Rapid Induction of Ageing Character in Brandy Products – Part III. Influence of Toasting

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As part of a broader study that investigated techniques for the rapid induction of the needed ageing character in brandy products, the effect of oak wood toasting on quality and chemical composition of oak wood extracts and matured and unmatured pot-still brandy, is reported on. Extracts, prepared from oak chips supplied by a South African cooper, and from commercially obtained oak, and representing different oak types and levels of toasting (i.e. untoasted, light, medium and heavy), were added to 70% (v/v) unmatured pot-still brandy and stored for eight months in glass containers (Schott bottles) at room temperature, or in the case of controls, below 0°C. Matured and unmatured (control) pot-still brandy samples were analysed for wood-derived congeners by means of HPLC and GC. Toasted, as opposed to untoasted oak, gave acceptable extracts, the best overall quality pot-still brandies and generally higher concentrations of volatile (GC-determined) and less volatile (HPLC-determined) wood-derived congeners. Toasting provoked an important separation as indicated by discriminant analysis.

Oak wood chemistry is complex because different flavour-affecting compounds are produced at different temperatures. Oak types also toast differently. American oak, which is denser and has coarse grain, toasts faster than fine grained European oaks (Caputo, 2004). Barrel making involves oak seasoning and toasting, which ensure the structural integrity of the barrel. Seasoning prevents the wood from shrinking after barrel construction and hence leakage, while firing is applied to stabilise the curved shape of the barrel. Both these fabrication steps also play important roles in oak flavour development. The application of coopering heat disrupts chemical bonds within the wood polymers cellulose, hemicellulose and lignin and results in degradation or compositional changes by pyrolysis and hydrothermolysis. These changes can influence the flavour of wine and spirits significantly. In addition, major changes take place in the structure and level of oak tannins (Hale *et al.*, 1999; Martricardi & Waterhouse, 1999). Observations that the intensity of the heat naturally affects the compounds produced during the degradation of wood macromolecules, has led to an increased interest in the ability to control the process more precisely. The traditional method of heating over an open flame using oak chips is still used today, while the temperature and duration of heating is more carefully controlled. Alternative methods of heating wooden barrels include the use of radio-frequency and infrared radiation (Mosedale & Puech, 1998).

The effects of, and time required for maturation of brandy, are highly variable and are influenced by a wide range of factors. Interest in methods of predicting, controlling and simulating the effects of maturation has increased as understanding of the process has evolved. The prestigious image associated with many alcoholic beverages depends largely on maintaining traditional methods of production. Much of the research on the application of accelerated maturation methods has been conducted in Eastern Europe where the production of brandy and other spirits is significant, but controlled by fewer regulations defining the method of maturation (Mosedale & Puech, 1998).

The designations light, medium and heavy toasts are traditional terms based on the visual appearance of the inner face of the staves. The term lightly toasted implies a mild visual darkening. Medium toasted staves are similar to the colour of toasted bread. Additional levels of toasting are often offered, light char and medium-plus being the most common. There is no industry standard on toast level. Many cooperages take pride in the uniqueness of their toasting. Temperature variability during toasting is high, due to variation in the intensity of the fire and the convective movement of the air. Heat intensity during toasting is controlled by the cooper who judges it by visually inspecting the wood and by feeling the temperature of the barrel. These methods do not provide precise control over the extent of toasting, but are the only procedures available to cooperages at present. A consequence is that barrels which are described as having the same toast level may vary considerably. To compound the problem, there is no universal definition of toasting levels, either physical or chemical (Matricardi & Waterhouse, 1999).

This is the third in a series of articles. The first article focused on the importance of extracts in brandy maturation, the preparation of oak extracts, the influence of different extraction conditions, mediums and suppliers of oak chips on chemical and sensory profiles. The second paper discussed the influence of oak type

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on the sensory and chemical profiles of fortified extracts and unmatured and matured pot-still brandy. This third article focuses on the impact of toasting on quality and chemical profiles. Interactive aspects of the collected data will be discussed in a subsequent, overview, article.

MATERIALS AND METHODS

Materials and methods have already been described in part I (Van Jaarsveld *et al.*, 2009). Briefly, American and French oak chips, representing different levels of toasting and obtained from a cooper and a commercial supplier, were placed in either water or a 55% (v/v) ethanol medium and boiled under reflux with backflow cooling for 5 hours. Either open concentration or concentration under reduced pressure using a Buchi rotavapour, followed. Extracts were fortified to 40% (v/v). Fortified extracts were added to 70% (v/v) unmatured pot-still brandy (procured as one batch) at 60 mL/L and stored for eight months in glass containers (Schott bottles) at room temperature, or in the case of controls, below 0°C. Samples were subjected to sensory and compositional analysis. Selected chemical variables encompass a wide range of compounds considered to contribute to the flavour of the maturing distillate. Volatile flavour constituents extracted into the distilled spirit from oak chips were separated, identified and quantified by gas chromatography (GC) and the non-volatile and less volatile compounds by using high-performance liquid chromatography (HPLC). Fortified (40%, v/v) extracts were sensorially evaluated in duplicate by a panel of seven judges for acceptability for brandy production in terms of a yes or no response. The number of yes scores for each extract evaluated was expressed as a percentage of the total number of evaluations per extract. Pot-still brandy samples were also sensorially evaluated for overall quality by a panel of seven experienced judges. A line method was used, i.e. evaluating the wine characteristics by making a mark on an unstructured, straight 10 cm line. The left and right-hand ends of the line were indicated by the terms, “not detectable” and “prominent”, respectively.

The variables measured were subjected to Analysis of Variance (ANOVA), using General Linear Models (GLM) procedure of SAS statistical software version 8.2 (SAS Institute Inc., Cary, NC, USA) (SAS, 2000). The Shapiro-Wilk test was performed to test for normality (Shapiro & Wilk, 1965). Fisher’s t-least significant difference (LSD) was calculated at the 5% probability level to facilitate comparison between treatment means. Values that differed at $p \leq 0.05$ were considered to be significantly different. Multivariate analysis of variance (MANOVA) was also performed at the 95% confidence level. Discriminant analysis (DA) was used as the pattern recognition tool, using the full dataset or dataset comprising the 65% (v/v) concentration level. Discriminant analysis by the forward stepwise method was used to select the most powerfull discriminators or variables most effective at separating the factors. The ellipses around each grouping represent the 95% confidence limit for that grouping. All computations were carried out with the package XLSTAT 2008 [Pro] (Win).

RESULTS AND DISCUSSION

From the analysis of variance the effects of the different treatments and their interactions were determined.

Extracts

Sensory evaluation of fortified oak extracts prepared from medium and heavy toasted (cooper and commercial French oak), light toasted (commercial French) and toasted (commercial American) oak chips, showed these extracts to be more acceptable than those prepared from premium (commercial American), special (commercial American oak) and untoasted (cooper) oak chips (Figs 1 to 4).

Unmatured pot-still brandy

Medium to heavy toasted oak chips generally yielded higher concentrations of volatile wood-derived congeners in unmatured pot-still brandy than untoasted oak (Table 1). The concentrations of less volatile vanillic acid, syringic acid, *p*-coumaric acid, syringaldehyde, ellagic acid, coniferaldehyde and synapaldehyde

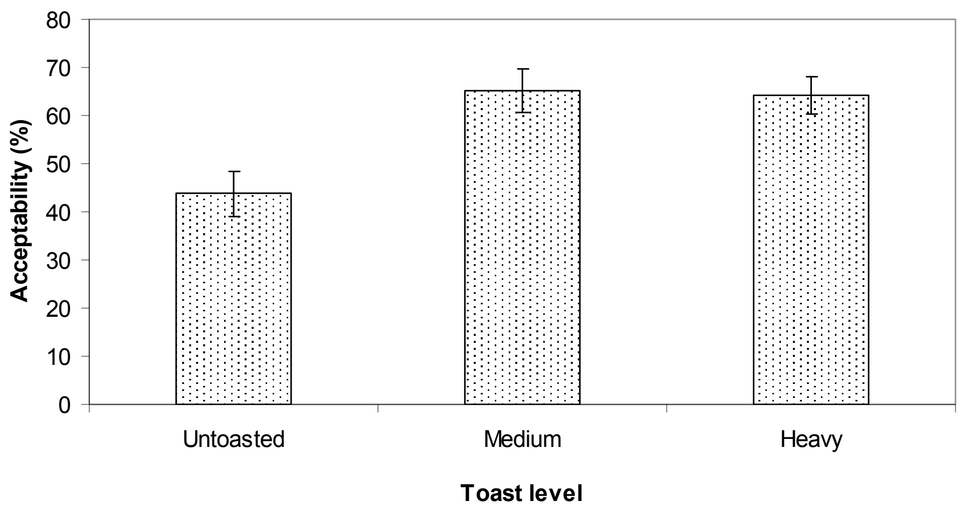

FIGURE 1

Percentage sensory acceptability of oak extracts prepared from chips of different types of differently toasted oak obtained from a cooper and subjected to various treatments. Only the 65% (v/v) concentration level was considered in the statistical evaluation. Refer to part I in this series for more detail regarding the various treatments. Error bars represent the standard error of the mean.

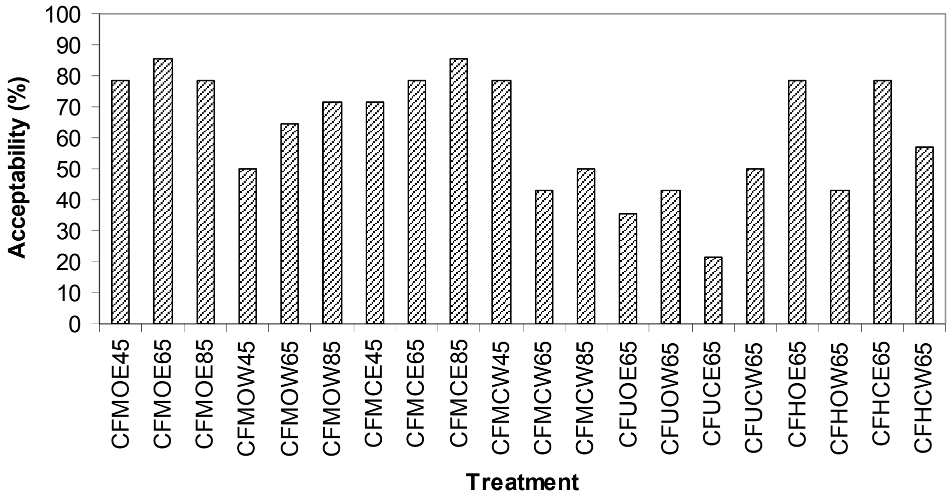

FIGURE 2

Sensory acceptability of extracts prepared from French oak as conditioned by a cooper. Preparation included boiling under reflux in either an ethanol or a water medium, followed by either open or closed concentration by 45, 65 or 85% (v/v). Refer to part I in this series for more detail regarding the various treatments. Treatment: CFMOE45: Cooper, French, medium, open, ethanol; CFMOE65: Cooper, French, medium, open, ethanol; CFMOE85: Cooper, French, medium, open, ethanol; CFMOW45: Cooper, French, medium, open, water; CFMOW65: Cooper, French, medium, open, water; CFMOW85: Cooper, French, medium, open, water; CFMCE45: Cooper, French, medium, closed, ethanol; CFMCE65: Cooper, French, medium, closed, ethanol; CFMCE85: Cooper, French, medium, closed, ethanol; CFMCW45: Cooper, French, medium, closed, water; CFMCW65: Cooper, French, medium, closed, water; CFMCW85: Cooper, French, medium, closed, water; CFUOE65: Cooper, French, untoasted, open, ethanol; CFUOW65: Cooper, French, untoasted, open, water; CFUCE65: Cooper, French, untoasted, closed, ethanol; CFUCW65: Cooper, French, untoasted, closed, water; CFHOE65: Cooper, French, heavy, open, ethanol; CFHOW65: Cooper, French, heavy, open, water; CFHCE65: Cooper, French, heavy, closed, ethanol and CFHCW65: Cooper, French, heavy, closed, water.

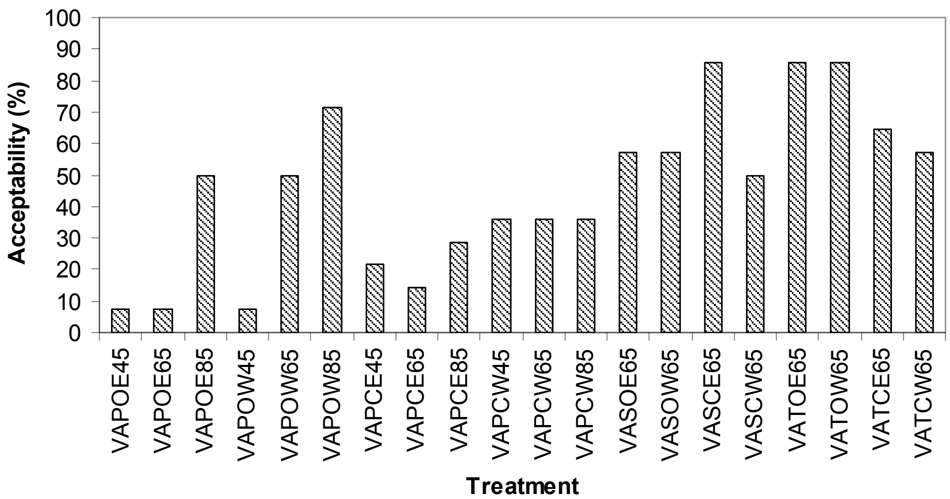

FIGURE 3

Sensory acceptability of extracts prepared from American oak obtained from a commercial supplier. Preparation included boiling under reflux in either an ethanol or a water medium, followed by either open or closed concentration by 45, 65 or 85% (v/v). Refer to part I in this series for more detail regarding the various treatments. Treatment: VAPOE45: Commercial, American, premium, open, ethanol; VAPOE65: Commercial, American, premium, open, ethanol; VAPOE85: Commercial, American, premium, open, ethanol; VAPOW45: Commercial, American, premium, open, water; VAPOW65: Commercial, American, premium, open, water; CAPOW85: Commercial, American, premium, open, water; VAPCE45: Commercial, American, premium, closed, ethanol; VAPCE65: Commercial, American, premium, closed, ethanol; VAPCE85: Commercial, American, premium, closed, ethanol; VAPCW45: Commercial, American, premium, closed, water; VAPCW65: Commercial, American, premium, closed, water; VAPCW85: Commercial, American, premium, closed, water; VASOE65: Commercial, American, special, open, ethanol; VASOW65: Commercial, American, special, open, water; VASCE65: Commercial, American, special, closed, ethanol; VASCW65: Commercial, American, special, closed, water; VATOE65: Commercial, American, toasted, open, ethanol; VATOW65: Commercial, American, toasted, open, water; VATCE65: Commercial, American, toasted, closed, ethanol and VATCW65: Commercial, American, toasted, closed, water.

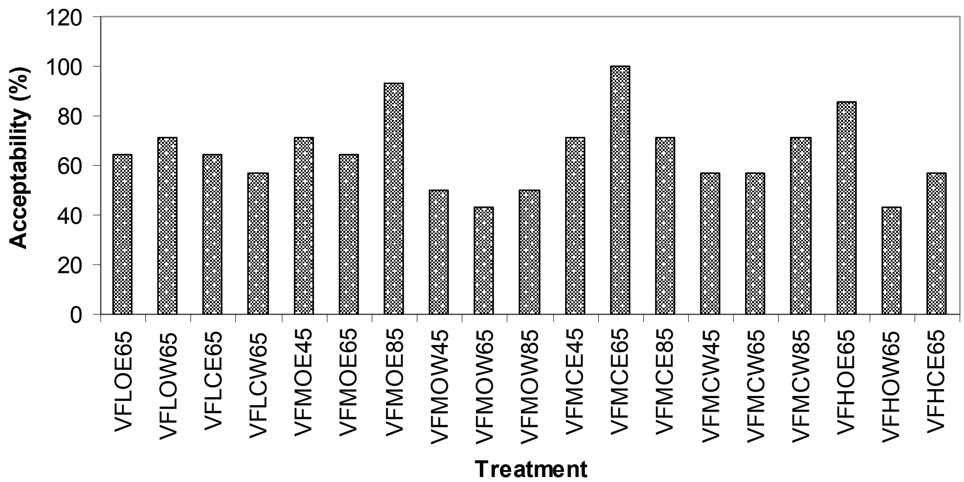

FIGURE 4

Sensory acceptability of extracts prepared from French oak obtained from a commercial supplier. Preparation included boiling under reflux in either an ethanol or a water medium, followed by either open or closed concentration by 45, 65 or 85% (v/v). Refer to part I in this series for more detail regarding the various treatments. Treatment: VFLOE65: Commercial, French, light, open, ethanol; VFLOW65: Commercial, French, light, open, water; VFLCE65: Commercial, French, light, closed, ethanol; VFLCW65: Commercial, French, light, closed, water; VFMOE45: Commercial, French, medium, open, ethanol; VFMOE65: Commercial, French, medium, open, ethanol; VFMOE85: Commercial, French, medium, open, ethanol; VFMOW45: Commercial, French, medium, open, water; VFMOW65: Commercial, French, medium, open, water; VFMOW85: Commercial, French, medium, open, water; VFMCE45: Commercial, French, medium, closed, ethanol; VFMCE65: Commercial, French, medium, closed, ethanol; VFMCE85: Commercial, French, medium, closed, ethanol; VFMCW45: Commercial, French, medium, closed, water; VFMCW65: Commercial, French, medium, closed, water; VFMCW85: Commercial, French, medium, closed, water; VFHOE65: Commercial, French, heavy, open, ethanol; VFHOW65: Commercial, French, heavy, open, water and VFHCE65: Commercial, French, heavy, closed, ethanol.

TABLE 1

Gas Chromatographic determination of unmatured pot-still brandy prepared from oak (cooper only) extracts.

Type/ Treatment	n*	Mean (mg/L)						
		Furfural	**5-Methyl-furfural**	**Guaiacol**	***trans*-Oak lactone**	***cis*-Oak lactone**	***o*-Cresol**	**Phenol**
Untoasted	12	21.323[a] (3.452)**	0.000[b] (0.000)	1.004[a] (0.158)	nd***	0.019[c] (0.019)	nd	nd
Medium	12	28.294[a] (3.620)	1.284[a] (0.403)	1.114[a] (0.187)	nd	0.078[a] (0.053)	nd	nd
Heavy	12	28.476[a] (3.473)	1.305[a] (0.383)	1.118[a] (0.192)	nd	0.064[b] (0.064)	nd	nd

Type/ Treatment	n*	Ethyl guaiacol	*p*-Cresol	Eugenol	2,6-Dimethoxy-phenol	5-Hydroxymethyl furfural	Vanillin
Untoasted	12	0.008[a] (0.008)	nd	0.031[a] (0.031)	31.982[a] (1.842)	1.926[a] (0.909)	4.370[a] (2.866)
Medium	12	0.000[a] (0.000)	nd	0.000[a] (0.000)	31.664[a] (1.373)	7.945[a] (1.677)	6.764[a] (3.622)
Heavy	12	0.000[a] (0.000)	nd	0.000[a] (0.000)	32.052[a] (1.198)	9.332[a] (2.875)	10.679[a] (4.719)

*Number of evaluations of samples; **standard error of the mean; ***not detected. Treatments with the same superscript within columns do not differ significantly ($p \geq 0.05$). Data representative of extracts concentrated by 65% (v/v).

in unmatured pot-still brandy, prepared from extracts from untoasted oak chips, tended to be sligthtly (*p*-coumaric acid) or significantly lower than those prepared from medium to high toast levels, with only those of gallic acid, catechin and *m*-coumaric acid being significantly higher (Table 2).

Large variation in the data may be attributed to factors that impact on the type and number of compounds extracted from the wood (Van Jaarsveld *et al.*, 2009). In addition, each oak species displays high variation between individual trees. Such differences are likely to influence the properties of the wood to a greater extent than any subsequent treatment (Mosedale & Puech, 1998).

Eight-month matured pot-still brandy

Generally, eight-month matured pot-still brandy prepared from extracts using medium and heavy toasted oak chips, tended to be sensorially more acceptable than those prepared from untoasted or lightly toasted chips (Figs 5 and 6). Sensory preferences for brandies stored in barrels of higher toasting compared to those of lesser toasting have been reported by Clyne *et al.* (1993) and Canas *et al.* (1999).

Generally, matured pot-still brandy prepared from toasted chips had significantly ($p \leq 0.05$) higher woody (Bosso *et al.*, 2004), toasted (Dennison, 1999) and sweet associated aromas compared to untoasted chips, thus confirming the important role toasting has on quality (Fig. 5).

The concentrations of the volatile, wood-derived congeners in eight-month matured pot-still brandy made from extracts prepared from toasted (medium, heavy) chips were generally higher than compared to lightly or untoasted chips (Tables 3 and 4). Similarly, the concentrations of the less volatile wood-derived congeners in eight-month matured pot-still brandy made from extracts prepared

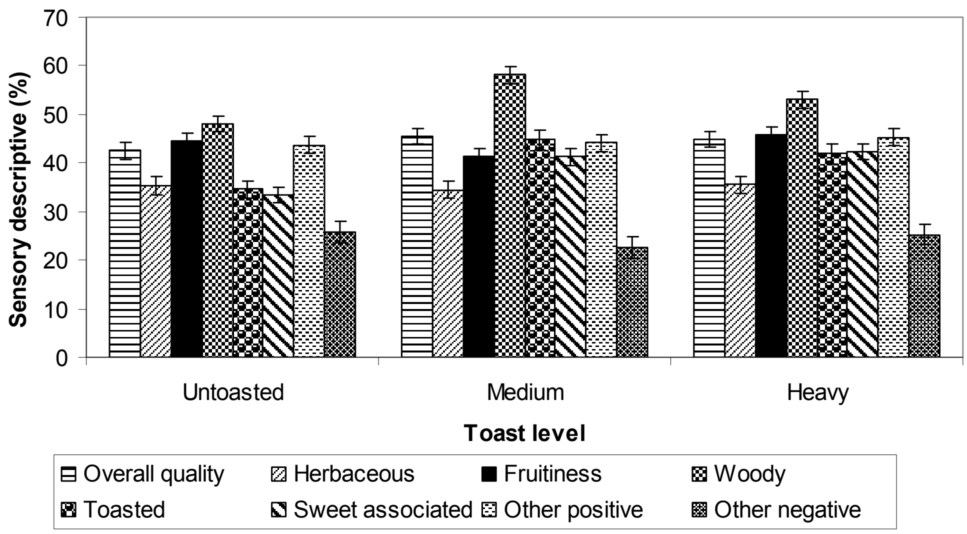

FIGURE 5

Overall sensory quality and other sensory descriptors of eight-month matured pot-still brandy from oak extracts prepared from chips of different types of oak (American and French) obtained from a cooper and subjected to various treatments. Only the 65% concentration level considered in the statistical evaluation. Refer to part I in this series for more detail regarding the various treatments. Error bars represent the standard error of the mean.

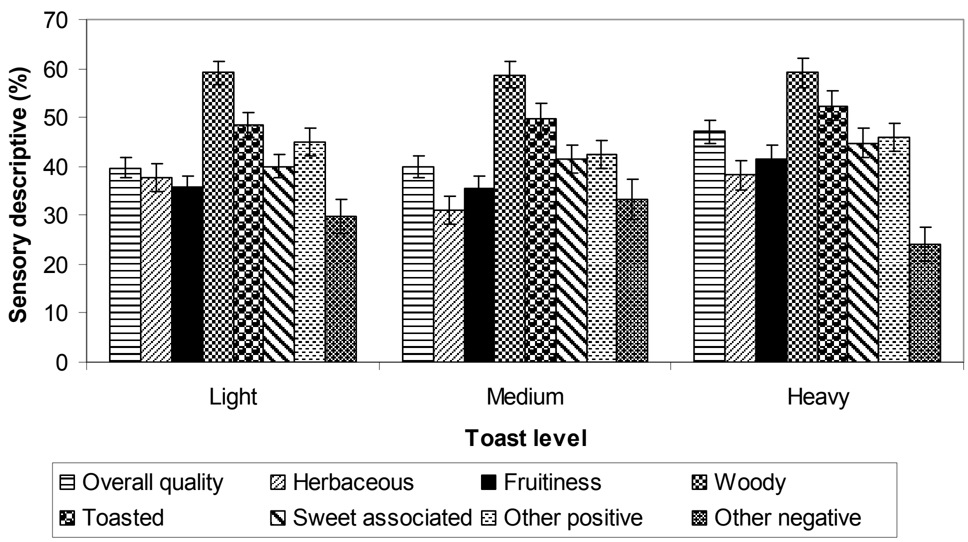

FIGURE 6

Overall sensory quality and other sensory descriptors of eight-month matured pot-still brandy from oak extracts prepared from French oak chips obtained from a commercial supplier and subjected to various treatments. Oak toast levels: Light, medium and heavy. Only the 65% concentration level was considered in the statistical evaluation. Refer to part I in this series for more detail regarding the various treatments. Error bars represent the standard error of the mean.

TABLE 2

High-performance Liquid Chromatographic determination of unmatured pot-still brandy prepared from oak (cooper only) extracts.

Type/Treatment	n*	Mean (mg/L)				
		Gallic acid	Catechin	Vanillic acid	Syringic acid	*p*-Coumaric acid
Untoasted	12	6.708[a] (1.237)**	6.352[a] (1.928)	0.074[c] (0.074)	0.898[c] (0.125)	0.287[a] (0.132)
Medium	12	5.910[ab] (1.078)	1.451[b] (0.631)	1.572[b] (0.254)	3.578[b] (0.462)	0.343[a] (0.166)
Heavy	12	4.623[b] (0.781)	0.913[b] (0.633)	2.279[a] (0.220)	5.589[a] (0.496)	0.359[a] (0.172)
		Syringaldehyde	*m*-Coumaric acid	Ellagic acid	Coniferaldehyde	Sinapaldehyde
Untoasted	12	1.000[c] (0.305)	0.469[a] (0.096)	20.316[b] (2.752)	2.711[b] (1.610)	11.006[b] (2.806)
Medium	12	6.754[b] (0.942)	0.141[b] (0.051)	39.497[a] (9.920)	10.922[a] (1.741)	36.568[a] (6.479)
Heavy	12	9.851[a] (1.006)	0.099[b] (0.037)	44.198[a] (11.252)	10.403[a] (1.574)	40.258[a] (6.460)

*Number of evaluations of samples; **standard error of the mean. Treatments with the same superscript within columns do not differ significantly (p ≥ 0.05). Data representative of extracts concentrated by 65% (v/v).

TABLE 3

Gas Chromatographic analysis of eight-month matured pot-still brandy prepared using oak extracts from different species of oak chips specially prepared by a cooper.

Type/ Treatment	n*	Mean (mg/L)						
		Furfural	5-Methyl-furfural	Guaiacol	*trans*-oak Lactone	*cis*-oak Lactone	Phenol	
Untoasted	32	14.313[b] (0.343)**	0.024[b] (0.017)	0.502[a] (0.061)	0.074[a] (0.044)	0.174[a] (0.065)	0.018[a] (0.018)	
Medium	32	24.042[a] (2.096)	1.546[a] (0.283)	0.559[a] (0.053)	0.000[b] (0.000)	0.095[ab] (0.040)	0.000[a] (0.000)	
Heavy	32	22.656[a] (1.772)	1.333[a] (0.250)	0.545[a] (0.052)	0.000[b] (0.000)	0.041[b] (0.029)	0.000[a] (0.000)	
		o-Cresol	Ethyl guaiacol	*p*-Cresol	Eugenol	2,6-Dimethoxy-phenol	5-Hydroxy-methyl furfural	Vanillin
Untoasted	32	0.007[a] (0.007)	2.339[b] (0.112)	0.017[a] (0.017)	0.257[a] (0.064)	34.962[b] (0.692)	0.849[c] (0.265)	1.107[c] (0.491)
Medium	32	0.000[a] (0.000)	2.532[a] (0.044)	0.000[a] (0.000)	0.184[ab] (0.036)	36.021[a] (0.560)	5.812[a] (0.318)	3.886[b] (0.264)
Heavy	32	0.000[a] (0.000)	2.499[a] (0.038)	0.000[a] (0.000)	0.147[b] (0.029)	35.990[a] (0.477)	5.095[b] (0.249)	5.124[a] (0.293)

*Number of evaluations of samples; **standard error of the mean. Treatments with the same superscript within columns do not differ significantly (p ≥ 0.05). Data representative of extracts concentrated by 65% (v/v).

from toasted chips, were generally higher than from lightly or untoasted chips (Tables 5 and 6).

In previous studies conducted at ARC Infruitec-Nietvoorbij (van Jaarsveld, 2003), where a different method of extraction was used, similar results were obtained. Results showed pot-still brandies prepared from toasted oak chips to be sensorially more acceptable, with higher concentrations of congeners, than pot-still brandies prepared from untoasted chips. With the exception of 1,1-diethoxypropan-2-one, all congeners analysed by means of GC-MS at Stellenbosch University showed increased concentrations with toasting. Congeners included vanillin, acetovanillone, syringaldehyde, coniferaldehyde, sinapaldehyde, eugenol, *cis*-oak lactone, *trans*-oak lactone, ethyl syringate, ethylhomovanillate, ethylhomosyringate, 2-phenylethanol, 2-phenylethylacetate and diethyl succinate. In these previous collaborative studies, oak chips were placed in 55% (v/v) neutral wine spirits at 100 g/L for three months. After three months, extracts were added to 70% (v/v) unmatured pot-still brandy and aged for sixteen months at 20°C. Samples were drawn at six and twelve months for analysis. Ethylhomovanillate and ethylhomosyringate (Figs 7 & 8) were identified as important aroma compounds in mature brandy in this study.

General

Coopering processes such as toasting of wood, and wetting when heating the staves, have a substantial effect on the sensory characteristics, as well as on the various flavour compounds

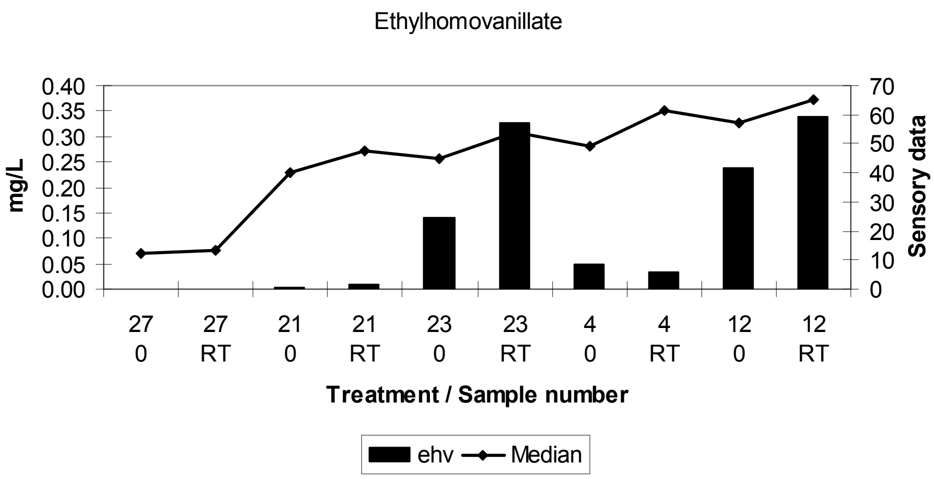

FIGURE 7

Median rating for the sensory overall impression plotted against the GC-MS determined concentration of ethylhomovanillate in matured pot-still brandy samples. Oak chips were placed in 55% (v/v) neutral wine spirits at 100 g/L for three months. After three months, extracts were added to 70% (v/v) unmatured pot-still brandy and aged for sixteen months at 20°C. Sample number 4 represent American oak toasted at 220°C; sample 12, French oak toasted at 230°C; sample 21, untoasted American oak from Columbit (code 45006 – “oak barrels”); sample 23, toasted American oak (code 56106, Columbit), and sample number 27, untoasted American oak from The Wine Cask, treated at high temperature. Samples 4 and 12 were prepared with oak extracts from oak staves obtained from overseas and toasted, chopped and milled in South Africa. Ehv, ethylhomovanillate concentration.

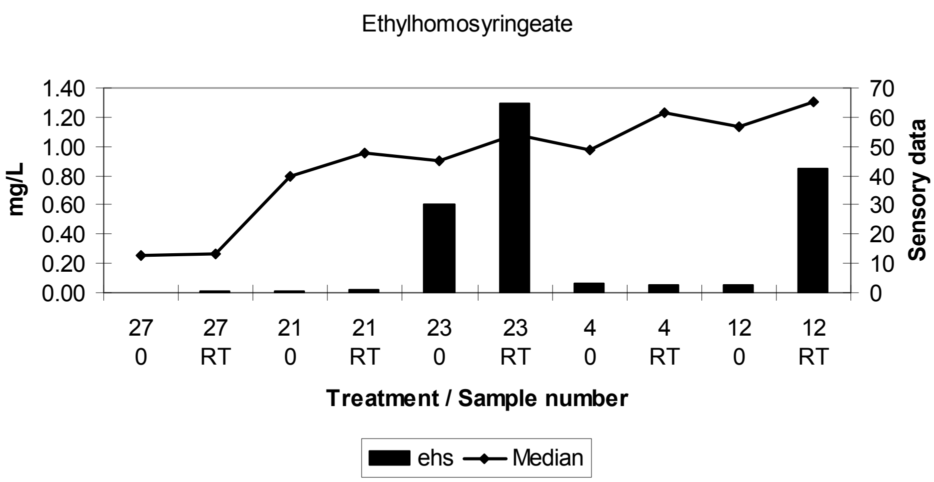

FIGURE 8

Median rating for the sensory overall impression plotted against the GC-MS determined concentration of ethylhomosyringeate in matured pot-still brandy samples. Oak chips were placed in 55% (v/v) neutral wine spirits at 100 g/L for three months. After three months, extracts were added to 70% (v/v) unmatured pot-still brandy and aged for sixteen months at 20°C. Sample number 4 represent American oak toasted at 220°C; sample 12, French oak toasted at 230°C; sample 21, untoasted American oak from Columbit (code 45006 – “oak barrels”); sample 23, toasted American oak (code 56106, Columbit), and sample number 27, untoasted American oak from The Wine Cask, treated at high temperature. Samples 4 and 12 were prepared with oak extracts from oak staves obtained from overseas and toasted, chopped and milled in South Africa. Ehs, ethylhomosyringeate concentration.

TABLE 4

Gas Chromatographic analysis of eight-month matured pot-still brandy prepared with extracts from chips obtained from a commercial supplier.

Type/Treatment	n*	Mean (mg/L)						
		Furfural	5-Methyl-furfural	Guaiacol	*trans*-oak Lactone	*cis*-oak Lactone	*o*-Cresol	Phenol
Light	16	15.209^{c} (0.336)**	0.179^{b} (0.062)	0.576^{a} (0.074)	0.000^{a} (0.000)	0.000^{a} (0.000)	nd***	nd
Medium	16	16.829^{a} (0.644)	0.521^{a} (0.139)	0.535^{a} (0.068)	0.146^{a} (0.102)	0.104^{a} (0.104)	nd	nd
Heavy	12	16.012^{b} (0.636)	0.477^{a} (0.105)	0.554^{a} (0.083)	0.128^{a} (0.128)	0.165^{a} (0.165)	nd	nd

Type/Treatment	n*	Ethyl guaiacol	*p*- Cresol	Eugenol	2,6-Dimethoxy-phenol	5-Hydroxy-methyl furfural	Vanillin
Light	16	2.614^{a} (0.049)	nd	0.322^{a} (0.169)	36.944^{a} (0.733)	3.967^{c} (0.280)	4.042^{b} (0.332)
Medium	16	2.466^{ab} (0.107)	nd	0.221^{a} (0.069)	35.748^{b} (0.869)	5.473^{a} (0.419)	8.166^{a} (0.920)
Heavy	12	2.350^{b} (0.142)	nd	0.243^{a} (0.090)	35.528^{b} (0.698)	4.994^{b} (0.408)	8.627^{a} (0.964)

*Number of evaluations of samples; **standard error of the mean; ***not detected. Treatments with the same superscript within columns do not differ significantly ($p \geq 0.05$). Data representative of French oak extracts concentrated by 65% (v/v).

TABLE 5

High-performance Liquid Chromatographic analysis of eight-month matured pot-still brandy prepared from oak (cooper only) extracts.

Type/Treatment	n*	Mean (mg/L)					
		Gallic acid	Protocatechuic acid	*p*-Hydroxy-benzoic acid	Catechin	Vanillic acid	Syringic acid
Untoasted	32	4.482^{a} (0.749)**	0.022^{b} (0.022)	0.838^{a} (0.220)	1.044^{b} (0.239)	1.092^{b} (0.317)	0.261^{c} (0.029)
Medium	32	4.393^{a} (0.667)	0.803^{a} (0.203)	0.244^{b} (0.091)	1.942^{a} (1.352)	1.267^{b} (0.139)	3.063^{b} (0.262)
Heavy	32	3.488^{b} (0.497)	0.650^{a} (0.205)	0.104^{b} (0.060)	0.621^{b} (0.307)	1.904^{a} (0.180)	4.894^{a} (0.308)
		p-Coumaric acid	Syringaldehyde	*m*-Coumaric acid	Ellagic acid	Coniferaldehyde	Sinapaldehyde
Untoasted	32	0.022^{a} (0.015)	1.678^{c} (0.154)	0.065^{a} (0.021)	0.407^{b} (0.120)	18.629^{b} (1.884)	0.477^{c} (0.171)
Medium	32	0.000^{a} (0.000)	10.973^{b} (0.920)	0.000^{b} (0.000)	7.243^{a} (0.653)	31.231^{a} (5.508)	21.465^{b} (2.179)
Heavy	32	0.000^{a} (0.000)	14.893^{a} (0.926)	0.000^{b} (0.000)	6.753^{a} (0.586)	34.663^{a} (6.049)	24.048^{a} (2.264)

*number of evaluations of samples; **standard error of the mean. Treatments with the same superscript within columns do not differ significantly ($p \geq 0.05$). Data representative of extracts concentrated by 65% (v/v).

imparted into wine and spirits (Singleton & Draper, 1961; Case & van Wyk, 1989; Litchev, 1989; Miller *et al.*, 1992; Sefton & Spillman, 1995; Singleton, 1995; Matricardi & Waterhouse, 1999). Considering the variables heating, wood origin, seasoning and country or region where seasoning take place, heating is the most important, producing the greatest sensory change (Sefton *et al.*, 1993). Few studies report the opposite. One such conflicting study (Francis *et al.*, 1992) reported that coopering practices such as the use of steam or fire heating for stave bending had little to no effect on beverage character. Heating will also influence the structure of the wood, forming small cracks on the inner surface of the stave, allowing brandy to penetrate deeper into the wood and extracting more compounds (László, 1995). The influence of heating on extraction of wood compounds and the universal differences in the definition of toasting levels (Matricardi & Waterhouse, 1999), may explain minor variations in chemical and sensorial behavioural trends sometimes observed between preparations with the same toast level obtained from a cooper and a commercial supplier.

In general, heating increased the intensity of the "woody", "toasted", "sweet associated" and "other positive" aromas (Figs 5 and 6). Similar effects of heating on sensory descriptors (i.e. smooth, vanilla, sweet, buttery, caramel, cedar, nutty, malty, spicy, fruity and floral) were reported by Clyne *et al.* (1993) and Singleton (1995). Francis *et al.* (1992) reported a major impact on aroma due to heat treatment and enhancement of highly rated vanilla, caramel, buttery, nutty and cedar attributes, as well as decreased raisin character. Generally, increases in the concentrations of oak lactones (ageing aroma substances derived from oak wood and accumulating in distilled liquor during the ageing process) were observed upon toasting in unmatured pot-still brandy (specifically the *cis*-isomeric form) (Table 1). As reported by Otsuka *et al.* (1974), higher concentrations of lactones coincided with improved quality (Table 1, Figs 1 to 4). Both increased, and decreased ($p \geq 0.05$) concentrations of *cis*- and *trans*-isomeric forms of oak lactones were observed in eight-month matured pot-still brandy prepared with extracts from chips obtained from a commercial supplier and a cooper, respectively (Tables 3 and 4). Conflicting reports, with increases, decreases and insignificant effects of concentration upon toasting have been reported (Sefton & Spillman, 1995; Singleton, 1995; Chatonnet, 1999; Godden *et al.*, 1999; Spillman *et al.*, 2004). Conflicting reports might be explained by the fact that, depending on the oak species, toasting has more of an effect on the rate of extraction than on the concentration as such, possibly due to a decrease in porosity of the wood (Godden *et al.*, 1999). Oak wood with small amounts of *cis*-oak lactone rely most on coopering heat for aroma generation, generally benefiting from medium toast and deep heating (Sefton & Spillman, 1995). Although concentrations of oak lactones generally increased with the level of toasting, heavy toasting resulted in some loss of these compounds (Tables 1, 3 and 4), as was also reported by Spillman *et al.* (2004).

The aromatic aldehydes, vanillin, furfural, 5-methylfurfural and 5-hydroxymethylfurfural and phenolic compound guaiacol, are primarily formed in wood during the toasting process. The concentrations of these compounds in wood and corresponding alcoholic beverages reflect the intensity of the toasting process (Sefton *et al.*, 1993; Canas *et al.*, 1999; Chatonnet, 1999; Spillman *et al.*, 2004), showing increased concentrations in brandy upon toasting (Tables 1, 3 and 4). As also reported by Godden *et al.* (1999), higher concentrations of guaiacol were generally associated with increased woody and toasty, and decreased fruity characters of matured pot-still brandy (Table 3; Fig. 5). The variations observed in vanillin concentration are, to some extent, due to the heat treatment applied by coopers/producers. Vanillin is known to be an important flavour compound in barrel-aged wines (Francis *et al.*, 1992; Clyne *et al.*, 1993; Sefton & Spillman, 1995). As reported by Chatonnet (1999) and Hale *et al.* (1999), volatile substances (i.e. caramelisation products such as furfural, 5-methylfurfural, 5-hydroxymethylfurfural) with a "toasty" aroma increased significantly and reached a maximum at medium toast, after which their concentration decreased rapidly at heavy toast, but still remained higher than untoasted and lightly toasted chips (Tables 3 & 4). This trend can be explained by the fact that heat treatment of oak is mainly associated with decreased hemicelluloses and increased furfural derivatives, unless the temperature is very high, in which case the furfurals polymerise, presumably with the lignin to produce phenol-aldehyde insoluble resins (Singleton, 1995; Matricardi & Waterhouse, 1999). The level of barrel toasting is more of a determining factor than oak source with regard to vanillin concentration (Godden *et al.*, 1999). The decrease of furanic derivatives in pot-still brandy prepared with heavy toasted oak wood could be attributed to their degradation and volatilisation at high temperatures during toasting (Canas *et al.*, 1999). 5-Hydroxymethylfurfural and 5-methylfurfural are derived from the hexoses of cellulose and furfural from the pentoses, which are the main constituents of hemicelluloses. That furfural exists in distilled liquid and that hemicelluloses are the most thermo sensitive polymers in wood, which are preferentially degraded during heat treatment, contributes to make furfural one of the main flavour constituents in toasted oak wood and in the corresponding brandies (Canas *et al.*, 1999) (Tables 1, 3 and 4).

The furanic derivatives, vanillic acid, syringic acid, ellagic acid and the phenolic compounds syringaldehyde, coniferaldehyde, sinapaldehyde and protochatechuic acid have also been found to increase in concentration upon toasting, or as the level of toasting increased (Tables 2, 5 and 6). Increases in ellagic acid (Matricardi & Waterhouse, 1999), vanillic acid (Canas *et al.*, 1999; Matricardi & Waterhouse, 1999), eugenol (Sefton *et al.*, 1993) and syringaldehyde (Clyne *et al.*, 1993) contents upon heat treatment have also been reported. Ellagic acid concentrations increased as a result of the degradation of ellagitannins (Matricardi & Waterhouse, 1999). Increases in syringic acid content are explained by concomitant increases in its main precursor, syringaldehyde (Tables 2, 5 and 6) (Canas *et al.*, 1999). The syringaldehyde:vanillin (Table 7a), gallic acid:vanillin (Table 7b), vanillic acid:vanillin (Table 7c) and syringic:vanillic acid (Table 7d) ratios in eight-month matured pot-still brandies generally increased as the level of toasting increased, particularly from untoasted and lightly toasted to medium toasted, with a decrease occasionally observed at higher toasting. Decreased gallic acid:vanillin and vanillic acid:vanillin ratios with increased toasting were evident for samples prepared from French oak from a commercial supplier. Puech (1988) reported syringic acid:vanillic acid ratios of 1.5 for Armagnac brandies, 1.1 to 7.17 for liquid wood extracts, and 1.6 to 3.5 for extracts in powder form. Syringaldehyde:vanillin ratios of 1.4 to 2.5, 1 and 1.90 to 2.76 (mean 2.41 ± 0.24), gallic acid:vanillin ratios of 0.09 to 0.27

(mean 0.17 ± 0.05) and vanillic acid:vanillin ratios close to one have been reported for Armagnac and wine spirit mixtures (Puech, 1981; Giménez Martínez *et al.*, 2001). A syringaldehyde:vanillin ratio of 1.4 to 2.5 reflects a balanced lignin composition and a ratio of one the lowest limit of this ratio (Giménez Martínez *et al.*, 2001). The effects of thermal treatment such as heating or toasting on the hydrolysis of oak wood, solubility, and the increase of various substances, i.e. aromatic aldehydes, phenolic compounds, acetals, acids and sugars, have been reported by Baldwin *et al.* (1967), Litchev (1989) and Miller *et al.* (1992). Matricardi & Waterhouse (1999) reported a reduction in or the loss of phenolic components upon toasting. Clyne *et al.* (1993) reported significantly lower

TABLE 6

High-performance Liquid Chromatographic analysis of eight-month matured pot-still brandy prepared from oak (commercial supplier only) extracts.

Type/Treatment	**n***	**Mean (mg/L)**					
		Gallic acid	**Protocatechuic acid**	***p*-Hydroxy-benzoic acid**	**Catechin**	**Vanillic acid**	**Syringic acid**
Light	16	5.047^{b} (0.150)**	0.000^{c} (0.000)	1.139^{a} (0.336)	2.593^{a} (0.545)	2.434^{a} (0.573)	1.895^{c} (0.113)
Medium	14	6.131^{a} (0.418)	0.864^{b} (0.395)	0.404^{b} (0.218)	2.862^{a} (1.002)	1.230^{b} (0.303)	5.775^{b} (0.629)
Heavy	12	4.524^{b} (0.264)	1.992^{a} (0.605)	0.452^{b} (0.193)	2.053^{a} (0.903)	1.173^{b} (0.376)	6.458^{a} (0.511)
		***p*-Coumaric acid**	**Syringaldehyde**	***m*-Coumaric acid**	**Ellagic acid**	**Coniferaldehyde**	**Sinapaldehyde**
Light	16	nd***	10.970^{b} (0.852)	0.039^{a} (0.018)	2.389^{c} (0.332)	52.558^{b} (6.277)	3.078^{c} (0.406)
Medium	14	nd	28.352^{a} (3.312)	0.017^{a} (0.017)	5.252^{b} (0.861)	82.359^{a} (16.198)	16.372^{b} (2.703)
Heavy	12	nd	28.027^{a} (2.644)	0.000^{a} (0.000)	6.689^{a} (0.829)	73.533^{a} (12.632)	19.734^{a} (2.800)

*Number of evaluations of samples; **standard error of the mean; ***not detected. Treatments with the same superscript within columns do not differ significantly ($p \geq 0.05$). Data representative of French oak extracts concentrated by 65% (v/v).

TABLE 7a

Syringaldehyde:vanillin ratios in matured pot-still brandies.

Supplier	**Type of oak**	**Toast level**	**n***	**Syringaldehyde:Vanillin ratio**		
				Minimum	**Maximum**	**Mean**
Cooper	American oak	Untoasted	4	0.30	0.78	0.55 (0.13)**
		Medium	16	1.66	4.08	2.62 (0.17)
		Heavy	16	2.00	4.19	2.80 (0.15)
	French oak	Untoasted	5	0.00	1.20	0.57 (0.29)
		Medium	16	1.63	6.39	3.08 (0.31)
		Heavy	16	1.94	5.64	3.12 (0.25)
Commercial supplier	French oak	Light	16	1.98	3.50	2.74 (0.09)
		Medium	14	2.58	6.77	3.57 (0.32)
		Heavy	12	2.72	4.75	3.34 (0.19)

*Number of evaluations of samples; **standard error of the mean. Data representative of brandies prepared with extracts from either French or American oak chips of different toast levels obtained from either a cooper or a commercial supplier. Extracts were prepared by boiling under reflux of oak chips in either water of ethanol medium, followed by either open or reduced pressure concentration (by 65%, v/v) and fortification. Samples were stored for eight months in glass containers at room temperature and below 0°C (control).

TABLE 7b

Gallic acid:vanillin ratios in matured pot-still brandies.

Supplier	Type of oak	Toast level	n*	Gallic acid:Vanillin ratio		
				Minimum	Maximum	Mean
Cooper	American oak	Untoasted	4	0.06	0.37	0.20 (0.07)**
		Medium	16	0.01	0.77	0.34 (0.05)
		Heavy	16	0.06	0.27	0.17 (0.02)
	French oak	Untoasted	4	0.03	4.04	1.80 (0.92)
		Medium	16	0.82	3.51	1.90 (0.17)
		Heavy	16	0.54	1.94	1.24 (0.10)
Commercial supplier	French oak	Light	16	0.80	2.24	1.37 (0.11)
		Medium	14	0.41	1.17	0.83 (0.06)
		Heavy	12	0.33	0.87	0.58 (0.05)

*Number of evaluations of samples; **standard error of the mean. Data representative of brandies prepared with extracts from either French or American oak chips of different toast levels obtained from either a cooper or a commercial supplier. Extracts were prepared by boiling under reflux of oak chips in either water of ethanol medium, followed by either open or reduced pressure concentration (by 65%, v/v) and fortification. Samples were stored for eight months in glass containers at room temperature and below 0°C (control).

TABLE 7c

Vanillic acid:vanillin ratios in matured pot-still brandies.

Supplier	Type of oak	Toast level	n*	Vanillic acid:Vanillin ratio		
				Minimum	Maximum	Mean
Cooper	American oak	Untoasted	4	0.00	0.00	0.00 (0)**
		Medium	16	0.09	1.01	0.37 (0.06)
		Heavy	16	0.11	0.67	0.35 (0.05)
	French oak	Untoasted	5	0.00	0.04	0.01 (0.01)
		Medium	16	0.10	0.61	0.32 (0.04)
		Heavy	16	0.13	0.89	0.45 (0.06)
Commercial supplier	French oak	Light	16	0.00	1.32	0.63 (0.15)
		Medium	14	0.00	0.54	0.19 (0.05)
		Heavy	12	0.02	0.68	0.20 (0.08)

*Number of evaluations of samples; **standard error of the mean. Data representative of brandies prepared with extracts from either French or American oak chips of different toast levels obtained from either a cooper or a commercial supplier. Extracts were prepared by boiling under reflux of oak chips in either water of ethanol medium, followed by either open or reduced pressure concentration (by 65%, v/v) and fortification. Samples were stored for eight months in glass containers at room temperature and below 0°C (control).

concentrations of vanillic acid, coniferaldehyde and sinapaldehyde in whiskey stored for three years in charred, as opposed to uncharred, American oak barrels. Other compounds that were lost or decreased in concentration with higher toast levels were eugenol, *o*-cresol, phenol, ethyl guaiacol, *p*-cresol, gallic acid, catechin and *m*-coumaric acid (Tables 1-6). Degradation of gallic acid upon toasting was also reported by Matricardi & Waterhouse (1999) and Giménez Martínez *et al.* (2001). Giménez Martínez *et al.* (2001) reported a decrease in gallic acid concentration with heating times above 185°C, thus explaining the decreases observed in the present study with heavy toast performed at 200°C for 150 minutes. Hale *et al.* (1999) reported similar behaviour for ellagic acid in their study, with maximum concentrations for medium toasted samples, with somewhat lower concentrations in heavy toasted samples (Table 5).

Heat treatment, therefore, has a significant influence on the majority of low molecular weight extractable compounds. Medium toast yielded the highest concentration of extractables, with light toast less effective and with the heat generated in heavy toast destroying a portion of the compounds (Hale *et al.*, 1999).

TABLE 7d

Syringic acid:vanillic acid ratios in matured pot-still brandies.

Supplier	Type of oak	Toast level	n*	Syringic:Vanillic acid ratio		
				Minimum	Maximum	Mean
Cooper	American oak	Untoasted		–	–	–
		Medium	16	0.96	9.17	3.24 (0.62)**
		Heavy	16	1.30	9.25	3.70 (0.65)
	French oak	Untoasted	11	0.00	0.38	0.14 (0.04)
		Medium	16	0.82	5.52	3.09 (0.34)
		Heavy	16	0.91	5.44	2.89 (0.33)
Commercial supplier	French oak	Light	13	0.34	7.25	2.22 (0.71)
		Medium	13	1.02	17.19	8.11 (1.47)
		Heavy	12	1.08	27.54	13.85 (2.79)

*Number of evaluations of samples; **standard error of the mean. Data representative of brandies prepared with extracts from either French or American oak chips of different toast levels obtained from either a cooper or a commercial supplier. Extracts were prepared by boiling under reflux of oak chips in either water of ethanol medium, followed by either open or reduced pressure concentration (by 65%, v/v) and fortification. Samples were stored for eight months in glass containers at room temperature and below 0°C (control).

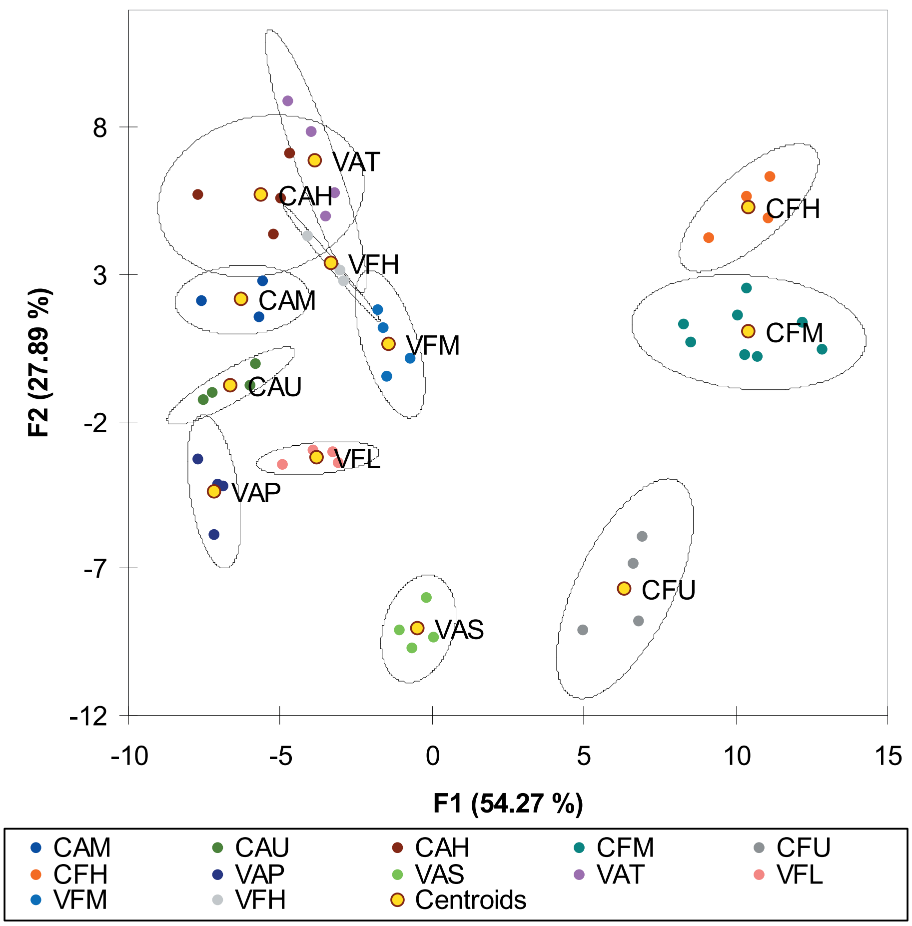

FIGURE 9

Plot of discriminant scores of the first two discriminant factors (F1, F2) of unmatured extract/pot-still brandies for the treatment classes CFU, CFM, CFH, CAU, CAM, CAH, VFL, VFM, VFH, VAS, VAP and VAT, defined by the variables percent acceptability, furfural, 5-methylfurfural, guaiacol, *trans*-oak lactone, *cis*-oak lactone, eugenol, 2,6-dimethoxyphenol, 5-hydroxymethyl furfural, vanillin, gallic acid, catechin, vanillic acid, syringic acid, *p*-coumaric acid, syringaldehyde, *m*-coumaric acid, ellagic acid, coniferaldehyde and sinapaldehyde. Abbreviations: CFU, cooper, French oak, untoasted; CFM, cooper, French oak, medium toast; CFH, cooper, French oak, heavy toast; CAU, cooper, American oak, untoasted; CAM, cooper, American oak, medium toast; CAH, cooper, American oak, heavy toasted; VFL, commercial supplier, French oak, light toast; VFM, commercial supplier, French oak, medium toast; VFH, commercial supplier, French oak, heavy toast; VAS, commercial supplier, American oak, special toast; VAP, commercial supplier, American oak, premium toast and VAT, commercial supplier, American oak, toasted.

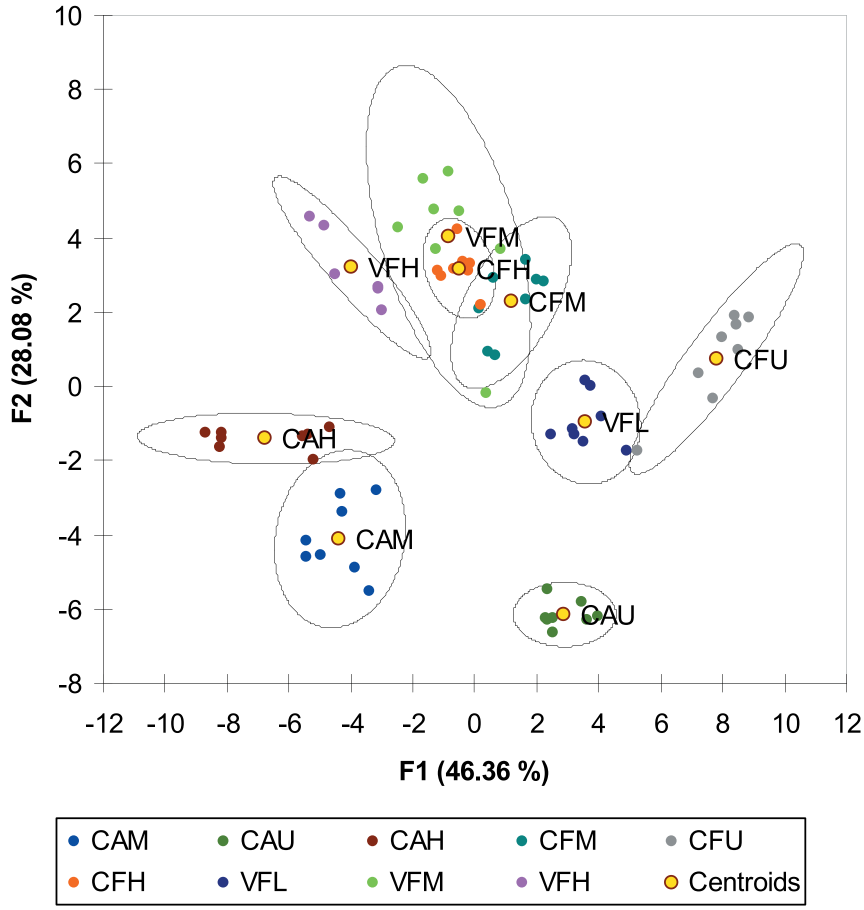

FIGURE 10

Plot of discriminant scores of the first two discriminant factors (F1, F2) of eight-month matured pot-still brandies for the treatment classes CFU, CFM, CFH, CAU, CAM, CAH, VFL, VFM and VFH, defined by the variables herbaceous, woody, overall quality. 5-methylfurfural, 2,6-dimethoxyphenol, 5-hydroxymethyl furfural, gallic acid, syringic acid, syringaldehyde, ellagic acid, coniferaldehyde, sinapaldehyde and *p*-hydroxybenzoic acid. Abbreviations: CFU, cooper, French oak, untoasted; CFM, cooper, French oak, medium toast; CFH, cooper, French oak, heavy toast; CAU, cooper, American oak, untoasted; CAM, cooper, American oak, medium toast; CAH, cooper, American oak, heavy toasted; VFL, commercial supplier, French oak, light toast; VFM, commercial supplier, French oak, medium toast and VFH, commercial supplier, French oak, heavy toast.

Medium strong toast is usually the recommended and/or specified toasting level (Hacker, 1991; Sefton & Spillman, 1995). Medium toast is recommended because it yields high quality products, and chemical breakdown or decreased concentrations of aroma compounds is observed at higher toasting levels. Higher toasting levels can also lead to charring and a coal taste that does not disappear with maturation. Intense toasting also promotes the formation of unwanted polycyclic hydrocarbons, resulting in the immediate degradation and loss of certain compounds (Tables 1 to 6). Economic advantages relate to less time and fewer materials spent on medium instead of high toasting. At low temperatures (light toasting), the pyrolysis of lignin is too slow and in untoasted oak the tannins are broken down slowly. The influence of toasting on the release of low molecular weight extractable compounds in brandy, and on subsequent sensory profiles, can be explained by (1) the increase in wood permeability caused by the fragmentation of the cell structures and reorganisation of the lignocellulose network, resulting in improved access of brandy to wood extraction sites, (2) the degradation of tannins, lignins, hemicelluloses and cellulose, and (3) the formation of heat treatment compounds, i.e. 5-hydroxymethylfurfural and 5-methylfurfural and their release into the brandy.

Discriminant analysis provided separation of sample groups representing the different toast levels. Pot-still brandy samples are grouped together into three clusters. Clusters related to the toast levels are separated mainly by the second discriminant function, whereas clusters related to oak, in particular French oak, are separated by the first discriminant function, expressing 28% and 54% in unmatured, and 28 and 46% of the variance in matured samples, respectively. Some heterogeneity in the boundary area between some of the clusters could be observed (Figs 9 and 10).

CONCLUSIONS

The treatments that yielded the highest observed quality were those that made use of toasted oak as opposed to untoasted oak. There is a definite relationship between treatment, wood-derived congener concentrations and pot-still brandy quality. Although recommendations regarding single treatments can be made, it must be borne in mind that it is not always a single treatment, but a combination of various practices, that yield the best quality products.

Medium toast is the recommended toasting strength. Medium toast yields high quality products and minimises chemical breakdown of aroma compounds, relative to heavy toast. Higher toasting levels can also lead to charring, the formation of unwanted polycyclic hydrocarbons, and immediate degradation and loss of flavour compounds. Certain economic advantages accrue from the use of medium instead of high toasting levels. These mainly relate to reduced time and materials usage.

LITERATURE CITED

Baldwin, S., Black, R.A., Andreasen, A.A. & Adams, S.L., 1967. Aromatic congener formation in maturation of alcoholic distillates. J. Agr. Food Chem. 15, 381-385.

Bosso, A., Panero, L. & Follis, R., 2004. The influence of wood chips utilisation before and after alcoholic fermentation on wines sensory characteristics. Riv. Vitic. Enol. 57, 33-62.

Canas, S., Leandro, M.C., Spranger, M.I. & Belchior, A.P., 1999. Low molecular weight organic compounds of chestnut wood (*Castanea sativa* L.) and corresponding aged brandies. J. Agric. Food Chem. 47, 5023-5030.

Caputo, T., 2004. Getting barrel toast levels right. Wines & Vines 85, 26-35.

Case, M. & van Wyk, C.J., 1989. Die effek van die oorsprong en die roosterintensiteit op die aroma samestelling en aanvaarbaarheid van eikehout vir wynveroudering. Wynboer, November, 6.

Chatonnet, P., 1999. Volatile and odoriferous compounds in barrel-aged wines: impact of cooperage techniques and aging conditions. In: Waterhouse, A.L. & Ebeler, S.E., (eds). Chemistry of Wine Flavor. 1999 - ACS Symposium Series no. 714. American Chemical Society, Washington D.C. pp. 180-207.

Clyne, J., Conner, J.M., Paterson, A. & Piggott, J.R., 1993. The effect of cask charring on Scotch whisky maturation. Int. J. Food Sci. Tech. 28, 69-81.

Dennison, C., 1999. A version of the invention of barrels and barrel alternatives. Am. J. Enol. Vitic. 50, 539-540.

Francis, I.J., Sefton, M.A. & Williams, P.J., 1992. A Study by sensory descriptive analysis of the effects of oak origin, seasoning, and heating on the aromas of oak model wine extracts. Am. J. Enol. Vitic. 43, 23-30.

Giménez Martínez, R., López García De La Serrana, H., Villalón Mir, M., Navarro Alarcón, M., Olalla Herrera, M., Cabrera Vique, C. & López Martínez, M.C., 2001. Study of vanillin syringaldehyde and gallic acid content in oak wood and wine spirits mixtures: Influence of heat treatment and chip size. J. Wine Res. 12, 175-182.

Godden, P.W., Pollnitz, A.P., Osicka, S., Sykes, M., Liacopoulos, D., Pardon, K.H., Spillman, P.J., Gawel, R., Jones, G.P., Skluoumounis, G.K. & Sefton, M.A., 1999. Measuring desirable oakwood components in wine. In: Blair, R.J., Sas, A.N., Hayes, P.F. & Høj, P.B. (eds). Proc. 10th Australian Wine Ind. Tech. Conf., 2-5 August 1998, Sydney, New South Wales, Australia. pp. 121-124.

Hacker, W.F., 1991. Die houtvat – sy eienskappe en invloed op wyn. Wynboer, October, 6-7.

Hale, M.D., McCafferty, K., Larmie, E., Newton, J. & Swan, J.S., 1999. The influence of oak seasoning and toasting parameters on the composition and quality of wine. Am. J. Enol. Vitic. 50, 495-502.

László, J., 1995. Wine-maturation in oak barrels in South Africa. Wynboer, September, 40-44.

Litchev, V., 1989. Influence of oxidation processes on the development of the taste and flavor of wine distillates. Am. J. Enol. Vitic. 40, 31-35.

Matricardi, L. & Waterhouse, A.L., 1999. Influence of toasting technique on color and ellagitannins of oak wood in barrel making. Am. J. Enol. Vitic. 50, 519-526.

Miller, D.P., Howell, G.S., Michaelis, C.S. & Dickmann, D.I., 1992. The content of phenolic acid and aldehyde flavor components of white oak as affected by site and species. Am. J. Enol. Vitic. 43, 333-338.

Mosedale, J.R. & Puech, J-L., 1998. Wood maturation of distilled beverages. Trends in Food Sci. and Technol. 9, 95-101.

Otsuka, K-I., Zenibayashi, Y., Itoh, M. & Totsuka, A., 1974. Presence and Significance of Two Diastereomers of β-Methyl-γ-octalactone in Aged Distilled Liquors. Agr. Biol. Chem. 38, 485-490.

Puech, J-L., 1981. Extraction and evolution of lignin products in Armagnac matured in oak. Am. J. Enol. Vitic. 32, 111-114.

Puech, J-L., 1988. Phenolic compounds in oak wood extracts used in the ageing of brandies. J. Sci. Food Agric. 42, 165-172.

SAS, 2000. SAS/STAT Users Guide, Version 8, First Edition, Volume 2. SAS Institute Inc., Cary, NC, USA.

Sefton, M.A. & Spillman, P.J., 1995. The influences of oak, coopering heat and microbial activity on oak-derived wine aroma. In: Stockley, C.S., Sas, A.N., Johnstone, R.S. & Lee, T.H. (eds.). Proc. 9th Aust. Wine Ind. Tech. Conf., 16-19 July 1995, Adelaide, South Australia. pp. 66-71.

Sefton, M.A., Spillman, P.J., Pocock, K.F., Francis, L.L. & Williams, P.J., 1993. The influence of oak origin, seasoning, and other industry practices on the sensory characteristics and composition of oak extracts and barrel-aged white wines. Aust. Grapegrow. Winemaker No. 355, July, 17-25.

Shapiro, S.S. & Wilk, M.B., 1965. An analysis of Variance Test for Normality (complete samples), Biometrika 52, 591-611.

Singleton, V.L., 1995. Maturation of wines and spirits: comparisons, facts, and hypothesis. Am. J. Enol. Vitic. 46, 98-115.

Singleton, V.L. & Draper, D.E., 1961. Wood chips and wine treatment: The nature of aqueous alcohol extracts. Am. J. Enol. Vitic. 12, 152-158.

Spillman, P.J., Sefton, M.A. & Gawel, R., 2004. The effect of oak wood source, location of seasoning and cooperage on the composition of volatile compounds in oak-matured wines. Aust. J. Grape Wine Res. 10, 216-226.

Van Jaarsveld, F.P., 2003. Developing techniques to rapidly induce the needed ageing character in brandy products. Project WW 09/05, 2002/2003-Progress report, ARC Infruitec-Nietvoorbij, Private Bag X5026, Stellenbosch, 7599, South Africa (unpublished).

Van Jaarsveld, F.P., Minnaar, P., Blom, M. & Hattingh, S., 2009. Rapid induction of ageing character in brandy products – Part I. Effects of extraction media and preparation conditions. S. Afr. J. Enol. Vitic. 30, 1-15.