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Did the Addition of Olive Cake in the Finishing Diet of Bísaro Pigs Affect the Volatile Compounds and Sensory Characteristics of Dry-Cured Loin and Dry-Cured "Cachaço"?

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Abstract: This study was conducted to determine the effect of different types of olive cake (five treatments) in the diet of Bísaro pigs on the volatile compounds and sensory characteristics in the dry-cured loin and dry-cured "cachaço" (2 processed meat products). The treatments applied are based on the type of extraction and this may imply differences in their water and fat content. Eighty compounds were identified, and grouped into eight chemical classes: hydrocarbons, aldehydes, esters, alcohols, ketones, acids, furans, and other compounds. Aldehydes and alcohols were the major groups of compounds, representing 57.06-66.07 % and 68.67-75.61 % for the loin and "cachaço", respectively. There were no significant differences between treatments for any of the volatile compounds identified. The major aldehydes were hexanal, heptanal, pentanal, and propanal. These compounds were significantly higher ($p<0.001$) in "cachaço". This significant difference between the two types of dry-cured products is directly related to the amount of total fat content. The major alcohols were 2.3-butanediol, 1-octen-3-ol, 1-butanol, 3-methyl, 1-hexanol, benzyl-alcohol and glycidol. Except for compounds 2,3-butanediol and benzyl-alcohol, the majority in this group are significantly different in terms of the type of dry-cured product. As for the sensory evaluation, for both dry-cured products, the trained tasters did not detect significant differences between the different treatments, which is in line with the results obtained for volatile compounds.

Keywords: Bísaro breed; dry-cured products; olive cake; volatile compounds; sensory

1. Introduction

Bísaro pig is a native breed from Trás-os-Montes region (Portugal). Despite being one of the most emblematic native Portuguese breeds, it is little known in Portugal. There were several reasons why there was a decline of the breed, leading to its near extinction (modern society´s eating habits, opting for lean meats; rural exodus, etc.). In the last decades we have observed a great valorization of

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extensive agriculture, associated with the production of high-quality products. This change of paradigm allowed the Bísaro pig breed to assume decisive importance in the rural development of Trás-os-Montes, contributing to maintaining the typical products of the region as well as traditional production systems.

The dry-cured loin is one of the most consumed products in the world, made from a noble part of the pork carcass. The dry-cured "cachaço" is a product made from the anterior part of the *Longissimus thoracis et lumborum* muscle and it is not as well known worldwide. Compared to other pig breeds such as the Iberian breed, processed products from the Bísaro breed do not have the same commercial value or worldwide recognition. The quality of the dry-cured products is due to the characteristics of the raw material (feeding characteristics, age of animals, and pig breed) [1].

According to Dominguez *et al.* [2] the volatile composition of a dry-cured product results from a set of reactions that are crucial in determining their sensory attributes. These reactions consist of the oxidative decomposition of lipids and degradation reactions of amino acids. The different compounds derived from the curing process and the addition of ingredients contribute to the formation of a peculiar aroma, characteristic of these products [2]. Paprika and garlic are two spices with a very important performance in terms of the taste of dry-cured products, due to volatile compounds (mainly terpenes and sulfide compounds), furthermore, they are important antioxidant agents [3]. The curing process generates a set of chemical changes that lead to the development of volatile compounds, mainly esters and furan compounds [4]. These chemical and biochemical changes lead to changes in the taste as well as in the odor of the final product, which will influence the flavor and consumers' acceptability.

The dry-cured loin is a highly appreciated product worldwide. There are multiple studies concerning its physicochemical and sensory characteristics [1,3]. Nevertheless, the Bísaro breed is not disseminated worldwide as some other breeds. Therefore, the present study makes it possible to complete the little existing information about this breed and its processed products.

On the other hand, as it is known, there are large amounts of waste that are generated during olive processing [5,6]. Since one of the major environmental objectives is the circular economy, these residues could be part of the diet of some animals. Studies were carried out with the inclusion of chestnut at the end of the diet, which altered the content of volatile compounds in the final product (dry-cured ham) [7]. However, studies carried out on lamb meat with the introduction of olive cake and linseed did not produce appreciable changes in the volatile compound of the meat [8].

So, the aim of this study was to evaluate the effect of the inclusion of different types of olive cake (T1—Basic diet and commercial feed; T2—Basic diet + 10% crude olive cake; T3—Basic diet + 10% olive cake in two phases; T4—Basic diet + 10% exhausted olive cake; T5—Basic diet + 10% exhausted olive cake + 1% olive oil) in the finishing diet on volatile compounds of dry-cured loin and dry-cured "cachaço" from Bísaro pig.

2. Materials and Methods

2.1. Animals management and diets

To carry out this study, animals of the Bísaro breed (*Sus scrofa*) with a live weight of 100 kg were used. These animals were randomly divided into 5 groups (of 8 animals each), one of them being control (basic diet) and the other four groups with different olive cakes (in combination with the basic diet) (T1—Basic diet and commercial feed; T2—Basic diet + 10% crude olive cake; T3—Basic diet + 10% olive cake in two phases; T4—Basic diet + 10% exhausted olive cake; T5—Basic diet + 10% exhausted olive cake + 1% olive oil). The animals come from the farm (Bísaro Salsicharia Tradicional®), raised in an extensive production system. In total, forty animals of the Bísaro breed were used. This feed was applied to all groups simultaneously and under the same conditions (feed level was "ad libitum" with an average consumption of 3 kg per day). The fatty acid profile of the diets used were made in Meat Technology Center of Galicia, Ourense – Spain. From the analysis of the fatty acid profile of the different diet, there were no statistically significant differences for MUFA, PUFA and SFA content of the diets incorporating olive cakes in relation to the control diet (T1).

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However, treatment with the T4, incorporating exhausted olive cake, showed the fatty acid profile most similar to T1. For SFA the values ranged from 20.03 to 21.54 % for all treatments. As for MUFA, they varied between 28.0 and 42.36 %. As for PUFA, the values ranged between 37.61 and 50.46 %. C16:0, C18:1n-9 and C18:2n-6 are the major fatty acids of SFA, MUFA and PUFA, respectively, in the different treatments used for the diet of the Bisaro pig animals. The chemical composition and the fatty acid profile of the diet applied to the animals were made according to Leite et al. [9].

Different types of olive oil cakes were used from different industries in the sector that receive olives from all over the Northeast of Portugal. The experimental feed (for 90 days) trial was carried out on Trás-os-Montes e Alto Douro University, Vila Real – Portugal.

The Bísaro pigs were slaughtered at the Municipal Slaughterhouse of Bragança as described by Álvarez-Rodríguez and Teixeira [10] and in accordance with applicable legislation [11].

2.2. Dry-cured Bísaro loin and Cachaço manufacture

Forty loins and forty "cachaços" from the forty slaughtered animals were used. The curing process was carried out at the company Bísaro – Salsicharia Tradicional, Lda. The dry-cured loin was made from one of the most valuable joints of pork carcass, the lumbar and thoracic parts of *Longissimus thoracis et lumborum* (LTL) muscle. The dry-cured "cachaço" was made from the proximal part of *Longissimus thoracis et lumborum* (LTL) muscle from the column's cervical part noticeable beneath the scapula until the fifth thoracic vertebra. The production process for the loin is the same as the "cachaço", only the size and location of the piece varies (vertebral from the end of the joint of the "cachaço" to the last lumbar vertebra). To elaborate this type of product, a series of steps were applied that led to the formation of the cured product. The following ingredients were added: 1.5% of salt, 0.5% of paprika, 0.5% of garlic, and 0.1% of oregano. The total curing time for both products was approximately 60 days.

After extracting the muscles from animal carcasses, the pieces (loin and "cachaço") were refrigerated in a chamber between 2 and 5 ºC. The next step was the salting and seasoning phase. Before starting this rotating process (for 30 minutes), all the ingredients were added. When the mixing is complete, the pieces are placed in a refrigeration chamber between 2-4 ºC for 4 days. The next phase is stuffing into collagen casings. The last phase is drying – curing, in which the most important biochemical changes take place. The temperature and relative humidity change as curing time progress, increasing temperature and decreasing relative humidity. In the first 15 days, the cuts were submitted to a temperature between 4-8ºC with a relative humidity between 80-90%. After this period, the product was submitted to a temperature between 8-12ºC and relative humidity between 70-80% for another 15 days. Finally, for the last 20 days, the product was submitted to a temperature between 12-18ºC and a relative humidity between 60-70%. The curing method applied was done according to Leite et al. [12].

2.3. Volatile compounds

The volatile compounds of 1 g of sample were analyzed using the SPME-gas chromatography-mass spectrometry technique (Agilent Technologies, Santa Clara, CA, USA), following the procedure described by Dominguez et al. [2]. The volatile results were expressed as area units per gram of sample (AU × 10^5/g of sample).

2.4. Sensorial Analysis

The dry-cured loin and dry-cured "cachaço" samples were evaluated by a trained taste panel. The training was done according to the Portuguese Standard [13] in the Sensory Analysis Laboratory at the Polytechnic Institute of Bragança. This panel (made up of nine tasters) was created after the recruitment, selection, and training phases. The conditions of the test room where the evaluation took place followed standard guidelines [14]. The light in the room and each booth was white light on to facilitate evaluation. The panelists were given water and unsalted toasts to cleanse the palate and remove residual flavors at the beginning of the session and between samples. Samples of dry-cured

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loin and dry-cured "cachaço" were divided into 1.5 mm thick slices, placed in a plate at room temperature, and evaluated. For both loin, and cachaço, two samples of each treatment were evaluated in each session, in 3 sessions per product. Each treatment was evaluated 6 times by each taster. Samples were evaluated for quantitative attributes related to appearance, odor, texture, taste, and flavor. A 9-point scale, in which 1 represents the minimum (very weak intensity) and 9 the maximum (very strong intensity), was used for the quantitative attributes considering a quantitative descriptive analysis.

2.5. Statistical Analysis

Data were tested for normal distribution and variance homogeneity by Shapiro–Wilk test. Then, the effect of product, treatment and the interaction between product x treatment (data not shown for the latter once all interaction were not significant) on volatile compounds contents was examined using analysis of variance (ANOVA) with the General Lineal Model (GLM) procedure, where these parameters was set as dependent variables and product and treatments as fixed effect. Results were given in terms of mean values and standard error of the mean (SEM). When a significant effect ($p < 0.05$) was detected, means were compared while using the student test.

Simple means were used to develop a sensory profile for the dry-cured loin and dry-cured "cachaço". A non-parametric analysis of variance was performed for sensory data, treatments were compared by Friedman´s test using SPSS software (IBM SPSS Statistics). Using the XLStat program (Addinsoft, New York, NY, USA), a generalized procrustean analysis (GPA) was used to minimize the differences between assessors, identify agreement between them, and summarizes the sets of 3-dimensional data.

3. Results and Discussion

3.1. Volatile compounds of dry-cured loin and dry-cured "cachaço"

The different locations of the same muscle used to make these two types of dry-cured products significantly affected a large proportion of the volatile organic compounds (VOC) detected. These compounds have been grouped into eight chemical classes: Hydrocarbons (21), aldehydes (18), esters (13), alcohols (12), ketones (12), acids (4), furans (4) and other compounds (4). According to some authors [15,16], the composition of volatile compounds is due to the smoking process, the added seasonings, and the reactions between lipids, proteins, and carbohydrates caused by microbial enzymes and oxidative processes.

Aldehydes were the main volatiles found in all treatments (Table 1), representing 34.84-46.45% and 50.77-55.68% of total VOC in dry-cured loin and dry-cured "cachaço", respectively. The aldehydes can be divided into two groups: linear aldehydes and branched aldehydes. The linear aldehydes derive mainly from lipid oxidation and the branched aldehydes are related to amino acid degradation and proteolysis [2,17,18]. The major aldehydes were hexanal, heptanal, pentanal, and propanal. These compounds were significantly higher ($p<0.001$) in the dry-cured "cachaço". This significant difference between the two types of dry-cured products is directly related to the amount of total fat content. According to Leite et al. [12] the dry-cured "cachaço" has a considerably higher amount (~45%) of total fat than the dry-cured loin (~20%). The linear aldehydes are typical products of lipid oxidation and are responsible for the fat odor. The main linear aldehyde was hexanal, as observed by other authors in other products: polish dry-cured loin [19], dry-fermented deer sausage [20], dry-cured traditional Istriam ham [21], cecinas [22], and fermented sausages [23]. Hexanal and pentanal derived from the oxidation of unsaturated fatty acids, namely through the lipoxygenase pathway (LOX), from linoleic, linolenic, and arachidonic fatty acids while heptanal derived from oleic acid [17,19,21,24]. As could be seen in Table 1, hexanal and pentanal contents were not influenced by the treatments used in the animal feeding. This fact could be related, and as was reported in a previous study [12], to the inclusion of olive cakes did not produce changes in the fatty acids profile of these products, thus, the oxidative degradations could be similar in both. According to Domínguez et al [2] the hexanal present a rancid aroma at high amounts, while in low content it gives a pleasant

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grassy aroma. Górska et al [19] described the hexanal aroma as green, grassy, fatty, rancid strong, unpleasant hot, and nauseating. With this in mind, it is expected that low-medium levels of this aldehyde produce a desirable flavor in the final product, related to the "curing" process and appreciated by the consumer.

In addition to these compounds, other important aldehydes (branched and aromatic) were also found in dry-cured loin and dry-cured "cachaço" of Bisaro pork, namely 3-methyl-butanal, benzaldehyde and benzeneacetaldehyde. These compounds were also found in dry-fermented deer sausage [20], dry-cured meat [2], fermented sausages [25], and cecinas [22]. The origin of 3-methyl-butanal, is the deamination-decarboxylation of the amino acid leucine, whereas benzaldehyde and benzeneacetaldehyde are derived from the Streaker degradation of some amino acids such as leucine or phenylalanine [2,5,18]. In this sense, aromatic aldehydes (benzaldehyde and benzeneacetaldehyde) possess floral, bitter almond notes, rancid and pungent aroma, while 3-methyl-butanal is an important compound for the dry-cured products since it has a typical "ripened flavor" [2].

Alcohols were the second major group in the volatile profile (Table 1) and represented between 19.06-19.62% and 17.90-19.93% of total VOC in dry-cured loin and dry-cured "cachaço", respectively. The major alcohols were 2,3-butanediol, 1-octen-3-ol, 1-butanol, 3-methyl, 1-hexanol, benzyl-alcohol and glycidol. These compounds were also observed in fermented sausages [2,20,25]. With the exception of compounds 2,3-butanediol and benzyl-alcohol, the majority in this group are significantly different in terms of the type of dry-cured product. As for the different treatments, and in line with what happened with the aldehydes group, there were no significant differences in the major compounds. Of the major compounds in this group of volatiles, 1-octen-3-ol stands out in the dry-cured "cachaço" while 1-butanol, 3-methyl, 1-hexanol, and glycidol are in greater proportion in the dry-cured loin. According to the other authors [2,18] the 1-octen-3-ol, derived from the oxidation process of linoleic acid and this compound is described as a very important compound for contributing a dry-cured aroma to the products. According to Leite et al. [12] the dry-cured "cachaço" contained a significantly higher linoleic acid content than that observed in the dry-cured loin, which may explain the presence of this compound with greater proportion in the dry-cured "cachaço". The major content of 1-butanol, 3-methyl can be due to the activity of the microorganisms present in the dry-cured loin. According to Muriel et al. [26] the microorganisms can act on butanal, 3-methyl formed by the degradation of amino acids during proteolysis to originate 1-butanol, 3-methyl.

Four compounds were isolated in the group of acids (Table 1): butanoic acid, 3-methyl, butanoic acid, hexanoic acid, and acetic acid. The acids represented 0.87-1.97% and 3.45-5.80% of total VOC in dry-cured loin and dry-cured "cachaço", respectively. Regarding to butanoic acid, hexanoic acid, and acetic acid compounds were significantly different ($p<0.001$) with regard to the type of product. Again, the treatment has no influence on any of the products studied. The highest amounts of butanoic acid and hexanoic acid were observed in the dry-cured "cachaço". These compounds have also been found in dry-fermented deer sausages [20], dry-cured loin and dry-cured shoulders [2], and cecina [2,22]. Butanoic acid, 3-methyl was also found in dry-cured ham [7] and dry-cured loin [19]. Regarding to some authors [19], butanoic acid, 3-methyl could be generated from leucine by the functions of some Staphylococcus. The aroma of this compound is described as cheese, feet, fatty, rancid and may contribute to the lower overall quality of products [19]. The acetic acid gives notes of ripened [20]. The most probable origin of hexanoic acid is the carbohydrate fermentation induced by microorganism such as lactic bacteria and staphylococci [27,28].

For these 3 groups of volatile compounds, no significant differences ($p > 0,05$) were detected between the treatments. In addition, no significant differences ($p > 0,05$) were also obtained between product and treatment interaction (data not shown). In previous studies [12], the fatty acid fractions obtained for these products were not affected by the introduction of olive cake in the animal feed. Considering that some of the mechanisms for the formation of volatile compounds, namely through the lipoxygenase pathway, are conducted at the level of fatty acids, we can say that it was possible that there were no differences at the level of the different treatments and their interaction with the type of product.

Table 1. Volatile compounds (expressed as AU 10^5/g) of dry-cured loin and dry-cured "cachaço". Effect of treatment with olive cake, product and interaction between product and treatment.

COMPOUNDS INFORMATION			**DRY-CURED LOIN**					**DRY-CURED "CACHAÇO"**					**SEM**	**Sig. Product**	**Sig. Treat.**
NAME	LRI	m/z	T1	T2	T3	T4	T5	T1	T2	T3	T4	T5			
Glycidol	512	43	6.64	9.40	9.97	11.40	8.60	5.91	6.94	6.45	6.76	6.49	0.586	0.025	0.648
2-Butanol, (R)-	610	59	0.56	0.36	0.93	0.52	12.99	0.25	1.01	0.17	0.36	0.83	1.203	0.292	0.343
1-Butanol	706	56	0.33	0.45	0.34	0.34	0.49	0.77	0.92	0.76	0.88	0.97	0.032	<0.001	0.308
2-Pentanol	749	45	2.59	2.13	1.58	1.99	2.24	1.20	0.73	1.00	0.94	1.01	0.136	<0.001	0.690
1-Butanol, 3-methyl-	805	55	12.36	16.23	13.67	14,78	10.69	3.12	3.11	2.68	4.79	2.65	1.133	<0.001	0.884
1-Pentanol	842	55	6.43	9.25	6.60	6.45	12.58	23.77	26.02	21.78	29.04	27.49	1.069	<0.001	0.429
2,3-Butanediol	911	45	41.95	20.62	30.50	38.45	19.30	37.27	51.67	41.90	47.41	20.78	3.934	0.211	0.392
1-Hexanol	947	56	12.66	25.09	17.64	18.68	29.29	8.16	11.00	9,35	9.18	10.70	1.489	<0.001	0.257
1-Octen-3-ol	1040	57	19.39	18.36	14.90	13.29	22.92	45.80	43.51	33.14	52.12	52.98	1.962	<0.001	0.273
Benzyl alcohol	1111	108	11.28	11.94	10.18	9.62	10.02	7.47	8.98	10.84	8.85	8.17	0.514	0.094	0.834
Terpinen-4-ol	1191	111	0.68	0.56	0.43	0.54	0.50	0.16	0.13	0.14	0.15	0.13	0.017	<0.001	0.140
Thymol	1290	150	0.09	0.09	0.07	0.12	0.08	0.02	0.02	0.01	0.01	0.02	0.004	<0.001	0.164
TOTAL ALCOHOLS			**114.96**	**114.48**	**106.81**	**116.18**	**129.70**	**133.90**	**154.04**	**128.22**	**160.49**	**132.22**			
Propanal	536	58	2.68	2.17	1.79	1.66	3.16	15.45	14.05	14.73	16.93	14.40	0.648	<0.001	0.960
Propanal, 2-methyl-	565	72	0.87	0.94	1.00	1.24	0.79	0.57	1.05	0.81	0.83	0.74	0.061	0.213	0.341
Butanal, 3-methyl-	660	58	7.75	8.84	11.65	12.75	7.37	2.65	5.05	4.69	4.71	3.45	0.875	0.003	0.595
Pentanal	727	58	9.01	8.21	6.10	6.46	11.51	24.34	25.60	21.57	27.44	26.35	0.881	<0.001	0.484
2-Butenal, 2-methyl-	798	84	1.04	1.16	1.22	1.87	1.26	0.51	1.29	0.90	0.92	1.16	0.153	0.275	0.737
Hexanal	842	55	162.54	153.55	140.13	130.40	183	291.52	320.50	287.24	325.08	302.33	9.878	<0.001	0.904
2-Hexenal, (E)-	931	83	0.19	0.20	0.17	0.15	0.26	1.34	0.97	0.96	1.30	1.37	0.050	<0.001	0.440
Heptanal	965	70	10.33	10.11	8.90	9.45	11.12	14.12	17.39	14.33	16.26	15.13	0.702	<0.001	0.887
Methional	990	104	0.37	0.40	0.50	0.52	0.38	0.05	0.17	0.14	0.08	0.10	0.021	<0.001	0.481
Furfural	993	96	0.02	0.02	0.02	0.01	0.05	0.18	0.15	0.16	0.21	0.17	0.008	<0.001	0.814
Benzaldehyde	1034	106	5.39	5.45	8.06	4.13	9.97	4.08	13.23	4.83	8.47	11.16	1.060	0.382	0.410
Octanal	1054	56	5.03	4.68	3.57	4.23	3.92	5.13	5.57	4.69	5.20	5.40	0.234	0.075	0.651
2,4-Heptadienal, (E,E)-	1067	81	0.05	0.03	0.03	0.03	0.06	1.67	1.23	1.33	2.07	1.77	0.088	<0.001	0.638
Benzeneacetaldehyde	1106	91	18.11	30.04	69.21	28.55	77.19	2.96	21.59	9.30	11.67	13.77	7.735	0.0391	0.615
2-Octenal, (E)-	1109	55	0.40	0.35	0.31	0.27	0.53	4.37	3.93	3.04	5.39	5.07	0.231	<0.001	0.548

Nonanal	1133	57	5.24	5.21	4.06	4.05	5.02	7.27	8.74	7.20	8.32	8.06	0.281	<0.001	0.653
2-Nonenal, (E)-	1184	70	0.47	0.35	0.29	0.35	0.38	0.72	0.76	0.68	0.82	0.66	0.035	<0.001	0.883
2-Decenal, (E)-	1253	70	0.20	0.21	0.15	0.17	0.18	0.30	0.29	0.25	0.30	0.27	0.014	<0.001	0.719
TOTAL ALDEHYDES			**229.69**	**231.92**	**257.16**	**206.29**	**316.15**	**377.23**	**441.56**	**376.85**	**436.00**	**411.36**			
Acetic acid	689	60	0.30	0.19	0.17	0.23	0.20	0.49	0.31	0.40	0.34	0.23	0.018	<0.001	0.248
Butanoic acid	910	60	2.60	2.06	3.33	3.86	3.51	21.59	15.85	22.42	19.87	13.90	0.834	<0.001	0.358
Butanoic acid, 3-methyl-	959	60	5.65	1.97	2.93	6.68	1.93	5.56	4.03	1.49	5.50	1.51	0.828	0.899	0.337
Hexanoic acid	1071	60	0.95	0.88	0.60	0.92	1.46	15.45	7.79	7.81	14.73	16.75	1.224	<0.001	0.617
TOTAL ACIDS			**9.50**	**5.10**	**7.03**	**11.69**	**7.10**	**43.09**	**27.98**	**32.12**	**40.44**	**32.39**			

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Hydrocarbons were the third largest compounds found in these types of dry-cured products and represented 13.68-20.37% and 6.78-9.34% of total VOC in dry-cured loin and dry-cured "cachaço", respectively (Table 2). The major hydrocarbons were octane, 2,2,4,4-tetramethyloctane, and heptane. These compounds were significantly higher in dry-cured loin, with no influence of the treatments applied to the animals feeding. The heptane and octane compounds (linear alkanes) have also been described by other authors in relation to Iberian ham and dry-cured loin [7,15,24,29]. The 2,2,4,4-tetramethyloctane (branched alkanes) were also found in dry-cured products such as ham, salchichón, shoulder, and cecina [2]. The appearance of these branched alkanes, especially methyl hydrocarbons, is related to the activity of molds, which synthesized these compounds as a product of the secondary degradation of triglycerides [30,31].

As for the ketones (Table 2), there were no significant differences between the two types of products, and no significant differences between the treatments ($p>0.05$). The ketones represented 6.79-11.60% and 5.92-8.93% of total VOC in dry-cured loin and dry-cured "cachaço", respectively. The most abundant ketone was acetoin, followed by 2-heptanone. Acetoin was the major ketone in dry-cured products such as loin, salchichón, shoulder, and chorizo [2,20]. As with the dry-cured loin and dry-cured "cachaço" of this work, the dry-cured shoulder [2] also obtained similar values for the compound 2-heptanone. The origin of ketones can be diverse [2], but according to Pérez-Santaescolástica et al. [31] the acetoin is formed through Maillard reactions. According to Sidira et al. [32], acetoin has a buttery, sweet odor, whit a very low odor threshold, and contributes to the typical flavor of dry-cured meat products. Regarding to 2-heptanone, and accordance to García-González et al., [34] this compound contributes to spicy, blue cheese and acorn sensory notes, and the release of 2-ketones is related to oxidative processes of lipids.

Regarding esters (Table 2), butanoic acid, ethyl ester was the most abundant compounds in the two types of products. The butanoic acid, 3 methyl, ethyl ester also has a higher amount compared to the dry-cured loin of Bísaro pork. The esters represented 7.93-10.81% and 3.40-5.68% of total VOC in dry-cured loin and dry-cured "cachaço", respectively. For the most abundant compound of this group (butanoic acid, ethyl ester) there were no significant differences between products or treatments. Regarding to butanoic acid, 3 methyl, ethyl ester, this compound were significant differences between both products, being higher in cachaço than the case of dry-cured loin. Petričević et al. [34] report that the main origin of esters in meat products is the esterification of carboxylic acids and alcohols. On the other hand, Marco et al. [35] and Akköse et al. [36] describe that low molecular weight esters can also be a product of carbohydrate metabolism. In this regard, the action of some microorganisms can promote the enzymatic esterification of fatty acids and alcohols due to their high esterase activity [2].

Four compounds were also found in the furan group (Table 2) (furan, 2-ethyl, 2-n-butyl furan, furan, 2-pentyl, and furan, 2-propyl). The furans represented 1.10-1.72% and 1.53-2.05% of total VOC in dry-cured loin and dry-cured "cachaço", respectively. With the exception of furan, 2-pentyl, the furans were significantly different ($p<0.001$) with regard to the type of product. Again, the treatment has no influence on any of the products studied. In other studies [2] no furans were found in dry-cured loin. In contrast, the same furans were found in other types of products such as cecina [2] and dry-cured ham of Celta pig [7]. Furan, 2-pentyl was also found in dry-fermented deer sausages [20] and dry-cured loin of Iberian pig [26]. The furans are described as compounds generated during heating [7]; however, they have already been found in other types of dry-cured products such as ham and dry-cured loin. According to Ruiz et al. [37], Akköse et al [36], and Lorenzo et al. [7] this VOC is a compound derived from linolenic and other n-6 fatty acids oxidation reactions. Due to their low odor threshold values, furans play an important role in all meat products. The furan, 2-pentyl, and furan 2-ethyl provide a pleasant aroma [1]

Four compounds were detected in the dry-cured loin and dry-cured "cachaço" but were not included in the previous groups (Table 2). The other compounds represented 0.94-2.59 % and 0.75-1.29 % in dry-cured loin and dry-cured "cachaço", respectively. These compounds found were sulfide, allyl methyl, 1H-pryrrole, 3-methyl, 1,3-Benzenediol, monobenzoate, pyrazine, 2,6-dimethyl. The compound sulfide, allyl methyl was the most expressive in this group, with no significant

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differences between the two types of products nor the difference between the types of treatments. This compound was described in samples of low-fat fermented sausages [25] and "chorizo" [38]. According to other authors [38] detection of compounds with "sulphur" and "allyl" could be related to the use of garlic as an ingredient. Therefore, they are organosulfur compounds derived from garlic. These compounds are components of the aroma of onion, garlic, and other Allium species [2].

For the groups of volatile compounds mentioned in Table 2, it was also found that there were no significant differences ($p > 0,05$) between the various types of treatments. Similarly, the interaction between treatments and products (data not shown) also had no significant difference ($p > 0,05$). The introduction of olive cake into the diet in the animals had no impact on the volatile compounds of the products studies. The fact that olive cake from various industries in the region was used may have approximated their composition in terms of volatile compounds, also reflected in the final product. Another possible reason why no significant differences were detected between the treatments may be due to the time period this diet was applied, proving insufficient for the formation of volatile compounds in the dry-cured products.

Table 2. Volatile compounds (expressed as AU 10^5/g) of dry-cured loin and dry-cured "cachaço". Effect of treatment with olive cake, product and interaction between product and treatment.

COMPOUND INFORMATION			DRY-CURED LOIN					DRY-CURED "CACHAÇO"					SEM	Sig. Product	Sig. Treat.
NAME	LRI	m/z	T1	T2	T3	T4	T5	T1	T2	T3	T4	T5			
Pentane	527	57	0.66	1.37	0.67	0.79	1.48	3.15	2.25	2.49	2.31	2.14	0.152	<0.001	0.874
1,4-Pentadiene	539	67	1.05	2.28	1.04	2.05	2.37	1.87	1.21	1.77	2.57	1.16	0.242	0.981	0.734
Cyclopentane, 1,2-dimethyl	665	56	0.29	0.31	0.16	0.20	0.26	0.28	0.26	0.25	0.26	0.24	0.020	0.649	0.666
Heptane	673	71	10.90	17.59	11.79	13.99	13.58	10.19	9.17	10.51	6.98	8.02	0.757	0.003	0.697
Octane	817	85	36.06	49.53	35.05	42.34	43.57	29.16	28.19	28.87	21.62	23.48	1.932	<0.001	0.735
2-Octene, (E)-	828	55	0.17	0.20	0.15	0.16	0.29	0.18	0.17	0.18	0.19	0.22	0.010	0.934	0.051
Nonane	927	57	0.88	0.89	0.64	0.79	0.58	0.61	0.46	0.55	0.43	0.72	0.039	0.010	0.758
α-Phellandrene	961	93	4.90	4.16	3.36	5.39	3.58	1.78	1.26	1.45	1.72	1.29	0.202	<0.001	0.219
Hexane, 3,3-dimethyl	927	85	0.48	0.46	0.35	0.44	0.35	0.32	0.25	0.28	0.24	0.37	0.020	0.003	0.696
Butane, 2,2,3-trimethyl-	994	57	0.39	0.35	0.21	0.32	0.21	0.16	0.15	0.15	0.14	0.16	0.016	<0.001	0.306
Nonane, 5-methylene-	999	56	0.48	0.47	0.28	0.37	0.23	0.18	0.17	0.18	0.16	0.18	0.022	<0.001	0.332
Decane	1018	57	2.13	3.68	1.89	2.03	1.52	1.02	0.69	0.51	0.80	0.97	0.153	<0.001	0.248
β-Myrcene	1021	93	1.16	1.09	0.73	1.30	0.73	0.35	0.24	0.26	0.38	0.27	0.054	<0.001	0.166
(Z)-4-Methyl-2-hexene	1043	98	0.83	0.75	0.54	0.68	0.44	0.26	0.30	0.28	0.27	0.29	0.036	<0.001	0.488
2,2,4,4-Tetramethyloctane	1048	57	33.59	31.00	23.97	30.09	19.66	14.07	15.60	15.46	13.73	14.59	1.376	<0.001	0.526
γ-Terpinene	1078	93	3.55	2.66	2.41	3.91	2.33	0.89	0.69	0.80	1.00	0.77	0.142	<0.001	0.153
Undecane	1098	85	0.51	0.49	0.39	0.47	0.34	0.28	0.26	0.25	0.24	0.29	0.019	<0.001	0.613
Dodecane, 2,6,10-trimethyl-	1171	57	0.61	0.57	0.47	0.58	0.48	0.32	0.34	0.30	0.30	0.38	0.027	0.001	0.914
Dodecane	1171	71	0.46	0.45	0.34	0.42	0.35	0.25	0.25	0.20	0.24	0.26	0.019	<0.001	0.594
2-Heptene, 3-methyl	1184	83	0.34	0.30	0.24	0.27	0.30	0.58	0.55	0.51	0.72	0.60	0.031	<0.001	0.886
Cyclododecane	1206	83	0.57	0.53	0.42	0.53	0.43	0.29	0.32	0.25	0.29	0.32	0.023	<0.001	0.668
TOTAL HYDROCARBONS			**100.01**	**119.13**	**85.10**	**107.12**	**93.08**	**66.19**	**62.78**	**65.50**	**54.59**	**56.72**			
2,3-Butanedione	594	86	0.76	0.25	0.32	0.61	0.30	0.92	1.39	1.15	0.90	0.54	0.082	0.002	0.470
2-Butanone	599	72	1.48	2.04	2.92	2.84	18.00	1.66	4.43	3.35	2.57	2.79	1.536	0.413	0.379
1-Penten-3-one	715	55	0.06	0.04	0.04	0.04	0.08	0.89	0.61	0.62	0.80	0.97	0.037	<0.001	0.334
2-Pentanone	719	86	5.51	5.17	3.41	5.00	4.07	4.94	6.67	4.98	3.61	3.31	0.629	0.918	0.810
2,3-Pentanedione	735	57	4.42	3.04	3.37	14.81	9.85	5.97	6.41	7.58	6.25	6.25	1.412	0.848	0.651
Acetoin	786	45	19.38	8.32	10.23	24.94	8.93	17.72	27.18	22.85	14.72	6.06	2.058	0.424	0.345
2-Hexanone	854	58	1.60	1.94	1.16	1.51	1.15	1.02	1.17	0.91	0.73	0.79	0.136	0.052	0.658
2-Heptanone	959	58	14.02	18.12	12.61	14.78	13.59	17.53	17.70	9.86	15.38	16.08	1.335	0.784	0.620
4-Hexen-3-one, 5-methyl-	1031	83	0.28	0.38	0.30	0.36	0.35	0.30	0.30	0.27	0.30	0.41	0.024	0.513	0.675

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Butyrolactone	1034	86	0.77	0.64	0.81	1.07	0.71	1.89	1.76	1.72	1.96	1.67	0.048	<0.001	0.334
3,5-Octadien-2-one	1121	95	0.72	0.41	0.46	0.46	0.83	2.05	1.73	1.78	2.62	2.37	0.141	<0.001	0.662
2-Nonanone	1126	58	2.07	2.29	2.00	2.23	1.69	2.66	2.98	0.87	2.60	2.46	0.284	0.654	0.710
TOTAL KETONES			**51.07**	**42.64**	**37.63**	**68.65**	**59.55**	**57.55**	**72.33**	**55.94**	**52.44**	**43.70**			
Acetic acid, methyl ester	547	74	0.30	0.19	0.17	0.23	0.20	0.49	0.31	0.40	0.34	0.23	0.025	0.008	0.188
Ethyl Acetate	603	61	10.70	9.71	8.21	9.11	10.78	5.10	3.91	2.26	4.00	4.53	0.578	<0.001	0.605
Propanoic acid, ethyl ester	735	102	0.52	0.28	0.30	2.81	1.63	0.23	0.15	0.11	0.27	0.22	0.291	0.123	0.555
n-Propyl acetate	742	61	0.15	0.18	0.17	0.25	0.62	0.08	0.13	0.06	0.09	0.09	0.042	0.029	0.317
Propanoic acid, 2-methyl-, ethyl ester	798	71	3.36	2.65	1.92	2.55	1.91	0.90	0.60	0.41	0.93	0.61	0.236	<0.001	0.698
Butanoic acid, ethyl ester	850	71	10.68	11.53	9.61	11.64	11.15	14.04	8.08	8.05	12.13	12.72	1.017	0.956	0.753
Butanoic acid, 2-methyl-, ethyl ester	902	102	7.83	5.11	3.49	5.09	3.41	1.72	1.57	0.94	2.00	1.33	0.578	0.004	0.632
Butanoic acid, 3-methyl-, ethyl ester	906	88	15.81	9.00	7.01	9.89	6.78	3.08	3.53	1.93	3.49	2.31	1.147	0.004	0.642
1-Butanol, 3-methyl-, acetate	932	70	0.63	0.43	0.41	0.41	0.93	0.65	0.50	0.43	0.70	0.59	0.049	0.938	0.191
1-Butanol, 2-methyl-, acetate	935	70	0.25	0.08	0.09	0.11	0.21	0.03	0.06	0.07	0.03	0.02	0.014	<0.001	0.508
Hexanoic acid, ethyl ester	1039	88	8.15	11.98	7.60	10.20	12.60	8.57	7.47	6.38	9.59	12.74	0.848	0.372	0.294
Octanoic acid, ethyl ester	1187	88	3.38	4.79	3.08	3.53	5.54	3.39	3.19	1.95	3.93	4.41	0.348	0.294	0.262
Decanoic acid, ethyl ester	1314	88	2.16	3.04	1.87	1.98	3.12	2.14	1.75	0.84	2.31	2.20	0.231	0.197	0.465
TOTAL ESTERS			**63.92**	**58.97**	**43.93**	**57.80**	**58.88**	**40.42**	**31.25**	**23.83**	**39.81**	**42**			
Furan, 2-ethyl	702	81	0.86	0.80	0.33	0.70	0.82	1.50	1.34	1.28	1.60	1.22	0.082	<0.001	0.640
2-n-Butyl furan	935	81	0.85	0.74	0.53	0.66	0.80	1.70	1.33	1.05	1.78	1.40	0.086	<0.001	0.411
Furan, 2-pentyl	1026	81	8.27	7.25	5.11	7.48	7..92	10.00	9.30	6.80	10.64	9.49	0.781	0.207	0.696
Furan, 2-propyl	1078	81	0.17	0.15	0.14	0.14	0.26	1.83	1.72	1.62	2.46	2.49	0.098	<0.001	0.410
TOTAL FURAN			**10.15**	**8.94**	**6.11**	**8.98**	**9.80**	**15.03**	**13.69**	**10.75**	**16.48**	**14.60**			
Sulfide, allyl methyl	699	88	8.03	4.91	7.04	11.53	3.39	6.27	4.52	5.40	3.38	3.70	0.967	0.253	0.644
1H-Pyrrole, 3-methyl	792	81	0.13	0.14	0.09	0.13	0.12	0.17	0.16	0.21	0.13	0.10	0.009	0.089	0.555
1,3-Benzenediol, monobenzoate	829	105	0.85	1.18	0.96	1.03	1.06	0.48	0.34	0.29	0.53	0.58	0.055	<0.001	0.780
Pyrazine, 2,6-dimethyl-	969	108	2.64	2.51	2.23	2.64	1.80	2.66	1.65	2.37	2.02	1.42	0.227	0.486	0.664
OTHERS COMPOUND			**11.65**	**8.74**	**10.32**	**15.33**	**6.37**	**9.58**	**6.67**	**8.27**	**6.06**	**5.80**			

Sig.: significance; ns—not significant; SEM—Standard Error of the Mean; LRI: linear retention index calculated for DB-624 capillary column installed on a gas chromatograph equipped with a mass selective detector; m/z: quantifier ion. Treatments: T1—Basic diet and commercial feed; T2—Basic diet + 10% crude olive cake; T3—Basic diet + 10% olive cake in two phases; T4—Basic diet + 10% exhausted olive cake; T5—Basic diet + 10% exhausted olive cake + 1% olive oil.

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3.2. Sensory Characteristics

In Figures 1a and 2a we can observe the average scores obtained in the sensory analysis of the dry-cured loin and dry-cured "cachaço". For these products, the panel tasters evaluated 16 quantitative sensory attributes (muscle color, flavor persistency, flavor intensity, bitterness, acidity, sweetness, saltiness, chewiness, juiciness, hardness, skatole odor, androsterone odor, odor intensity, fat distribution, muscle/fat and fat color). The obtained results indicated that all dry-cured loin and dry-cured "cachaço" were characterized by relatively high overall quality. For both dry-cured products, the different treatments applied to the animals´ feeding had no significant effects on the taster's evaluation of the attributes described above.´ Concerning dry-cured "cachaço", the flavor intensity (5.96-6.36), flavor persistency (5.78-6.05), muscle color (5.50-6.00), odor intensity (5.94-6.30) and juiciness (5.80-6.19), were the attributes with the highest scores by the panel of tasters in the different treatments. As for the dry-cured loin, the most outstanding attributes were flavor intensity (5.78-6.05), flavor persistency (5.36-5.81), muscle/fat ratio (6.69-7.03), heterogeneous fat distribution (6.21-6.56) and odor intensity (5.76-6.01). On the other hand, the panel of tasters attributed the lowest values, for both dry-cured products of Bísaro pork, in the attributes of acidity, bitterness, androsterone and skatole odors. For the flavor intensity attribute, an average score of 5.93 – 6.36 for the dry-cured "cachaço" and 5.78 – 6.05 for the dry-cured loin. Lower values were also obtained by other authors for this attribute in dry-cured loin from the different lines of Iberian pig (5.22-5.67) [3]. Regarding odor intensity slightly lower values were obtained in dry-cured loin of Entrepelado and Retinto lines for the intensity odor [3]. The values obtained for the juiciness of the dry-cured loin in this work are in line with the values obtained for the dry-cured loin of different lines of Iberian pigs with a commercial base diet [3]. The attributes of muscle/fat, hardness and juiciness are directly related. It is important to mention that the flavor intensity for both products in this work was superior to those obtained by other authors in dry-cured loin of Iberian pork [3]. The dry-cured "cachaço" had a lower hardness score, and a higher proportion of muscle/fat and juiciness score when compared to the dry-cured loin. According to Leite et al. [12] the dry-cured "cachaço" has a higher ?? total amount of fat, which will be directly related to the sensory assessment by the panel tasters. This fact was also verified in the composition of the volatiles. The major aldehydes were significantly higher in the dry-cured "cachaço", and these compounds were directly related to the amount of total fat. The sensory characteristics indicated that the use of different treatments in the animals feeding resulted in higher acceptability of the final product. These results are in accordance with the ones reported by Fortin et al [39] in which juiciness, tenderness, flavor and absence of off-flavors were the most important attributes comprising the sensory experience during meat consumption.

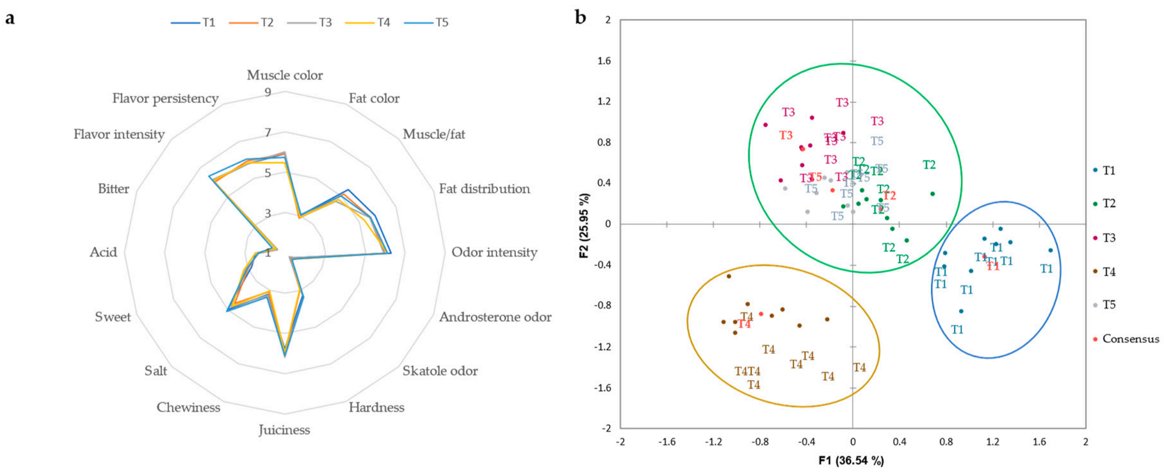

Figure 1. Sensory profile (a) and consensus configuration from GPA (b) of dry-cured "cachaço"of Bísaro pork.

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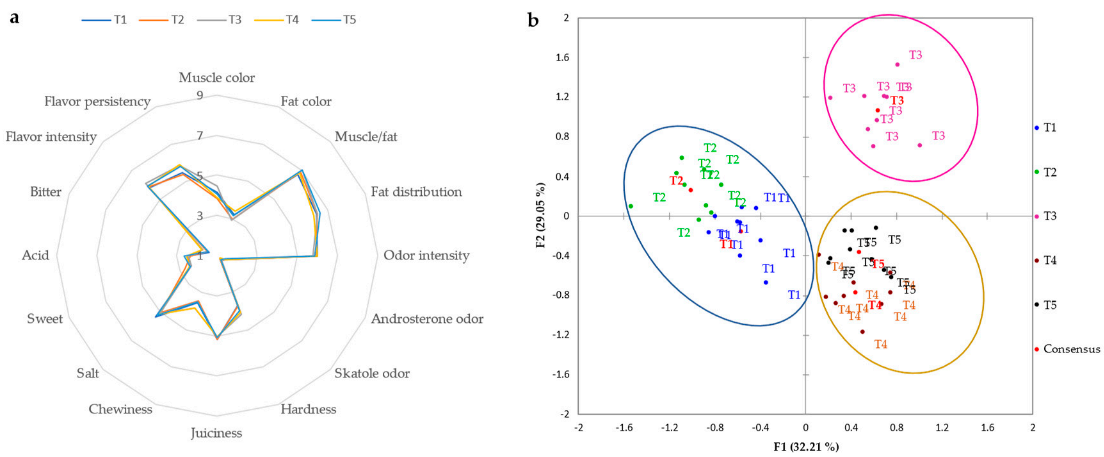

Figure 2. Sensory profile (a) and consensus configuration from GPA (b) of dry-cured Loin of Bísaro pork.

In the present research, in order to minimize the differences between testers, the GPA was used to find a consensus. Figures 1b and 2b show the biplot of the consensus configuration with the correlations between the sensory attributes, treatments and coordinates of the dry-cured "cachaço" and dry-cured loin. For the dry-cured "cachaço" (Figure 1b) F1 and F2 together explained 62.49 % of the total variability. Regarding dry-cured loin (Figure 2b) F1 and F2 together explained 61.26 % of the total variability. According to the coordinates of the different types of treatments and the correlation of the sensory attributes, panel tasters separated the treatments in three groups for both products, although they do not have the same trends. Concerning to dry-cured "cachaço" the taster panel clearly separated the treatment T1 (Basic diet and commercial feed) and T4 (Basic diet + 10% exhausted olive cake). The treatments T2 (Basic diet + 10% crude olive cake), T3 (Basic diet + 10% olive cake in two phases), and T5 (Basic diet + 10% exhausted olive cake + 1% olive oil) were grouped into one group. As far as the dry-cured "cachaço" is concerned, the tasters were able, though the 16 attributes studied, to group the T4 and T1 treatments separately. As for the other treatments, the various sensory attributes grouped them together. For the dry-cured loin, and across the 16 sensory attributes, treatments T4 and T5 were grouped together. This makes perfect sense, since these treatments concern the same type of olive cake except for the 1 % fat added in T5. There was another group joining T2 and T1. Finally, T3 was separated from the other treatments.

4. Conclusions

The results obtained in the present study allow us to conclude that the introduction of olive industry by-products, namely olive cake, does not significantly influence two major acceptability parameters of dry-cured loin and dry-cured "cachaço": sensory analysis and volatile profile. Regarding sensory analysis, the panelists were not able to detect differences among the olive cakes introduced in the pig's diet. This observation is directly related to the volatile profile, which was not significantly affected. These results also show that the addition of olive cake in the pigs' diet is a triple positive strategy: 1. It is possible to reduce costs with pigs' diet, by introducing smaller percentages of olive cake (a by-product with residual costs and available in high amounts, promoting in this way the circular economy); 2. The use of olive cake will open a new line of research for its use and will reduce the quantity of this olive waste to process, being highly beneficial for the environment (since the olive cake is a hazardous and toxic by-product); 3. The olive cake does not negatively affect the sensory and volatile components of the processed meat products, maintaining them appealing to the consumer.

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