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Fatty acid profile and glycerol concentration in *cachaças* aged in different wood barrels

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The objective of this study was to evaluate the fatty acid profile and glycerol concentration in *cachaças* aged for 3 years in barrels made from different types of tropical woods and European oak. Glycerol gives body to spirits, whereas the fatty acid profile influences the spirit sensory characteristics. The concentration of these compounds in aged spirits depends on the wood composition. Tropical wood barrels made from amendoim (*Pterogyne nitens Tul*), araruva (*Centrolobium tomentosum*), cabreúva (*Myrocarpus frondosus*), cerejeira (*Amburana cearensis*), grápia (*Apuleia leiocarpa*), ipê roxo (*Tabebuia heptaphylla*), jequitibá (*Cariniana estrellensis*), jequitibá rosa (*Cariniana legalis*) and pereira (*Platycyamus regnellii*), as well as European oak (*Quercus petraea*), were used in this study. Glycerol concentrations in aged *cachaças* were measured using colorimetric methods, and fatty acid profiles were determined by GC-MS analysis. The main fatty acids found were propanoic (C3:0), butanoic (C4:0), pentanoic (C5:0), octanoic (C8:0), decanoic (C10:0), dodecanoic (C12:0), hexadecanoic (C16:0), octadecanoic (C18:0) and octadecenoic (C18:1^9) acids. Araruva barrels stood out as the major supplier of C4:0 and cabreúva barrels provided greater amounts of C18:0 to *cachaça*. Carvalho and araruva barrels conferred more complete fatty acid profiles and higher concentrations of glycerol.

Keywords: Brazilian sugar cane spirit; wood; glycerol; fatty acid profile; GC-MS

Introduction

Cachaça is the typical Brazilian sugar cane spirit. Its alcohol content must be between 38 and 48% by volume at 20 °C. It should be obtained by distillation of fermented sugar cane juice *(1)*. Therefore, the production of *cachaça* is carried out by extraction of sugar cane juice followed by fermentation and distillation. *Cachaça* is composed of water, ethanol and some volatile compounds that are responsible for its sensory attributes *(2)*. Although not mandatory for *cachaça*, the aging process in wooden barrels refines the sensory profile and improves the quality of this beverage. The evolution of phenolic compounds, oxidation, stabilization of colour, flavour and appearance of woody characteristics all contribute to the richness and complexity of the aromatic bouquet and, consequently, results in higher quality spirits *(3)*.

Organic acids in spirits correspond mainly to acetic acid and some fatty acids resulting from fermentation and aging processes *(4)*. Fatty acids contribute to the development of important sensory features (e.g. flavour, aroma, taste and colour), stability and acceptability *(5)*. Fatty acids and esters, which are commonly found in spirits, have already been determined in mezcal and tequila *(6)*, whiskies *(7)* and Scotch whisky *(8)*.

Volatile congeners and short-chain fatty acids (C3 to C5) are produced by yeast (*Saccharomyces cerevisiae*) during alcoholic fermentation. The biochemical pathway of yeast generates glycerol, fatty acids, esters, aldehydes, alcohols and ketones. Fatty acids synthesised from pyruvate and other α-ketoacids, which are activated by coenzyme A (CoA), form acetyl-CoA derivatives. The synthesis of fatty acids and other organic acids occurs through the intermediary metabolism of amino acids from α-keto acids and carbohydrate metabolism in the Krebs cycle *(9)*. During distillation, volatile compounds, including short-chain fatty acids, are released in the alcoholic liquid according to their boiling point and affinity to ethanol. Pure spirits normally exhibit low concentrations of glycerol owing to their high boiling point (290 °C), although it may be generated by transesterification of wood triglycerides with ethanol, yielding fatty acid ethyl esters and free glycerol during the maturation process *(7)*.

Long-chain fatty acids (≥C8) originate from the degradation of wood structure. Wood is a complex source of extractable compounds primarily consisting of cellulose, hemicellulose, lignin and secondary constituents such as triacylglycerols and tannins *(9)*. The aging process is a complex system involving many reactions, especially those regarding the extraction of molecules from wood and their interactions with the distillate constituents *(10)*. Aging is the main factor in the characterization of the spirit, since approximately 60% of the aromatic compounds present in this beverage come from the wood or wood interaction with the spirit *(11)*. Aging congeners (e.g. gallic acid, vanillin, vanillic acid, syringic acid, syringaldehyde, sinapaldehyde and coniferaldehyde) derive from extraction and ethanolysis reactions of wood macromolecules *(12)*. The concentrations of compounds such as aldehydes, fatty acids, esters and carbohydrates increase during aging. Glycerol and fatty acids (capric, lauric, myristic and palmitic acids), generally found in aged spirits, are generated from hydrolysis of wood triglycerides and contribute to the aroma and the body (oiliness and viscosity) of the beverage *(13)*. Moreover, ethanol can react with pyruvic acid derived from acids such as lactic acid and acetic acid, as well as with other organic acids such as butyric, caproic,

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capric and lauric acids. Ester formation also occurs during aging, mainly owing to esterification reactions between alcohols and acids *(14)*. Ethyl acetate is the predominant ester in *cachaça*, corresponding to approximately 80% of the total esters of this spirit. It derives from the esterification of ethanol and acetic acid, and is also formed during the aging period. However, several other esters are also present in *cachaça*, including some derived from fatty acids *(15)*.

Globally, oak wood species are the most widely used to age spirits. Nevertheless, different species of wood are commonly used in Brazilian cooperages because they are economically more feasible and easier to obtain. Furthermore, the different types of Brazilian wood confer *cachaça* characteristic compounds for each species, thereby enabling diversification of the final product, because each type of wood has different properties and composition *(16)*.

This study examined the behaviour of *cachaça* aged in different wooden barrels of interest, especially compared with oak barrels. The physicochemical properties of wood influence the maturation process. Spirits are directly affected by the wood species used in the barrels, and are indirectly affected by wood maturation methods, form of construction of the barrels, heat treatment and the finishing procedures during cooperage *(17)*.

Given that Brazil possesses a vast diversity of flora, it is very interesting to study the chemical profiles of *cachaças* aged in different native wood barrels and compare them with those obtained in oak barrels. Fatty acids and glycerol can contribute to quality and sensory properties of distilled spirits. Therefore, the objective of this study was to evaluate the fatty acid profile and glycerol concentration in *cachaças* aged for 3 years in barrels made from different types of tropical wood, namely amendoim (*Pterogyne nitens Tul*), araruva (*Centrolobium tomentosum*), cabreúva (*Myrocarpus frondosus*), cerejeira (*Amburana cearensis*), grápia (*Apuleia leiocarpa*), ipê roxo (*Tabebuia heptaphylla*), jequitibá (*Cariniana estrellensis*), jequitibá rosa (*Cariniana legalis*) and pereira (*Platycyamus regnellii*), as well as European oak (*Quercus petraea*).

Materials and methods

Production of sugar cane spirit and aging process in wooden casks

The wort was prepared using sugar cane stalks from variety SP 83-2847. The sugar cane juice was extracted using a stainless steel presser. After that, it underwent a thermal treatment (105 °C) to eliminate contaminating microorganisms and decantation for 2 h for colloidal precipitation. Fermentation was performed in 4 m^3 tanks using *Saccharomyces cerevisiae* strain CA-11 (LNF Latinoamericana, Bento Gonçalves, RS, Brazil) and distillation was carried out in columns. The *cachaça* so obtained was stored in a 5 m^3 stainless steel container until further use.

The volume of the sugar cane spirit necessary to conduct this study was homogenized and distributed into casks made from 10 types of wood: amendoim, araruva, cabreúva, cerejeira, grápia, ipê roxo, jequitibá, jequitibá rosa, pereira and European oak. The sugar cane spirit (61% ethanol, v/v) was aged in the wooden casks for 3 years, in triplicate, at room temperature (22 ± 5 ° C) and a relative humidity of 55 ± 10% and protected from vibrations.

The casks were constructed in the shape of a frustum of a cone, with an inner base diameter of 66 cm, a height of 86 cm and an inner lid diameter of 54 cm, resulting in an average cask volume of 245 L, an internal contact area with the sugar cane spirit of 196 dm^2 (excluding the lid) and, consequently, a volume/surface area ratio of 1.25 L/dm. The casks were not charred after construction. Before the experiment, the casks were washed with steam, hot water and cold water, filled with sugar cane spirit and stored for 24 months. After that, they were exhausted and washed with cold water to be ready for the aging process.

Physicochemical and chromatographic analyses

Glycerol determination. Aged spirits were appropriately diluted with potable water to 41% (v/v) ethanol. Glycerol was determined according to Amerine and Ough *(18)*. The colourimetric method is based on the oxidation of glycerol with metaperiodate releasing formaldehyde, which is subsequently condensed with acetylacetone and ammonia, forming a coloured complex with maximum absorption at $\lambda = 420$ nm. Glycerin (Merck) was used as a standard reagent for the construction of the calibration curve ($y = -44{,}271x + 88.651$, $r^2 = 0.9993$).

Identification of fatty acids using gas chromatography-mass spectrometry (GC-MS scan). Samples were previously filtered in Millex-HV filters (Millipore) with a polyvinylidene difluoride membrane (13 mm diameter, 0.45 μm pore size) and injected directly into the chromatograph, in triplicate. The chromatographic method does not require pre-treatment because the fatty acids are dissolved in the spirit and other interfering compounds are eliminated at the time of injection, which is shown by the boiling point, and do not interact with the chromatographic column used. Separation of compounds was performed using a Shimadzu GC 2010 gas chromatograph with mass detector Shimadzu QP-2010 plus, in scan mode ($m/z = 40$–400), equipped with a capillary column with polar phase (esterified polyethylene glycol) HP-FFAP (49 m × 0.20 mm × 0.33 μm). The injector and detector temperatures were 180 and 220 °C, respectively. The following temperature programme was applied: starting at 35 °C for 3 min; increasing to 250 °C at a rate of 15 °C/min; and remaining at this temperature for 5 min. The duration of the analyses was 65 min. The injected volume was 2.0 μL in a split ratio 1:5, with automatic injection. The carrier gas was helium at a flow rate of 1 mL/min. The identification of compounds was based on the similarity index (SI ≥ 85%) to the libraries 'Flavours and Fragrances of Natural and Synthetic Compounds' – Mass Spectral Database (FFNSC) and Wiley Registry of Mass Spectral Data 8. The 10 most abundant fatty acids were selected for each of the 10 types of wood used. The percentage of each fatty acid was determined according to the peak area in the chromatograms obtained.

Results and discussion

The presence of glycerol in wines and its sensory contribution to the wine's sweet taste and viscosity are well known *(19–21)*. Although pure spirits exhibit low concentrations of glycerol, some researchers have found a particular role for this compound in *cachaça* and bagaceira *(22)*, cognac and cognac spirits *(23)* and sugar cane spirits *(24)*.

Fatty acids originate fatty acid ester and enanthic ester components that are relevant to the sensory characteristics of spirits, such as ethyl octanoate and ethyl decanoate *(4)*. Fatty acid ester and enanthic ester components are present in spirits, conferring both positive and negative features, for example, the soapy characteristic found in French cognacs owing to the presence of enanthic esters *(23)*.

Table 1. Mean and standard deviation for the glycerol content in *cachaças* aged for 3 years in barrels made from different types of wood

Wood	Glycerol (mg/L)
Control	$1.39^{d} \pm 0.11$
Amendoim	$15.79^{b} \pm 0.04$
Araruva	$22.99^{a} \pm 0,29$
Cabreúva	$16.20^{b} \pm 0.49$
Cerejeira	$11.51^{c} \pm 0.05$
Grápia	$10.79^{c} \pm 0.05$
Ipê roxo	$17.35^{b} \pm 0.53$
Jequitibá	$12.50^{b} \pm 0.05$
Jequitibá rosa	$15.16^{b} \pm 1.56$
Pereira	$13.11^{b} \pm 0.93$
Oak	$21.94^{a} \pm 0.55$
Average	15.73

Different letters in the column indicate significant statistical difference by the Tukey test ($p < 0.05$).

The total ester content in *cachaças* ranges from 200 to 470 mg/100 mL of anhydrous alcohol. In a study by Nascimento et al. *(25)*, ethyl acetate was the major ester, with a median content of 22.6 mg/100 mL of anhydrous alcohol, followed by ethyl lactate, with a median content of 8.32 mg/100 mL of anhydrous alcohol. Comparing ester profiles (content of ethyl octanoate, ethyl hexanoate, ethyl butyrate, ethyl nonanoate, ethyl decanoate and ethyl laurate) of *cachaças*, whiskey and rum, the major complexity was found in the whiskey and rum samples *(25)*, probably owing to mandatory aging processes.

Glycerol

The content of glycerol in the aged *cachaças* is shown in Table 1. *Cachaça* aged in araruva and oak barrels presented the highest content of glycerol. Amendoim, cabreúva, ipê roxo, jequitibá, jequitibá rosa and pereira had a medium potential to generate glycerol in sugar cane spirits. The lowest content of glycerol was extracted from the grápia and cerejeira.

The average glycerol concentration in the aged *cachaças* was 15.73 mg/L and the control (fresh distilled *cachaça* before aging) presented 1.39 mg/L. The comparison of the glycerol content in freshly distilled and aged *cachaças* can provide some information regarding the amount of this compound incorporated by the contact of the spirit with the internal surface of the wood barrel. In a Brazilian study, the amount of glycerol in aged and non-aged *cachaças* was compared and the former exhibited contents were 10-fold higher than the latter (1.75 and 17.5 mg/L) *(24)*. Another study evaluated glycerol content in *cachaças* and *bagaceira* (Portuguese spirit), which ranged from 36.3 to 2150 mg/L in *cachaça* and from 44 to 88 mg/L in *bagaceira (22)*.

Table 2. Retention time (RT), significant ions of the mass spectrum (*m/z*) and similarity index (SI ≥ 85%) to flavour libraries of fatty acids present in *cachaças* aged for 3 years in barrels made from different types of wood

Fatty acid	RT (min)	*m/z*	SI
Propanoic acid (C3:0)	6.60	M^+ 88(100) 94(86) 87(19) 57(13) 73(7)	85
Butanoic acid (C4:0)	8.78	M^+ 89(100) 60(99) 206(50) 87(6)	89
Pentanoic acid (C5:0)	10.28	M^+ 60(100)	100
Octanoic acid (C8:0)	13.98	M^+ 88(100) 70(25) 101(36) 57(34) 127(30) 60(20) 55(19) 129(14)	90
Decanoic acid (C10:0)	21.46	M^+ 88(100) 101(37) 55(18) 70(19) 73(18) 157(17) 155(16) 89(10)	94
Dodecanoic acid (C12:0)	30.10	M^+ 88(100) 101(44) 55(18) 70(17) 73(16) 157(15) 183(9)	92
Hexadecanoic acid (C16:0)	43.75	M^+ 88(100) 101(51) 55(19) 157(18) 89(18) 157(18) 57(16) 70(15) 73(13)	95
Octadecenoic acid (C18:1^{9})	49.15	M^+ 55(100) 96(86) 68(78) 97(73) 88(62) 101(60) 98(60) 110(42) 137(43)	88
Octadecanoic acid (C18:0)	49.73	M^+ 88(100) 101(67) 55(26) 157(25) 70(23) 312(15) 73(12)	97

(C*x*:*y*): fatty acids classification – *x* is molecular number of carbons and *y* is molecular number of unsaturated bond between carbons. SI: Index of Similarity concerning to the libraries 'Flavours and Fragrances of Natural and Synthetic Compounds' – Mass Spectral Database (FFNSC) and the Wiley Registry of Mass Spectral Data 8.

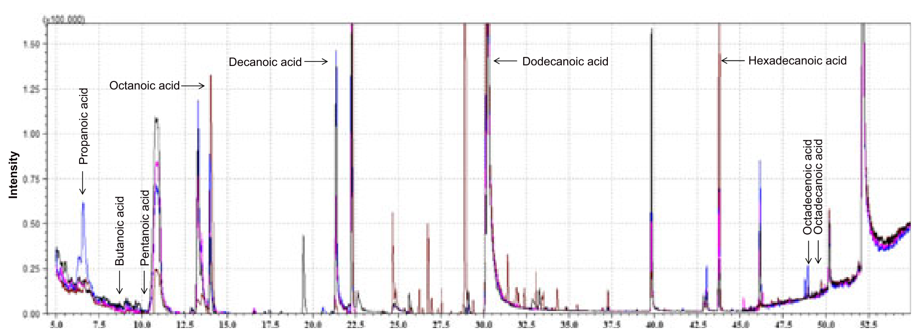

Figure 1. Chromatographic profile of recovered samples of *cachaças* aged for 3 years in barrels made from different types of wood.

Characterization of fatty acid profile using GC-MS

The fatty acids identified by gas chromatography–mass spectrometry (GC-MS) in aged *cachaças* are shown in Table 2. A typical chromatogram is presented in Fig. 1. The *cachaças* presented different ratios of fatty acids and some specificity according to the type of wood used in the barrel in which they were aged (Table 3).

Decanoic acid is the fatty acid found in highest amounts in Scotch whiskies, followed by octanoic and dodecanoic acids *(11)*. The concentrations of short-chain fatty acids found in rum were as follows: propionic acid, 0.5–4.2 mg/L; butyric acid, 0.4–2.6 mg/L; and hexanoic acid, 0.3–1.3 mg/L *(26)*.

The fatty acid composition of high quality *cachaça* had averages of 2.57 mg/L of propanoic acid and 0.45 mg/L of hexanoic acid. In the olfactometry analysis, butyric, pentanoic, hexanoic, decanoic and dodecanoic acids received the aromatic description of 'cheese' and 'fatty acid' *(27)*. Normally, fatty acids have pleasant aromas of butter, cheese and almonds. However, when present in high concentrations, certain fatty acids cause sensory defects such as 'manure-like' odours *(28)*.

The analysis of volatile compounds after fermentation and after distillation of Armagnac revealed that the continuous distillation still (column coupled with six plates) provided a 25% recovery of hexanoic acid, 35% of octanoic acid and 99% of decanoic acid *(29)*.

Brazilian woods, namely amburana, bálsamo, jequitibá, jatobá and ipê, incorporated some phenolic compounds to sugar cane spirits that are typically found in spirits aged in oak barrels. However, the prevalence and concentration of these compounds varied during the aging process *(29)*. The following fatty acids were identified in aged sugar cane spirits: butyric, decanoic, dodecanoic, heptanoic, hexanoic, hexadecanoic, isobutanoic, isopentanoic, isovaleric, octanoic, pentanoic, propanoic and tetradecanoic *(17)*.

The fatty acids present in many samples of aged *cachaças* were octanoic (C8:0), decanoic (C10:0) and dodecanoic (C12:0), whereas those found in a smaller number of samples were propanoic (C3:0) and pentanoic (C5:0). Propanoic acid was detected only in sugar cane spirit aged in ipê roxo barrels and pentanoic acid was detected only in *cachaças* aged in grápia barrels. Another less abundant fatty acid was butanoic (C4:0), found only in suga rcane spirits aged in araruva and oak barrels. Octadecenoic acid ($C18:1^9$) was observed in *cachaças* aged in barrels made from ipê roxo, jequitibá and jequitibá rosa (Fig. 2). The influence of the wood species was a determining factor to the profile obtained.

Cachaças aged in araruva, grápia, ipê roxo, jequitibá, jequitibá rosa and oak barrels had five different fatty acids. At least three fatty acids were observed in the sugar cane spirits aged in amendoim, cabreúva, cerejeira and pereira barrels.

Araruva provided about three times more butanoic acid (C4:0) to the suga rcane spirit than oak. Cabreúva was the only type of wood studied that supplied octadecenoic acid ($C18:1^9$) to aged *cachaças*. Araruva and oak might be considered the most complex woods regarding the fatty acid composition for spirit aging. *Cachaças* aged in jequitibá and jequitibá rosa barrels presented very similar fatty acid profiles.

In a similar study, the glycerol content and fatty acid profile were investigated and compared between commercial aged and non-aged sugar cane spirits *(24)*. For aged spirits,

Table 3. Fatty acid composition (percentage and area) present in *cachaças* aged for 3 years in barrels made from different types of wood

Fatty acid	Type of wood																					
	Control		Amendoim		Araruva		Cabreúva		Cerejeira		Grápia		Ipê roxo		Jequitibá		Jequitibá rosa		Pereira		Oak	
	%	Area	%	Area	%	Area	%	Area	%	Area	%	Area	%	Area	%	Area	%	Area	%	Area	%	Area
Propanoic (C3:0)	—	—	—	—	—	—	—	—	—	—	—	—	11.55	1.55	—	—	—	—	—	—	—	—
Butanoic (C4:0)	—	—	—	—	36.07	6.67	—	—	—	—	—	—	—	—	—	—	—	—	—	—	14.34	2.39
Pentanoic (C5:0)	—	—	—	—	—	—	—	—	—	—	9.52	1.45	—	—	—	—	—	—	—	—	—	—
Octanoic (C8:0)	49.95	1.83	28.26	2.66	14.55	2.69	—	—	38.33	2.22	27.48	4.20	27.20	3.65	18.39	3.04	23.57	3.83	40.81	4.37	24.44	4.08
Decanoic (C10:0)	55.04	2.24	40.0	3.77	19.33	3.57	38.65	5.42	47.40	2.75	38.48	5.89	32.15	4.31	27.39	4.53	30.10	4.89	—	—	35.09	5.86
Dodecanoic (C12:0)	—	—	18.90	1.78	13.47	2.49	—	—	14.26	0.82	15.29	2.34	18.86	2.53	14.09	2.33	11.39	1.85	12.11	1.29	17.82	2.98
Hexadecanoic (C16:0)	—	—	12.82	1.21	16.56	3.06	34.12	4.78	—	—	9.21	1.41	—	—	30.05	4.97	24.42	3.97	47.06	5.04	8.28	1.38
Octadecenoic ($C18:1^9$)	—	—	—	—	—	—	27.21	3.82	—	—	—	—	—	—	—	—	—	—	—	—	—	—
Octadecanoic (C18:0)	—	—	—	—	—	—	—	—	—	—	—	—	10.21	1.37	10.05	1.66	10.48	1.70	—	—	—	—

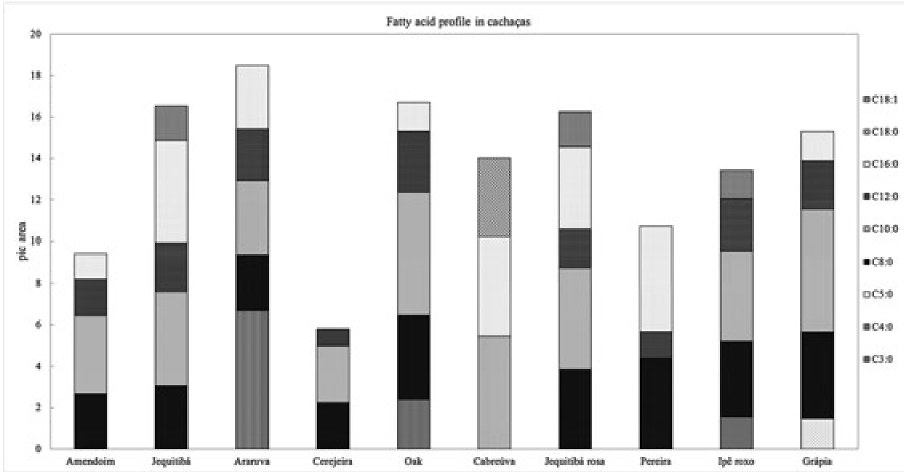

Figure 2. Fatty acid profile of *cachaças* aged for 3 years in barrels made from different types of wood.

glycerol concentration varied according to the type of wood used in the barrels. Amburana supplied more glycerol (42 mg/L) than oak, jatobá, jequitibá and amendoim. As observed in the current research, higher concentrations of decanoic acid than other fatty acids were detected in all samples. Dodecanoic, tretradecanoic and hexadecanoic acids were found in smaller quantities and with smaller variations between samples.

Conclusions

The species of wood influenced the profile of the fatty acids found in aged *cachaças*. Araruva, grápia, ipê roxo, jequitibá, jequitibá rosa and oak conferred greater complexity to the fatty acid profile.

The relevance of this study lies in the improvement of the knowledge of the chemical composition of *cachaças* aged in Brazilian wood barrels compared with oak wood. This analysis can also serve as a basis for more accurate conclusions about the development of sensory characteristics of each type of wood and formulation of blends in order to add complexity to the chemical composition and sensory quality of *cachaças*.

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