Packed bed techniques for accelerated aging of rum

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RESUMEN. El ron es la bebida alcohólica a partir de la caña de azúcar, obtenida por la destilación de las melazas fermentadas y posterior añejamiento en barriles de roble, donde adquiere su aroma y sabor característicos. Se evaluó la tecnología de cama empacada para acelerar el añejamiento de muestras de ron. Esto incluyó tres tratamientos primarios: ron en contacto con la madera natural, con madera tostada y con madera tratada químicamente, en todos los casos, en forma de pequeños cubos durante 90 d y cuatro tratamientos secundarios (calentamiento ocasional y aireación). Se analizaron los índices físicos y químicos, compuestos fenólicos y furánicos por CLAR, las lactonas de roble por microextracción en fase sólida del espacio de cabeza combinada con cromatografía de gases, los resultados del uso de una nariz electrónica usando como sensor un detector de espectrometría de masas y análisis sensorial para cada muestra. Los resultados mostraron que las variaciones en la composición química son similares a las que tienen lugar en el añejamiento tradicional. Se apreciaron diferencias en la composición debidas al tratamiento de la madera, con la mejor composición desde el punto de vista sensorial en las variantes con la madera tostada o tratada químicamente. Las muestras añejadas con la madera tostada o tratada químicamente presentaron mayores concentraciones de fenoles, compuestos furánicos y lactonas de roble que las expuestas a la madera sin tratar. Se logró la diferenciación de las muestras con diferente tratamiento de la madera mediante el análisis de componentes principales de los resultados obtenidos con la nariz electrónica. El análisis sensorial reveló una mayor intensidad de las notas añejas en los rones expuestos a la madera tostada o tratada químicamente. El ron en contacto con la madera tostada por 90 d y aireado (por 3 min con 7,5 L de aire por litro de ron por períodos de 30 y 60 d) mostró la nota más añeja.

ABSTRACT. Rum is a sugar cane spirit obtained by the distillation of molasses through yeast fermentation and subsequent aging in wooden barrels, where the spirit acquires its special flavor and aroma. Packed bed schemes were applied to the accelerated aging of rum samples. These involved maturation in contact with natural, toasted or chemically treated oak wood chips for 90 d and four secondary treatments (occasional heating and aerating). The physical and chemical indexes, phenolic and furanic composition by HPLC, oak lactones by HS-SPME-GC, HS-MS e-nose and sensory analysis were determined for each sample. Results show that variations in chemical composition are similar to those occurring in traditional aging. Differences in rum composition due to wood treatment were observed, with the best compositional data from a sensory point of view corresponding to samples aged with either toasted or chemically treated wood. Samples aged with toasted or chemically treated wood present higher concentrations of phenolic and furanic compounds and oak lactones than those exposed to untreated wood. Differentiation by wood treatment was made by principal component analysis applied to HS-MS e-nose data. Sensory evaluation revealed more mature notes in rums aged with toasted or chemically treated oak woods than in those exposed to untreated wood. The rum that had been in contact with toasted wood for 90 d and aerated (for 3 min with 7.5 L of air per liter of rum for periods of 30 and 60 d) showed the fullest mature note.

INTRODUCTION

Rum is a sugar cane spirit obtained by the distillation of molasses (by-product of sugar cane processing) through yeast fermentation and subsequent aging in wooden barrels, normally of American or French oak, where the spirit acquires its special flavor and aroma. In this stage, also called maturation, the complexity of composition is increased because of the extraction by the spirit of compounds present in the oak wood, though reactions involving only spirit compounds and evaporation of volatile compounds can also occur.[1,2]

Among the compounds released from oak wood, the most important from a sensory point of view are the oak lactones (*cis*- and *trans*-β-methyl-γ-octalactones) present in green wood.[3-5] They arise from lipid oxidation and are known to increase their concentrations after the wood is toasted.[6,7] Other important compounds are phenols, like guaiacol

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and 4-methylguaiacol, with a smoky aroma, both lignin degradation products formed during toasting, and vanillin, a compound normally present in green oak wood but whose concentration is increased by seasoning and toasting.[5-9] Other compounds that can be of sensory importance are the furfuryl compounds (2-furfural and 5-methyl-2-furfural), with a pleasant aroma, which are formed from the degradation of hemicellulose during toasting.[5,7,9-11]

The fundamental process by which the rum acquires its unique organoleptic characteristics however, incurs substantial maintenance overheads, arising from nonsaleable stocks, the annual rotation of casks, insurance of premises and merchandise, storage costs and evaporation losses.[12]

Therefore, from the economic point of view, accelerated aging of rum is an interesting technological option. Three kinds of maturation effects attributed to barrels (extraction, oxidation, and component reaction) would be intensified with increased wood surface in contact with a unit of spirit using a packed bed system, with the advantage of minimal evaporation. Information about the use of this system in rum aging is not available due to commercial protection.

The objective of this study was to ascertain the effects of packed bed system on the volatile compounds of rum.

MATERIALS AND METHODS

Accelerated aging treatments

White cane spirit was used to manufacture rum, which was then subjected to the accelerated aging treatments. Each liter contained 125 mL fresh cane distillate, 875 mL of 60 % alcohol and 3 g refined sugar. Fresh cane distillate and alcohol were treated with 2.5 or 0.5 g of dust-activated charcoal per liter of liquid, respectively.

Rums were manufactured via accelerated aging, for three months, in a packed bed device containing American (*Quercus alba*) oak chips previously subjected to one of the following primary treatments: toasting at 180 °C for 2 h (Treatment 1); no treatment (natural) (Treatment 2) and a chemical treatment (Treatment 3). The latter involved soaking the chips in hot water for 8 h, then in 1 % sodium carbonate solution for 3 h, next in 1 % sulfuric acid solution for 3 h, and washing the chips until neutral pH. Oak chips in all the treatments were cubes with a 1-cm edge and a surface/volume ratio of 90 cm^2/L (15 chips/L), which corresponds to the ratio in a 180-200 L barrel, the most common in Cuban distilleries. All the experiments were carried out in 10 L glass flasks.

With each primary treatment, once the oak chips were in contact with the rum four technological actions (secondary treatments) were performed: simple contact (A); heating at 45 °C (B); aerating for 3 min with 7.5 L of air per liter of rum (C) and heating and then aerating for 24 h at the same rate as in C (D). Each treatment was completed at 30 and 60 d of aging.

All rum samples were analyzed at 15, 30, 60 and 90 d of aging.

Physicochemical analysis

Samples were subjected to the following standard analytical procedures: alcoholic content, total acidity, non volatile acidity, total esters, total aldehydes, pH and dry extract.[13]

Determination of phenolic and furanic compounds

High-performance liquid chromatography (HPLC) was used to determine the phenolic and furanic composition by direct sample injection at 0 and 90 d of aging. Prior to HPLC, the samples were filtered through cellulose membranes of 0.45 mm pore diameter. The liquid chromatograph was a Knauer model WellChrom (Germany) with an analytical pump K-1001, and a diode array detector K-2800 set at 280 nm . The column used was a Hypersil 5 ODS (25 cm x 4.6 mm i.d.). Separation was made isocratically using as solvent a mixture of sulfuric acid-methanol-propanol (180 : 30 : 5) at a flow-rate of 1mL/min.[14] Standard solutions of syringic acid, galic acid, vanillic acid, vanillin, syringaldehyde and furfural were used for calibration curves. Analyses were made in triplicate.

Determination of oak lactones

Oak lactones were determined by headspace solid-phase microextraction (HS-SPME) analysis at 0 and 90 d of aging. The SPME holder for manual sampling and fibers were purchased from Supelco (Bellefonte, USA). Rum samples were diluted to 12% ethanol with deionized water. Dilution delayed ethanol adsorption onto the fibre. Three stationary phase fiber coatings, 100 µm polydimethylsiloxane (PDMS), divinylbenzene (DVB) and polyacrylate (PA) were considered for this research. However, preliminary tests (not presented) indicated that DVB and PA fibers have less affinity for oak lactones, so they were excluded from the experiments. The different parameters that influence the HS-SPME analyses were selected from a previous study of fatty acid esters in rum.[15] Conditions were as follows: for each analysis, a 5 mL sample was put in a 10-mL glass vial with 0.88 g NaCl, 0.1 mL of 0.1 % γ-dodecalactone in ethanol as internal standard, and a little magnetic stirring bar. Then, the vial was tightly capped with a silicon septum and pre-stabilized for 15 min in a thermostatic bath at 40 °C. Afterwards, the stainless steel needle housing the fiber was pushed through the vial septum, and then the fiber was pushed out of its housing and exposed, for 30 min at 40 °C, to the headspace generated in the sample vial. After extraction, the fiber was pulled into the housing and the SPME device was removed from the vial and inserted into the GC injection port for thermal desorption of the analytes. Analyses were made in triplicate.

GC-FID analyses were performed with a Konik 4000A gas chromatograph (Barcelona, Spain) equipped with a flame ionization detector (FID). The injection was made in the splitless mode at 250 °C for 1 min, using an inlet liner of 0.75 mm i.d. Chromatographic separations were performed using a DB-Wax (30 m X 0.25 mm i.d., 0.25 mm film thickness) fused silica capillary column with hydrogen as carrier gas at a flow-rate of 1 mL/min . The temperature program was 40 °C at the outset, raised after 1 min at 4 °C/min to 250 °C, then held for 20 min. The FID temperature was set to 250 °C . Quantification was performed by a standard internal method from electronically measured peak areas. Standard solutions of oak lactones were used for calibration curves. The relative standard deviation estimated by performing seven replicate analyses were 3.0 and 2.8 % for the *cis*- and the *trans*-isomer, respectively, while the linearity coefficients for the calibration curves were 0.999 for both isomers.

GC-MS analysis was carried out using a Hewlett-Packard 6890 gas chromatograph coupled to a HP-5973 mass selective detector for identification of oak lactones. Separation was achieved under the same conditions described before, with helium as carrier gas at a flow-rate of

1 mL/min . The mass spectrometer was operated in the electron impact ionization mode at 70 eV . Interface, source, and quadrupole temperatures were 200, 230, and 150 °C, respectively. Mass range was from 35 to 400 amu.

HS-MS e-nose

The rums were diluted to 40 % (v/v), which is the usual alcoholic content of most alcoholic beverages. Diluted samples were analyzed in triplicate by a HS-MS e-nose composed of an HP 7694 static headspace sampler, an HP 6890 gas chromatograph and an HP 5973 quadrupole mass spectrometer from Hewlett-Packard (Waldbronn, Germany). With this setup, the gas chromatograph's function was to transfer the volatiles to the MS, not to resolve the peaks chromatographically.

In the HS-MS analysis, 5 mL of the diluted spirit and 0.6 g NaCl were placed in a 10 mL vial that was hermetically capped with a PTFE/silicone septum. The sample was held at 65 °C for 1 h under constant stirring. Afterwards, the headspace of the sample was introduced into the gas chromatograph's injection port. The loop and transfer line temperatures were 90 and 105 °C, respectively, and the pressurization and injection times were 0.30 and 0.60 min, respectively. Chromatographic injection was made in splitless mode for 1.6 min at 200 °C using a 1.5 mm i.d inlet. To transfer the volatile compounds to the MS a HP-5MS chromatographic column (30 m X 0.25 mm i.d., 0.25 mm film thickness) was used, and the oven temperature program was 70 °C at the outset, raised after 1 min at 70 °C/min to 180 °C, the held for 2.5 min . The carrier gas was helium, with a flow rate of 1.8 mL/min . With these strong temperature and flow conditions, the transfer of the volatile compounds to the MS detector was achieved in only 5 min . The mass spectrum obtained was due to the fragmentation of the compounds that were inside the mass spectrometer's ionization chamber for this time. Mass spectra were recorded by electronic impact (EI) ionization at 70 eV . The mass-to-charge ratio (m/z) range used was 50-250 amu. The ion source and mass quadrupole temperatures were 230 °C and 150 °C, respectively.

The final response data matrix was then made up with the abundance data 156 objects (51 spirits analyzed in triplicate, from three primary treatments x four secondary treatments x four sampling times and the three analysis of the fresh white cane spirit) at 201 variables (m/z ratios).

Sensory evaluation

Ranking sensory difference tests as to mature flavor intensity were carried out by a panel of five trained judges, during three replicate tasting sessions. Only rums aged for 90 d and diluted to 38 % ethanol were selected for this evaluation. Each wood treatment was evaluated first. Samples for ranking were from the technological treatments. Another ranking sensory difference evaluation was made to the selected samples of each primary treatment group. The Friedman test was used at the 95 % confidence level.[16]

Statistical analysis

General descriptive statistics were processed using Statistica 7.0 from SatSoft Inc. (Tulsa, USA), and the principal component analysis (PCA) was made by means of Pirouette 2.6 from Infometrix Inc. (Woodinville, USA).

RESULTS AND DISCUSSION

Physicochemical analysis

Some important differences were found among rums with different aging treatments (Tables 1 and 2):

Alcohol. The analysis of variance revealed that ethanol content decreased during the experiment, particularly in those samples where heating was used (1B, 1D, 2B, 2D, 3B, and 3D). Such differences are due to the more intense evaporation the latter undergo, in comparison with samples from the non-heating treatments. Nevertheless, ethanol loss is less important than when aging is performed in wooden barrels.[12] No significant differences were observed between 90-d samples from the wood treatments (T-1, T-2, and T-3).

Total acidity. The analysis of variance indicated that total acidity increased during the experiment. Higher values were found in samples exposed to toasted wood. The three

Table 1. Alcohol, total acidity and non volatile acidity of aged rums in packed bed system.[1]

Treatment	Alcohol (%)				Total acidity (mg/100 mL a.a.)				Non volatile acidity (mg/100 mL)			
	Días											
	15	30	60	90	15	30	60	90	15	30	60	90
1A	59.2a	59.1a	58.7b	58.3c	29.5a	33.8b	39.6c	41.9d	11.9a	18.9b	19.8c	21.0d
1B	59.2a	59.1a	58.8b	57.7c	26.6e	32.6a	38.6c	40.3d	13.5e	17.1f	17.7f	19.5c
1C	59.5a	59.2a	58.7b	58.1c	29.5a	33.8b	36.2c	40.0d	11.6a	16.0e	16.8f	19.7c
1D	59.3a	59.2a	58.4b	57.6c	28.6a	34.8b	38.5c	41.4d	11.4a	17.4f	17.9f	20.8d
2A	59.3a	59.3a	58.8b	57.7c	20.4f	26.3e	27.6e	31.3a	11.0a	16.9e	17.5f	17.9f
2B	59.3a	59.1a	58.3b	57.7c	20.4f	29.3a	29.4a	31.8a	11.4a	15.9e	17.4f	19.1g
2C	59.5a	59.2a	58.8b	57.8c	21.4f	27.4e	29.7a	30.7a	10.6a	16.4e	17.1f	19.1g
2D	59.5a	59.3a	58.6b	58.1c	22.3f	27.4e	31.4a	31.8a	10.7a	16.0e	16.7f	19.1g
3A	59.5a	59.2a	58.8b	58.3c	22.4f	29.5a	33.3b	34.5b	13.2e	16.7f	16.9f	17.2f
3B	59.4a	59.3a	58.6b	58.0c	23.2f	30.6a	33.8b	36.9b	11.6a	15.9e	16.5e	17.9f
3C	59.5a	59.1a	58.9b	58.3c	21.4f	21.7f	33.9b	35.4b	11.2a	17.8f	17.9f	19.2g
3D	59.5a	59.4a	59.0b	58.6c	26.5e	29.4a	33.2b	35.4b	11.4a	16.6f	17.5f	19.2g

[1] White cane spirit: 59.6 % alcohol, 11.0 mg/100 mL a.a. total acidity and 0 mg/100 mL non volatile acidity. Values followed by the same letter are not significantly different at $P \leq 0.05$.

Table 2. Total esters, total aldehydes, pH value and dry extract of aged rums in packed bed system.[1]

Treatment	Total esters (mg/100 mL a.a.)				Total aldehydes (mg/100 mL a.a.)				pH				Dry extract (g/L)			
	Days															
	15	30	60	90	15	30	60	90	15	30	60	90	15	30	60	90
1A	7.7a	9.2b	10.8c	11.7d	2.8a	2.8a	2.6a	3.1b	4.7a	4.4b	4.4b	4.3b	0.349a	0.364b	0.383a	0.403c
1B	7.7a	9.2b	11.1c	12.2d	2.0c	2.9a	3.1b	3.1b	4.7a	4.5b	4.4b	4.3b	0.353a	0.372b	0.400c	0.442c
1C	8.1a	9.2b	10.7c	11.8d	2.4a	2.4a	3.1b	3.3b	4.8a	4.5b	4.4b	4.3b	0.350a	0.370b	0.375b	0.395c
1D	9.2b	10.7c	11.0c	12.5d	1.8c	3.0a	3.2b	3.3b	4.8a	4.6b	4.4b	4.3b	0.358a	0.378b	0.384c	0.420c
2A	7.7a	9.2b	10.8c	12.8d	1.4c	1.9c	2.3a	2.8a	4.8a	4.6b	4.4b	4.3b	0.343a	0.365b	0.379b	0.395c
2B	8.1a	9.2b	10.8c	12.4d	1.8c	2.8a	2.9a	2.9a	4.9a	4.7a	4.5b	4.4b	0.352a	0.373b	0.379b	0.403c
2C	7.7a	8.1a	10.4c	11.9d	1.9c	2.7a	2.8a	2.9a	4.8a	4.7a	4.5b	4.4b	0.349a	0.373b	0.377b	0.397c
2D	8.1a	9.2b	10.7c	12.3d	2.1c	2.4a	2.8a	2.8a	4.9a	4.8a	4.6b	4.5b	0.355a	0.379b	0.385c	0.404c
3A	9.1b	9.2b	10.7c	15.8e	2.2c	2.7a	3.2b	3.7d	4.1c	4.0c	4.0c	3.8d	0.335a	0.357b	0.364b	0.380c
3B	9.2b	10.7c	12.4d	15.5e	1.9c	2.0c	2.7a	3.7d	4.1c	3.9cd	3.8d	3.8d	0.337a	0.349a	0.359b	0.386c
3C	7.6a	10.2c	10.7c	15.7e	2.6a	2.8a	3.0a	3.9d	4.1c	4.1c	3.9cd	3.8d	0.355a	0.377b	0.384c	0.395c
3D	10.6c	11.7d	12.4d	15.3e	2.6a	2.8a	3.0a	3.8d	4.2c	3.9cd	3.8d	3.8d	0.355a	0.380b	0.381c	0.413c

[1] White cane spirit: 7.7 mg/100 mL a.a. total ester, 1.6 mg/100 mL a.a. total aldehydes, 5.1 pH, and 0.308 g/L dry extract. Values followed by the same letter are not significantly different at $P \leq 0.05$.

wood treatments were significantly discriminated (at 95 % confidence) in rum samples at 90-d aging, with values in the following order: samples exposed to toasted wood > chemically treated wood > untreated wood.

Non volatile acidity. In a similar way to total acidity, the analysis of variance revealed an increase in non volatile acidity during the experiment. At 90-d aging, samples that were in contact with toasted wood significantly differ at 95 % confidence from those in which untreated (natural) wood or chemically treated wood was used, while there is no difference between samples exposed to chemically treated wood and untreated wood.

Total esters. The ester content significantly increased with aging (at 95 % confidence). At 90-d aging, samples aged with chemically treated wood significantly differ from those that had undergone contact with untreated wood or toasted wood, but there is no difference between the latter two samples.

Total aldehydes. The total aldehydes content significantly increased with aging (at 95 % confidence). At 90-d aging, samples exposed to chemically treated wood significantly differ from those in which natural wood or toasted wood was used, while there is no difference between the latter two.

pH. The pH value significantly decreased during the first 60 d of aging, not so in the interval between 60 and 90 d, in which the rum samples that had undergone contact with chemically treated wood had the lowest values, followed by those exposed to toasted wood and natural wood (at 95 % confidence level).

Dry extract. The analysis of variance indicated that dry extract increased during the experiment. No significant differences were observed between 90-d samples from all wood treatments.

Evolution of phenolic and furanic compounds

It is apparent from HPLC results (Table 3) that the differences between the means are sufficient to distinguish the different wood treatments, but not the different technological treatments between 90-d samples (at 95 % confidence level). The concentration of these compounds is significantly higher in rums that were in contact with toasted wood than in samples exposed to chemically treated wood. The same is true of the latter in comparison with rums matured with natural wood (at 95 % confidence level). This increase is especially apparent in the cases of vanillin and syringaldehyde, as these aromatic

Table 3. Concentrations of phenolic and furanic compounds at 90-d aging.

Treatment	Furfural	Gallic acid	Vanillic acid	Syringic acid	Vanillin	Syringaldehyde
	(mg/L)					
1A	4.7a	4.2a	3.0a	7.4a	6.3a	7.2a
1B	4.5a	3.7b	3.1a	7.5a	5.5a	6.1a
1C	4.7a	3.0c	2.9a	7.0a	5.0a	5.2a
1D	4.7a	4.2a	2.2a	6.1a	5.5a	6.2a
2A	–	3.5b	–	–	–	–
2B	0.2b	2.7c	–	–	–	–
2C	0.2b	2.9c	–	–	–	–
2D	–	3.0c	–	–	–	–
3A	3.9c	1.9d	–	3.2b	2.6b	1.2b
3B	3.3c	2.0d	–	3.2b	3.0b	1.5b
3C	3.9c	2.1d	–	3.0b	2.9b	1.9b
3D	3.5c	2.0d	–	2.8b	2.6b	1.8b

– Not detected. Values followed by the same letter in a same column are not significantly different at $P \leq 0.05$.

aldehydes are characteristic of aging in oak wood barrels.[9-12]

Evolution of oak lactones

It is apparent from the GC results (Table 4) that the differences between the means are sufficient to distinguish the different wood treatments, but not the different technological treatments between 90-d samples (at 95 % confidence level). The concentrations of *cis*- and *trans*-oak lactones are significantly higher in rums exposed to toasted wood than in samples that were in contact with chemically treated wood, and also higher in the latter than in rums exposed to natural wood (at 95 % confidence level).

Analysis of samples by HS-MS e-nose

The whole set of 51 rum samples (48 aged samples and three white cane spirits) was subjected to PCA from the correlation matrix. It was very difficult to appreciate any grouping tendency (results not shown), either for the wood treatment (1 to 3) or the technological treatment (A to D).

Evaluation of the profile of several ions in the mass spectrum allowed selection of four ions that seem to differ more between the rum samples according to wood treatment: 95, 96, 109 and 110. Figure 1 shows the results of applying PCA to all the samples with these four ions, which explains 99.6 % of the total variance. There are three groups, corresponding to wood treatment (1 to 3).

The analysis of the factor loadings of each original variable revealed that ions 95 and 96 separate the rum samples matured in natural wood and white cane spirits from the samples that were matured in toasted or chemically treated wood (the last two presenting stronger signatures of these ions), while ions 109 and 110 discriminate the samples aged in toasted wood (showing a stronger signature of these ions) from those exposed to chemically treated wood.

These ions could be assigned to furfural (whose mass spectrum displays intense signatures in m/z 95 and 96) and 5-methylfurfural (with ions 109 and 110 as the most intense). Both furanic compounds, of pleasant scent, are degradation products of hemicellulose and it is known that their concentrations increase with toasting of the oak wood.[7,9,11]

Table 4. Concentration of oak lactones at 90-d aging.

Treatment	*Trans*-oak lactone	*Cis*-oak lactone
	(mg/L)	
1A	0.36a	0.41a
1B	0.39a	0.47a
1C	0.35a	0.43a
1D	0.36a	0.47a
2A	0.29b	0.36b
2B	0.29b	0.39b
2C	0.28b	0.36b
2D	0.29b	0.39b
3A	0.31c	0.40c
3B	0.31c	0.40c
3C	0.32c	0.41c
3D	0.32c	0.41c

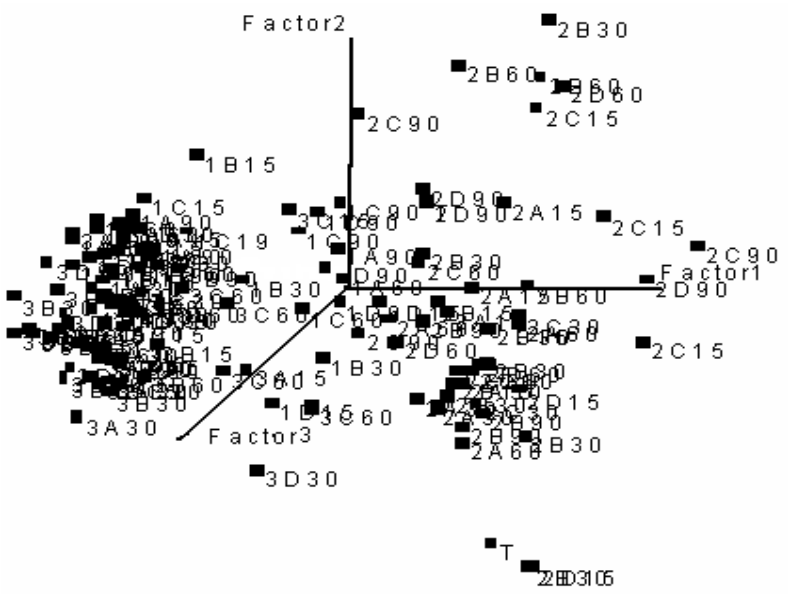

Fig 1. *PCA on the data obtained from the analysis of rums by HS-MS e-nose (ions 95, 96, 109 and 110 m/z).*

Sensory analysis

Table 5 presents the results of the ranking difference sensory analysis according to the mature flavor intensity with 90-d aging. According to the Friedman test (at 95 % confidence level), there are significant statistical differences between the samples in each wood treatment. In rums in contact with toasted wood or natural wood, technological treatment C (aerated) is significantly better in mature flavor intensity than the others. Nevertheless, in rums in contact with chemically treated wood, technological treatments B and C (aerated, and heated and aerated, respectively) were similar and significantly

Table 5. Results of the ranking difference sensory analysis between wood treatments.

Treatment	A	B	C	D
1	23a	16b	37c	24a
2	16a	22b	37c	25b
3	13a	31b	37c	19a

Values followed by the same letter in a same row are not significantly different at $P \leq 0.05$.

better in mature flavor intensity than the samples with the other treatments.

A comparison of the four best samples (1A, 1B, 3B and 3C) was made (Table 6). According to the Friedman test (at 95 % confidence level), the rum matured with toasted wood and aerated (treatment 1C) has a significantly stronger mature flavor at 90-d aging than samples from the other treatments.

Table 6. Results of the ranking difference sensory analysis between best primary and secondary treatments.

Treatment	1C	2C	3B	3C
Value	40a	16b	19b	25c

Values followed by the same letter are not significantly different at $P \leq 0.05$.

CONCLUSIONS

Variations in chemical composition generated by 90-d aging in a packed bed system are similar to those occurring in traditional aging. Differences in rum composition related to wood treatment were observed, with the best compositional data from a sensory point of view corresponding to samples that had been exposed to either toasted or chemically treated wood.

Higher concentrations of phenolic and furanic compounds and *cis*- and *trans*- oak lactones were found in samples matured with toasted or chemically treated wood, in comparison with samples that had been exposed to untreated wood.

The use of a HS-MS e-nose allowed discrimination of the samples matured in natural wood and the white cane spirit on one hand and those matured in toasted and chemically treated wood on the other, using only the m/z 95 and 96 ions corresponding to furfural, while the m/z 109 and 110 ions corresponding to 5-methylfurfural discriminate the samples matured in toasted wood from those in which chemically treated wood was used. Sensory evaluation showed that both toasted and chemically treated oak wood yield rums with higher mature notes than untreated wood. Besides, rums aged in contact with toasted wood for a 90-d period and aerated (with 7.5 L of air per liter of rum for 3 min, with both 30 and 60-d aging) showed the fullest mature note.

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