,56	1,57	1,4	1,29	0,6	0,62	0,92
apparent β -sitosterol	93,44	93,84	94,34	94,52	94,72	95,43	95,19	94,86
Δ - 7 - stigmasterol	0,16	0,17	0,18	0,16	0,24	0,2	0,19	0,22
Eritrodiol + Uvaol	1,21	1,46	1,85	1,83	1,74	1,07	1,08	1,56
Total Sterols	2508,4	2115,8	1545,59	1595,95	1588,7	1503,67	1672,25	1619,08

classification with one exception. For the olive oil obtained from the Press system the acidity value is above 2 in the last three years of analysis and therefore the resulting oil should be classified as "lampante" olive oil (Regulation CE nº1989/2003). These results are hard to understand since acidity with press extraction is usually not affected but, in this case, it was obtained in three of the studied years and so it is most likely related to the system used.

The above assumptions were taken only from an analytical point of view and were not based on any statistical treatment. To allow reliable conclusions an ANOVA treatment was applied. Extraction technologies as well as replicates were the target variable. The interaction extraction*replicates were also tested. The statistical analysis was made using the software Minitab the ANOVA General Linear Model, with a significance level of 95%. In Table 9

only the interactions where significant differences were detected are presented. From this table we could conclude that data replicates did not show significant differences, with an exception in acidity values, probably due to prolonged contact with water coming from the separation process, which occurs when the pressing technology is used. The separation time is also longer when the press system is used which might also account for the differences observed.

As can be observed, extraction technology has had a significant impact on some of the data obtained.

For sterols analysis, extraction technology has a significant impact on the results only for the Uvaol+Eritrodiol and total sterols. The slight differences verified in individual data (table 8) are common in the same variety (Alves *et al.*, 2005).

For fatty acids (Table 4) only the heptadecenoic acid (C17:1) presents results beyond regulation limits for extra virgin olive oils. These values were detected for only one extraction system (two phase decanter) although only in the year 2004, but if we considered the covariance for the heptadecenoic acid (C17:1), we verify that it is not higher than 5% for the highest permitted value according to regulation (Regulation (CE) n° 1989/2003). In Table 4, trans-palmitoleic acid is also above the regulated value for all the oils, however, when this value is considered in combination with trans-linolenic acid the final value falls within the allowed range for extra virgin olive oil.

In Table 9 significant differences, due to extraction system are shown for C16:1, C17:0, C17:1, C18:1, C18:2, C20:0 e C18:3 acids.

Table 5 shows significant differences for the C40, C42, C44 as well as for Total Wax content, however Total Wax is always < 250 mg/kg, thus the resulting oils can always be classified as extra virgin.

For the triacylglycerols the impact of the extraction procedure was significant for some data (PoOL, OOL+LnPP, POL+StLL, OOO, POO, PPO and StOO+StOSt) (Table 6). These results are in accordance with others previously reported, in which, in addition to cultivar, other rather important

Table 9

Results of the application of ANOVA General Linear Model, Software Minitab, with a significance level of 95%, the analytical data collected. (Replicates and extraction technology were the variables)

	Source	DF	F	P
Acidity %	Extraction	1	155,53	0,003
	Repetition	2	4,87	0,028
	Extraction*Repetition	2	3,92	0,047
K232 (Abs.)	Extraction	1	25,39	0,003
K270 (Abs.)	Extraction	1	5,48	0,038
IP (mEq O2/kg)	Extraction	1	17,97	0,004
Cholesterol	Extraction*Repetition	2	6	0,016
	Extraction	1	18,06	0,004
Stigmasterol	Extraction*Repetition	2	4,13	0,042
Uvaol+Eritrodiol	Extraction	1	54,78	0,003
Total Sterols	Extraction	1	24,53	0,003
C16:1	Extraction	1	17,3	0,001
C17:0	Extraction	1	5,73	0,037
C17:1	Extraction	1	6,76	0,026
C18:0	Extraction	1	41,02	0,003
C18:1	Extraction	1	14,58	0,005
C18:2	Extraction	1	15,39	0,005
C20:0	Extraction	1	8,38	0,016
C18:3	Extraction	1	7,34	0,022
OLLn	Extraction	1	14,96	0,005
OLL	Extraction	1	42,1	0,003
PoOL	Extraction	1	55,85	0,003
OOL+LnPP	Extraction	1	29,34	0,003
POL+StLL	Extraction	1	34,96	0,003
OOO	Extraction	1	30,19	0,003
POO	Extraction	1	49	0,003
PPO	Extraction	1	25,9	0,003
StOO+StOSt	Extraction	1	21,39	0,004
Polyphenols	Extraction	1	6,22	0,031
	Repetition	2	1,83	0,205

factors affecting total fatty acid composition, and especially oleic acid content include latitude, climatic conditions, and the ripening stage of the fruit at harvest (Ranalli *et al.*, 1997; Aparicio and Luna, 2004, Aranda *et al.*, 2004) as well as extraction method (Salvador *et al.*, 2001).

4. CONCLUSIONS

From these results we can conclude that extraction methods might be responsible for some of the differences observed in olive oil quality since, for the same raw material, different final products are obtained. Taking into account the olive oil classification from EC Regulation N° 1989/2003 from November 6th, it is clear, however, that besides acidity, all the significant differences among the parameters studied are not sufficient to change the oil classification. Acidity differences are enough to depreciate the oil in terms of classification. The high acidity obtained for the oil from press extraction is probably due to an extreme hydrolysis of fat due to the excessive contact time of the oil with vegetation water during the decanting step.

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