Journal of CO2 Utilization 70 (2023) 102455

Contents lists available at ScienceDirect

Journal of CO2 Utilization

journal homepage: www.elsevier.com/locate/jcou

Supercritical co2 deodorization of dried pork liver

B. Abril [a], J.M. Lorenzo [b,c], J.V. García-Pérez [a], M. Contreras [a], J. Benedito [a,*]

[a] Department of Food Technology, (Universitat Politècnica de València, Valencia, Spain
[b] Centro Tecnológico de la Carne de Galicia, Rúa Galicia No. 4, Parque Tecnológico de Galicia, San Cibrao das Viñas, Ourense, Spain
[c] Universidad de Vigo, Área de Tecnología de los Alimentos, Facultad de Ciencias de Ourense, 32004 Ourense, Spain

ARTICLE INFO

Keywords:
Pork liver
Deodorization
Steam distillation
Supercritical CO_2
Volatile
Off-flavour

ABSTRACT

Pork liver has excellent nutritional properties but is a highly perishable product often rejected by consumers due to its strong unpleasant flavour. The objective of this study was to analyze the feasibility of the deodorization and defatting of dried pork liver by means of supercritical CO_2 (SC-CO_2) and conventional vacuum steam distillation (VSD).

The results showed that both deodorization techniques were effective at reducing volatile organic compounds (VOCs). Through VSD, the VOC content was reduced by 67.6% UA, while an 81.3% UA reduction was achieved by SC-CO_2, with respect to dried pork liver. In addition, 3 characteristic compounds of raw pork liver were completely eliminated by applying SC-CO_2, which could potentially reduce the characteristic mushroom (1-octen-3-ol), fatty and green (1-nonanol), and fishy ((*E*,*E*)– 2,4-heptadienal) off-flavours. Therefore, SC-CO_2 could be considered a promising technique for the elimination of VOCs, and furthermore it leads to a reduction in the fat content (24.9%).

1. Introduction

Pork liver, like other meat co-products and pork viscera, is a co-product of the porcine industry with a high nutritional value, being an important source of proteins (22.05%), lipids (2.94%), minerals and vitamins [1]. However, the commercial value of pork liver is very low at this moment [2]. In this context, it is of interest to valorize animal co-products, such as pork liver, searching for new products or ingredients in order to achieve a more competitive and sustainable industry [3]. Different studies have reported the valorization of pork liver as a means of obtaining proteins and liver hydrolysates, which have excellent functional and technological (foaming, emulsifying and solubility) properties and biological activity as bioactive peptides [2,4].

Nowadays, pork liver is mainly used as an ingredient in pâté products and animal feed [3], what is linked to two main aspects. The first issue is its highly perishable nature, since it contains a high water content (73%) and many nutrients [5]. In order to address stability problems, air drying could be considered as a suitable preservation technique of moderate cost. In addition, dried pork liver facilitates storage and reduces weight and volume in liver processing and transportation [5]. The second thing that explains its low commercial value, is linked to its strong, characteristic flavour, which leads to consumer rejection. The pork liver off-flavour has been described as intensely fishy and metallic [6]. Several studies have aimed to improve liver acceptability using different culinary techniques to eliminate or mask liver's unpleasant odour [7]. Thereby, a deep understanding of pork liver deodorization, using conventional or emerging techniques, would be a matter of relevant research and necessary in order to improve further uses of its protein fraction.

The issue related to off-flavours in pork liver is common to other novel protein sources, such as in the case of legumes or other vegetables [8–11]. In addition to vegetable proteins, whey protein concentrates have also been associated with tastes that are unpleasant for the consumer [12]. For these reasons, unless the flavour of the new protein isolates is improved, their direct use in human foodstuffs will remain very limited.

Deodorization consists of reducing or completely eliminating the content of those Volatile Organic Compounds (VOCs) associated with an unpleasant odour. Previous studies have applied deodorization to oils, either oilseed-based [13] or fish-based [14]. Deodorization has also been applied to fish [15] and spices [16], among other foods. However, to our knowledge, the application of deodorization techniques to meat products has not been addressed to date. To date, the most frequently-applied and efficient deodorization technique for the extraction of VOCs is

* Corresponding author.
E-mail address: jjbenedi@tal.upv.es (J. Benedito).

https://doi.org/10.1016/j.jcou.2023.102455
Received 21 November 2022; Received in revised form 1 March 2023; Accepted 7 March 2023
Available online 14 March 2023

distillation, which is based on the use of water as an extraction medium, either as a liquid or as vapour. Specifically, deodorization by steam distillation is the most common method [16]; this consists of generating steam at a high or moderate temperature (with or without vacuum) and putting it in contact with the sample to be deodorized [13]. This method has been shown to be the most effective at deodorizing products, such as turmeric powder, reducing the odour to a greater extent than other methods, such as Kjeldahl, rotary evaporation or non-steam vacuum distillation [16].

The use of supercritical CO_2 (SC-CO_2) represents one of the few alternatives to distillation. The physical properties of SC-CO_2, including high compressibility, liquid-like density, low viscosity, and high diffusivity [17], allow its penetration into the solid matrix and the solubility of target compounds. Moreover, SC-CO_2 has other advantages: it has a low critical temperature (31ºC) and surface tension and better selectivity and is also non-toxic, non-flammable, economical and easily removable from the matrix to be deodorized [18]. SC-CO_2 has been used in the food industry for the extraction of different molecules, such as lipids and cholesterol [19], colorants such as tomato lycopene [20], caffeine from coffee or tea [21] and various components (squalene, lipids, vitamin A and β carotene) from liver [22–24]. In addition to extracting compounds of interest, SC-CO_2 has been used to reduce the fat content of high-protein food sources, such as meat and fish, preventing protein denaturation due to the mild temperatures used [25]. Although the application of this technique for deodorization purposes is relatively expensive, its numerous advantages are in line with the demand for clean and safe technologies [25]. SC-CO_2 has been applied for the deodorization of drinking water [26], custard apple seed powder [27], truffles [28], lavandin and thyme extracts [29], soy-protein isolate [30] and fish sauce [31]. However, so far, the use of SC-CO_2 for the extraction of VOCs in meat products has not been addressed. Therefore, the use of SC-CO_2 for the deodorization, and simultaneous defatting, of the pork liver would help to revalorize a product with a very relevant nutritional composition, taking advantage of its high protein content. Thus, the objective of this study was to analyze the feasibility of using supercritical CO_2 for the deodorization and defatting of dried pork liver comparing its performance with vacuum steam distillation.

2. Material and methods

2.1. Raw material and sample preparation

Raw pork livers (RPL), from an industrial slaughterhouse, were transported at 4ºC to the laboratory. Liver conditioning consisted of i) the separation of its 4 main lobes, ii) the splitting of each lobe into two parts, (iii) vacuum packaging (200 ×300 PA / PE, Sacoliva, Castellar del Vallès, Barcelona) and (iv) freezing (at −20ºC) until processing.

2.2. Drying process

Before drying, vacuum packaged samples were tempered at 2ºC for 2 h in order to facilitate further handling. Using a household device, cylinders (12.6 mm diameter x 15 mm height) were obtained to be dried. Drying was carried out at a high temperature (105ºC) using a convective oven (FD 56, Binder, Germany) with an air speed of 1.3 $m{\cdot}s^{-1}$ for 24 h until constant weight was reached (AOAC 950.46 B) [32]. Subsequently, the dried pork liver (DPL) samples were ground and mixed with the aim of obtaining a representative sample. Then, the dried pork liver samples were vacuum packaged in 200 × 300 PA / PE bags (Sacoliva, Castellar del Vallès, Barcelona) of approximately 3 ± 0.05 g for deodorization tests by vacuum steam distillation and 30 ± 0.05 g for deodorization tests by supercritical CO_2. Finally, the samples were stored in refrigeration at 4ºC until their subsequent deodorization.

2.3. Deodorization process

2.3.1. Vacuum steam distillation

Deodorization by vacuum steam distillation (VSD) was carried out using the experimental set-up shown in Fig. 1. The distillation balloon (2, Fig. 1), with 2 L of distilled water, was placed in the water bath (HB digital 115, IKA, Germany) (1, Fig. 1) which was kept at 80ºC. The balloon was attached to the distillation column (3, Fig. 1) in which 3 g of dried pork liver samples were placed into filter-paper cartridges. Thus, the steam generated rose in the column passing through the sample with a flow of 0.19 g/s. Afterwards, the steam passed through a condenser (4, Fig. 1), which was connected to a cooling unit (5, Fig. 1), and the condensate was collected in a small distillation balloon (6, Fig. 1). The entire system was connected to a vacuum pump (MZ 2 C, Vacuubrand, Germany) (7, Fig. 1) (0.07 mbar) that allowed steam generation at low temperature (42 ± 2ºC). A water trap (8, Fig. 1) was used to prevent the steam from reaching the vacuum pump. After the deodorization operation was completed, the volume of water collected was measured in order to compute the steam used (1100–1200 mL). The deodorization time was set at 90 min. Finally, the deodorized pork liver samples (DPL-VSD) were removed from the paper cartridge and stored frozen (−20ºC) until their VOCs analysis. Deodorization by VSD was replicated 3 times.

2.3.2. Supercritical CO_2

Pork liver was treated with supercritical carbon dioxide (SC-CO_2) for the purposes of removing the VOCs using laboratory-scale equipment. Fig. 2 shows the SC-CO_2 system, which consisted of a deodorization vessel (1, Fig. 2) into which a stainless steel cell, containing 30 g of dried pork liver, was placed (2, Fig. 2). The stainless steel cell had filters on both sides, that allowed the sample to be retained and the SC-CO_2 to circulate at a pre-set temperature, and in pre-established pressure and time conditions. The vessel containing the sample to be deodorized was immersed in a thermostatic water bath (3, Fig. 1) to maintain the temperature. In addition, the system was supplied with a separator tank (4, Fig. 1) into which 90 g of activated carbon was placed for its ability to absorb volatile compounds [33]. The CO_2 tank (5, Fig. 1) was connected to a cooling system, which maintained CO_2 at − 18ºC (6, Fig. 1) and a diaphragm dosing pump (LDB, LEWA, Tokyo, Japan) was used to achieve the desired pressure in the deodorization vessel (7, Fig. 1). A manometer and a K-type thermocouple were installed inside the vessel to measure the temperature and pressure of the sample throughout the process (8, Fig. 1).

Carbon dioxide was pumped from the cooling tank (6, Fig. 2) to the deodorization vessel (1, Fig. 2), where the sample to be deodorized was placed, changing states from liquid to supercritical. When the supercritical CO_2 leaves the deodorization vessel, it turns to vapour and passes through the separator tank containing active carbon (4, Fig. 2). The CO_2 flow rate was constant during all the experiments (1 ± 0.05 kg CO_2/h).

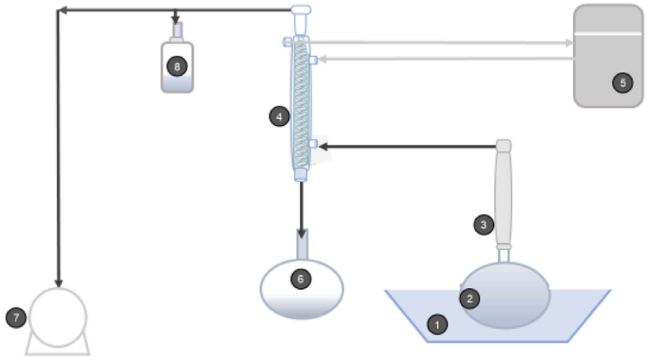

Fig. 1. Deodorization by vacuum steam distillation: 1- Water bath; 2-Distillation balloon; 3-Distillation column; 4-Condenser; 5-Cooling system; 6-Condensate balloon; 7-Vacuum pump; 8- Water trap.

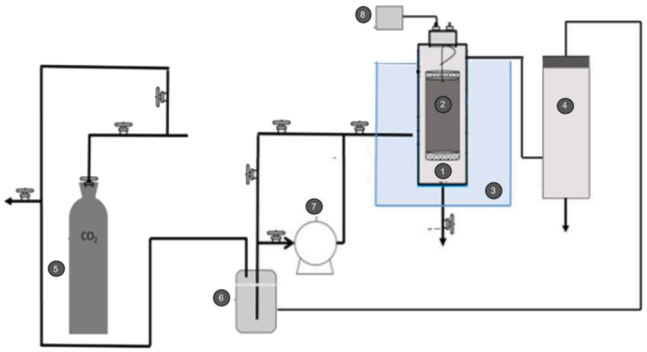

Fig. 2. Deodorization by supercritical CO_2: 1- Deodorization vessel, 2- Stainless steel cell containing the sample, 3- Bath, 4- Separator tank with active carbon, 5- CO_2 tank, 6-Reservoir/cooling tank, 7-Pump, 8- Manometer and a K-type thermocouple.

The circuit was closed when the CO_2 vapour turned into liquid by passing it through the cooling reservoir (6, Fig. 2) and recirculated again. Subsequently, the deodorized pork liver samples (DPL-SCCO$_2$) were extracted from the stainless steel container and stored frozen (−20ºC) until their VOC analysis.

A Box Behnken experimental design was carried out for the optimization of the deodorization conditions, including the following variables: pressure (200, 325 and 450 bar), temperature (35, 50 and 65ºC) and time (30, 60 and 90 min). The design involved three levels for each factor and three replicates at the centre point (15 experimental runs) (Table 1).

2.4. Volatile organic compound analysis

The volatile organic compounds (VOCs) were analyzed following the methodology previously described by Domínguez et al. [34] and consisting of solid phase microextraction with headspace (HS-SPME), coupled to gas chromatography (GC) and mass spectroscopy (MS) detection (HS-SPME-GC/MS).

For the HS-SPME, a 10 mm long molten silica fibre coated with a 50/30 mm thick layer of divinylbenzene, carboxene and polydimethylsiloxane (Supelco, Bellefonte, PA, USA) was used. Before carrying out the analysis, the fibre was conditioned by heating at 270ºC for 30 min. To perform the extraction, 1 ± 0.02 g were placed into 20 mL vials (Agilent Technologies, Santa Clara, CA, USA) and screwed with a laminated Teflon rubber disc. The samples were balanced for 15 min at 37ºC to ensure a homogeneous sample temperature and headspace. Then, the extractions were performed at 37ºC for 30 min

Once the extraction was completed, the fibre was transferred to the injection zone of the system consisting of the gas chromatograph and the mass spectrometer (GC-MS). The HS-SPME fibre was desorbed and maintained in the injection site (splitless mode and a helium pressure of 9.59 psi) at 260ºC for 8 min. After each injection, the fibre was washed and conditioned at 270ºC for 2 min to ensure that it was clean before the next extraction. Helium was used as the carrier gas at a constant flow rate of 1.2 mL/min (9.59 psi). The column that was used for the separation of the volatile components was a DB-624 capillary column 30 m long, 250 μm wide and with a film thickness of 1.4 μm (J&W Scientific, Folsom, CA, USA). A preheating was carried out to reach an isothermal temperature of 40ºC for 10 min, followed by a first heating step during which the temperature rose to 200ºC at 5ºC/min. Finally, there was a second heating step in which the temperature rose to 250ºC at 20ºC/min, which was maintained for 5 min. The analysis lasted for 49.5 min

As for the conditions of the MS, the transfer line was maintained at 260ºC. The ion source used was The Extraction Source, Xtr EI 350 (Agilent Technologies, Santa Clara, CA, USA). The mass spectrum was obtained using the selective mass detector, 5977B, working with an electronic energy of 70 eV, with an electron multiplier voltage of around 900 V (gain factor = 1) and obtaining 2.9 scanners/s in the m/z range of 40–550 in scan acquisition mode. The mass source was maintained at 230ºC while the mass quad was adjusted to 150ºC.

After the chromatographic analysis, all the data obtained were studied using the MassHunter Quantitative Analysis B.07.01 software and these were compared with bibliographic information. The integration of the peak areas was performed with the Agile2 algorithm, while the detection peak was obtained by deconvolution. The compounds were identified by comparing the mass spectra obtained with those published in the NIST14 database. The compounds were considered correctly identified when they had a matching factor of over 85%.

From the chromatograms, the content of each compound was determined by the area of its peak, which was expressed by units of area (UA). In order to calculate the percentage of VOCs for the different families of chemical compounds, the ratio between the UA for each VOC family and the total area (sum of the UA for all the VOCs detected) was computed for each of the analyzed samples: RPL, DPL, DPL-VSD and DPL-SCCO$_2$. In order to calculate how the deodorization affects VOC content, the loss (% UA) for each VOC family was computed as the ratio of the UA for each family of the deodorized sample (VSD and SC-CO$_2$) to the UA from dried pork liver (DPL).

Table 1
Box Behnken design for the SC-CO$_2$ deodorization experiments.

Treatment	Temperature (ºC)	Pressure (bar)	Time (min)
1	50	200	90
2	65	200	60
3	65	325	90
4	65	325	30
5	50	450	30
6	35	325	30
7	65	450	60
8	50	450	90
9	50	200	30
10	35	450	60
11	35	200	60
12	35	325	90
13	50	325	60
14	50	325	60
15	50	325	60

2.5. Chemical composition

To analyze the chemical composition (moisture, ash, protein, and fat contents) of DPL, DPL-VSD and DPL-SC-CO$_2$, the AOAC procedures [32] were followed. The moisture and ash contents were determined gravimetrically using a hot air oven and a muffle furnace, respectively. The protein content was estimated from the total Kjeldahl nitrogen (TKN x 6.25) by using a Gerhardt KB20 digestion system (C. Gerhardt GmbH & Co., Königswinter, Germany) and a Büchi K-314 distillation unit (Büchi Labortechnik AG, Flawil, Switzerland). The total fat content was determined gravimetrically by Sohxlet extraction with diethyl ether as a solvent. Each sample was analyzed in triplicate.

2.6. Statistical analysis

A response surface design (Box-Behnken) was employed using Statgraphics Centurion XVI (Statpoint Technologies Inc., Warrenton, VA, USA) to study how the temperature and pressure at which SC-CO$_2$ deodorization takes place and the length of time of the process (Section 2.3.2) influence the VOC contents. In addition, to evaluate the differences between the VOC content and chemical composition of the different samples (RPL, DPL, DPL-VSD and DPL-SCCO$_2$), an analysis of variance (ANOVA) was performed and the LSD (least significant differences) intervals were identified (95% confidence level) using Statgraphics Centurion XVI.

3. Results and discussion

3.1. Drying of raw pork liver

3.1.1. Impact on volatile organic compounds (VOCs)

In the present study, a total of 136 volatile organic compounds (VOCs) were identified (Table 2). In raw pork liver (RPL), 58 VOCs were detected, while in dried pork liver (DPL), the number of VOCs increased up to 116.

A significant variation in the results of the VOC quantification can be observed in Table 2. As illustrated in Fig. 3, the results of the quantitative analysis in terms of the percentages of units of area (UA) of the main VOC families in RPL were: hydrocarbons (46.6% UA), ketones (27.7% UA), aldehydes (9.6% UA), ethers (9.4% UA), alcohols (5.4% UA), sulphur compounds (0.8% UA), halogen compounds (0.2% UA), esters (0.2% UA), nitrogen compounds (0.06% UA) and furans (0.02% UA). As is illustrated in Fig. 3, there are great differences between the content of the main VOC families in RPL and DPL. Thus, the acid family was not detected in RPL. However, after the drying process, as a result of lipid hydrolysis [35] and from the degradation of the carbohydrates and aldehydes formed from the Strecker reaction [36], acids (15.9% UA) were formed [37].

As for the aliphatic alcohols, an increase was found when comparing RPL and DPL (from 5.4% to 9.5% UA). These compounds contribute to the flavour of the meat through unsaturated alcohols, and of these, 1-octen-3-ol must be highlighted. This was present in RPL and DPL, and its content increased after the drying process (from 3.16 to 53.01 UA) probably as a result of the degradation of fatty acids catalyzed by the lipoxygenase [38]. As in aliphatic alcohols, in aliphatic aldehydes, the VOC content increased in DPL compared to RPL (from 9.6% to 25.3% UA). In DPL, the content of every saturated aldehyde increased when compared to the content in RPL, such as pentanal (from 0.58 to 13.92 UA), hexanal (from 16.04 to 139.08 UA) and heptanal (from 0.47 to 3.59 UA). However, the content of unsaturated aldehydes did not increase in the same magnitude, in some cases even falling, such as 2-butenal, 2-methyl- (from 1.72 to 1.58 UA) or (*E*,*E*)− 2,4-heptadienal (from 5.63 to 2.26 UA). This was probably linked to the fact that unsaturated aldehydes are more reactive than saturated aldehydes and, therefore, more likely to participate in the Maillard reactions and give rise to other compounds [39].

Ketones are also one of the major flavour-related chemical families and are generated from lipid oxidation, alkane degradation and the dehydrogenation of alcohols by bacteria [40]. After drying, there was a smaller content of ketones (8.3% UA) than in RPL (27.7% UA).

One of the most noticeable changes in VOCs caused by drying was the increase in heterocyclic compounds, such as nitrogen compounds (pyrazines, 25.7% UA) and furans (0.7% UA) (Table 2). These heterocyclic compounds are generated through the Maillard reactions and are generally formed at high temperatures. Pyrazines contribute to the aroma of grilled meat [39,41]. As for furans, they were formed by reactions that take place at temperatures of over 100ºC, usually from fatty acid oxidation [42]. In the DPL, therefore, new compounds appeared, such as 3(2 H)-furanone, dihydro-2-methyl-, which present a sweet caramel odour [43].

In contrast, the content of other VOC families in DPL dropped after drying: halogenated compounds (from 0.21% to 0.03% UA), ethers (from 9.39% to 0.29% UA) and hydrocarbons (from 46.58% to 10.40% UA). This decrease is possibly due to the reactions that take place during drying, such as lipid oxidation, the participation of proteins and sugars in the Maillard reaction, protein degradation, and amino acid condensation with Maillard intermediates in Strecker degradation, among others.

3.1.2. Off-flavour removal

Im et al. [44] characterized the off-flavour of the VOCs present in pork liver as fishy, metallic and fatty, with hints of cardboard, nuts, such as almonds, oil, and nature, such as grass (green) and mushroom. The following compounds are those that contribute to the complexity of the unpleasant notes of pork liver:

- (*E*,*E*)− 2,4-heptadienal (Fishy odour)
- 1-octen-3-one and 1-hexanol (Metallic odour)
- (E)− 2-nonenal (Cardboard odour)
- Butanal, 2-methyl, benzaldehyde and pentanal (Almond odour)
- Propanal 2-methyl, hexanal and heptanal (Fatty odour)
- Hexanal, butanal, 2-butenal, 2-methyl- and 1-hexanol (Green-nature odour).
- 1-octen-3-ol (Mushroom-nature odour)

It has to be considered that the significant unpleasant aroma of pork liver is not only the result of a single odour, but the combination of the different ones, especially the metallic and fishy odours [6].

The drying process eliminated one of the characteristic compounds of the liver odour, the benzaldehyde (almond odour). However, the quantity of the two compounds to which the nutty odour is linked rose: butanal, 2-methyl- (from 19.04 to 30.55 UA) and pentanal (from 0.58 to 13.92 UA), (Table 2). Likewise, the units of the characteristic fatty odours increased: propanal 2-methyl from 1.39 to 8.69 UA, hexanal from 16.04 to 139.08 UA and heptanal from 0.47 to 3.59 UA (Table 2). As for the nature odour, identified as the green odour, two of the characteristic compounds of liver increased their content compared to RPL, hexanal (already shown) and butanal from 0.09 to 0.48 UA. However, 1-hexanol decreased from 3.79 to 0.86 UA and the content of 2-butenal, 2 methyl (1.58 UA) was similar to that of RPL (1.72 UA). In addition, 1-nonanol, a compound also characteristic to fatty, green odours that are undetected in RPL, was found in DPL. The units of the other characteristic nature odour, mushroom, attributed to 1-octen- 3-ol, increased from 3.16 to 53.01 UA after drying. This alcohol is usually described as an important volatile that contributes to the typical aroma of dry-cured meat products [45]. Finally, the fishy and metallic odours of the pork liver were not completely removed after the drying process. In this regard, (*E*,*E*)− 2,4-heptadienal, a compound related to a fishy odour, decreased in quantity from 5.63 UA in RPL to 2.26 UA in DPL and the content of 1-octen-3-one, related to a metallic odour, remained similar to that in RPL (2.25 UA in RPL and 1.82 UA in DPL).

3.2. Dried pork liver deodorization by vacuum steam distillation

3.2.1. Impact on volatile organic compounds (VOCs)

Overall, the VOC content detected in DPL-VSD fell if compared to DPL (Fig. 4): the content of acids dropped by 91.9% UA, alcohols by 87.7% UA, aldehydes by 72.6% UA, halogen compounds by 51.9% UA, nitrogen compounds by 89.8% UA, sulphur compounds by 84.5% UA, esters by 26% UA, furans by 93% UA and ketones by 81.5% UA. In the families of ethers and hydrocarbons however, there was an increase of 20% UA and 55.9% UA, respectively. The increase in hydrocarbons as a result of the VSD technique was also observed in the deodorization of fish sauce (9.4% UA) [46]. It should be noted that the compounds of heptane, 2,4-dimethyl; hexane, 2,4,4-trimethyl- (hydrocarbons) and tetrahydrofuran (furan) were eliminated from DPL after VSD deodorization (Table 2).

3.3. Supercritical CO_2 deodorization of dried pork liver

3.3.1. Impact on volatile organic compounds (VOCs)

The factors analyzed in SC-CO_2 deodorization (temperature, pressure and time) did not significantly ($P < 0.05$) affect the VOC content. Thus, the statistical model evaluated fitted (R^2 <40%) the experimental results poorly. Therefore, the average VOC values for the different experimental conditions analyzed were considered. Thereby, as illustrated in Fig. 4, SC-CO_2 deodorization resulted in the complete removal of halogen compounds, and the following VOC families were greatly reduced if

Table 2
Profile of VOCs (UA = units area x 10^5) in raw pork liver (RPL), dried pork liver (DPL) and dried pork deodorized by vacuum steam distillation (DPL-VSD) and supercritical CO_2 (DPL-$SCCO_2$).

COMPOUND	RPL	DPL	DPL-VSD	DPL-$SCCO_2$	ODOUR	COMPOUND	RPL	DPL	DPL-VSD	DPL-$SCCO_2$	ODOR
Acids						Sulphur compounds					
Acetic acid	nd_A	120.78 ± 24.87$_D$	9.19 ± 1.36$_C$	5.96 ± 0.71$_B$	sour, vinegar	Dimethyl sulfide	5.62 ± 2.70$_B$	nd_A	nd_A	nd_A	cabbage, sulfur, gasoline
Propanoic acid	nd_A	3.67 ± 0.73$_C$	1.15 ± 0.5$_B$	nd_A		Carbonyl sulfide	0.56 ± 0.34$_B$	0.38 ± 0.08$_B$	0.07 ± 0.01$_A$	0.06 ± 0.03$_A$	
Butanoic acid	nd_A	14.04 ± 3.48$_D$	0.84 ± 0.61$_C$	0.20 ± 0.01$_B$	cheese	Dimethyl disulfide	0.02 ± 0.01$_A$	2.95 ± 0.91$_D$	0.51 ± 0.01$_B$	0.91 ± 0.12$_C$	onion, cabbage, putrid
Alcohols						Acetamide	nd_A	3.63 ± 1.12$_C$	0.50 ± 0.09$_B$	0.45 ± 0.15$_B$	
1-Propanol	0.59 ± 0.19$_B$	nd_A	nd_A	nd_A		**Esters**					
Cyclopentanol	0.62 ± 0.22$_B$	nd_A	nd_A	nd_A		Acetic acid, methyl ester	nd_A	0.21 ± 0.08$_C$	0.33 ± 0.12$_C$	0.07 ± 0.05$_B$	
2-Butanol, 2,3-dimethyl-	0.43 ± 0.17$_B$	nd_A	nd_A	nd_A		Acetic acid ethenyl ester	nd_A	2.07 ± 0.55$_D$	0.04 ± 0.01$_B$	0.89 ± 012$_C$	
2-Pentanol, 2-methyl-	0.13 ± 0.16$_B$	nd_A	nd_A	nd_A		Propanoic acid, ethyl ester	1.84 ± 1.00$_B$	nd_A	nd_A	nd_A	
Glycidol	nd_A	6.72 ± 2.03$_C$	1.15 ± 0.50$_B$	1.14 ± 0.10$_B$		Formic acid, 2-propenyl ester	nd_A	21.88 ± 4.93$_B$	16.44 ± 0.74$_B$	nd_A	
1-Propanol, 2-methyl-	nd_A	0.17 ± 0.06$_C$	0.03 ± 0.01$_B$	nd_A		Ethyl Acetate	nd_A	0.73 ± 0.32$_C$	1.62 ± 0.04$_D$	0.32 ± 0.01$_B$	pineapple
1-Butanol	0.24 ± 0.03$_B$	0.42 ± 0.09$_C$	0.28 ± 0.12$_{BC}$	nd_A	medicine, fruit,wine	**Ethers**					
2-Propen-1-ol	nd_A	10.26 ± 2.41$_C$	0.21 ± 0.01$_B$	nd_A		Dimethyl ether	71.91 ± 45.21$_C$	2.55 ± 0.75$_A$	3.06 ± 0.61$_A$	8.14 ± 0.69$_B$	
1-Butanol, 3-methyl-	2.11 ± 1.77$_D$	0.21 ± 0.02$_C$	0.06 ± 0.0$_B$	nd_A	whiskey, malt, burnt	**Furanes**					
1-Butanol, 2-methyl-	0.37 ± 0.12$_D$	0.11 ± 0.01$_C$	0.04 ± 0.01$_B$	nd_A	malt, wine, onion	Furan, 3-methyl-	0.07 ± 0.04$_A$	0.74 ± 0.19$_C$	0.15 ± 0.04$_A$	0.25 ± 0.05$_B$	mint
1-Pentanol	0.53 ± 0.05$_B$	5.71 ± 1.02$_C$	0.37 ± 0.02$_A$	0.76 ± 0.45$_{AB}$	fruit, balsamic	Tetrahydrofuran	nd_A	0.54 ± 0.11$_C$	nd_A	0.04 ± 0.03$_B$	
1-Butanol, 2,3-dimethyl-	0.06 ± 0.01$_B$	nd_A	nd_A	nd_A		Furan, 2-ethyl-	0.06 ± 0.02$_B$	0.93 ± 0.17$_D$	0.14 ± 0.04$_C$	nd_A	
(S)-(+)− 1,2-Propanediol	nd_A	1.87 ± 0.25$_C$	1.18 ± 0.07$_B$	nd_A		3(2 H)-Furanone, dihydro-2-methyl-	nd_A	0.44 ± 0.06$_C$	0.02 ± 0.01$_B$	1.20 ± 0.18$_D$	caramel, sweet
2,3-Butanediol	27.39 ± 19.07$_C$	0.79 ± 0.10$_B$	0.06 ± 0.03$_B$	nd_A		3-Furanmethanol	nd_A	2.42 ± 0.24$_C$	0.06 ± 0.01$_B$	nd_A	
1-Pentanol, 2-methyl-	1.01 ± 0.66$_B$	nd_A	nd_A	nd_A	pungent	Ethanone, 1-(2-furanyl)-	nd_A	0.96 ± 0.15$_C$	0.05 ± 0.02$_B$	nd_A	
1-Butanol, 3-methyl-, acetate	0.07 ± 0.01$_C$	0.11 ± 0.02$_D$	0.03 ± 0.01$_B$	nd_A		**Hydrocarbures**					
1-Hexanol	3.79 ± 0.94$_C$	0.86 ± 0.08$_B$	0.13 ± 0.02$_A$	0.09 ± 0.02$_A$	resin, flower, green, metallic	Butane, 1-chloro-	nd_A	0.07 ± 0.02$_B$	0.04 ± 0.02$_B$	nd_A	
Phenylethyl Alcohol	1.07 ± 0.03$_B$	nd_A	nd_A	nd_A	honey, spice, rose, lilac	Pentane	nd_A	3.36 ± 0.68$_B$	4.24 ± 0.74$_B$	2.99 ± 1.09$_B$	alkane
Ethanol, 2-butoxy-	nd_A	0.41 ± 0.03$_C$	0.32 ± 0.01$_B$	nd_A		Pentane, 3-methyl-	0.16 ± 0.07$_C$	0.07 ± 0.01$_B$	0.05 ± 0.01$_A$	0.09 ± 0.02$_{BC}$	
(S)-(+)− 5-Methyl-1-heptanol	nd_A	0.46 ± 0.13$_D$	0.02 ± 0.01$_B$	0.10 ± 0.04$_C$		Cyclopentane, methyl-	0.02 ± 0.01$_B$	0.12 ± 0.08$_C$	0.04 ± 0.01$_C$	nd_A	
1-Octen-3-ol	3.16 ± 0.71$_B$	53.01 ± 1.12$_C$	4.95 ± 1.22$_B$	nd_A	mushroom	Benzene, 1,3-dimethyl-	nd_A	nd_A	nd_A	0.21 ± 0.01$_B$	
Ethanol, 2,2'-oxybis-	nd_A	0.37 ± 0.06$_B$	0.42 ± 0.04$_B$	nd_A		Heptane, 3-methyl-	nd_A	1.10 ± 0.41$_C$	0.20 ± 0.02$_B$	nd_A	

(*continued on next page*)

Table 2 (*continued*)

COMPOUND	RPL	DPL	DPL-VSD	DPL-$SCCO_2$	ODOUR	COMPOUND	RPL	DPL	DPL-VSD	DPL-$SCCO_2$	ODOR
Acids						Sulphur compounds					
1,2-Propanediol, 1-phenyl-	nd_A	0.18 ± 0.06 $_C$	0.05 ± 0.01$_B$	nd_A		Undecane	nd_A	0.63 ± 0.12 $_C$	0.44 ± 0.15 $_C$	0.38 ± 0.03$_B$	
1-Nonanol	nd_A	0.25 ± 0.03$_B$	0.32 ± 0.01 $_C$	nd_A	fatty, green	Dodecane	nd_A	0.83 ± 0.12$_D$	0.05 ± 0.01$_B$	0.23 ± 0.11 $_C$	
1-Tetradecanol	nd_A	0.15 ± 0.02$_B$	0.21 ± 0.01 $_C$	nd_A	coconut	Cyclododecane	nd_A	nd_A	nd_A	0.25 ± 0.01$_B$	
2,2,4-Trimethyl-1,3-pentanediol diisobutyrate	nd_A	0.55 ± 0.06$_B$	0.34 ± 0.04$_B$	nd_A		Cyclopropane, pentyl-	nd_A	0.45 ± 0.03 $_C$	0.03 ± 0.01$_B$	nd_A	
Aldehydes						Octane	nd_A	7.93 ± 1.25 $_C$	1.26 ± 0.74$_B$	2.33 ± 0.66$_B$	
Propanal	3.17 ± 1.07$_B$	nd_A	nd_A	nd_A	solvent, pungent	2-Octene, (*E*)-	1.37 ± 0.97 $_C$	nd_A	nd_A	0.31 ± 0.12$_B$	
Propanal, 2-methyl-	1.39 ± 0.94 $_A$	8.69 ± 2.21 $_C$	5.22 ± 0.74$_B$	2.01 ± 1.12 $_A$	smoke, fatty	3-Octene, (*E*)-	0.67 ± 0.32 $_C$	nd_A	nd_A	0.16 ± 0.01$_B$	
Butanal	0.09 ± 0.07 $_A$	0.48 ± 0.13$_B$	0.16 ± 0.05 $_A$	0.20 ± 0.04 $_A$	pungent, green	Heptane, 2,4-dimethyl-	2.20 ± 1.01 $_C$	0.01 ± 0.01$_B$	nd_A	nd_A	
Butanal, 3-methyl-	15.57 ± 9.01$_{AB}$	17.38 ± 4.12$_B$	16.66 ± 3.97$_B$	7.12 ± 1.81 $_A$	malt	Hexane, 2,4,4-trimethyl-	nd_A	0.19 ± 0.02$_B$	nd_A	nd_A	
Butanal, 2-methyl-	19.04 ± 8.06$_{AB}$	30.55 ± 6.54$_B$	23.66 ± 5.72$_B$	9.63 ± 1.16 $_A$	cocoa, almond	Heptane, 2,2,4,6,6-pentamethyl-	333.45 ± 151.44$_D$	15.59 ± 1.02 $_A$	58.10 ± 1.02 $_C$	35.63 ± 1.95$_B$	
Pentanal	0.58 ± 0.18 $_A$	13.92 ± 2.84$_D$	2.12 ± 0.43$_B$	5.03 ± 1.19 $_C$	almond, malt, pungent	Decane	nd_A	0.39 ± 0.15$_B$	14.85 ± 1.41$_{AC}$	nd_A	
2-Butenal, 2-methyl-	1.72 ± 0.41 $_C$	1.58 ± 0.51 $_C$	0.37 ± 0.02$_B$	0.14 ± 0.06 $_A$	green, fruit	Nonane	0.31 ± 0.24$_B$	nd_A	nd_A	nd_A	
Hexanal	16.04 ± 4.33$_B$	139.08 ± 16.80$_D$	9.60 ± 2.04 $_A$	26.39 ± 2.48 $_C$	green, grassy, tallow, fatty	2,2,4,4-Tetramethyloctane	14.21 ± 8.92$_B$	3.46 ± 0.26 $_A$	11.84 ± 0.84$_B$	8.63 ± 0.33$_B$	
Heptanal	0.47 ± 0.29 $_A$	3.59 ± 0.29 $_C$	0.57 ± 0.02 $_A$	0.83 ± 0.06$_B$	fatty, citrus, rancid, unpleasant	Undecane, 5,5-dimethyl-	nd_A	0.83 ± 0.41$_B$	1.95 ± 0.06$_D$	1.57 ± 0.05 $_C$	
(E)— 2-Nonenal	0.61 ± 0.14$_B$	0.62 ± 0.12$_B$	0.12 ± 0.02 $_A$	0.18 ± 0.09 $_A$	cardboard	Undecane	nd_A	0.63 ± 0.23$_B$	0.44 ± 0.04$_B$	0.38 ± 0.02$_B$	
Benzaldehyde	9.05 ± 3.21 $_C$	nd_A	nd_A	0.70 ± 0.02$_B$	almond, burnt sugar	Decane, 2,3,6-trimethyl-	nd_A	0.28 ± 0.02$_B$	0.24 ± 0.02$_B$	nd_A	
2-Hexenal, 2-ethyl-	nd_A	1.34 ± 0.10 $_C$	0.06 ± 0.02$_B$	nd_A		Pentane, 3,3-diethyl-	nd_A	0.16 ± 0.02$_B$	0.34 ± 0.04 $_C$	0.15 ± 0.09$_B$	
2-Isopropyl-5-methylhex-2-enal	0.04 ± 0.02$_B$	1.55 ± 0.04$_D$	0.19 ± 0.02 $_C$	nd_A		Undecane, 3-methylene-	nd_A	0.17 ± 0.01$_B$	0.32 ± 0.12$_B$	nd_A	
(*E*,*E*)— 2,4- Heptadienal	5.63 ± 1.05 $_C$	2.26 ± 0.77$_B$	1.79 ± 0.63$_B$	nd_A	fishy	Hexane, 3,3-dimethyl-	nd_A	0.29 ± 0.01 $_C$	0.26 ± 0.01$_B$	nd_A	
Halogen compounds						Nonane, 5-(1-methylpropyl)-	nd_A	0.41 ± 0.12$_B$	0.39 ± 0.01$_B$	nd_A	
Methane, oxybis[dichloro-	1.63 ± 0.99 $_C$	0.27 ± 0.08$_B$	0.13 ± 0.04$_B$	nd_A		Cyclododecane	nd_A	0.10 ± 0.01$_B$	0.19 ± 0.01 $_C$	0.25 ± 0.02$_D$	
Nitrogen compounds						Benzene, 1,3-dimethyl-	0.16 ± 0.05 $_A$	13.94 ± 1.66 $_C$	0.94 ± 0.04$_B$	0.21 ± 0.02 $_A$	
Fumaronitrile	nd_A	0.68 ± 0.08$_D$	0.15 ± 0.04$_B$	0.47 ± 0.06 $_C$		3-Ethyl-4-methyl-2-pentene	nd_A	2.14 ± 0.78 $_C$	0.32 ± 0.06$_B$	nd_A	
Tetraethyl ammonium fluoride	nd_A	2.45 ± 0.91 $_C$	0.01 ± 0.0$_B$	nd_A		Trimethylene oxide	nd_A	5.52 ± 1.48 $_C$	0.63 ± 0.05$_B$	nd_A	
Propane, 2-nitro-	nd_A	0.81 ± 0.20 $_C$	0.21 ± 0.02$_B$	nd_A		Diazene, dimethyl-	nd_A	2.88 ± 0.67 $_C$	0.30 ± 0.02$_B$	nd_A	
Pyridine, 2-methyl-	nd_A	0.84 ± 0.07 $_C$	0.02 ± 0.00$_B$	nd_A		Borane, diethyl (decyloxy)	nd_A	nd_A	nd_A	0.24 ± 0.02$_B$	
Pyrazine, methyl-	0.09 ± 0.01$_B$	41.90 ± 5.48$_D$	1.24 ± 0.11 $_C$	nd_A	popcorn	2-Methyl-1-butene	nd_A	0.09 ± 0.04$_B$	0.11 ± 0.02$_B$	nd_A	

(*continued on next page*)

Table 2 (*continued*)

COMPOUND	RPL	DPL	DPL-VSD	DPL-$SCCO_2$	ODOUR	COMPOUND	RPL	DPL	DPL-VSD	DPL-$SCCO_2$	ODOR
Acids						Sulphur compounds					
Cyclopentanone, 2-methyl-	nd_A	$0.25 \pm 0.02_C$	$0.03 \pm 0.01_B$	nd_A		D-Limonene	$4.25 \pm 0.80_C$	$0.55 \pm 0.07_B$	$0.59 \pm 0.01_B$	nd_A	
2-Hexanone, 5-methyl-	nd_A	$2.92 \pm 0.36_C$	$0.17 \pm 0.02_B$	nd_A		**Ketones**					
Pyrazine, 2,6-dimethyl-	$0.14 \pm 0.10_A$	$49.36 \pm 4.58_D$	$2.21 \pm 0.24_C$	$0.86 \pm 0.09_B$	cocoa, roasted nut, roast beef, medicine	Acetone	$13.43 \pm 7.41_B$	$13.87 \pm 3.62_B$	$3.56 \pm 1.11_A$	$10.02 \pm 1.01_B$	
Pyrazine, ethyl-	nd_A	$4.22 \pm 0.27_C$	$0.12 \pm 0.02_B$	nd_A	peanut butter, wood	2-Butanone	$3.06 \pm 1.98_A$	$11.33 \pm 2.37_B$	$2.97 \pm 1.05_A$	$1.95 \pm 0.33_A$	blue cheese
Pyrazine, 2,3-dimethyl-	nd_A	$2.53 \pm 0.17_C$	$0.09 \pm 0.02_B$	nd_A		1-Penten-3-one	nd_A	$5.52 \pm 1.52_D$	$0.02 \pm 0.01_B$	$0.31 \pm 0.05_C$	
Pyrazine, 2-ethyl-6-methyl-	$0.14 \pm 0.07_A$	$30.29 \pm 0.79_C$	$1.07 \pm 0.55_B$	$0.41 \pm 0.08_B$	fruit, sweet	2-Pentanone	$0.26 \pm 0.04_B$	$0.36 \pm 0.06_B$	$0.09 \pm 0.01_A$	$0.10 \pm 0.02_A$	butter, spicy, blue cheese
Pyrazine, trimethyl-	nd_A	$11.23 \pm 0.58_D$	$0.33 \pm 0.12_C$	$0.14 \pm 0.04_B$	roast, potato, must	3-Pentanone	$0.29 \pm 0.04_B$	$9.14 \pm 2.31_D$	$1.15 \pm 0.06_C$	nd_A	
Pyrazine, 3-ethyl-2,5-dimethyl-	$0.02 \pm 0.02_A$	$14.84 \pm 0.56_D$	$0.98 \pm 0.02_C$	$0.26 \pm 0.11_B$	potato, roast	2,3-Pentanedione	nd_A	$5.97 \pm 1.46_D$	$0.14 \pm 0.02_B$	$0.80 \pm 0.03_C$	
Pyrazine, 3,5-diethyl-2-methyl-	nd_A	$3.10 \pm 0.24_C$	$0.23 \pm 0.06_B$	nd_A		Methyl Isobutyl Ketone	nd_A	$0.95 \pm 0.13_C$	$0.15 \pm 0.02_B$	nd_A	
Pyrazine, 2,5-dimethyl-3-(2-methylpropyl)-	$0.01 \pm 0.00_B$	$1.71 \pm 0.25_D$	$0.13 \pm 0.05_C$	nd_A		2-Hexanone	nd_A	$0.28 \pm 0.03_D$	$0.04 \pm 0.01_B$	$0.11 \pm 0.01_C$	ether, grape
2-Isoamyl-6-methylpyrazine	nd_A	$8.46 \pm 1.69_C$	$0.43 \pm 0.05_B$	nd_A		2-Heptanone	$0.35 \pm 0.01_A$	$16.73 \pm 1.80_D$	$1.31 \pm 0.04_C$	$0.97 \pm 0.01_B$	soap, spicy and blue cheese
Ethanone, 1-(1 H-pyrrol-2-yl)-	nd_A	$1.02 \pm 0.27_C$	$0.06 \pm 0.01_B$	nd_A		Butyrolactone	nd_A	$2.82 \pm 0.14_C$	$0.56 \pm 0.04_B$	nd_A	
Pantolactone	nd_A	$6.09 \pm 1.48_C$	$3.53 \pm 1.21_B$	nd_A		5-Hexen-3-one	nd_A	$0.22 \pm 0.04_B$	$0.40 \pm 0.08_C$	$0.33 \pm 0.09_B$	
2-Pyrrolidinone	nd_A	$0.96 \pm 0.31_C$	$0.23 \pm 0.02_B$	nd_A		1-Octen-3-one	$2.25 \pm 1.23_C$	$1.82 \pm 0.14_C$	$0.03 \pm 0.01_A$	$0.66 \pm 0.22_B$	metallic
2-Decanone	nd_A	$2.05 \pm 0.15_C$	$0.50 \pm 0.02_B$	nd_A		3,5-Octadien-2-one	nd_A	$2.88 \pm 0.10_D$	$0.02 \pm 0.01_B$	$0.44 \pm 0.04_C$	
2(4 H)-Benzofuranone, 5,6,7,7a-tetrahydro-4,4,7a-trimethyl-, (R)-	nd_A	$0.31 \pm 0.09_B$	$0.14 \pm 0.09_B$	nd_A		Acetoin	$192.49 \pm 67.46_C$	$0.05 \pm 0.10_A$	$1.54 \pm 0.01_B$	$0.02 \pm 0.01_A$	butter, cream
Pyrazine	$0.05 \pm 0.02_A$	$3.02 \pm 0.54_C$	$0.45 \pm 0.04_B$	$0.05 \pm 0.01_A$		Cyclobutanone, 2,3,3-trimethyl-	nd_A	$2.17 \pm 0.02_C$	$0.20 \pm 0.01_B$	$0.22 \pm 0.04_B$	
Pyrolo [3,2-*d*] pyrimidin-2,4 (1 H,3 H)-dione	nd_A	$5.83 \pm 0.57_B$	$5.94 \pm 0.74_B$	$4.25 \pm 0.88_B$		2-Heptanone, 6-methyl-	nd_A	$1.22 \pm 0.04_B$	$1.75 \pm 0.07_C$	$1.12 \pm 0.31_B$	
Methylamine, N,N-dimethyl-	nd_A	$30.10 \pm 7.22_D$	$4.72 \pm 0.71_B$	$13.54 \pm 0.94_C$							

*Odour description obtained from database available on the web at https://www.flavornet.org/flavornet.html.
*nd, not detected.
Average values ± LSD intervals are given (n = 5).
Different capital letters show homogeneous groups established from LSD

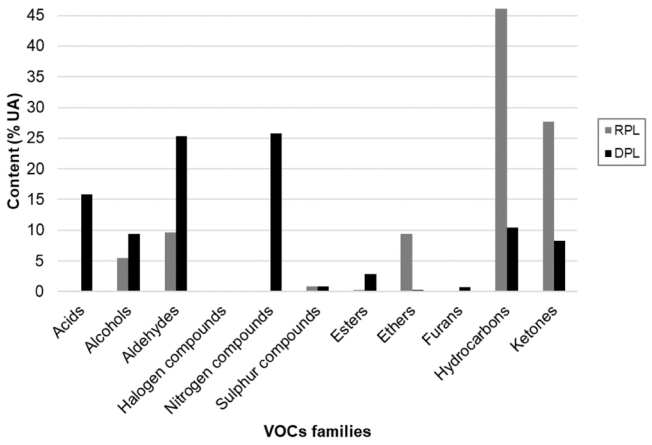

Fig. 3. Content (% UA) of the main VOC families detected in raw pork liver (RPL) and in dried pork liver (DPL).

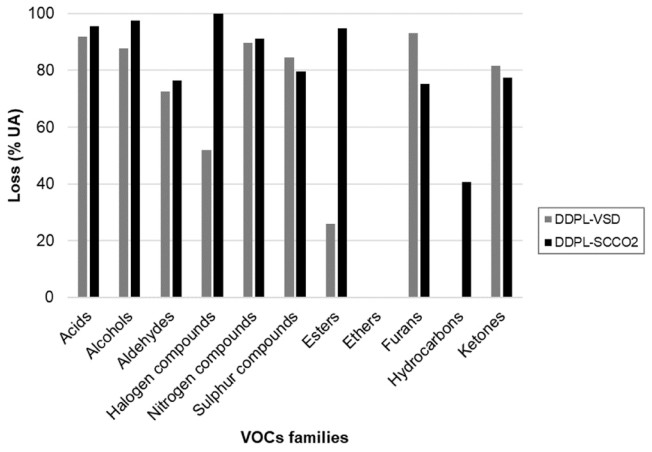

Fig. 4. Loss (% UA) of the main VOC families in dried pork liver (DPL) after vacuum steam distillation (DPL-VSD) and supercritical CO_2 (DPL-$SCCO_2$) deodorization.

compared to DPL: acids by 95.6% UA, alcohols by 97.5% UA, aldehydes by 76.4% UA, nitrogen compounds by 91.1% UA, sulphur compounds by 79.6% UA, esters by 94.9% UA, furans by 75.3% UA, hydrocarbons by 40.6% UA and ketones by 81.5% UA.

As for the reduction of sulphur compounds, Shimoda et al. [31] carried out the SC-CO_2 deodorization of fish sauce and observed a remarkable reduction in sulphur compounds, which could contribute to the attenuation of the unpleasant odour produced by these compounds. Specifically, dimethyl disulphide in fish sauce was reduced by 37% UA when using SC-CO_2, while in the present study it was reduced by 69.2% UA (from 2.95 to 0.91 UA) (Table 2).

As for the ketones, 2-butanone and 2-pentanone were efficiently separated, and only 17.2% and 27.8% UA were found after the SC-CO_2 deodorization, respectively (Table 2). A similar reduction was observed in the deodorization of fish sauce, with a UA retention of 12% and 40%, respectively, after the SC-CO_2 deodorization [31]. However, the UA in dimethyl ether, the only ether compound, increased fourfold compared to its value in DPL (from 2.55 to 8.14 UA) (Table 2), which could be linked to the chemical reactions taking place in the SC-CO_2 medium. Therefore, the high diffusivity of SC-CO_2 allowed its penetration through the liver matrix and the dissolution of extractable soluble VOCs. Also a small amount of compounds with low affinity for SC-CO_2 could be washed out of the deodorization vessel. When the mixture of VOCs and SC-CO_2 arrived to the separator tank, CO_2 turned to vapour and the VOCs where retained in the active carbon, allowing the CO_2 to be recirculated to continue the deodorization process.

Table 3
Proximate analysis of dried pork liver (DPL) and dried pork deodorized by vacuum steam distillation (DPL-VSD) supercritical CO_2 (DPL-$SCCO_2$).

Samples	Moisture (%)	Protein (%)	Fat (%)	Ash (%)
DPL	$2.77 \pm 0.18_{C}$	$73.18 \pm 0.07_{B}$	$19.33 \pm 0.10_{A}$	$2.69 \pm 0.21_{B}$
DPL-VSD	$45.00 \pm 0.31_{A}$	$35.79 \pm 1.01_{C}$	$17.15 \pm 0.64_{B}$	$2.29 \pm 0.4_{B}$
DPL-$SCCO_2$	$3.56 \pm 0.21_{B}$	$75.71 \pm 0.44_{A}$	$14.52 \pm 0.70_{C}$	$5.02 \pm 0.22_{A}$

%: chemical composition (moisture, protein, fat and ash) g / 100 g product. Average values ± LSD intervals are given.
Different capital letters show homogeneous groups established from LSD intervals ($p < 0.05$) for moisture, protein, fat and ash content.

3.3.2. Chemical composition

As in the VOC analysis, no significant ($P > 0.05$) effect was found for any of the factors (temperature, time and pressure) considered in the Box Behnken design. Therefore, an average value for all the SC-CO_2 treated samples was also considered and compared with DPL and DPL-VSD (Table 3).

The fat content of the samples treated with SC-CO_2 was significantly ($P < 0.05$) lower than in the untreated samples, decreasing from 19.33% in DPL to 14.52% in DPL-$SCCO_2$. SC-CO_2 has been used for fat removal in foods with a high protein content, as an alternative to other conventional techniques using undesirable organic solvents [17]. Thus, it has been used for the extraction of lipids in ground beef (39% lipids extracted, 172 bar and 50ºC) [47]. In bovine heart, the SC-CO_2 treatment (at 40 MPa and 40ºC) reduced the fat content from 154.22 to 9.87 g/kg [48]. In the case of lamb meat, SC-CO_2 (45ºC, 500 bar, 3 mL CO_2/min) allowed the initial fat content to be reduced by 87.4% [49]. Compared to these previous results, the fat content reduction in the DPL treated by SC-CO_2 was smaller (24.9%) than in the analyses mentioned above. This could be linked to the experimental conditions employed and probably to the flow rate and extraction time used in the present study, which were not optimum for fat removal purposes.

As a result of the fat content reduction in the DPL-$SCCO_2$, the content of the rest of the components was different from that in DPL and DPL-VSD (Table 3). Thus, the protein content increased from 73.18% in DPL to 75.71% in DPL-$SCCO_2$. However, in DPL-VSD, the protein content was significantly lower (35.79%), mainly due to the increase in moisture (45.0%), caused by water adsorption during VSD, in comparison to DPL (2.77%) and DPL- $SCCO_2$ (3.56%). VSD would require further drying in order to reduce its moisture content to values close to DPL. As for the ash content, there were no significant differences ($P < 0.05$) between DPL and DPL-VSD (2.69% in DPL and 2.29% in DPL-VSD); however, in DPL-$SCCO_2$, the ash content increased by 5.02%.

3.4. Comparison between vacuum steam distillation and supercritical CO_2 deodorization techniques

If the two deodorization techniques are compared, SC-CO_2 allowed for the complete removal of a greater number of VOCs than VSD, as illustrated in Table 2. In addition, some VOC families presented a greater % loss after SC-CO_2 deodorization than the sample deodorized by VSD: acids (3.6% UA), alcohols (9.8% UA), aldehydes (3.8% UA), halogenated compounds (48.2% UA), nitrogenous compounds (1.3% UA), esters (69.9% UA) and hydrocarbons (40.6% UA). Otherwise, VSD was more effective at reducing sulphur compounds, presenting a greater % loss than the sample deodorized by SC-CO_2 (4.9% UA), furans (17.7% UA) and ketones (4.1% UA) (Fig. 4). However, neither of the two techniques was able to reduce dimethyl ether, whose content increased after the deodorization processes compared to DPL (20% UA in DPL-VSD

and 219.2% UA in DPL- SCCO$_2$).

Both techniques, VSD and SC-CO$_2$, led to a relevant modification of the dried pork liver off-flavour (Table 2). SC-CO$_2$ treatment removed 3 characteristic compounds from the typical odour of pork liver, unlike VSD: 1-octen-3-ol, characteristic of the mushroom odour, 1-nonanol, a compound attributed to a fatty, green odour, and the (*E*,*E*)− 2,4-heptadienal with a fishy odour. However, after SC-CO$_2$ deodorization, benzaldehyde was formed, a compound that had been eliminated by drying. This compound is attributed to the almond odour. In this sense, the content of the almond-like odour characteristic of pentanal was 137.3% UA higher and that of 2-methylbutanal was 59.3% UA lower in SC-CO$_2$ deodorization, compared to deodorization by VSD. In relation to the cardboard odour, (E)− 2-nonenal content was 50% UA higher compared to DPL-VSD. Regarding the fatty odour, typical of hexanal and heptanal, its content after deodorization by SC-CO$_2$ was 174.9% UA and 45.6% UA, higher, respectively, compared to DPL-VSD; however, the propanal 2-methyl content was 61.5% UA lower. Finally, as regards the nature aroma (green odour), the VOC content of 1-hexanol, and 2-butenal-2-methyl-, was 30.8% UA and 62.2% UA lower, respectively using SC-CO$_2$, if compared to VSD. However, the butenal and hexanal content after SC-CO$_2$ deodorization was 25% and 174.9% UA higher, respectively, than in VSD. In this regard, high levels of hexanal can be associated with a rancid off-flavour, while low levels of hexanal have been associated with a green and nature aromatic note [50]. Therefore, after the application of SC-CO$_2$, the typical metallic and fishy odours [6,44] were reduced to metallic, leaving the pork liver without a fishy odour. The effect of VSD and SC-CO$_2$ treatment on the deodorization of meat products has not been previously addressed.

4. Conclusions

Deodorization by vacuum steam distillation (VSD) and supercritical CO$_2$ (SC-CO$_2$) have proven to be reliable techniques for the reduction and removal of VOCs in dried pork liver. The VSD showed more affinity to eliminate or reduce the content of sulphur compounds, furans and ketones, while SC-CO$_2$ showed a higher capacity for the elimination or reduction of acids, alcohols, aldehydes, halogenated compounds, nitrogenous compounds, esters and hydrocarbons. Thereby, it has been demonstrated that compounds responsible for the liver off-flavour, such as (*E*,*E*)- 2,4-heptadienal (fishy), 1-octen-3-ol (mushroom) and 1-nonanol (fatty and green), can be efficiently removed by SC-CO$_2$. Moreover, it should be noted that SC-CO$_2$ led to a remarkable reduction in fat and a slight reduction in the moisture content compared to dried pork liver. Thus, further studies should assess the cost of SC-CO$_2$ deodorization in order to both evaluate its implementation in the industrial recovery of pork liver proteins or in protein isolates as a strategy to mitigate undesirable flavours, as well as look at its potential application in other matrices.

CRediT authorship contribution statement

B. Abril: Conceptualization, Methodology, Investigation, Formal analysis, Writing – original draft, **J.M.Lorenzo:** Conceptualization, Methodology, Formal analysis, Writing – review & editing, **J.V. García-Pérez:** Conceptualization, Methodology, Formal analysis, Writing – review & editing, Supervision, Project administration, **M. Contreras:** Formal analysis, Writing – original draft, **J. Benedito:** Conceptualization, Methodology, Formal analysis, Writing – review & editing, Supervision, Project administration.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data Availability

Data will be made available on request.

Acknowledgements

The authors acknowledge the financial support from the “Ministerio de Economía y Competitividad (MINECO) and Instituto Nacional de Investigación y Tecnología Agraria y Alimentaria (INIA)” in Spain (Project RTA2017-00024-C04-03). The authors acknowledge the contribution of the slaughterhouse “Carnes de Teruel S.A.”, (D.O. Jamón de Teruel, Spain) for the supply of pork livers.

References

[1] P.N. Seong, K.M. Park, S.H. Cho, S.M. Kang, G.H. Kang, B.Y. Park, H. Van Ba, Characterization of edible pork by-products by means of yield and nutritional composition, Korean J. Food Sci. Anim. Resour. 34 (3) (2014) 297, https://doi.org/10.5851/kosfa.2014.34.3.297.

[2] P.N. Seong, K.M. Park, S.H. Cho, S.M. Kang, G.H. Kang, B.Y. Park, H. Van Ba, Values-added utilization of protein and hydrolysates from animal processing by-product livers: A review, Trends Food Sci. Technol. 110 (2021) 432–442, https://doi.org/10.1016/j.tifs.2021.02.033.

[3] S.A. Lynch, A.M. Mullen, E. O'Neill, L. Drummond, C. Álvarez, Opportunities and perspectives for utilisation of co-products in the meat industry, Meat Sci. 144 (2018) 62–73, https://doi.org/10.1016/j.meatsci.2018.06.019.

[4] M. López-Pedrouso, P. Borrajo, M. Pateiro, J.M. Lorenzo, D. Franco, Antioxidant activity and peptidomic analysis of porcine liver hydrolysates using alcalase, bromelain, flavourzyme and papain enzymes, Food Res. Int. 137 (2020), 109389, https://doi.org/10.1016/j.foodres.2020.109389.

[5] E.A. Sánchez-Torres, B. Abril, J. Benedito, J. Bon, J.V. García-Pérez, Water desorption isotherms of pork liver and thermodynamic properties, LWT 149 (2021), 111857, https://doi.org/10.1016/j.lwt.2021.111857.

[6] S. Im, T. Kurata, Characterization of off-flavors in porcine liver collected by SDE, Food Sci. Technol. Res. 9 (4) (2003) 338–341, https://doi.org/10.3136/fstr.9.338.

[7] T. Kimura, M. Kagaya, Y. Fukuya, S. Kosugi, Properties of chicken liver preserved in Miso, J. Home Econ. Jpn. 41 (7) (1990) 629–636, https://doi.org/10.11428/jhej1987.41.629.

[8] P. Kumar, M.K. Chatli, N. Mehta, P. Singh, O.P. Malav, A.K. Verma, Meat analogues: Health promising sustainable meat substitutes, Crit. Rev. Food Sci. Nutr. 57 (5) (2017) 923–932, https://doi.org/10.1080/10408398.2014.939739.

[9] S. Damodaran, A. Arora, Off-flavor precursors in soy protein isolate and novel strategies for their removal, Annu. Rev. Food Sci. Technol. 4 (1) (2013) 327–346, https://doi.org/10.1146/annurev-food-030212-182650.

[10] B. Wang, Q. Zhang, N. Zhang, K.H. Bak, O.P. Soladoye, R.E. Aluko, Y. Zhang, Insights into formation, detection and removal of the beany flavor in soybean protein, Trends Food Sci. Technol. 112 (2021) 336–347, https://doi.org/10.1016/j.tifs.2021.04.018.

[11] B. Guldiken, R. Green, M.T. Nickerson, The impact of different adsorbents on flavor characteristics of a lentil protein isolate, Eur. Food Res. Technol. 247 (3) (2021) 593–604, https://doi.org/10.1007/s00217-020-03648-z.

[12] M.C. Whetstine, A.E. Croissant, M.A. Drake, Characterization of dried whey protein concentrate and isolate flavor, J. Dairy Sci. 88 (11) (2005) 3826–3839, https://doi.org/10.3168/jds.S0022-0302(05)73068-X.

[13] C.T. Zehnder. Practical Handbook of Soybean Processing and Utilization. Chapter 14.Deodorization.(1995),239–257.https://doi.org/10.1016/B978–0-935315–63-9.50018–8.

[14] D.A. de Oliveira, M.G. Minozzo, S. Licodiedoff, N. Waszczynskyj, Physicochemical and sensory characterization of refined and deodorized tuna (Thunnus albacares) by-product oil obtained by enzymatic hydrolysis, Food Chem. 207 (2016) 187–194, https://doi.org/10.1016/j.foodchem.2016.03.069.

[15] Y. Li, W. Zhou, Y. Cao, X. Gong, J. Li, X. Lu, Y. Dai, Analysis of volatile components of Tilapia enzymolysis solution after different deodorization treatments, IOP Conf. Ser. Earth Environ. Sci. 571 (2020) 1, https://doi.org/10.1088/1755-1315/571/1/012121.

[16] L.V. Silva, D.L. Nelson, M.F.B. Drummond, L. Dufossé, M.B.A. Glória, Comparison of hydrodistillation methods for the deodorization of turmeric, Food Res. Int. 38 (8–9) (2005) 1087–1096, https://doi.org/10.1016/j.foodres.2005.02.025.

[17] A. Mouahid, C. Dufour, E. Badens, Supercritical CO$_2$ extraction from endemic Corsican plants; comparison of oil composition and extraction yield with hydrodistillation method, J. CO$_2$ Util. 20 (2017) 263–273, https://doi.org/10.1016/j.jcou.2017.06.003.

[18] E. Reverchon, I. De Marco, Supercritical fluid extraction and fractionation of natural matter, J. Supercrit. Fluids 38 (2) (2006) 146–166, https://doi.org/10.1016/j.supflu.2006.03.020.

[19] N. Vedaraman, C. Srinivasakannan, G. Brunner, B.V. Ramabrahmam, P.G. Rao, Experimental and modeling studies on extraction of cholesterol from cow brain using supercritical carbon dioxide, J. Supercrit. Fluids 34 (1) (2005) 27–34, https://doi.org/10.1016/j.supflu.2004.10.004.

[20] G. Vasapollo, L. Longo, L. Rescio, L. Ciurlia, Innovative supercritical CO$_2$ extraction of lycopene from tomato in the presence of vegetable oil as co-solvent, J. Supercrit. Fluids 29 (1–2) (2004) 87–96, https://doi.org/10.1016/S0896-8446(03)00039-1.

[21] G.L. Zabot, Decaffeination using supercritical carbon dioxide (pags), Green Sustain. Process Chem. Environ. Eng. Sci. (2020) 255–278, https://doi.org/10.1016/B978-0-12-817388-6.00011-8.
[22] S.C. Lee, D. Surendhiran, B.S. Chun, Extraction and encapsulation of squalene-rich cod liver oil using supercritical CO_2 process for enhanced oxidative stability, J. CO_2 Util. 62 (2022), 102104, https://doi.org/10.1016/j.jcou.2022.102104.
[23] S.W. Kang, H.M. Kim, M.S. Rahman, A.N. Kim, H.S. Yang, S.G. Choi, Nutritional quality and physicochemical characteristics of defatted bovine liver treated by supercritical carbon dioxide and organic solvent, Korean J. Food Sci. Anim. Resour. 37 (1) (2017) 29, https://doi.org/10.5851/kosfa.2017.37.1.29.
[24] B.J. Burri, T.R. Neidlinger, A.O. Lo, C. Kwan, M.R. Wong, Supercritical fluid extraction and reversed-phase liquid chromatography methods for vitamin A and beta-carotene Heterogeneous distribution of vitamin A in the liver, J. Chromatogr. A 762 (1997) 201–206.
[25] J.W. King, Modern supercritical fluid technology for food applications, Annu. Rev. Food Sci. Technol. 5 (2014) 215–238, https://doi.org/10.1146/annurev-food-030713-092447.
[26] F. Kobayashi, Y. Hayata, K. Kohara, N. Muto, M. Miyake, Y. Osajima, Application of supercritical CO_2 bubbling to deodorizing of drinking water, Food Sci. Technol. Res. 12 (2) (2006) 119–124, https://doi.org/10.3136/fstr.12.119.
[27] D. Panadare, G. Dialani, V. Rathod, Extraction of volatile and non-volatile components from custard apple seed powder using supercritical CO_2 extraction system and its inventory analysis, Process Biochem. 100 (2021) 224–230, https://doi.org/10.1016/j.procbio.2020.09.030.
[28] E. Tejedor-Calvo, S. García-Barreda, S. Sánchez, D. Morales, C. Soler-Rivas, A. Ruiz-Rodriguez, P. Marco, Supercritical CO_2 extraction method of aromatic compounds from truffles, LWT 150 (2021), 111954, https://doi.org/10.1016/j.lwt.2021.111954.
[29] M. Oszagyan, B. Simandi, J. Sawinsky, A. Kery, E. Lemberkovics, J. Fekete, Supercritical fluid extraction of volatile compounds from lavandin and thyme, Flavour Fragr. J. 11 (3) (1996) 157–165, https://doi.org/10.1002/(SICI)1099-1026(199605)11:3<157::AID-FFJ559>3.0.CO;2-6.
[30] P. Maheshwari, E.T. Ooi, Z.L. Nikolov, Off-flavor removal from soy-protein isolate by using liquid and supercritical carbon dioxide, J. Am. Oil Chem. Soc. 72 (10) (1995) 1107–1115, https://doi.org/10.1007/BF02540975.
[31] M. Shimoda, Y. Yamamoto, J. Cocunubo-Castellanos, T. Yoshimura, M. Miyake, H. Ishikawa, Y. Osajima, Deodorization of fish sauce by continuous-flow extraction with microbubbles of supercritical carbon dioxide, J. Food Sci. 65 (2000) 1349–1351, https://doi.org/10.1111/j.1365-2621.2000.tb10610.x.
[32] AOAC, G. (2016). Official methods of analysis of AOAC International. Rockville, MD: AOAC International, ISBN: 978–0-935584–87-5.
[33] W.K. Pui, R. Yusoff, M.K. Aroua, A review on activated carbon adsorption for volatile organic compounds (VOCs, Rev. Chem. Eng. 35 (5) (2019) 649–668, https://doi.org/10.1515/revce-2017-0057.
[34] R. Domínguez, L. Purriños, C. Pérez-Santaescolástica, M. Pateiro, F.J. Barba, I. Tomasevic, J.M. Lorenzo, Characterization of volatile compounds of dry-cured meat products using HS-SPME-GC/MS technique, Food Anal. Methods 12 (6) (2019) 1263–1284, https://doi.org/10.1007/s12161-019-01491-x.
[35] C. Pugliese, F. Sirtori, M. Škrlep, E. Piasentier, L. Calamai, O. Franci, M. Čandek-Potokar, The effect of ripening time on the chemical, textural, volatile and sensorial traits of Bicep femoris and Semimembranosus muscles of the Slovenian dry-cured ham, Meat Sci. 100 (2015) 58–68, https://doi.org/10.1016/j.meatsci.2014.09.012.
[36] T.C. Merlo, J.M. Lorenzo, E. Saldaña, I. Patinho, A.C. Oliveira, B.S. Menegali, C. J. Contreras-Castillo, Relationship between volatile organic compounds, free amino acids, and sensory profile of smoked bacon, Meat Sci. 181 (2021), 108596, https://doi.org/10.1016/j.meatsci.2021.108596.
[37] G. Johansson, J.L. Berdagué, M. Larsson, N. Tran, E. Borch, Lipolysis, proteolysis and formation of volatile components during ripening of a fermented sausage with Pediococcus pentosaceus and Staphylococcus xylosus as starter cultures, Meat Sci. 38 (2) (1994) 203–218, https://doi.org/10.1016/0309-1740(94)90110-4.
[38] L. Sun, G. Xin, Z. Hou, X. Zhao, H. Xu, X. Bao, L. Li, Biosynthetic mechanism of key volatile biomarkers of harvested lentinula edodes triggered by spore release, J. Agric. Food Chem. 69 (32) (2021) 9350–9361, https://doi.org/10.1021/acs.jafc.1c02410.
[39] C.K. Wei, Z.J. Ni, K. Thakur, A.M. Liao, J.H. Huang, Z.J. Wei, Aromatic effects of immobilized enzymatic oxidation of chicken fat on flaxseed (Linum usitatissimum L.) derived Maillard reaction products, Food Chem. 306 (2020), 125560, https://doi.org/10.1016/j.foodchem.2019.125560.
[40] A. Casaburi, P. Piombino, G.J. Nychas, F. Villani, D. Ercolini, Bacterial populations and the volatilome associated to meat spoilage, Food Microbiol. 45 (2015) 83–102, https://doi.org/10.1016/j.fm.2014.02.002.
[41] M. Cardinal, M. Chaussy, C. Donnay-Moreno, J. Cornet, C. Rannou, C. Fillonneau, P. Courcoux, Use of random forest methodology to link aroma profiles to volatile compounds: application to enzymatic hydrolysis of Atlantic salmon (Salmo salar) by-products combined with Maillard reactions, Food Res. Int. 134 (2020), 109254, https://doi.org/10.1016/j.foodres.2020.109254.
[42] J. Liu, M. Liu, C. He, H. Song, F. Chen, Effect of thermal treatment on the flavor generation from Maillard reaction of xylose and chicken peptide, LWT-Food Sci. Technol. 64 (1) (2015) 316–325, https://doi.org/10.1016/j.lwt.2015.05.061.
[43] X. Li, J. Zhu, C. Li, H. Ye, Z. Wang, X. Wu, B. Xu, Evolution of volatile compounds and spoilage bacteria in smoked bacon during refrigeration using an E-Nose and GC-MS combined with partial least squares regression, Molecules 23 (12) (2018) 3286, https://doi.org/10.3390/molecules23123286.
[44] S. Im, F. Hayakawa, T. Kurata, Identification and sensory evaluation of volatile compounds in oxidized porcine liver, J. Agric. Food Chem. 52 (2) (2004) 300–305, https://doi.org/10.1021/jf030337v.
[45] M. Narváez-Rivas, E. Gallardo, & M. León-Camacho. Analysis of volatile compounds from Iberian hams: a review.Grasas y aceites,63(4),(2012),432–454.
[46] G. Song, M. Zhang, X. Peng, X. Yu, Z. Dai, Q. Shen, Effect of deodorization method on the chemical and nutritional properties of fish oil during refining, LWT 96 (2018) 560–567, https://doi.org/10.1016/j.lwt.2018.06.004.
[47] R.R. Chao, S.J. Mulvaney, M.E. Bailey, L.N. Fernando, Supercritical CO_2 conditions affecting extraction of lipid and cholesterol from ground beef, J. Food Sci. 56 (1) (1991) 183–187, https://doi.org/10.1111/j.1365-2621.1991.tb08007.x.
[48] M.S. Rahman, K. Gul, H.S. Yang, J. Chun, W.L. Kerr, S.G. Choi, Thermal and functional characteristics of defatted bovine heart using supercritical CO_2 and organic solvent, J. Sci. Food Agric. 99 (2) (2019) 816–823, https://doi.org/10.1002/jsfa.9250.
[49] H. Taher, S. Al-Zuhair, A. AlMarzouqui, I. Hashim, I, Extracted fat from lamb meat by supercritical CO_2 as feedstock for biodiesel production, Biochem. Eng. J. 55 (1) (2011) 23–31, https://doi.org/10.1016/j.bej.2011.03.003.
[50] M. Flores, & A. Olivares. Flavor. Handbook of fermented meat and poultry, (2014),217–225.https://doi.org/10.1002/9781118522653.ch25.