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Production, chemical characterization, and sensory profile of a novel spirit elaborated from spent coffee ground

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ABSTRACT

This study describes a process for the production of a spirit from spent coffee ground (SCG), the chemical composition, and sensory profile of this distillate. The process consisted in three steps starting with the extraction of aroma compounds by hydrothermal treatment of SCG, followed by the fermentation of this extract supplemented with sucrose to ethanol, and the fermented broth distillation. Seventeen volatile compounds were identified in the distillate (including alcohols, esters, aldehydes, and acids), all of them in concentrations able to promote pleasant characteristics to the product. Based on the chemical composition, SCG spirit was considered as having organoleptic quality acceptable for human consumption. Twelve descriptors defined the sensory characteristics of this product, including clarity and brilliance (visual analysis), coffee, roasted, alcohol, elegance and frankly (olfactory analysis), bitter, astringent and pungent (gustatory analysis), and finesse (olfactory and gustatory analyses). Coffee was the most representative aroma by olfactory analysis. Based on the sensory analysis, SCG spirit was considered as having features of a pleasant beverage, with smell and taste of coffee.

1. Introduction

Spirits are alcoholic beverages obtained by distillation of a fermented broth usually produced from grains or fruits. Traditional examples of spirits commercially found in the market include the Scottish “whisky” (Jack & Steele, 2002), Brazilian “cachaça” (Oliveira, Cardello, Jerônimo, Souza, & Serra, 2005), Italian “grappa” (Bovo et al., 2009), French “eau-de-vie de marc” (Hang & Woodams, 2010), Spanish “orujo” (Diéguez, de la Peña, & Gómez, 2005), Portuguese “bagaceira” (Silva, Malcata, & de Revel, 1996), Greek “tsipouro” (Apostolopoulou, Flouros, Demertzis, & Akrida-Demertzi, 2005), Cuban “rum” (Pino, Tolle, Gök, & Winterhalter, 2012), and the Cypriote “zivania” (Ballabio, Kokkinofta, Todeschini, & Theocharis, 2007).

During the last years, the industry of distillates has demonstrated large interest in producing new products from unusual raw materials that enable acquisition of different flavors, attracting new markets. Based on this interest, several researches have been focused in finding potential raw materials for the production of these alcoholic beverages. Agricultural residues derived from industrial activities are potential raw materials for this purpose due to their low cost, characteristic aroma, and presence of sugars that can be converted to ethanol. Recently, new spirits have been produced from the by-product of jabuticaba fermentation (skin and pulp) (Asquieri, Silva, & Cândido, 2009), and from the residues obtained during the production of concentrated orange juice (Roçafa Junior, Padovan, & Faria, 2005). Spent coffee ground (SCG), the solid residue obtained during the processing of coffee powder with hot water or steam to prepare instant coffee, is a coffee industry residue generated in large amounts but practically unused (Mussatto, Carneiro, Silva, Roberto, & Teixeira, 2011). This residue presents a remarkable residual aroma of roasted coffee beans, being an interesting feedstock for the production of a new spirit. Alcoholic beverages produced from coffee beans can be found in the market, but a spirit produced from SCG can be considered as a novel beverage.

Distilled alcoholic beverages are characterized by the presence of a variety of volatile compounds that arise during fermentation, distillation and storage processes. The composition and concentration levels of these compounds may vary widely to each beverage. While some of them appear in high concentrations (hundreds of mg/L), a large part may appear at significantly lower levels (even as low as ng/L) (Plutowska & Wardencki, 2008), but all of them significantly contribute to the quality of the final product.

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Therefore, the identification of these compounds is of large importance to determine the flavor characteristics of the drink, being also useful to detect illicit spirits, and to identify anomalies that are indicative of inconsistent manufacturing practices (Fitzgerald, James, MacNamara, & Stack, 2000). The sensory attributes are also one of the most important aspects to be evaluated when elaborating a new product, since they reveal the global character of the product and give a perspective of its acceptability in the market. Sensory descriptive analysis methods have been used to characterize spirits and wines (Dragone, Mussatto, Oliveira, & Teixeira, 2009; Vilanova, Zamuz, Tardáguila, & Masa, 2008). Quantitative Descriptive Analysis (QDA) is the most used method in sensory analysis for product characterization. This method allows identifying and quantifying attributes in the products by persons with training and experience. The qualitative component comprises the descriptive terms, attributes, which define the sensory profile of the product (Carlucci & Monteleone, 2001), while the quantitative component measures the intensity and frequency of each descriptor perceived (Meilgaard, Civille, & Carr, 1999). By using this method, it is possible to identify which sensory attributes are important for the product acceptance (Lawless & Heymann, 1998).

The present study describes a process for the elaboration of a spirit from SCG. The produced distillate was submitted to a chemical characterization to identify the volatile compounds present, as well as to determine the flavor characteristics. A quantitative descriptive analysis was also used to evaluate the sensory profile, and the most important aroma descriptors contributing to the sensory quality of this novel spirit were identified.

2. Materials and methods

2.1. Raw material

Spent coffee ground (SCG) was supplied by NovaDelta – Comércio e Indústria de Cafés, Lda (Campo Maior, Portugal). As soon as obtained, the material was dried at 60 °C to a moisture content of 10 g/100 g dry matter, to be stored. Chemical composition of SCG, which was determined according to Mussatto et al. (2011), consisted of (g/100 g): glucan (8.6), arabinan (1.7), galactan (13.8), mannan (21.2), protein (13.6), lignin (32.1), ashes (1.6), acetyl groups (2.2), and extractives (5.2).

2.2. Hydrothermal process and fermentation medium

A hydrothermal process was initially performed aiming to extract aroma compounds from SCG. Solid-liquid extraction conditions consisted in using water and SCG in the ratio of 1 g/10 mL, at 163 °C during 45 min. At the end of the reaction, the residual solid material was separated by centrifugation (5000 g, 15 min) and the liquid fraction (SCG extract) was stored at 4 °C. To be used as fermentation medium, SCG extract was supplemented with 180 g/L sucrose and 175 mg/L potassium metabisulfite. Then, the pH of the fermentation medium was adjusted to 5 by addition of calcium carbonate, and the remaining solid residue was removed by centrifugation (5000 *g*, 15 min).

2.3. Microorganism and inoculum

Saccharomyces cerevisiae (RL-11), supplied by University of Lavras (Department of Biology, Brazil) was the yeast strain used in the experiments. This yeast was selected among other strains due to its high capacity of producing ethanol (Dias, Schwan, & Lima, 2003). Cultures of this yeast were maintained at 4 °C on malt extract agar medium whose composition consisted in (g/L): yeast extract (3.0), malt extract (3.0), peptone (5.0), glucose (10.0), and agar (20.0).

For the inoculum preparation, cells of the yeast grown for 24 h in the maintenance medium were transferred to 500-mL Erlenmeyer flasks containing 200 mL of culture medium composed by (g/L): glucose (30.0), $(NH_4)_2HPO_4$ (3.0), $MgSO_4 \times 7H_2O$ (1.0), and yeast extract (3.0). Concentrated solutions of each compound were prepared separately and sterilized in an autoclave at 121 °C for 20 min, with exception of the glucose and yeast extract that were autoclaved at 112 °C for 15 min. The solutions were mixed aseptically in order to obtain the desired concentration of each nutrient in the culture medium. The inoculated flasks were incubated in a rotary shaker (Certomat H, B. Braun Biotech International, Melsungen, Germany) at 30 °C, 200 rpm, for 24 h. After this time, the cells were recovered by centrifugation (4000 *g*, 20 min) and resuspended in the fermentation medium.

2.4. Fermentation and distillation conditions

Fermentation assays were performed in a 6.5-L bioreactor (B. Braun Biotech International, Melsungen, Germany) containing 3.5 L of fermentation medium (180 g/L sucrose) inoculated with an initial cell concentration of 1 g/L. Fermentations were maintained at 30 °C and 150 rpm. Samples were periodically withdrawn to determine the total sugars concentration in order to determine the fermentation end. At the end of the fermentation, the fermented broth was centrifuged (5000 *g*, 10 °C, 15 min) to separate the biomass, and the liquid phase was stored at 4 °C for further distillation. The fermentation runs were performed in triplicate.

The free-biomass fermented broth was distilled in a system comprising a vigreux column of 36 cm of length, a condenser, and a 4 L flask filled with 2–2.5 L of fermented broth. During the fermented broth distillation, samples of approximately 20 mL were recovered and the ethanol content in each one of them was determined. Then, the samples were blended in order to obtain three fractions according to their ethanol content: the foreshot or "head" (>70 mL/100 mL), the middle cut or "heart" (70–40 mL/100 mL), and the feints or "tail" (<40 mL/100 mL). The fraction corresponding to the heart had the ethanol concentration corrected to 40 mL/100 mL by adding ultrapure water, and was stored in glass bottles with caps and plastic coverings at room temperature for later sampling, chemical and sensory analyses.

2.5. Analytical methods

The cell concentration was determined by dry weight per vol (g/L). Samples of the fermented broth were filtrated by suction through pre-weighed 0.45 μm membranes, washed with distilled water, and dried at 105 °C to constant weight. Total sugar content was determined by the anthrone method (Dreywood, 1946). Ethanol concentration was determined by high-performance liquid chromatography (HPLC) on a Jasco chromatograph (Jasco, Tokio, Japan) equipped with a refractive index detector and a Metacarb 67H (300 × 6.5 mm) column at 60 °C, using 0.005 mol/L sulfuric acid as eluent in a flow rate of 0.7 mL/min. The alcohol content (mL/100 mL) was calculated by the ratio between product formed (ethanol, g/L) and the density of ethanol (0.789 g/mL).

Major volatile compounds were analyzed in a Chrompack CP-9000 gas chromatograph (Chrompack, Middelburg, The Netherlands) equipped with a split/splitless injector and a flame ionization detector (FID). A capillary column coated with CP-Wax 57 CB (50 m × 0.25 mm i.d., 0.2 μm film thickness; Chrompack) was used. The temperature of the injector and the detector were both set to 250 °C. The oven temperature was held at 60 °C for

5 min, then programmed to rise from 60 °C to 220 °C at 3 °C/min, and finally held at 220 °C for 10 min. Helium at 103 kPa was used as carrier gas, and the split vent was set to 13 mL/min. 4-nonanol was used as internal standard. The quantification of the volatile compounds as 4-nonanol equivalents was performed by comparing the retention indices with those of pure standard compounds (purchased from both, Sigma–Aldrich, St. Louis, MO, USA, and Merck, Darmstadt, Germany).

Minor volatile constituents were analyzed in a GC–MS Varian Saturn 2000 gas chromatograph (Varian Inc., Walnut Creek, CA, USA) equipped with an ion-trap mass detector (IT-MS) (electronic impact mode of 70 eV; acquisition range from m/z 35 to m/z 260). A capillary column coated with VF-Wax ms (30 m × 0.15 mm i.d., 0.15 μm film thickness) was used. The temperature of the injector (SPI – septum-equipped programmable temperature) and the detector were both set to 250 °C. The oven temperature was held at 60 °C for 2 min, then programmed to rise from 60 °C to 234 °C at 3 °C/min, then programmed to rise from 234 °C to 250 °C at 10 °C/min, and finally maintained at 250 °C for 10 min. Helium at 103 kPa was used as carrier gas, and the split vent was set to 13 mL/min. 4-nonanol was used as internal standard. The quantification of the volatile compounds as 4-nonanol equivalents was performed by comparing the retention indices with those of pure standard compounds.

All the analytical determinations were carried out in triplicate.

2.6. Sensory analysis

The sensory analysis was developed by eight trained panelists from Apellation Orujo de Galicia (Galicia, Spain), six male and two female with ages between 35 and 55 years old. All the judges were experienced spirit tasters and have previously participated in similar studies. The spirit evaluation by sensory analysis was performed by QDA methodology (Lawless & Heymann, 1998), and sensory descriptors in visual, olfactory and gustatory phases were generated. To create an adequate environment for the identification of descriptors, the sensory analysis was performed in a professional-standard room, in accordance to ISO Norm 8589 (1988).

A constant sample volume of 30 mL was evaluated in spirit-taster glasses at 12 °C. During the analysis, the judges smelled and tasted the sample and indicated if the different descriptors were perceived. The intensity of each sensory attribute was rated on a 10-point scale, where 0 indicated that the descriptor was not perceived, and 9 indicated a very high intensity. The descriptors were classified by using the comparison of Geometric Means (GM), according to the International Organization for Standardization (ISO Norm 11035, 1994). GM was calculated as $GM^2 = F \times I$, where F or frequency, is the number of times that the descriptor was mentioned, divided by the total number of times that the descriptor could be mentioned, expressed as a percentage; and I or intensity, is the sum of the intensities given by the whole panel for a descriptor divided by the maximum possible intensity for this descriptor, expressed as a percentage.

GM values were used to classify the descriptors, and the classification of them made it possible to eliminate those whose geometric means were relatively low. This method allowed taking into account descriptors which were rarely mentioned but which were very important in terms of the perceived intensity, and descriptors with a low perceived intensity but which are mentioned often. Sensory data of the descriptive analysis were analyzed using XLstat-Pro (Addinsoft, New York, EEUU). GM data were statistically analyzed by using multivariate techniques.

Pearson correlations among all sensory attributes were calculated. Then, Principal Component Analysis (PCA) on sensory descriptors of the spirit and panelists was applied. PCA is used as a tool for screening, extracting and compressing data. PCA employs a mathematical procedure that transforms a set of possibly correlated response variables into new set of no correlated variables called principal components.

To show the relationship between sensory variables with GM > 10% and volatile compounds with odor activity value (OAV, as the compound concentration/perception threshold) > 0.2, Partial Least Squares Regression (PLS1) was applied. PLS1 shows the relationship between X data (volatile compounds) and Y data (sensory descriptor). The X data are actively used in estimating the latent variables to ensure that the first components are those that are most relevant for predicting the Y variable. This is a data reduction technique since it reduces the X variables to a set of no correlated factors that describe the variation in the data.

3. Results and discussion

3.1. Spirit production

SCG was initially submitted to a hydrothermal process aiming to extract aroma compounds. The process conditions used at this stage were based on a previous study that selected the best acid hydrolysis conditions for hemicellulose sugars extraction from SCG (Mussatto et al., 2011). However, since it was desired the use of the extract as fermentation medium for the production of a beverage, the use of the acid catalyst was omitted, being maintained the other hydrolysis conditions previously established. By performing this hydrothermal process, an extract containing only 3.4 g/L total reducing sugars was obtained. This value is about 10-fold lower than that obtained by acid hydrolysis of SCG (≅35 g/L). This lower sugar recovery was expected since the hydrolysis conditions have been optimized considering the use of H_2SO_4 as catalyst, and such reactive presented strong influence on the sugars recovery from the raw material structure (Mussatto et al., 2011). Nevertheless, the SCG extract obtained by hydrothermal treatment presented a remarkable and pleasant aroma of coffee, very attractive for the spirit production.

Additional efforts were not made in order to find a hydrolysis condition able to maximize the sugars extraction from SCG because even if 35 g sugars/L were recovered, this would be a low sugar concentration for use as fermentation medium. Consequently, a subsequent step for concentration of the extract previous its use as fermentation medium would be required to attain higher initial sugar concentration, and such step would increase the spirit production costs. Thus, the fermentation medium was formulated with the aromatic SCG extract supplemented with sucrose. The kinetic behavior of sucrose consumption, ethanol production and cell growth of *S. cerevisiae* RL-11 cultivated in this medium is shown in Fig. 1. As can be seen, the yeast was able to growth and produce ethanol from this extract with elevated yield (0.42 g ethanol/g sucrose) and productivity (0.77 g/L.h), as it was expected since this yeast strain is reported to have great ability to convert sugars to ethanol, being recommended for the production of alcoholic beverages (Dias et al., 2003). These results also revealed that the compounds extracted during the hydrothermal process of SCG were not able to negatively affect the yeast bioconversion performance.

At the end of fermentation, a fermented broth containing an ethanol concentration of 9.0 mL/100 mL was obtained. This ethanol concentration can be well compared to other values obtained by using this yeast strain for the production of alcoholic beverages from yellow mombin (*Spondias mombin*) and from pejibaye or peach palm (*Bactris gasipaes* Kunth), which yielded ethanol concentration of 12 and 9.4 mL/100 mL, respectively, from 240 g/L total

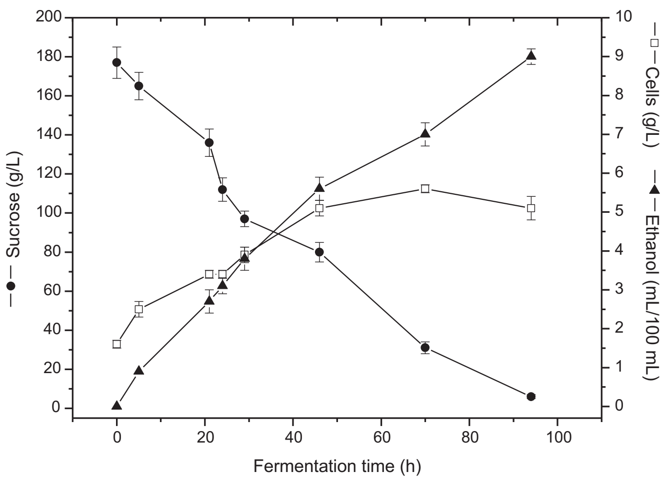

Fig. 1. Fermentation profile of ethanol production, sucrose consumption and cell growth of *Saccharomyces cerevisiae* (RL-11) from spent coffee ground extract. Data correspond to mean values (±SD) for three independent experiments.

Table 1
Volatile compounds present in spent coffee ground (SCG) spirit.[a]

Major volatile compounds		Concentration (mg/L)
Aldehydes	Acetaldehyde	80 ± 24
Esters	Ethyl acetate	38 ± 9
Alcohols	1-Propanol	44 ± 5
	2-Methyl-1-propanol (isobutyl alcohol)	269 ± 64
	2-Methyl-1-butanol (isopentyl alcohol)	185 ± 47
	3-Methyl-1-butanol (isoamyl alcohol)	810 ± 201
	2-Phenylethanol	8 ± 3
	Methanol	11 ± 3
Minor volatile compounds		Concentration (μg/L)
Esters	Ethyl butanoate (ethyl butyrate)	74 ± 3
	3-Methylbutyl acetate (isoamyl acetate)	188 ± 17
	Ethyl hexanoate (ethyl caproate)	337 ± 37
	Ethyl octanoate (ethyl caprilate)	842 ± 83
	Phenylethyl acetate	130 ± 13
Alcohols	1-Hexanol	465 ± 34
Acids	Hexanoic acid (caproic acid)	947 ± 53
	Octanoic acid (caprylic acid)	4369 ± 572
	Dodecanoic acid (lauric acid)	930 ± 102

[a] Data correspond to mean values (±standard deviation) for three independent experiments, which were also analyzed in triplicate.

sugars (Dias et al., 2003; Oliveira et al., 2001). The efficient conversion of sugars to ethanol by the yeast is advantageous for the process since the greater the ethanol content in the fermented broth, the greater the volume of spirit that can be obtained, and the lower the costs of distillation.

3.2. Volatile compounds in SCG spirit

Distilled beverages are usually composed by a number of volatile compounds that are largely responsible for their aroma and flavor. Major volatile compounds are usually formed during the fermentation process, and their formation is influenced by the cultivation conditions. On the other hand, minor volatile components are mainly derived from the raw material used. All these compounds are of great importance for the drink, and must be present in appropriate concentrations to provide pleasant flavor and aroma to the distillate; otherwise, when present in high concentrations, many of them provide an unpleasant taste to the final product.

SCG spirit obtained by distillation of the fermented broth and subsequent correction to an ethanol concentration of 40 mL/100 mL was subjected to a chemical characterization to determine the volatile compounds present. Seventeen volatile compounds were identified in this spirit, alcohols and esters being the most abundant among the major and minor volatile compounds, respectively (Table 1). The higher alcohols 3-methyl-1-butanol (isoamyl alcohol), 2-methyl-1-propanol (isobutyl alcohol), and 2-methyl-1-butanol (isopentyl alcohol) were the major volatile components found in the highest concentrations, being thus responsible for the greater proportion of the aroma of the distillate. These same higher alcohols are also reported to be the most abundant volatile compounds in many other spirits, including orujo (Cortés, Gil, & Fernández, 2005) and cheese whey spirit (Dragone et al., 2009). According to Rapp and Versini (1995) higher alcohols positively contribute to the aroma of the beverage when present in concentrations higher than 300 mg/L, as observed in SCG spirit. Additionally, the relations between the alcohols 3-methyl-1-butanol/2-methyl-1-propanol, and 2-methyl-1-propanol/1-propanol, are considered indicatives of the quality of the drink, and must be greater than one unit (Rodríguez Mendiola & de la Serra Torroba, 2000). Considering this aspect, the distilled beverage produced from SCG may be considered as having organoleptic quality acceptable for human consumption.

Following the higher alcohols, acetaldehyde was the major volatile compound present in the highest concentration in SCG distillate (80 mg/L). The concentration of this compound in the distillate was higher than the value reported for cheese whey spirit (36.7 mg/L) (Dragone et al., 2009), but was much less than the amount found in bagaceiras (600 mg/L) (Silva et al., 1996), and orujo (262 mg/L) (Cortés et al., 2005), for example. The concentration of acetaldehyde in SCG spirit is in the range reported to promote pleasant characteristics, such as aroma of walnuts, sherry and ripe apples. Concentrations of this compound higher than 125 mg/L adversely affect the organoleptic property of the beverage (Geroyiannaki et al., 2007). Ethyl acetate was also present in SCG spirit in a concentration level considered to positively contribute to the final aroma of the beverages (between 50 and 80 mg/L) (Steger & Lambrechts, 2000). Low concentrations of this compound confers aroma of fruit to the drink (Sánchez-Palomo, Díaz-Maroto, González Viñaz, Soriano-Pérez, & Pérez-Coello, 2007), while concentration values above 150 mg/L provide features of deterioration to the beverage (Apostolopoulou et al., 2005).

Other major volatile compounds, including 1-propanol, 2-phenylethanol, and methanol were also identified in SCG distillate but at low concentration values. Low concentrations of 1-propanol promote a pleasant sweet odor, but very high concentrations of this compound exhale an odor of "solvent" that does not allow to detect the positive odors of the distillate (Fundira, Blom, Pretorius, & van Rensburg, 2002). Additionally, high concentrations of 1-propanol can be an indicator of possible microbial spoilage during the fermentation or storage of the fermented broth before distillation (Apostolopoulou et al., 2005). The concentration of this compound in SCG distillate (44 mg/L) was unable to negatively affect the odor of the distillate, and demonstrates that the fermentation and storage of the fermented broth were appropriately performed.

2-phenylethanol was the major volatile compound present in the lowest concentration in SCG spirit (8 mg/L), but its concentration was close similar to values found in other distillates, such as in spirits produced from cheese whey (7.8 mg/L) (Dragone et al., 2009) or grape bagasse (10 mg/L) (Silva et al., 1996). Low concentrations of this compound provide sweet and roses aroma to the distillate (Falqué, Fernandez, &Dudourdieu, 2001). Methanol was also found in the composition of SCG spirit, but in a very low concentration (11 mg/L), which is a positive aspect due to the toxicity of this compound. Many distillates contain this compound at low concentration values. Orujo, for example, contains 704 mg/L methanol

(Cortés et al., 2005). Methanol can be harmful to the human health when present in high concentrations (>4000 mg/L).

Table 1 also summarizes the minor volatile compounds identified in SCG spirit and their concentrations. Although these compounds are found in low concentration in distilled beverages, they are of great importance to their aroma. In fact, compounds appearing in trace quantities in alcoholic beverages, quite frequently have a greater influence on their sensory properties than those compounds that appear in high concentrations (Plutowska & Wardencki, 2008).

Among the minor volatile compounds identified in SCG spirit, esters were the most abundant. These compounds contribute with a pleasant fruity flavor and floral aroma to the drink (Escudero et al., 2004). 1-Hexanol is an alcohol that has a positive influence in the aroma when in concentrations up to 20 mg/L. But increased concentrations contribute negatively to the product aroma with characteristics of “coconut-like”, “harsh” and “pungent” (Falqué et al., 2001). The concentration of this alcohol in SCG distillate was low and can be considered to positively affect the flavor of the product. Hexanoic, octanoic, and dodecanoic acids were also present in SCG distillate, but these acids are reported to have low flavor effect in the distillates (Soufleros, Mygdalia, & Natskoulis, 2004).

3.3. Sensory analysis

Sensory evaluation of the SCG spirit was carried out by a panel of trained judges to establish visual, olfactory, and gustatory descriptors. Table 2 shows the sensory descriptors identified in SCG spirit and their correspondent means of frequency (F) and intensity (I) obtained by the tasting panels. SCG spirit was characterized with 21 sensory descriptors, two in visual analysis, eleven in the olfactory analysis and eight in gustatory analysis. In the visual analysis, the two descriptors (clarity and brilliance) showed high intensity (86%) and frequency (100%). The highest intensity for olfactory analysis was found for coffee descriptor (61%), and the highest frequency (100%) was for coffee, finesse, elegance and frankly. Pungent and bitter showed the highest values for intensity (61% and 57%, respectively) and frequency (88%) for gustatory analysis. Such characteristics, mainly pungent, are typical of newly distilled spirits, and can be improved with the aging of the distillate (Clyne, Conner, Paterson, & Piggott, 1993; Monica Lee, Paterson, & Piggott, 2001).

The geometric mean (GM, Table 2) was obtained through the values of intensity and frequency of each attribute. The two descriptors that had the highest geometric mean were the visual descriptors. Coffee, alcohol, finesse, elegance and frankly showed geometric means greater than 65% for olfactory analysis. Pungent and bitter were the descriptors with the highest geometric mean for the gustatory analysis. Descriptors with geometric mean greater than 50% were considered the descriptors with the highest contribution in this study. Thus, twelve descriptors (with GM>50%) defined the sensory characteristics of SCG spirit, including clarity and brilliance (visual analysis), coffee, roasted, alcohol, elegance and frankly (olfactory analysis), bitter, astringent and pungent (gustatory analysis), and finesse (olfactory and gustatory analyses). Roasted and alcohol were also found as sensory descriptors in different Galician orujo spirits (Cortes, Fernández, Otero, & Salgado, 2009; Diéguez et al., 2005).

Pearson correlations (pair-wise) were then calculated from the GM values, and the significant correlations among the sensory descriptors of the SCG spirit are shown in Table 3. Note in this table that among the descriptors, several of them were correlated with high r value ($r > 0.8$), and particularly, correlation between nuts and tobacco showed the highest positive value ($r = 1$, or 100%). In visual phase, clarity and brilliance are descriptors very close and therefore they showed a very high correlation ($r = 0.910$). Similar situation was observed for finesse and elegance in olfactory phase ($r = 0.944$). Astringency and bitter are two descriptors with the same origin; the flavonol polymers (Lesschaeve & Noble, 2005) and their sensation are also very close, which explains the very high correlation between both ($r = 0.901$). Finally, good correlation was shown between toasted (olfactory phase) and bitter (gustatory phase) ($r = 0.880$).

Principal Component Analysis (PCA) was applied for evaluation of spirit descriptors by each one panelist (Fig. 2). The PCA was performed with the values of the descriptors with GM > 50% and the eight panelists that carry out the sensory analysis. The first two principal components, F1 and F2 accounted for 72.86% of total variance (42.87 and 29.99%, respectively). In the positive side of the first component (F1), two panelists selected clarity and brilliance in visual phase and coffee aroma as the most important descriptors in the spirit. Another one panelist selected elegance and frankly aroma and finesse in olfactory and gustatory phases in the negative side on F1. Two panelists sited in the positive side of F2 selected astringency, bitter, and pungent in gustatory phase and alcohol and roasted in olfactory phase, as the most important descriptors in spirit. The last three panelists, sited in the negative side of the F2, did not select any descriptor as important in spirit coffee.

Studies performed by Prescott (1998) revealed few cross-cultural differences in the perceptions of the panels, implicating

Table 2
Intensity (I), frequency (F), and geometric mean (GM) to each descriptor of spent coffee ground (SCG) spirit.[a]

Descriptors	I (%)	F (%)	GM (%)
Visual analysis			
Clarity	86	100	93
Brilliance	86	100	93
Olfactory analysis			
Coffee	61	100	78
Frankly	56	100	75
Elegance	50	100	71
Alcohol	54	88	69
Finesse	43	100	66
Roasted	36	75	52
Cacao	22	63	37
Licorice	22	50	33
Fruity	8	38	18
Nuts	8	13	10
Tobacco	4	13	7
Gustatory analysis			
Pungent	61	88	73
Bitter	57	88	71
Astringent	56	75	65
Finesse	36	75	52
Acid	38	63	48
Salt	39	75	44
Alcoholic	29	50	38
Sweet	18	63	34

[a] $GM^2 = F \times I$, where F is the number of times that the descriptor was mentioned, divided by the total number of times that the descriptor could be mentioned, in percentage; and I is the sum of the intensities given by the whole panel for a descriptor divided by the maximum possible intensity for this descriptor, in percentage.

Table 3
Pearson correlation matrix (r) for sensory descriptors of the spent coffee ground (SCG) spirit. V: visual analysis; O: olfactory analysis; G: gustatory analysis.

Variables	Clarity (V)	Roasted (V)	Tobacco (O)	Nuts (O)	Finesse (O)	Bitter (G)
Brilliance (V)	0.910	–	–	–	–	–
Nuts (O)	–	–	1.000	1.000	–	–
Finesse (O)	–	–	0.866	0.866	1.000	–
Elegance (O)	–	–	0.882	0.882	0.944	–
Bitter (G)	–	0.880	–	–	–	1.000
Alcoholic (G)	–	–	–	–	0.806	–
Astringent (G)	–	0.838	–	–	–	0.901

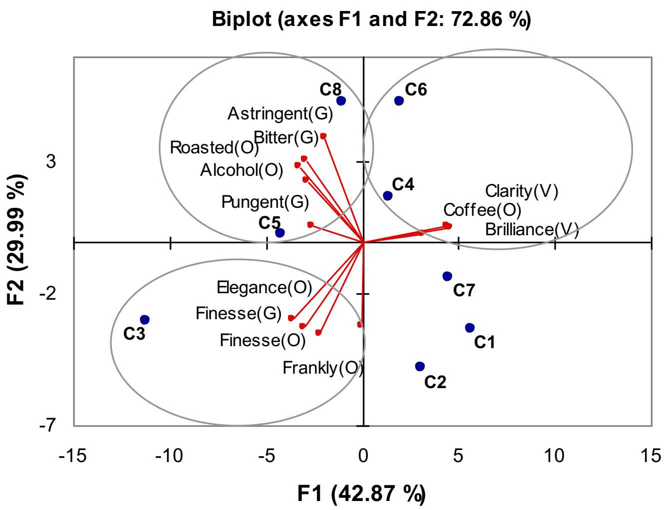

Fig. 2. Principal Component Analysis of preference descriptors (GM > 50%) by taster. C: panelist; V: visual analysis; O: olfactory analysis; G: gustatory analysis.

an important role for dietary experience in differences in evaluation. There was also little evidence for cross-cultural influences on panelists' assessment behavior, such as scale usage. Studies of predominantly sweet and salty foods from both Australia and Japan illustrated the importance of familiarity with the overall product as an influence on the assessment of individual sensory characteristics. In our study, the differences found in the evaluation of descriptors among panelists are due to the degree of familiarity with the product, because SCG spirit is a new product for all tasters; however coffee spirit is also elaborated in Galicia region but with different process. Five panelists evaluated in different form the descriptors with GM > 50% and another three tasters did not show differences in evaluation of descriptors. These results also indicate that the panelists rated in different ways for the most of the sensory descriptors. Several authors considered that this is usual in descriptive analysis, because different tasters use different parts of the scale (Douglas, Cliff, & Reynols, 2001; Poste, Mackie, Butler, & Larmond, 1991; Tang, Hsieh, Heymann, & Huff, 1999).

Finally, a PLSR analysis was performed taking into account the volatile compounds analyzed and aroma descriptors with GM > 50%, considering the mean of all panelists. For a better understanding about the relationship between sensory variables and volatile compounds, the data were standardized to get all of them in approximately the same scale. Considering that the olfactory descriptors finesse, frankly and elegance are those more closely related with the global quality of a product, and considering also that in the present study, the Pearson's correlations showed high correlation (more than 80%) among these three descriptors, it was randomly chosen to perform the PLSR with the finesse aroma descriptor (Fig. 3). According to the loading weight, finesse descriptor was mainly predicted by the following compounds: 1-hexanol, methanol, 2-phenylethanol, 3-methylbutyl acetate, and phenylethyl acetate. Coffee and roasted were correlated with decanoic acid and alcohol with acetaldehyde, 1-propanol, ethyl acetate, 2-methyl-1-propanol, 2-methyl-1-butanol, 3-methyl-1-butanol and ethyl butyrate.

In brief, it is known that the quality of distilled alcoholic beverages varies according to the used raw material, fermentation (including yeast strain) and distillation conditions, aging process, etc (Oliveira et al., 2005). Based on the sensory analysis results here presented, it was concluded that the process conditions used for the SCG spirit elaboration were able to produce a beverage with acceptable organoleptic characteristics, and with smell and taste of coffee.

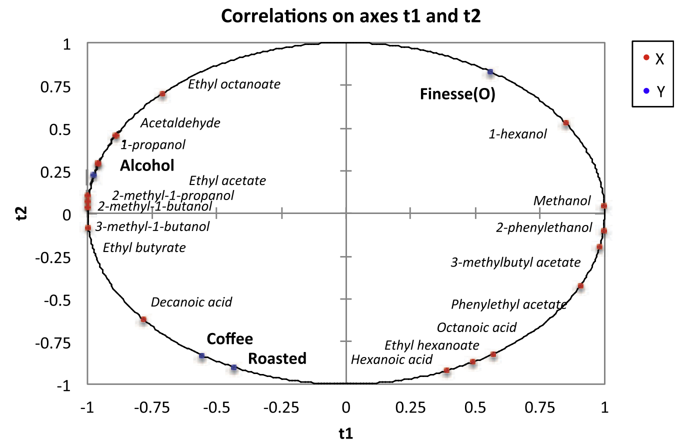

Fig. 3. Partial Least Square Regression between sensory aroma descriptor (finesse) and volatile compounds.

4. Conclusions

SCG can be used as raw material for the production of a distilled beverage. The process to obtain this product consisted in three main steps, namely a hydrothermal extraction, fermentation and distillation, among of which, the first stage for the extraction of aroma compounds from SCG was the main difference of this procedure when compared to the steps involved in the production of the spirits commercially found. Seventeen volatile compounds were identified in the composition of the SCG spirit, all of them in concentration values acceptable for human consumption and able to promote pleasant characteristics to the aroma and flavor of this distillate. Twelve descriptors defined the sensory characteristic of this product, coffee being the most representative aroma by olfactory analysis, as it was desired. Despite the pungent taste, due to being a "new spirit", sensory analysis revealed features of a pleasant beverage, which could be further improved by aging the distillate. In brief, SCG was successfully used for the production of a novel spirit, which presented a remarkable aroma of coffee derived from the raw material, and quality acceptable for human consumption. Production of this spirit is an interesting alternative for the SCG reuse and to expand the distillates' market. Additionally, since the residual solid material obtained after the hydrothermal process is rich in sugars; it could be further reused as raw material for the production of other valuable products, which would give additional value to SCG in a biorefinery concept.

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