UNIVERSIDADE ESTADUAL DE CAMPINAS
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VINHO SYRAH DO VALE DO SÃO FRANCISCO: CARACTERIZAÇÃO FÍSICO - QUÍMICA, PERFIL SENSORIAL E ESTUDO DE CONSUMIDOR

SYRAH WINE FROM SÃO FRANCISCO VALLEY: PHYSICO-CHEMICAL CHARACTERIZATION, SENSORY PROFILE AND CONSUMER STUDY

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Tese de Doutorado apresentada à Faculdade de Engenharia de Alimentos da Universidade Estadual de Campinas como parte dos requisitos exigidos para a obtenção do título de Doutora em Alimentos e Nutrição, na área de Consumo e Qualidade de Alimentos.

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Orientador: Prof. Dr. Jorge Herman Behrens
Co-orientadora: Drª. Aline Camarão Telles Biasoto

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Ata de defesa, com as respectivas assinaturas dos membros encontra-se no processo de vida acadêmica do aluno.

Dedico este trabalho aos enólogos e colaboradores das vinícolas localizadas na região do Vale do São Francisco – Brasil, com objetivo de colaborar com informações relevantes para vinificação de vinhos tintos da uva Syrah produzidos nessa região.

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RESUMO

O Brasil faz parte do "novo mundo dos vinhos" e, atualmente, o País tem buscado melhorar a qualidade dos produtos, aumentar o consumo interno e promover os vinhos e espumantes no mercado global. Nesse contexto, as vinícolas brasileiras procuram consolidar identidades regionais, certificações de qualidade e principalmente investir em regiões vitivinícolas emergentes como o Vale do São Francisco, nos estados da Bahia e Pernambuco, na região nordeste do País. Essa região é semiárida, caracterizada por altas temperaturas e taxas de insolação, ausência de inverno e água fornecida pelo rio São Francisco. A uva *Vits Vinífera* Syrah está bem adaptada à região e os vinhos produzidos a partir dessa variedade apresentam alta concentração de compostos fenólicos, o que os torna apropriados ao envelhecimento em madeira. Na região utiliza-se o envelhecimento em barril, no entanto, a adição de chip de carvalho, por ser um método de baixo custo e rápido envelhecimento, pode ser uma alternativa interessante para os produtores locais com o objetivo de melhorar a qualidade sensorial e estabilidade química das bebidas, e até o momento, não existem relatos de trabalhos brasileitos que apresentem o perfil sensorial e físico-químico de vinhos envelhecidos com a utilização de chip de carvalho, a partir de uvas Syrah cultivada no Vale do São Francisco. O objetivo deste trabalho foi caracterizar físico - química e sensorialmente o vinho Syrah envelhecido com chip de carvalho Americano (*Quercus Alba*) e/ou Francês (*Quercus Petrae*) durante a fermentação alcoólica e /ou malolática. Os vinhos foram produzidos a partir do varietal Syrah cultivado no campo experimental da Embrapa Semi-árido situadas a 09 ° 09 'S, 40 ° 22' W, 365,5 m no Vale do São Francisco, Pernambuco, Brasil. As uvas foram colhidas manualmente no vinhedo em julho de 2015 em seu estágio de amadurecimento ótimo (26º Brix). Após a colheita, as uvas foram mantidas em uma câmara fria durante 10 horas a 10° C para diminuir e estabilizar a temperatura. Sequencialmente, as uvas foram desengaçadas e foram transferidas para um tanque de aço inoxidável. Seis lotes de uvas foram processados em escala semi-industrial; a maceração (30 dias) começou com a adição de metabissulfito de potássio (0,10 g L^{-1}). A enzima pectinolítica Everinetec® (0,008 mL L^{-1}), a levedura comercial Maurivin Consistent Quality® Saccharomyces cerevisiae (0,20 g L^{-1}) e o fosfato de amônio Coatec® ativado (0,20 g L^{-1}) foram adicionados ao mosto para iniciar a fermentação alcoólica sob temperatura controlada (24 °C ± 2 ºC), e 4 g L^{-1} de chips de carvalho americano e/ou francês foram adicionados no mosto na fermentação alcóolica e malolática ou apenas na fermentação malolática. A fermentação malolática iniciou-se espontaneamente e o final da mesma foi identificado por cromatografia em papel, análise utilizada para identificar a degradação do ácido málico. Posteriormente, o vinho foi submetido filtração, à estabilização a frio durante 10 dias a 0 ºC e engarrafado, sendo armazenado em adega na posição horizontal com temperatura controlada (18 °C) durante 30 dias. Avaliou-se os compostos fenólicos, atividade antioxidante, intensidade de cor, perfil sensorial descritivo, aceitabilidade dos vinhos controle e envelhecido com chip de carvalho. Durante o período de maceração prolongada observou-se que entre o 15º e 20º dia houve maior extração de compostos fenólicos permanecendo os mesmos estáveis até o 30º dia. Os vinhos apresentaram concentrações mais elevadas de compostos fenólicos, como ácido gálico, ácido clorogênico, (-) - epicatequina, (+) - catequina, (-) - galato epigalocatequina procianidina A2, B1 e B2, isorhamnetin-3-O-glicosídeo, quercetina 3--β -D- glicosídeo, quercetina, kaempferol-3-O-glicosídeo, rutina, miricetina, cianidina-3-O-glicosídeo, peonidina-3-O-glicosídeo,

petunidina-3-O-glicosídeo, delfinidina-3-O-glicosídeo, malvidina-3-O-glicosídeo, pelargonidina-3-O-glicosídeo. O vinho com chip de carvalho francês adicionado na fermentação malolática mostrou maior atividade antioxidante de acordo com o método ORAC. Na análise sensorial observou-se que a maior intensidade de cor e aromas de café, madeira e doce/caramelizado, gosto doce e sabor de madeira foram observados nos vinhos envelhecidos com chip em comparação com o controle. No teste de aceitação foram identificados dois segmentos de consumidores, um grupo (n=60) que rejeitou as amostras de vinhos desenvolvidas e outro grupo (n=69) que aceitou moderadamente a bebida. Para ambos os grupos o descritor de aroma e sabor de madeira foi importante para a aceitabilidade dos vinhos. Como conclusão, a região semiárida brasileira apresenta potencial para produção de vinhos com maceração prolongada e envelhecidos com chips de madeira, sendo o uso de chip de carvalho americano com tosta média o mais promissor para se obter vinhos de boa aceitação pelos consumidores.

Palavras-chave: Vinho e vinificação – Brasil, Análise descritiva, Consumidor, Antioxidantes, Compostos fenólicos, Avaliação sensorial.

ABSTRACT

Brazil is part of the "new world of wines". At the same time, the country has sought to improve the quality of wines, increase domestic consumption and promote wines in the global market. In this context, Brazilian wineries seek to consolidate regional identities of the beverage, quality certifications and mainly invest in local emerging wine regions such as the Valley of the São Francisco, in the states of Bahia and Pernambuco, in the northeast region of the country. This region is semi-arid, characterized by high temperatures and insolation rates, absence of winter and water provided by the São Francisco river. Grape *Vits Vinífera* Syrah is well adapted in the region and wines produced from this variety have high concentration of phenolic compounds, which makes it favorable to aging in wood. Aging in barrels is currently used by local wineries; however, oak chip addition has been proposed as a low cost and fast aging method that has been applied in international studies. It can be an interesting alternative for local producers with the aim of improving sensory quality and chemical stability of beverages, and to date there are no reports on the sensory and physico-chemical characteristics of wines produced in the São Francisco Valley and aged with oak chips. The objective of this work was to characterize chemically and sensorially Syrah wine aged with American oak (*Quercus Alba*) and / or French (*Quercus Petrae*) chips during alcoholic and / or malolactic fermentation. The wines were produced from grapes cultivated in the experimental field of Embrapa Semi-arid located at 09 ° 09'S, 40 ° 22 'W, 365.5 m in the Valley of São Francisco, Pernambuco, Brazil. They were harvested manually in the vineyard in July 2015 at its optimum ripening stage (26 ° Brix). After harvest, the grapes were kept in a cold room for 10 hours at 10 ° C to lower and stabilize the temperature. Sequentially, the grapes were destemmed and transferred to a stainless steel tank. Six lots of grapes were processed on a semi-industrial scale; maceration (30 days) started with the addition of potassium metabisulfite (0.10 g L^{-1}). The pectinolytic enzyme Everinetec® (0.008 mL L^{-1}), commercial yeast Maurivin Consistent Quality Saccharomyces cerevisiae (0.20 g L^{-1}) and activated ammonium phosphate Coatec® (0.20 g L^{-1}) were added to the must to start alcoholic fermentation under controlled temperature (24 ° C ± 2 ºC), and 4 g L^{-1} of American and / or French oak chips were added to the must in alcoholic and malolactic fermentation or in malolactic fermentation solemly. The malolactic fermentation started spontaneously and the end of it was identified by paper chromatography, an analysis used to identify the degradation of malic acid. The resulting wine was subjected to cold stabilization for 10 days at 0 ºC and bottled afterwards, being stored in a cellar with controlled temperature (18 ° C) horizontally for 30 days. Phenolic compounds, antioxidant activity, color intensity, descriptive sensorial profile, acceptability and perceptions in the control and aged wines were assessed. During the period of prolonged maceration it was observed that during the 15 th to the 20 th day there was a higher extraction of phenolic compounds, remaining stable until the 30th day. Syrah wines presented higher concentrations of phenolic compounds such as gallic acid, chlorogenic acid, (-) - epicatechin, (+) - catechin, (-) - epigallocatechin gallate procyanidin A2, B1 and B2, isorhamnetin -3-O-glucoside , quercetin -3-β-D-glucoside, quercetin, kaempferol -3-O-glucoside, rutin, myricetin, cyanidin-3-O-glucoside, peonidin -3-O-glucoside, petunidine -3-O-glucoside, delphinidin -3-O-glucoside, malvidin -3-O-glucoside, pelargonidin-3-O-glucoside. The wine with French oak chips added during the malolactic fermentation showed greater antioxidant activity for the ORAC. In the sensory analysis it was observed higher color intensity

and aromas of coffee, wood and sweet / caramelized, sweet taste and wood flavor in the wines aged with chips as compared to the control wine . In the acceptance test two consumer segments were identified, one group (n = 60) that rejected the samples and another group (n = 69) that moderately accepted the beverage. For both groups the wood character was identified as important for the acceptability of the wines. As a conclusion, the Brazilian semi-arid region presents potential for the production of wines with prolonged maceration and aged with wood chips, and the use of American oak chip with medium toast showed to be the most promising to obtain wines that are well accepted by consumers.

Keywords: Wine and wine making – Brazil, Descriptive analysis, Consumer, Antioxidants, Phenolic compounds, Sensory evaluation.

SÚMARIO

INTRODUÇÃO

A prática da viticultura no mundo foi difundida pelos gregos no século IV A.C., que cultivavam as vinhas ao longo do seu país, e também propagaram a prática da viticultura no sul da Itália, no sul da França, e em suas colônias no mar negro (RENFREW, 2003).

No Brasil, a viticultura chegou com os portugueses nos primeiros tempos de colônia, porém, somente se difundiu significativamente com a vinda de imigrantes italianos no século XIX que se instalaram no centro-sul do país. Na década de 1970, amparada por estudos realizados pela Secretária de Estado de Agricultura, a produção de viníferas ampliou-se no Estado do Rio Grande do Sul, mais precisamente na Serra Gaúcha e, assim, a produção de vinhos finos, notadamente dos varietais Riesling Itálico, Semillón, Cabernet Franc e Merlot (LONA, 1996).

A vitivinicultura brasileira é caracterizada pela introdução de certificações de qualidade e busca consolidar identidades regionais. Assim, algumas vinícolas tem se alocado em novas regiões como a Campanha Gaúcha e o Vale do São Francisco, a fim de produzir vinhos com qualidade diferenciada e redução dos custos pela ampliação de escala de produção, mecanização e qualidade sanitária de solos (NIERDELE; VITROLLES, 2010). Desta forma, o setor vitivinícola brasileiro tem avançado significativamente nos últimos anos, através da produção integrada de uvas de mesa, produção de vinhos finos e suco de uva (CAMARGO; TONIETTO; HOFFMAN, 2011), graças à cooperação entre centros de pesquisa, vinícolas e agricultores familiares (JEZIORMY; ORTEGA, 2013).

As inovações no setor vitivinícola levaram à inserção do Brasil no cenário da competição global. No ano de 2016, o mercado interno brasileiro registrou a comercialização de 15 milhões de litros de vinhos tintos. Entretanto, o Instituto Brasileiro do Vinho divulgou no primeiro trimestre do ano de 2017 uma queda de 30,5% na comercialização de vinho comparado ao mesmo período de 2016, enquanto a participação de rótulos importados no mercado brasileiro no ano de 2016 foi de 87,8 milhões de litros, sendo o Chile o principal país que comercializa rótulos no Brasil com 43,5 milhões de litros (IBRAVIN, 2017a; IBRAVIN, 2017b). Esses números indicam, por um lado, a predominância de vinhos estrangeiros no mercado nacional, mas, sob outro ponto de vista, há espaço para crescimento de consumo do produto nacional.

Além do Rio Grande do Sul, novas regiões mostram-se promissoras como produtoras de uvas e vinhos pertencentes ao gênero *Vitis vinifera* e, dentre elas, o Vale Submédio do São Francisco, situado em uma região de clima tropical semiárido entre os estados da Bahia e

Pernambuco. O clima dessa região possibilita o escalonamento da produção de uvas para vinhos ao longo do ano, devido às altas temperaturas, altos índices de insolação e água disponível em abundância para irrigação proveniente do rio São Francisco (PEREIRA, 2013).

As uvas cultivadas na região do Vale Submédio do São Francisco vêm passando por processo de adaptação de clima e solo. São produzidos, nessa região, vinhos jovens e de boa qualidade (DIAS et al., 2013). As uvas tintas mais adaptadas à região são Syrah, Cabernet Sauvignon, Alicante Bouschet, Barbera, Touriga Nacional, Petit Verdot, Ruby Cabernet e Tempranillo (PADILHA et al., 2017). Segundo LEÃO et al. (2011), os vinhos produzidos na região do Vale do São Francisco apresentam características marcantes e exóticas, de forma que os consumidores são receptivos a conhecer esses vinhos. No entanto, até o momento, são escassos os estudos conduzidos com vinhos tintos do Vale Submédio do São Francisco (BELMIRO; PEREIRA; PAIM, 2017; DE OLIVEIRA et al., 2017; LAGO et al., 2017; LUCENA et al., 2010; PADILHA et al., 2017).

O vinho produzido a partir da variedade de uva Syrah cultivada no Vale do São Francisco, em particular, tem mostrado alto teor de compostos fenólicos e elevada capacidade antioxidante (LUCENA et al., 2010; PADILHA et al., 2017; ANDRADE et al., 2013; DE OLIVEIRA et al., 2017). Do ponto de vista sensorial, os compostos fenólicos impactam na cor e no sabor da bebida e, por outro lado, são cada vez mais reconhecidos os benefícios de alguns desses compostos à saúde, pois eles atuam como antioxidantes em sistemas biológicos (ARCARI et al., 2013; GARRIDO; BORGES, 2013).

Atualmente, novos métodos de envelhecimento do vinho estão sendo utilizados, como, por exemplo, os chips (cavacos) de diferentes espécies de madeiras. O envelhecimento com chip é um método rápido se comparado ao tradicional barril, com impacto sensorial similar à bebida. Em geral, a adição de chips é realizada durante a fermentação alcoólica, malolática, ou ambas as fermentações (GÓMEZ GARCÍA-CARPINTERO; SÁNCHEZ-PALOMO; GONZÁLEZ VIÑAS, 2014). Os chips de carvalho são autorizados para prática enológica pela *International Organisation of Vine and Wine* sendo exclusivamente do gênero *Quercus*, no estado natural ou com tosta fraca, média ou alta (OIV, 2007).

A utilização do chip de madeira promove alguns benefícios à bebida como a estabilização da cor (GORTZI et al., 2013; GORDILLO et al., 2016; CEJUDO-BASTANTE; RIVERO-GRANADOS; HEREDIA, 2017) e melhora do perfil de aroma dos vinhos (SCHUMACHER et al., 2013; GALLEGO et al., 2015).

No Brasil, estudos foram conduzidos para avaliar a aplicação de chip de carvalho (madeiras coletadas nas florestas *Allier*, *Vosges* e *Nièvre,* na França) com diferentes níveis de

tosta em envelhecimento de cachaça (BORTOLETTO; ALCARDE, 2015) e chip de carvalho francês em cerveja do tipo Lager (WYLER et al., 2015). Entretanto, não existem estudos na literatura sobre a utilização do chip de madeira para vinhos produzidos no Brasil.

Neste trabalho, propôs-se o estudo do vinho Syrah do Vale Submédio do São Francisco, com maceração por 30 dias e adicionado com chip de carvalho Americano (*Quercus Alba*) e francês (*Quercus Petrae*).

OBJETIVOS

O objetivo geral desse trabalho foi caracterizar química e sensorialmente o vinho Syrah adicionado com chip de carvalho americano (*Quercus Alba*) e/ou francês (*Quercus Petrae*) durante a fermentação alcoólica e /ou malolática.

Especificamente cada artigo desta tese teve como objetivo:

1. Avaliar os compostos fenólicos, atividade antioxidante, e intensidade de cor durante 30 dias de maceração, e o perfil sensorial do vinho produzido com uva Syrah (*Vitis vinifera L.*) cultivada no Vale do Francisco, Brasil.
2. Caracterizar o perfil dos compostos fenólicos e a atividade antioxidante dos vinhos produzidos adicionados com chip de carvalho francês e americano colocados em diferentes estágios do processo de vinificação.
3. Determinar o perfil sensorial descritivo, avaliar a aceitabilidade e caracterizar as percepções dos consumidores do vinho Syrah do Vale do São Francisco elaborado com a adição de chips de carvalho americano e francês.

CAPÍTULO 1 - REVISÃO BIBLIOGRÁFICA

1. Origens do Vinho

Evidências arqueológicas sugerem que o vinho tenha surgido há mais de 7.500 anos no norte das montanhas de Zagros, no Irã (JACKSON, 2008). Posteriormente, registros egípcios referem-se à utilização da uva para fabricação de vinho e inúmeras referências bíblicas já relatavam a importância da bebida na Antiguidade (HORNSEY, 2007). As videiras (*Vitis Vinifera)* são originárias da região do Oriente Médio, mais precisamente da região do Cáucaso, entre o Mar Negro e o Mar Cáspio e posteriormente expandiu-se o cultivo para outras regiões do mundo. Atualmente a maioria dos vinhos modernos e de qualidade superior é produzida a partir dessa espécie de videira (HORNSEY, 2007).

Por volta dos anos 200 a 400 D.C. houve avanços nos métodos de produção e cultivo da videira, porém a produção de restringiu-se aos monastérios da Europa Ocidental durante a Idade Média (THE AUSTRALIAN WINE RESEARCH INSTITUTE, 2010; TOUSSAINT-SAMAT, 2009).

A partir dessas informações, acredita-se que a fermentação do vinho seja um dos processos mais antigos da humanidade e desde o início da civilização a bebida tem sido associada à cultura, à arte e à religião, por seu papel vital em cerimônias. Por isso, os mosteiros tiveram grande importância na fabricação e fornecimento de vinhos para celebrações religiosas e outras atividades culturais (HUI et al., 2004).

A partir do século XX, a produção de vinho seguiu novos caminhos com o desenvolvimento da viticultura e da enologia, por meio de cantinas com modernas instalações de vinificação, estocagem, envelhecimento e engarrafamento de vinhos; colheita mecanizada de uvas e fermentação a frio, além da introdução de novos varietais (LONA, 1996). Esse desenvolvimento permitiu que a cultura do vinho se expandisse do velho mundo (Eurásia) ao novo mundo (Américas, África e Oceania).

Tradicionalmente, França, Itália, Espanha, Portugal, são considerados o “velho mundo”, e nessas regiões a característica mais importante para a vitivinicultura é o *terroir* (relação entre solo, micro-clima, que produz uvas de qualidade e tipicidade). No “novo mundo” dos vinhos, caracterizado por países como Austrália, Nova Zelândia, Estados Unidos, Chile, Argentina e Brasil, ciência e inovação desempenham um papel importante na vitivinicultura (FLINT; SIGNORI; GOLICIC, 2016).

Atualmente, o vinho é uma bebida alcoólica tradicional e culturalmente inserida em hábitos alimentares da população ao redor do mundo. Particularmente, o vinho é o mais preferido pelos consumidores ocidentais e asiáticos devido aos benefícios à saúde associados ao seu consumo (YOO et al., 2013). Além do fator saúde, os consumidores consideram importante sua origem, variedade da uva, sabor e o preço (BARREIRO-HURLE; COLOMBOA; CANTOS-VILLAR, 2008; KALLAS; ESCOBAR; GIL, 2013).

2. Vitivinicultura

2.1 Vitivinicultura no mundo e no Brasil

Durante o último terço do século XX, o mercado mundial do vinho tornou-se mais competitivo, principalmente com a inserção de países como Estados Unidos, Austrália e Chile na competição pela produção de vinhos de qualidade (BISSON et al., 2002).

Nos últimos anos, o consumo da bebida cresceu na América do Norte, na Ásia e no Reino Unido, mas de forma também expressiva em mercados emergentes como Brasil, Índia, Taiwan e a República da Coréia que tornaram-se importantes importadores de vinhos (INSEL, 2014).

Com a expansão e segmentação do mercado internacional criaram-se oportunidades para a comercialização de vinhos do Mediterrâneo e de regiões periféricas do Atlântico. Além disso, o vinho europeu passou a competir com vinhos americanos e australianos (PAN-MONTOJO, 2009). Em geral, o comércio internacional de vinhos tem crescido com tendências positivas, principalmente considerando Austrália e Chile, que apresentam os melhores desempenhos entre os exportadores mundiais (CASSI; MORRISON; RABELLOTTI, 2014). Os mercados da Europa Ocidental estão em declínio, enquanto China, Brasil, Rússia e, em menor grau, a Índia, apresentam-se em expansão (LOCKSHIN; CORSI, 2012).

A produção mundial de vinhos no ano de 2016 foi de 267 milhões de hectolitros, um declínio de 9,3 milhões de hectolitros em comparação a produção de 2015. Os cincos principais produtores no mundo em 2016 foram Itália (50,9 milhões de hectolitros), França (43,5 milhões de hectolitros), Espanha (39,3 milhões de hectolitros), Estados Unidos (23,9 milhões de hectolitros) e Austrália (13,0 milhões de hectolitros) (OIV, 2017).

Na Tabela 1 está representado o consumo mundial de vinho e nela é possível observar que o consumo em 2016 foi de 242 milhões de hectolitros, um sutil aumento de 0,9 milhões de hectolitros em comparação com o ano de 2015. Os Estados Unidos, com o consumo estimado

em 31,8 milhões de hectolitros, confirmam sua posição como principal consumidor. Isso ocorre devido à crescente demanda doméstica, enquanto houve uma ligeira queda na Europa, como pode ser observado na Espanha e Portugal. Na América do Sul, o consumo de vinho foi menor em 2016 quando comparado a 2015, principalmente na Argentina e no Brasil. Entretanto, no Chile, o consumo cresceu em torno de 5% (OIV, 2017).

A vitivinicultura no Brasil ocupa uma área de aproximadamente 83,7 mil hectares, com vinhedos estabelecidos desde o extremo sul do país até regiões situadas próximas a linha do Equador. Em todo o território brasileiro existem mais de 1,1 mil vinícolas, sendo a maioria instalada em pequenas propriedades com área de 2 hectares (INSTITUTO BRASILEIRO DE VINHO, [s.d.]). Desde o ano 2000, as vinícolas brasileiras tornaram-se conglomerados industriais e têm investido em tecnologia e produtos voltados para diversos públicos e mercados (ATHIA; COSTA, 2009). A cultura da videira está satisfatoriamente adaptada ao país, inclusive em regiões tropicais, o que é comprovado pelas exportações de uva de mesa (ROSA; SIMÕES, 2004).

No país a viticultura está concentrada nos estados do Rio Grande do Sul, Santa Catarina, Paraná, São Paulo, Minas Gerais e no Vale do Submédio do São Francisco, em Pernambuco (CAMARGO; TONIETTO; HOFFMAN, 2011).

A região do Vale do São Francisco tem avançado tecnologicamente na produção de vinhos e, consequentemente, aumentou o portfólio de produtos da região, tanto na diversificação dos vinhos para o mercado interno quanto para exportação (PROTAS, 2011).

Tabela 1: Consumo mundial de vinho[a].

mhl	2012	2013	2014	2015[b]	2016[c]	2016/2015 Volume em variação	2016/2015 Variação em %
Estados Unidos	30,0	30,2	30,4	31,0	31,8	0,8	2,5
França	28,0	27,8	27,5	27,2	27,0	-0,2	-0,7
Itália	21,6	20,8	19,5	21,4	22,5	1,1	5,3
Alemanha	20,3	20,4	20,2	20,6	20,2	-0,4	-1,8
China*	17,1	16,5	15,5	16,2	17,3	1,1	6,9
Reino Unido	12,8	12,7	12,6	12,7	12,9	0,2	1,4
Espanha	9,9	9,8	9,9	10,0	9,9	0,0	-0,4
Argentina	10,1	10,4	9,9	10,3	9,4	-0,9	-8,3
Rússia	11,3	10,4	9,6	9,3	9,3	0,0	0,3
Austrália	5,4	5,4	5,4	5,3	5,4	0,1	2,4
Canadá	4,9	4,9	4,7	4,9	5,0	0,1	3,1
Portugal	5,0	4,8	4,7	4,8	4,8	0,0	0,1
África do Sul	3,6	3,7	4,0	4,2	4,4	0,1	3,1
Romênia	4,3	4,6	4,7	3,9	3,8	-0,2	-4,5
Japão	3,1	3,4	3,5	3,5	3,5	0,0	-0,3
Países baixos	3,5	3,5	3,4	3,5	3,4	-0,1	-2,3
Bélgica	2,9	2,9	2,7	3,0	3,0	0,0	1,1
Brasil	3,2	3,5	3,5	3,3	2,9	-0,4	-12,0
Suíça	2,7	2,7	2,8	2,9	2,8	-0,1	-1,8
Áustria	2,7	2,8	3,0	2,4	2,4	0,0	2,0
Sérvia	2,3	2,3	2,4	2,4	2,4	0,0	-0,9
Suécia	2,3	2,4	2,3	2,4	2,3	-0,1	-3,3
Grécia	3,1	3,0	2,6	2,4	2,3	-0,1	-4,4
Chile	3,2	2,9	3,0	2,1	2,2	0,1	4,8
Hungria	2,0	1,9	2,2	2,2	1,9	-0,3	-12,7
Dinamarca	1,5	1,6	1,6	1,6	1,6	0,0	0,0
Croácia	1,4	1,4	1,2	1,1	1,2	0,1	6,7
Polônia	0,9	0,9	1,0	1,1	1,1	0,1	4,9
Bulgária	1,0	0,8	0,9	1,0	1,0	0,0	3,4
World total	244	243	240	241	242	0,9	0,4

Nota: [a]: Países para os quais a informação foi fornecida com consumo de vinho superior a 1 milhão de hectolitros. [b]dados provisórios. [c]: dados previstos. * Consumo aparente calculado pelos dados "Produção + Importações - Exportações" para 2015 e 2016.
Fonte: ORGANISATION INTERNATIONALE DE LA VIGNE ET DU VIN, 2017.

2.2 Produção de vinhos na região do Vale do São Francisco

O Submédio Vale do São Francisco, região situada entre os estados de Pernambuco e Bahia (região semiárido tropical), é uma região caracterizada pelo clima seco e quente e que atualmente emerge na produção de vinhos (Figura 1). Por lá, cada videira gera duas safras por ano, em ciclos de 120 a 130 dias, e são irrigadas com a água do Rio São Francisco. Atualmente, o Vale do São Francisco é responsável pela produção de 5 milhões de litros de vinho por ano (IBRAVIN, [s.d.]).

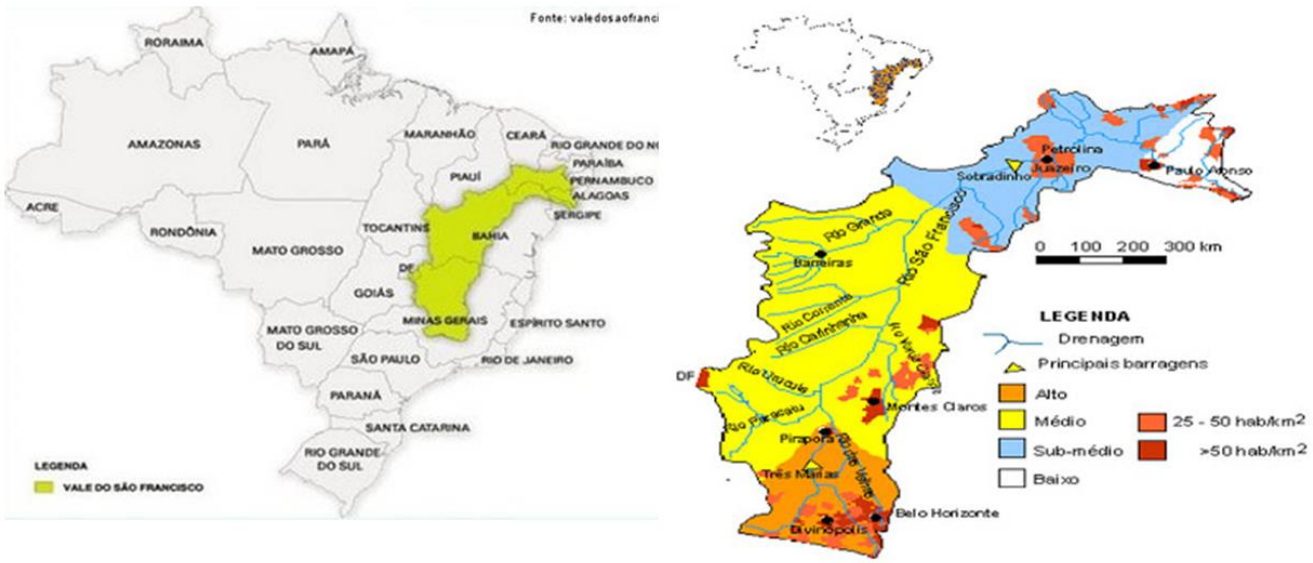

Figura 1. Mapa da região do Vale do São Francisco
Fonte: SOUZA, 2011.

Na região do Vale Submédio do São Francisco, algumas variedades de uvas foram satisfatoriamente adaptadas como as uvas tintas Syrah, Cabernet sauvignon e Alicante Bouschet e as brancas Moscato Canelli, Chenin Blanc e Sauvignon Blanc (GUERRA et al., 2006). A uva Syrah se destaca por ser a que melhor se adaptou a região do semiárido brasileiro (FERREIRA, 2008), sendo o período de maio a setembro o mais favorável para a produção dessa uva, por apresentar menor risco de chuvas e temperaturas mais amenas, com a possibilidade de controle da disponibilidade hídrica do solo pela irrigação (TONIETTO; TEIXEIRA, 2007). O clima e o solo da região propiciam vinhos jovens frutados e aromáticos (FIALHO, 2004).

A viticultura foi introduzida nessa região na década de 1950 pela Companhia de Abastecimento do Vale do São Francisco. Investimentos públicos e privados iniciaram a atividade por meio de plantio de cem mil mudas híbridas de uvas para vinho na região de Petrolândia - PE. No ano seguinte, iniciou-se o plantio das variedades de uva Moscato Italiano, Peverella, Trebbiano, Moscatel de Alexandria, Ferral Preta, Alphonso Lavallee e Alicante Preta no município de Belém de São Francisco - PE (LEÃO, 2010). Na época, verificou-se que as

uvas cultivadas no sul do País poderiam ser cultivadas sob altas temperaturas e condições do semiárido brasileiro no nordeste. Dessa forma, surgiu a uva tropical de alta qualidade e extremamente doce (MINISTÉRIO DA AGRICULTURA, 2014; PEREIRA, 2007).

No ano de 1960, videiras provenientes da Estação Experimental de São Roque, do Instituto Agronômico de Campinas, foram implantadas na região de Petrolina, dando origem a duas estações experimentais, localizadas em Petrolina - PE e Juazeiro - BA (LEÃO, 2010). Em 1975 a Embrapa Semiárido passou a contribuir com o desenvolvimento de novas tecnologias, permitindo o incremento da produção e da qualidade de uvas (PEREIRA, 2007) promovendo, assim, a transformação do cenário agrícola da região. No mesmo ano, dois empreendimentos comerciais, Fazenda Milano e Fazenda Ouro Verde, para produção de vinho e suco de uva foram instalados na região (LEÃO, 2010). Na década de 1990, houve uma melhora na infraestrutura das vinícolas, melhoria do sistema rodoviário e portuário e formação de cooperativas (LEÃO, 2010). O Vale do São Francisco é o segundo maior produtor de vinho no Brasil, atividade que emprega cerca de 30 mil pessoas (PEREIRA, 2007).

O sistema produtivo do vinho no Vale do São Francisco apresenta alguns fatores que contribuem para a vitivinicultura nessa região são: I) disponibilidade de terras para o cultivo de uva para vinho, com produção de 5 safras a cada dois anos; II) base tecnológica de produção irrigada; III) pessoal disponível, qualificado e de baixo custo que atende ao setor; IV) cultura de produção de vinhos trazida há mais de 20 anos da região Sul; V) existência de uma cultura de produção de vinhos jovens trazida da Califórnia – EUA; VI) entrada de capitais externos através de investimentos de empresários na vitivinicultura; VII) distribuição dos vinhos em rede nacional e internacional; VIII) apoio do governo; IX) forte organização e cooperação de entidades de representação técnica e política, que buscam fortalecer esse mercado (VITAL, 2009).

A vitivinicultura no semiárido apresenta um papel importante econômico e social para a região à medida que envolve anualmente negócios voltados para o mercado interno e externo, além de gerar empregos diretos e indiretos (SILVA; COELHO, 2010). A região do Vale do São Francisco tem mostrado vocação para o enoturismo, pois vinculado às vinícolas encontram-se atrativos naturais que refletem a cultura local, como o artesanato e a culinária típica (ZANINI; ROCHA, 2010).

Uma característica importante dos vinhos produzidos no Vale do São Francisco é o elevado teor de compostos fenólicos e alta capacidade antioxidante quando comparado a vinhos de outras regiões do mundo (OLIVEIRA et al., 2017; PADILHA et al., 2017). O vinho da variedade Syrah, em especial, apresenta altas concentrações de antocianina individual e total

(92.1 mg L^{-1}) em comparação, por exemplo, ao vinho Syrah produzido no Chile (74,5 mg L^{-1}) (ANDRADE et al., 2013). Além de antocianinas, o vinho Syrah do São Francisco apresenta também maior concentração de compostos fenólicos como catequina (29,85–94,63 mg L^{-1}), quercitina-3-glicosídeo (34,65-125,52 mg L^{-1}) e resveratrol (0,96-7.41 mg L^{-1}), que o vinho do mesmo varietal produzido na Argentina: catequina (80.04–14,07–45,64 mg L^{-1}), quercitina-3-glicosídeo (7,86–15,72mg L^{-1}) e resveratrol (0.50–2,71mg L^{-1}) (BELMIRO; PEREIRA; PAIM, 2017). Assim, os estudos sugerem que houve uma boa adaptação da Syrah no semiárido nordestino.

2.3 A uva Syrah

Na família das *Vitacea*, as duas espécies mais importantes são *V.vinifera e V.labrusca,* como mostrado no Quadro 1. A maior parte dos vinhos produzidos no mundo é proveniente de *V. vinifera L.*, parreira nativa da Europa e da Ásia Central, tendo-se como exemplos as variedades Chardonnay, Cabernet Sauvignon, Merlot, Riesling, Sauvignon Blanc, Zinfandel e Syrah, entre outras (MACKNEIL, 2003). Dentre as viníferas americanas e seus híbridos estão muito difundidas no Brasil as variedades Concord e Isabel (LONA, 1996).

Quando 1: Classificação botânica da uva.

Ordem: RAMNIDAS	
Família: Vitacea	
Subfamília: AMPELIDEAS	
Gênero: Vitis	Espécie AMERICANA
Subgênero: EUVITIS	Espécie EUROPÉIA – *Vitis vinifera*
	Espécie Asiática

Fonte: LONA, 1996.

A uva Syrah é proveniente do norte do vale do Ródano, no sudeste da França, região onde se concentram pequenos distritos vinícolas. No século XVII, os franceses levaram a uva para o Cabo da Boa Esperança, onde foi denominada Shiraz. Da África do Sul foi levada para a Austrália, onde também é chamada Shiraz. No início da década de 1980, a Syrah foi levada para Califórnia, local promissor para seu cultivo (MACKNEIL, 2003).

Apesar de sua origem em zona de clima temperado, houve a inserção do cultivo da Syrah em países de clima tropical. Os fatores que favorecem o cultivo na área tropical são resistência

a doenças fúngicas, adaptação a condições climáticas extremas e alto rendimento da uva (KOK, 2014). A concentração de compostos fenólicos na uva Syrah é influenciada pelo clima da região produtora, ou seja, em regiões onde ocorre maior radiação solar na videira, encontra-se maior concentração de compostos fenólicos e o vinho apresenta maior atividade antioxidante (SARTOR et al., 2017). Deste modo, o Brasil é um país com bom potencial para produção de uva Syrah de qualidade e fonte de compostos bioativos (SARTOR et al., 2017). O consumo regular desses compostos auxilia na prevenção de várias doenças crônico-degenerativas, incluindo o câncer e as doenças inflamatórias, em função das propriedades neuroprotetoras, antimicrobianas e anti-mutagênicas desses compostos (FOLMER et al., 2014; NILE; PARK, 2014; PEÑA-NEIRA, 2017).

O consumo moderado de vinho (0,6 g/Kg peso corporal) vem sendo associado à melhora da saúde cardiovascular e diminuição da pressão arterial devido à presença dos polifenóis no vinho, que agem através de ação antioxidante para reduzir os danos provocados pelas doenças coronarianas (CHEYNIER, 2005; FIGUEIREDO et al., 2017; GERMAN; WALZEM, 2000).

Os flavonóides compreendem parte dos polifenóis presentes no vinho tinto e são extraídos da casca, semente de uvas durante o processo de maceração (WATERHOUSE, 2005). A semente da uva Syrah é uma importante fonte de compostos fenólicos (282,22 mg $GAEg^{-1}$) (TOUNSI et al., 2009). Além disso, apresenta altas concentrações de antocianinas (403 mg/L), sendo a malvidina a principal espécie presente na casca da uva. A presença das antocianinas influi na densidade da cor e maior concentração de taninos (26 mg/L) nos vinhos produzidos (KILMISTER et al., 2014; SHI et al., 2016).

Segundo GRANATO; KATAYAMA; CASTRO (2012) os vinhos de Syrah e Cabernet Sauvignon produzidos na América do Sul mostram-se superiores em atividade antioxidante com inibição de 64,75 e 66,44% para DPPH e ORAC entre 29801 – 29594 µmol TE^{-1}. Por outro lado, esses vinhos apresentam boa qualidade sensorial quando avaliado o parâmetro de qualidade global da bebida em comparação aos vinhos *Vitis labrusca,* que apresentaram atividade antioxidante de 41,35-45,35 % para inibição de DPPH e ORAC 13285 – 18017 µmol TE^{-1} e menor aceitação global.

GRIS et al. (2013) verificaram que vinho da uva Syrah produzido na região de São Joaquim (SC) apresentou alta concentração de flavonóis, entre 2732,2 e 2790,5 mg L^{-1}, principalmente quercetina e miricetina.

Em outro estudo brasileiro, a uva Syrah cultivada no Vale do São Francisco apresentou concentrações mais elevadas de antocianinas totais (92,1-386 mg L^{-1}), em comparação à uva Syrah cultivada no Chile (75,5 – 199 mg L^{-1}) (ANDRADE et al., 2013).

2.4 Compostos Fenólicos

Os compostos fenólicos presentes no vinho são responsáveis pela cor e sabor, principalmente em vinhos tintos, além de conferir à bebida propriedades que promovem benefícios à saúde. Os compostos fenólicos do vinho dividem-se em dois grupamentos principais, flavonoides e não flavonoides. Os principais flavonóides do vinho são os flavan-3-óis ou flavanóis (taninos, catequina, epicatequina), os flavonóis (miricitina, quercetina, rutina e kaempferol) e as antocianinas (cianidina-3-O-glucosídeo, malvidina-3-O-glucosídeo, peonidina-3-O-glucosídeo, petunidina-3-O-glucosídeo e delfinidina-3-O-glucosídeo). Uma vez que os flavonóis são encontrados na casca e na semente das uvas, sua transferência para o mosto do vinho é determinada principalmente pelo período do tempo maceração no processo de vinificação. A maceração a baixas temperaturas antes da fermentação inicia a extração de antocianinas e procianidinas da casca da uva (CHEYNIER, 2005). Os compostos fenólicos classificados como não flavonoides no vinho são derivados principalmente do ácido benzoico (ácidos vanílico, siringico, gálico e *ρ*-hidroxibenzóico) e do ácido cinâmico (ácidos ρ-cumárico, ferrúlico, cafeico e sinápico). Fazem parte também do grupo dos não-flavonóides os estilbenos (trans-resveratrol), os fenólicos voláteis (eugenol e guaiacol) e os taninos hidrolisáveis, que correspondem a ésteres de ácido gálico e ácido elágico com glicose, extraídos da madeira durante o envelhecimento do vinho (MORENO; PEINADO, 2012; RIBEREAU-GAYON et al., 2006). Para a quantificação da capacidade redutora dos polifenóis em sua totalidade no vinho tinto, englobando taninos condensados e procianidinas, a metodologia mundialmente utilizada normalmente é o método com reagente Folin-Ciocalteu, desenvolvido por Rossi & Singleton (1965).

Dentre os flavonóides, destacam-se as antocianinas, primariamente responsáveis pela cor vermelha característica e que podem ser quantificadas por cromatografia líquida de alta performance (HPLC) (NATIVIDADE et al., 2013; RIBEREAU-GAYON et al., 2006).

Os taninos presentes na casca e semente da uva são polifenóis que conferem adstringência e gosto amargo à bebida, quando utilizadas uvas que não estão maduras para vinificação. A maturação fenólica é um importante estágio na elaboração do vinho. Trata-se da determinação quantitativa e qualitativa dos principais polifenóis de importância enológica da baga da uva. Esse processo serve para definir a época de colheita e o potencial de qualidade de uma determinada safra ou região de cultivo (GUERRA; ZANUS, 2003). A maturação fenólica da uva ocorre quando o teor de antocianinas e taninos da casca é máximo e teor de taninos da

semente começa a decair e se torna relativamente constante. Neste momento, ocorre o equilíbrio entre os taninos da casca e da semente, o que caracterizará adstringência e amargor equilibrados na bebida. Após este ponto, o teor de antocianinas começa a decair e os teores de taninos da casca e da semente permanecem constantes. Se a uva for colhida antes de sua maturação fenólica ideal, provavelmente o vinho originado terá menor intensidade de cor, uma vez que as antocianinas se acumulam por ocasião da maturação. Além disso, o vinho será mais rico em taninos da semente, conferindo elevada adstringência e sensação de "secura" após a ingestão da bebida. Na maturação fenólica coleta-se uma amostra representativa do vinhedo. Separam-se as sementes e as cascas; das primeiras são extraídos os taninos via processo de extração sólido/liquido com auxílio de um solvente orgânico. Posteriormente, são quantificados os taninos totais. Das cascas extraem-se taninos e antocianinas, procedendo-se a análise de taninos totais e antocianinas monoméricas. (GUERRA; ZANUS, 2003).

Os ácidos orgânicos, por sua vez, apresentam grande contribuição para composição, estabilização e qualidade sensorial dos vinhos. Dentre os ácidos orgânicos destacam-se o ácidos acético, málico e tartárico. O ácido acético é um ácido volátil produzido durante a fermentação alcoólica; porém, em níveis elevados, produz alterações sensoriais como aroma avinagrado. O ácido málico presente no vinho deve ser, ao final da fermentação alcoólica, convertido em ácido láctico, pois sua presença na bebida é caracterizada por elevada acidez e intenso amargor (LIMA et al., 2015; RODRÍGUEZ-BENCOMO; POZO-BAYÓN; MORENO-ARRIBAS, 2012).

No vinho, ocorre também precipitação do ácido tartárico na forma de tartarato ácido de potássio, sal pouco solúvel em álcool. Durante o resfriamento após a fermentação alcóolica, ocorre a formação de cristais que aderem às paredes do tanque formando camadas espessas de bitartarato de potássio e tartarato neutro de cálcio (CORDONNIER, 2007). Em vinhos tintos, menores concentrações de ácidos promovem melhor estabilização durante o envelhecimento (RIBÉREAU-GAYON et al., 2006b).

2.5 Elaboração do vinho tinto

Os vinhos finos elaborados com uvas do gênero *Vitis* podem ser classificados como monovarietal ou de corte. O vinho de corte é um vinho proveniente da mistura de mostos ou vinhos de diferentes variedades de uvas, podendo abranger diferentes colheitas. Já o vinho monovarietal é preparado com uvas de uma só variedade (RAY, 2001). De acordo com a legislação brasileira, o vinho pode ser classificado como de mesa, leve, fino, espumante,

frisante, gaseificado, licoroso, composto, e por cor, tinto, rosado ou rosé ou clarete e branco, como exemplificado no Quadro 2 (BRASIL, 2014).

Na elaboração do vinho tinto ocorre a maceração, ou seja, as cascas das uvas permanecem por certo tempo em contato com o mosto. Esta etapa é de grande importância para extração das substâncias de cor e dos taninos presentes na casca da uva e semente, respectivamente. Assim, o processo de maceração possibilita que o enólogo personalize o vinho aplicando tempos de maceração diferenciados para cada vinho produzido (RIBÉREAU-GAYON et al., 2006a).

A qualidade da uva influencia diretamente na qualidade do vinho, assim fatores como técnicas de cultivo, maturação da uva e condições fitossanitárias devem ser consideradas no momento da colheita do fruto. Para realizar o controle do amadurecimento das uvas, 50 bagas de diferentes cachos são colhidas e seu mosto analisado quanto ao ºBrix, acidez e pH. Assim, quando as uvas chegam ao amadurecimento necessário, começa a colheita ou vindima (BARBA; LIZARRITURRY, 2007).

Quadro 2: Classificação dos vinhos comercializados no Brasil.

Classificação do vinho	Definição
Mesa	Vinho com teor alcoólico de 8,6% a 14% e volume podendo conter até uma atmosfera de pressão a 20ºC.
Leve	Vinho leve é o vinho com teor alcoólico de 7% a 8,5% em volume, obtido exclusivamente da fermentação dos açúcares naturais da uva, produzido durante a safra nas zonas de produção, vedada sua elaboração a partir de vinho de mesa.
Fino	Vinho de teor alcoólico de 8,6% a 14% em volume, elaborado mediante processos tecnológicos adequados que assegurem a otimização de suas características sensoriais e exclusivamente de variedades Vitis vinifera do grupo nobres.
Espumante	Vinho cujo anidrido carbônico provém da fermentação em recipiente fechado, de mosto ou de mosto conservado de uva moscatel, com uma pressão mínima de 4 atmosferas a 20ºC, e com um teor alcoólico de 7% a 10% em volume, e no mínimo 20 gramas de açúcar remanescente.
Frisante	Vinho com teor alcoólico de 7% a 14% em volume, e uma pressão mínima de 1,1 a 2,0 atmosferas a 20ºC, natural ou gaseificado.
Gaseificado	Vinho resultante da introdução de anidrido carbônico puro, por qualquer processo, devendo apresentar um teor alcoólico de 7% a 14% em volume, e uma pressão mínima de 2,1 a 3,9 atmosferas a 20ºC.
Licoroso	Vinho resultante da introdução de anidrido carbônico puro, por qualquer processo, devendo apresentar um teor alcoólico de 7% a 14% em volume, e uma pressão mínima de 2,1 a 3,9 atmosferas a 20ºC.
Composto	Bebida com teor alcoólico de 14% a 20% em volume, elaborado pela adição ao vinho de mesa de macerados ou concentrados de plantas amargas ou aromáticas, substâncias de origem animal ou mineral, álcool etílico potável de origem agrícola, açúcar, caramelo e mistela simples.

Fonte: BRASIL, 2004.

As etapas básicas para vinificação (RIBÉREAU-GAYON et al., 2006a) que serão descritas nessa seção são:

- Colheita, desengace, esmagamento e enchimento do tanque;
- Maceração e fermentação alcoólica;
- Prensagem (separação das bagas do mosto);
- Fermentação malolática;
- Estabilização;
- Engarrafamento

O processo de elaboração do vinho está exemplificado na Figura 2.

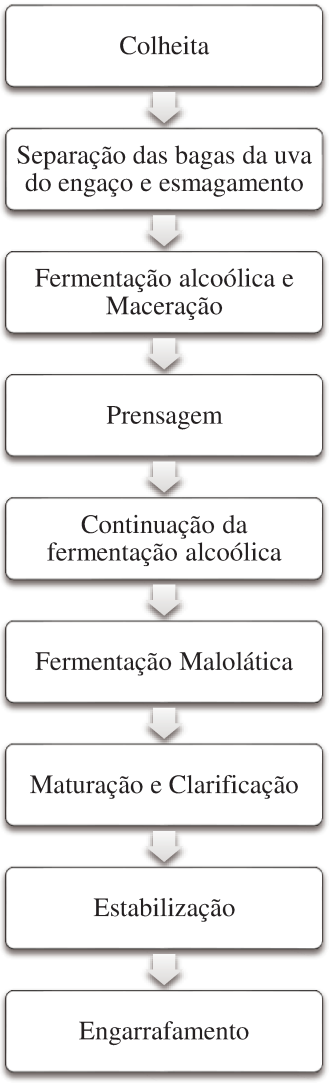

Figura 2. Fluxograma do processo de vinificação.

Fonte: JACKSON, 2008.

Os cuidados básicos para produção dos vinhos começam no momento da colheita e no pós-colheita das uvas, que devem ser colhidas no período da manhã, preservando-se sua

integridade física em caixas contentoras higienizadas. Também devem ser selecionados cachos sadios e frescos, excluindo uvas com contaminação microbiana, mantidos em caixas plásticas contentoras, sob refrigeração, da chegada até seu processamento em no máximo 12 horas após a colheita (LONA, 1996; RIBÉREAU-GAYON et al., 2006a).

A separação das bagas da uva do engaço é a primeira etapa do processo de vinificação (WUCHERPFENNIG, 2003). Nesse momento, o equipamento também promove o esmagamento, que consiste no rompimento da película da baga e tem como objetivo liberar o mosto contido na polpa para que este entre em contato com o ar e as leveduras presentes na superfície da película. Dessa maneira, as uvas são parcialmente ou completamente esmagadas contribuindo para o início da fermentação alcoólica e a extração dos pigmentos (RIBÉREAU-GAYON et al., 2006a).

Após, o mosto é acondicionado em barris de carvalho ou tanques de inox durante sete dias, e no primeiro dia é realizada a inoculação da levedura *Saccharomyces cerevisiae*. Esta etapa é denominada maceração, sendo essencial para a extração de aromas, antocianinas e taninos, provenientes da semente da uva, polpa e casca. Concomitantemente a esse processo, ocorre a fermentação alcoólica (HARTMEIER; REISS, 2002; JACKSON, 2014; WUCHERPFENNIG, 2003). Após o vinho ser acondicionado no tanque é adicionado dióxido de enxofre com o intuito de inibir o crescimento de microrganismos deteriorantes, prevenir a oxidação e escurecimento da bebida, a quantidade recomendada varia de 50 a 100 mg/L (RIBÉREAU-GAYON et al., 2006a).

A fermentação alcoólica consiste em transformar glicose e frutose em álcool. Os dois açúcares fermentáveis são transformados em etanol e gás carbônico conforme a equação global (1):

$$C_6H_{12}O_6 \rightarrow 2CH_3CH_2OH + 2CO_2 + 2ATP \quad (1)$$

Cerca de 1% dos açúcares é reservado à respiração quando o meio ainda contém oxigênio e outros 10% seguem a via de fermentação gliceropirúvica e promovem a formação de produtos secundários. O balanço da glicólise se traduz pela formação de duas moléculas de ATP originadas da fermentação alcoólica. Essa bioenergia é disponibilizada para o crescimento das leveduras e síntese de seus componentes celulares (anabolismo). Nesse nível, são formados compostos voláteis, como álcoois superiores e ésteres. A multiplicação celular é acompanhada e seguida pela fase catabólica da autólise da biomassa formada. O teor alcoólico do vinho varia de 9 a 15% em volume para taxas de glicerol de 6 a 12 gramas (CORDONNIER, 2007; MACNEIL, 2003). A fermentação alcoólica é esquematizada na Figura 3.

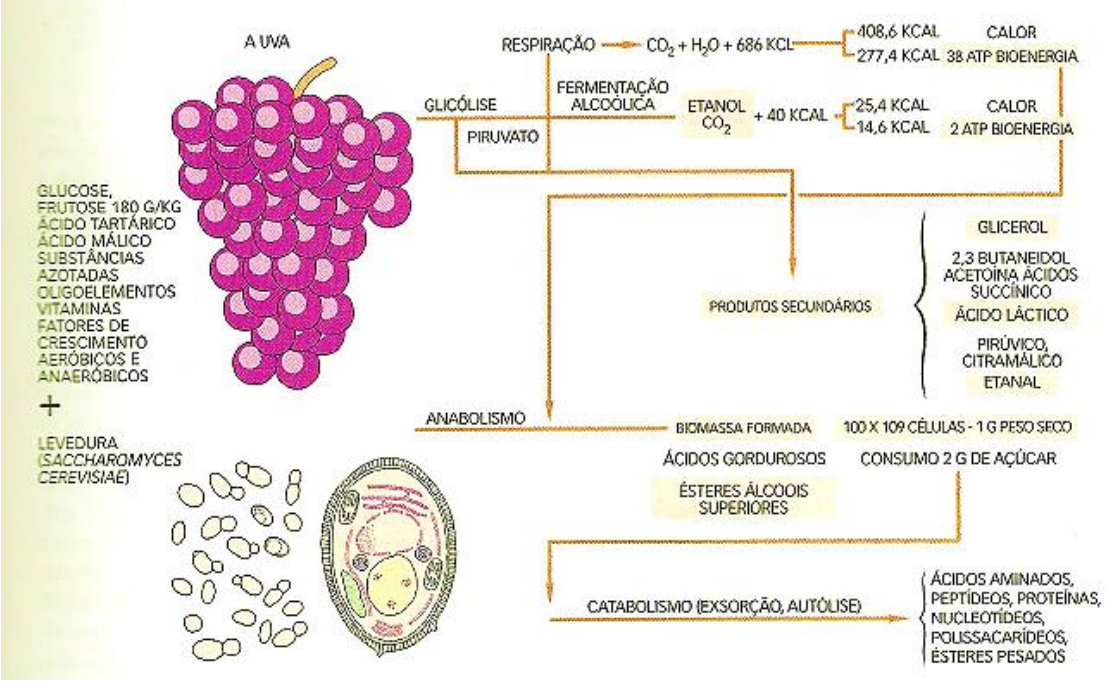

Figura 3. Fermentação alcoólica

Fonte: CORDONNIER, 2007.

Durante a fermentação alcoólica o controle de temperatura é essencial na vinificação. Mostos com elevada concentração de açúcar, ou para serem bebidos jovens devem ter a temperatura controlada e relativamente constante entre 25 – 28 ºC. Essa temperatura favorece a coloração do vinho, além de promover aroma frutado para a bebida. Nessa etapa do processo, é importante que se realize no tanque de fermentação bombeamentos diários do mosto, processo denominado aeração e que consiste em oxigenar o mosto que está sendo fermentado. A esquematização do processo pode ser observada na Figura 4. Além da oxigenação o processo promove outros benefícios como: homogeneização da temperatura, do açúcar e das leveduras, promove maior extração de antocianinas e taninos, melhorando o processo de maceração, evita a formação de aromas indesejáveis (DAY et al., 2015; RIBÉREAU-GAYON et al., 2006a).

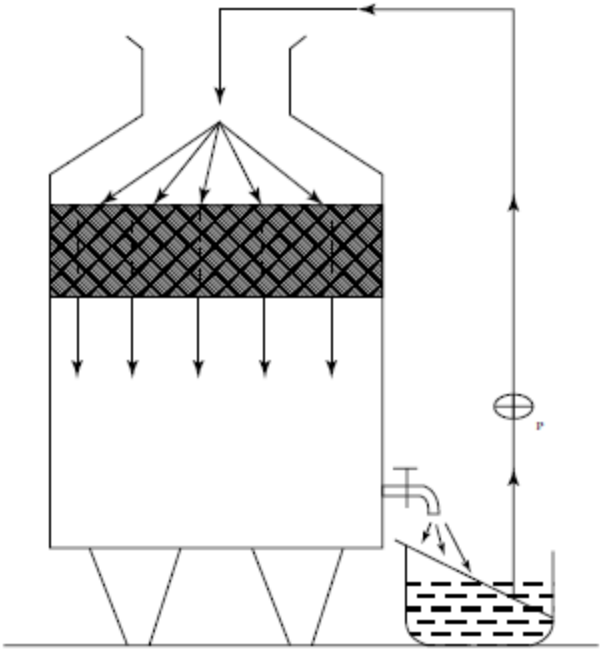

Figura 4. Operação de bombeamento, mostrando a aeração do mosto (P=bomba).
Fonte: RIBÉREAU-GAYON et al., 2006.

Durante a fermentação alcoólica, alguns controles de processo são necessários como a verificação da densidade. A fermentação é finalizada quando a densidade do vinho está em torno de 0.991 - 0.996 g / cm^3. Além disso, é importante verificar a quantidade de açúcar presente no vinho, no qual deve ser inferior a 2 g / L (HARTMEIER; REISS, 2002; RIBÉREAU-GAYON et al., 2006a). Ao término da maceração, as bagas são prensadas e o vinho é então transferido para um novo tanque. Essa etapa do processo contribui para o aumento do volume final da bebida e quantidade de taninos (RIBÉREAU-GAYON et al., 2006a).

Ao final da fermentação alcoólica, o vinho apresenta excesso de acidez, que prejudica sua qualidade sensorial e é proveniente dos ácidos tartárico e málico, sendo que este último é degradado a ácido láctico (Figura 5) quase completamente através de uma segunda fermentação, chamada de fermentação malolática. Essa fermentação acontece de forma espontânea ou não e nesse estágio, a temperatura deve ser controlada entre 18 e 20 ºC. Para cessar a atividade das bactérias láticas, inibir o crescimento de microrganismos, impedir o escurecimento e a oxidação são adicionadas doses de dióxido de enxofre entre 30 e 100 ppm (CORDONNIER, 2007; MACNEIL, 2003).

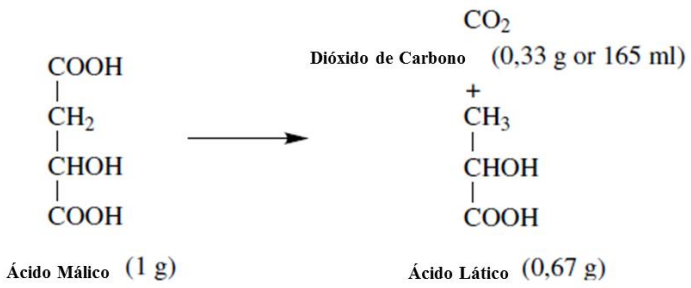

Figura 5. Reação da fermentação malolática.
Fonte: RIBÉREAU-GAYON et al., 2006.

Durante a fermentação malolática é importante determinar o início e final dessa fermentação e a depleção do ácido málico nos tanques, é analisado pela cromatografia em papel. Apesar de não ser um método muito preciso, ela é amplamente aplicada nas vinícolas, sendo monitorado diariamente a depleção de ácido málico (RIBÉREAU-GAYON et al., 2006a; AVILA; DAUDT, 1997).

Após o término da fermentação malolática, o vinho passa pelo processo de estabilização tartárica em tanque de aço inoxidável, sendo refrigerado e conservado durante 8 a 10 dias entre -3 a -4 ºC (CONDDONIER, 2007).

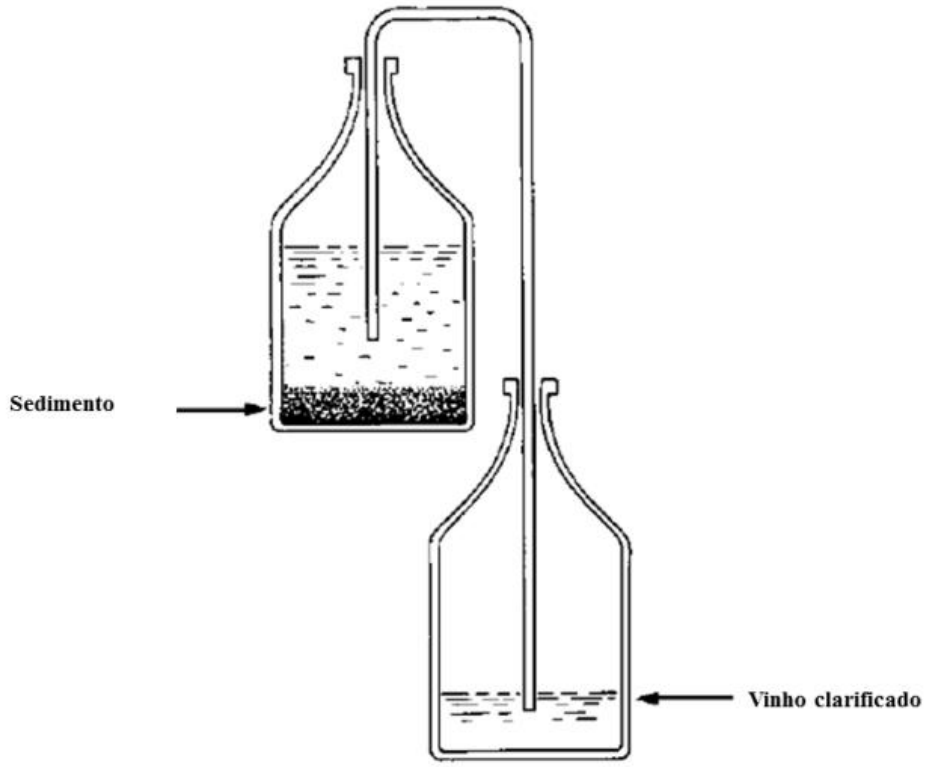

Figura 6. Processo de clarificação do vinho
RIBÉREAU-GAYON et al., 2006a.

Posteriormente o vinho é submetido à fase de maturação, envelhecimento ou guarda, na qual é fundamental a oxigenação para a polimerização dos taninos de baixo peso molecular. Tradicionalmente, a guarda do vinho é realizada em barris de carvalho – francês ou americano. A função principal do carvalho é promover a oxigenação natural e gradual do vinho e essa fase compreende um período de oito meses a um ano. A maturação promove a formação dos aromas do vinho (LONA, 1996). Ao final dessa fase, o vinho é submetido ao processo de clarificação através de técnicas de centrifugação e/ou filtragem, a fim de reter as leveduras e partículas em suspensão (Figura 6) (HARTMEIER; REISS, 2002; JACKSON, 2014; RIBÉREAU-GAYON et al., 2006a; WUCHERPFENNIG, 2003). Esta etapa do processo pode provocar perdas no conteúdo de polifenóis extraídos na etapa de maceração e também reduzir a intensidade da cor do vinho (GARRIDO; BORGES, 2013).

Finalmente o vinho é envasado a frio, de forma estéril para assegurar que a bebida não tenha contaminação microbiológica (HARTMEIER; REISS, 2002; JACKSON, 2014; MACNEIL, 2003; WUCHERPFENNIG, 2003).

2.5.1 Maceração

O processo de maceração realizado em vinhos tintos é responsável principalmente pela extração de compostos fenólicos (antocianinas e taninos) presentes na casca e semente da uva (Figura 7), os quais promovem benefícios sensoriais para bebida como cor, sabor e aroma (RIBÉREAU-GAYON et al., 2006a).

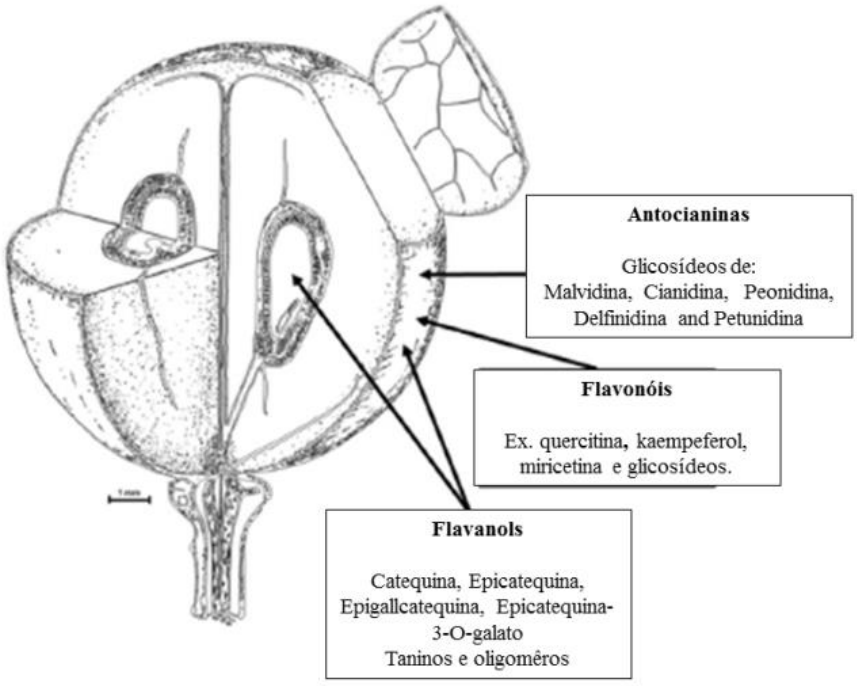

Figura 7. Localização dos compostos fenólicos na uva.
Fonte: SETFORD et al., 2017.

Tradicionalmente a maceração ocorre durante a fermentação alcoólica. Atualmente, existem vários tipos de maceração que são realizados diferenciando-se do método tradicional.

I. Termovinificação, que consiste na extração em alta temperatura antes da fermentação. O processo de maceração é realizado com as uvas intactas ou levemente esmagadas, aquecidas a 50-70 ºC por um curto período (60 °C por 30 minutos ou 80 °C por 3 minutos). Esse processo resulta no aumento da concentração de compostos fenólicos, especialmente o resveratrol, e da atividade antioxidante (CVEJIĆ; ATANACKOVIĆ, 2015; RIBÉREAU-GAYON et al., 2006b; SETFORD et al., 2017). O processo de termovinificação pode ocasionar degradação térmica de compostos aromáticos como

terpenóides, norisoprenóides e alguns polifenóis e o aumento de compostos aromáticos como α-terpineol, guaiacol e 2,6-dimetoxifeno (GEFFROY et al., 2015).

II. Extração em baixa temperatura antes da fermentação, com objetivo de promover vinhos com altas concentrações de compostos fenólicos e aromas complexos. Nesse processo os mostos são resfriados a 5 ºC durante 5-15 dias. O período de pré-fermentação promove um mosto com cor intensa. Posteriormente o vinho é colocado na temperatura tradicional e a fermentação alcoólica prossegue (JACKSON, 2008; RIBÉREAU-GAYON et al., 2006a; SETFORD et al., 2017).

III. Maceração pós fermentação, é realizada para melhorar a qualidade de vinhos tintos superiores. Ao término da fermentação, as cascas da uva permanecem em contato com o vinho podendo aumentar a temperatura do tanque e dessa forma proporcionar maior extração de compostos fenólicos (RIBÉREAU-GAYON et al., 2006a).

IV. Maceração carbônica, nesse processo as bagas da uva são mantidas em atmosfera de dióxido de carbono e a fermentação ocorre de maneira espontânea. Após o período de uma a duas semanas as uvas são prensadas e o mosto inoculado com levedura. Esse tipo de maceração aumenta a concentração de polifenóis totais do mosto (CVEJIĆ; ATANACKOVIĆ, 2015)

Na maceração, a extração de compostos fenólicos varia de acordo com o tempo, sendo maior a extração de compostos fenólicos e antocianinas (intensidade de cor) nos primeiros 8-10 dias do processo (Figura 3). Para maior extração dos taninos presentes na semente da uva, é necessária uma maceração mais longa (20 a 40 dias) (RIBÉREAU-GAYON et al., 2006a).

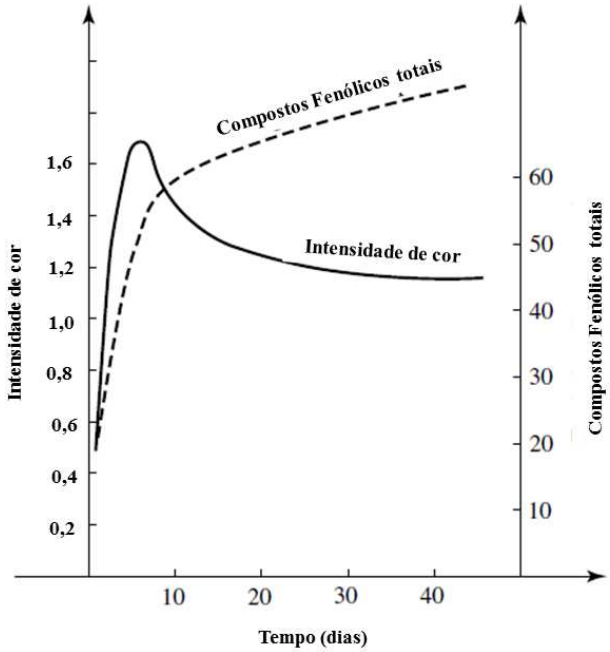

Figura 8. Evolução da intensidade de cor e concentração de compostos fenólicos durante o tempo de maceração.
Fonte: RIBÉREAU-GAYON et al., 2006.

A maceração prolongada pode aumentar a concentração de resveratrol; no entanto, é importante enfatizar que a concentração final de compostos fenólicos no vinho depende também da variedade da uva e do processo de vinificação (CVEJIĆ; ATANACKOVIĆ, 2015). Essa técnica também contribui para um incremento 11 a 18% da extração de taninos promovendo maior adstringência para o vinho. Nos primeiros dias de maceração, a maior contribuição da extração dos taninos é da casca da uva, enquanto ao final da fermentação e maceração prolongada, a extração de taninos é da semente. Nesse contexto, a maior extração de taninos durante a maceração estendida ocorre no período de 10 a 20 dias (SMITH; MCRAE; BINDON, 2015; WATERHOUSE; SACKS; JEFFERY, 2016).

Alguns aspectos da vinificação favorecem a extração de antocianinas durante a maceração, como por exemplo, o etanol formado na fermentação alcoólica, a temperatura de fermentação alcoólica (20 a 30ºC), aeração do vinho e a técnica de termovinificação que impulsionam a extração de antocianinas e promovem estabilização da cor da bebida (DURNER, 2016).

2.5.2 Envelhecimento do vinho

O envelhecimento em madeira para vinhos tintos é uma técnica muito tradicional e comumente aplicada para vinhos. O envelhecimento em madeira promove uma série de vantagens, sendo a principal a estabilidade da cor vermelha dos vinhos. A estabilidade de cor deve-se a compostos derivados da madeira (ácido gálico, ácido siríngico, ácido vanilico, ácido ferrulico, ácido elágico e elagitaninos), que migram da madeira para o vinho (DURNER, 2016). Por outro lado, também são extraídos os taninos hidrolisáveis, cuja quantidade irá depender do tempo de envelhecimento do vinho na madeira (SMITH; MCRAE; BINDON, 2015).

O envelhecimento em madeira é tipicamente realizado em barril, por no mínimo seis meses. Esse processo aumenta a concentração de muitos compostos de aroma importantes para características sensoriais do vinho, como as lactonas de carvalho (*Oak lactones)* que são os principais constituintes de aroma dessa madeira e são relacionadas com aromas característicos de frutas vermelhas, baunilha e coco (JACKSON, 2014).

Dois grandes produtores de madeira para barris são a França e os Estados Unidos. Na França, as espécies de madeira *Quercus robur* e *Quercus petraea* são produzidas sendo que a espécie *Quercus robur* é caracterizada por alta extração de fenólicos e baixa concentração de compostos odoríferos, enquanto a espécie *Quercus petraea* apresenta alto potencial aromático. Nos Estados Unidos a espécie dominante é o carvalho branco *Quercus alba*, cuja madeira é caracterizada por baixa concentração de fenólicos, e alta concentração de compostos de aroma, especialmente o composto metil-octalactona que influencia o aroma do vinho durante o envelhecimento (RIBÉREAU-GAYON et al., 2006b).

A madeira destinada para envelhecimento do vinho passa pelo processo de tosta, classificada como leve, média ou forte. Cada tipo de tosta pode influenciar o sabor e o aroma do vinho durante o envelhecimento. Além disso, a queima da madeira provocará degradação dos elagitaninos e ácido gálico, e isso ocorre devido a estes compostos serem sensíveis ao tratamento térmico (ALAÑÓN et al., 2011; CHIRA; TEISSEDRE, 2015; RIBÉREAU-GAYON et al., 2006b).

Atualmente, a adição de chip de carvalho tem sido estudada como alternativa econômica de substituição do tradicional processo em barril. Os valores sugeridos de adição de chip situam-se em torno de 10g / L / ano para vinhos brancos e duas vezes mais para vinhos tintos. Em geral, para chips com diâmetro ≤ 1mm cerca de 90% dos compostos são extraídos em 1 semana. Em geral, o envelhecimento com chip ocorre durante a fermentação. Dessa forma, ocorre uma rápida extração de taninos e fenólicos devido ao extenso contato do chip com o vinho (JACKSON, 2014).

A produção de vinhos em barril de carvalho e com a utilização de chip possui vantagens e desvantagens que são detalhadas no Quadro 3.

Quadro 3: Vantagens e desvantagens dos métodos de envelhecimento em barril de carvalho e com chip de carvalho.

Método de envelhecimento	Vantagem	Desvantagem
Barril de carvalho	Vinhos produzidos em barril apresentam aromas de carvalho mais suaves.	Possui custo elevado, pois o carvalho utilizado é produzido no meio-oeste dos EUA, denominado de carvalho americano ou produzido na parte central da França denominado de carvalho francês. Dessa forma, há a necessidade das vinícolas brasileiras importarem o barril. O encaixe das aduelas deve ser bem ajustado, para que evite vazamento ou processo oxidativo da bebida, pela entrada excessiva de oxigênio no barril. Os barris usados durante muitos anos tendem a transferir menor quantidade de aromas e sabores do carvalho.
Chip de Carvalho	Chip de carvalho que utiliza pequenos fragmentos de madeira de carvalho. Baixo custo comparado ao barril. Aumento da transferência de compostos de aroma. Em curto período os vinhos desenvolvem aromas típicos da madeira semelhantes a vinhos maturados em barril de carvalho	Quando não se tem o controle do processo, o excesso de madeira promove efeitos negativos como excesso de aroma e sabor de madeira.

Fonte: CEJUDO-BASTANTE; HERMOSÍN-GUTIÉRREZ; PÉREZ-COELLO, 2011a, 2011b; MACNEIL, 2003; TAO; GARCÍA; SUN, 2014.

A utilização de chip de carvalho para envelhecimento de vinho é autorizada de acordo com as especificações do Codex Internacional Enológico desde o ano de 2005 (*Resolution oeno 3/2005*), sendo considerada uma alternativa de envelhecimento substituindo o barril. No entanto, os pedaços de madeira devem ser exclusivamente do gênero *Quercus* (OIV, 2007).

A literatura apresenta vários estudos que testam a quantidade de chip de madeira a ser utilizada e a etapa de introdução durante o processo de vinificação. Em geral, a quantidade utilizada nos estudos com vinho tinto variam de 3-9 g / L (ALAÑÓN et al., 2013; CEJUDO-BASTANTE; HERMOSÍN-GUTIÉRREZ; PÉREZ-COELLO, 2011a; GÓMEZ GARCÍA-CARPINTERO; SÁNCHEZ-PALOMO; GONZÁLEZ VIÑAS, 2014; GORDILLO et al., 2013; HERNÁNDEZ-ORTE et al., 2014). O chip de carvalho pode ficar em contato com o vinho em diferentes estágios do processo vinificação podendo ser adicionado na fermentação alcoólica, malolática ou após os processos fermentativos do vinho (GALLEGO et al., 2015). A etapa de

adição do chip e a dose utilizada provocam significativa influência na composição química e sensorial do vinho (SÁNCHEZ-PALOMO et al., 2017). GARCÍA-CARPINTERO et al. (2012) observaram que vinhos fermentados com a mistura de chip de carvalho francês e americano durante a fermentação alcoólica mostraram concentrações mais altas de ésteres etílicos, enquanto maiores concentrações de compostos furânicos, lactonas de carvalho foram encontradas em envelhecidos durante a fermentação malolática.

A utilização do chip possibilita em curto período que os vinhos desenvolvam aromas típicos da madeira advindos de compostos fenólicos e cores semelhantes às de vinhos maturados em barril de carvalho (CEJUDO-BASTANTE; HERMOSÍN-GUTIÉRREZ; PÉREZ-COELLO, 2011a; EIRIZ; OLIVEIRA; CLÍMACO, 2007; KOUSSISSI et al., 2009; TAO; GARCÍA; SUN, 2014).

O chip de carvalho possibilita uma rápida e progressiva extração de compostos aromáticos se comparado ao processo em barril. Ao comparar o envelhecimento no barril com chip de carvalho, para que o composto aromático furfural seja extraído do chip na mesma concentração observada em barril, são necessários de cinco a seis dias, e ao final de 14 dias, a taxa de extração dobra. Esse exemplo mostra a rápida progressão de extração do chip de carvalho, favorecendo assim o rápido envelhecimento do vinho (ARAPITSAS et al., 2004).

Os vinhos envelhecidos em madeira, assim como os vinhos jovens, são muito apreciados pelos consumidores. A técnica de envelhecimento em madeira é utilizada com o objetivo de melhorar as características sensoriais da bebida. Assim o envelhecimento proporciona ao vinho um aumento da complexidade de aromas em comparação a vinhos jovens, com descritores de aroma que lembram cravo, baunilha e madeira. Quando são utilizadas madeiras com torra forte, aromas e sabor de fumaça e tabaco podem caracterizar a bebida, o que pode mascarar outros aromas e sabores do vinho menos intensos (SCHUMACHER et al., 2013). O envelhecimento contribui também para o aumento da doçura percebida no vinho, que pode estar associado à extração de vanilina (GONZÁLEZ-CENTENO; CHIRA; TEISSEDRE, 2016; KYRALEOU et al., 2015). Os métodos de envelhecimento em barril e chip promovem características sensoriais muito semelhantes, no entanto, o chip de carvalho apresenta a vantagem da maior velocidade de envelhecimento.

O chip de carvalho contribui para retardar a mudança de cor no vinho durante o envelhecimento (LIU et al., 2016), pois os elagintaninos e os compostos fenólicos liberados pelo chip de carvalho podem consumir o oxigênio durante o envelhecimento e, dessa forma, o contato da madeira desempenha um papel importante contra a oxidação do vinho (NAVARRO et al., 2016).

2.6 Padrões de Identidade de vinhos no Brasil

O Ministério da Agricultura Pecuária e Abastecimento (MAPA) é o órgão responsável no Brasil por regulamentar a elaboração e comercialização de vinhos. De acordo com a Instrução Normativa n° 14, de 08 de fevereiro de 2018, a composição de vinhos tintos de mesa ou finos deve respeitar os limites estabelecidos, como mostra a Tabela 2.

Tabela 2: Limites estabelecidos para comercialização de vinhos de mesa e finos.

Parâmetros	Mínimo	Máximo
Graduação alcoólica, em % vol/vol, a 20ºC	8,6	14,0
Acidez total, em mEq/l	40,0	130,0
Acidez volátil, em mEq/l	-	20,0
Sulfatos totais, expresso em sulfato de potássio, em g/l - para vinhos que passaram por, no mínimo 2 anos de envelhecimento	-	1,2
Cloretos totais, expresso cloreto de sódio, em g/l	-	0,2
Cinzas, em g/l:		
Vinho tinto de mesa de americanas ou híbridas	1,5	-
Vinho branco ou rosado de mesa de americanas ou híbridas	1,3	-
Vinho tinto de mesa de viníferas	1,5	-
Vinho branco ou rosado de mesa de viníferas	1,0	-
Álcool metílico, em mg/l	-	
Tinto		400,0
Brancoe rosado		300,00
Edulcorante	Ausência	
Corante artificial	Ausência	

Fonte: BRASIL, 2018.

2.7 Propriedades sensoriais e aceitabilidade do vinho

Atualmente são muitas as evidências científicas que apresentam benefícios do consumo moderado de vinho a saúde e, por isso, existe um crescente interesse dos consumidores (CHANG; LIZ THACH; OLSEN, 2016; VECCHIO et al., 2017; YOO; SALIBA; PRENZLER, 2010).

Dentre os vinhos tintos, a Cabernet Sauvignon é a variedade de uva mais preferida entre os consumidores. Outros fatores que influenciam na escolha do vinho são a experiência prévia com bebida, (KALLAS; ESCOBAR; GIL, 2013), a origem (SILLANI; MICCOLI; NASSIVERA, 2017) e a rotulagem que exerce influência sobre as expectativa do consumidor (LICK et al., 2017).

As propriedades sensoriais do vinho influenciam diretamente a aceitação da bebida. Dessa maneira, nas últimas décadas, a indústria vinícola e centros de pesquisa começaram a aplicar metodologias de análise sensorial para determinar o perfil sensorial de vinhos, além das preferências e percepções dos consumidores. A utilização dos testes sensoriais auxilia os enólogos a identificar atributos sensoriais que possam contribuir positiva ou negativamente para a aceitação da bebida e ajustar técnicas de vinificação tornando o vinho mais apreciável pelos consumidores (BAKER; CASTURA; ROSS, 2016; FRANCIS; WILLIAMSON, 2015).

Muitas técnicas de vinificação são utilizadas com o objetivo de melhorar as características sensoriais do vinho e, como exemplo dessas técnicas podem ser citados a maceração (prolongada, a frio, termovinificação) e o envelhecimento do vinho (BAIANO et al., 2016). A maceração prolongada (30 dias) pode causar cor marrom mais pronunciada, e resultar em vinhos mais adstringentes, devido à alta concentração de taninos (FEDERICO CASASSA et al., 2013). Outra técnica de vitivinicultura que interfere no perfil sensorial do vinho é a irrigação. Vinhos de uva Syrah produzidos com uvas de vinhedos com sistema de irrigação no norte da Grécia, região com clima semiárido, foram caracterizados como mais adstringentes por avaliadores treinados (KYRALEOU et al., 2016).

Os vinhos da variedade Syrah produzidos na América do Sul diferem no perfil sensorial em relação aos descritores de aroma e sabor e isto ocorre devido à influência do *terroir* no vinho. O solo, a topografia da região vitivinícola e o clima são elementos que interagem e determinam o microclima da região produtora (LLOBODANIN; BARROSO; CASTRO, 2014). Nesse contexto, cada região produzirá um vinho Syrah com características sensoriais muito específicas. Enquanto um vinho Syrah produzido na Austrália apresentou perfil sensorial caracterizado por aroma de frutas em compota, frutas vermelhas, baunilha, persistência gustativa, sabor de fruta e caramelo e gosto doce (LATTEY; BRAMLEY; FRANCIS, 2010), um vinho Syrah produzido na Grécia foi caracterizado pelos atributos aroma frutado, sabor frutado, atributos relacionados a aroma e sabor de madeira, aroma de baunilha e especiarias, encorpados e de sabor alcoólico (KOUSSISSI; PATERSON; CRISTOVAM, 2002). Outro estudo com vinhos Syrah californianos e italianos mostrou na análise sensorial descritiva que aroma de frutas e geléia caracterizaram o vinho italiano, ao passo que o vinho californiano foi caracterizado por aromas de chocolate e carvalho. No mesmo estudo verificou-se que os vinhos californianos foram significativamente preferidos ($P < 0{,}0001$) aos italianos (TORRI; NOBLE; HEYMANN, 2013). Assim, o *terroir* é considerado um fator importante na determinação do perfil sensorial dos vinhos.

Na comercialização de vinho, além dos aspectos sensoriais, é importante considerar as emoções sentidas pelos consumidores. No estudo desenvolvido por DANNER et al. (2016), um vinho Syrah australiano definido por provadores treinados como de alta qualidade foi o mais apreciado e provocou nos consumidores emoções positivas e intensas, por exemplo, contente, entusiasmado, feliz, otimista e apaixonado quando comparado a um vinho de baixa qualidade. Além disso, as emoções também influenciam na compra do vinho, mostrando que os consumidores estão dispostos a pagar mais por produtos que eliciam emoções positivas e intensas.

Alguns estudos apontam que parece não existir diferença na preferência dos consumidores quanto a vinhos envelhecidos em barril ou com chips de carvalho (PÉREZ-MAGARIÑO; ORTEGA-HERAS; GONZÁLEZ-SANJOSÉ, 2011). Dessa maneira, os dois tipos de envelhecidos podem coexistir no mercado uma vez que refletem diferentes processos tecnológicos e mesmo regionalidades (PÉREZ-MAGARIÑO; ORTEGA-HERAS; GONZÁLEZ-SANJOSÉ, 2011).

3. REFERENCIAS

ALAÑÓN, M. E. et al. A study of the antioxidant capacity of oak wood used in wine ageing and the correlation with polyphenol composition. **Food Chemistry**, v. 128, p. 997–1002, 2011.

ALAÑÓN, M. E. et al. Enological potential of chestnut wood for aging Tempranillo wines Part II: Phenolic compounds and chromatic characteristics. **Food Research International**, v. 51, n. 2, p. 536–543, 2013.

ANDRADE, R. H. S. DE et al. Anthocyanic composition of Brazilian red wines and use of HPLC-UV – Vis associated to chemometrics to distinguish wines from different regions. **Microchemical Journal**, v. 110, p. 256–262, 2013.

ARAPITSAS, P. et al. Artificial aging of wines using oak chips. **Food Chemistry**, v. 86, p. 563–570, 2004.

ARCARI, S. G. et al. Brazilian forti fi ed wines : Chemical composition , chromatic properties and antioxidant activity. **Food rearch international**, v. 53, n. 1, p. 164–173, 2013.

ATHIA, F.; COSTA, A. D. Empresas e tecnologias na nova conjuntura vinícola brasileira do início do século XXI. **Economia & Tecnologia**, v. 18, p. 147–156, 2009.

AVILA, L. D. DE; DAUDT, C. E. Indução da fermentação maloláctica em vinho tinto com a cultura láctica viniflora oenos. **Ciencia Rural**, v. 27, n. 2, p. 325–330, 1997.

BAIANO, A. et al. Wine Processing: A Critical Review of Physical, Chemical, and Sensory Implications of Innovative Vinification Procedures. **Critical Reviews in Food Science and Nutrition**, v. 56, n. 14, p. 2391–2407, 2016.

BAKER, A. K.; CASTURA, J. C.; ROSS, C. F. Temporal Check-All-That-Apply Characterization of Syrah Wine. **Journal of Food Science**, v. 81, n. 6, p. 1521–1529, 2016.

BARBA, L. M.; LIZARRITURRY, M. **Elaboração do vinho**. Barcelona: Folio, 2007.

BARREIRO-HURLE, J.; COLOMBOA, S.; CANTOS-VILLAR, E. s there a market for functional wines? Consumer preferences and willingness to pay for resveratrol-enriched red wine. **Food Quality and Preference**, v. 19, p. 360–371, 2008.

BELMIRO, T. M. C.; PEREIRA, C. F.; PAIM, A. P. S. Red wines from South America: Content of phenolic compounds and chemometric distinction by origin. **Microchemical Journal**, v. 133, p. 114–120, 2017.

BISSON, L. F. et al. The present and future of the international wine industry. **Nature**, v. 418, n. 6898, p. 696–699, 8 ago. 2002.

BORTOLETTO, A. M.; ALCARDE, A. R. Aging marker profile in cachaça is influenced by toasted oak chips. **Journal of the Institute of Brewing**, v. 121, n. 1, p. 70–77, 2015.

BRASIL. **Lei nº 10970, de 12 de novembro de 2004. Dispõe sobre a produção, circulação e**

comercialização do vinho e derivados da uva e do vinho, e dá outras providências. Brasilia: [s.n.].

BRASIL. **INSTRUÇÃO NORMATIVA N° 14, DE 08 DE FEVEREIRO DE 2018. Complementação dos Padrões de Identidade e Qualidade do Vinho e Derivados da Uva e do Vinho.** Brasilia: [s.n.].

BRASIL. **DECRETO Nº 8.198, DE 20 DE FEVEREIRO DE 2014. Regulamenta a Lei no 7.678, de 8 de novembro de 1988, que dispõe sobre a produção, circulação e comercialização do vinho e derivados da uva e do vinho.** Brasilia: [s.n.].

CAMARGO, U. A.; TONIETTO, J.; HOFFMAN, A. Progressos na viticultura brasileira. **Revista Brasileira de Fruticultura**, v. 33, 2011.

CASSI, L.; MORRISON, A.; RABELLOTTI, R. Proximity and Scientific Collaboration: Evidence from the global wine industry. **Wine Economics**, v. 149, p. 1–28, 2014.

CEJUDO-BASTANTE, M. J.; HERMOSÍN-GUTIÉRREZ, I.; PÉREZ-COELLO, M. S. Micro-oxygenation and oak chip treatments of red wines : Effects on colour-related phenolics , volatile composition and sensory characteristics . Part II : Merlot wines. **Food Chemistry**, v. 124, p. 738–748, 2011a.

CEJUDO-BASTANTE, M. J.; HERMOSÍN-GUTIÉRREZ, I.; PÉREZ-COELLO, M. S. Micro-oxygenation and oak chip treatments of red wines : Effects on colour-related phenolics , volatile composition and sensory characteristics . Part II : Merlot wines. **Food Chemistry**, v. 124, p. 738–748, 2011b.

CEJUDO-BASTANTE, M. J.; RIVERO-GRANADOS, F. J.; HEREDIA, F. J. Improving the color and aging aptitude of Syrah wines in warm climate by wood-grape mix maceration. **European Food Research and Technology**, v. 243, n. 4, p. 575–582, 2017.

CHANG, K. J.; LIZ THACH, M.; OLSEN, J. Wine and health perceptions: Exploring the impact of gender, age and ethnicity on consumer perceptions of wine and health. **Wine Economics and Policy**, v. 5, n. 2, p. 105–113, 2016.

CHEYNIER, V. Flavonoids in Wine. In: ANDERSEN, O.; MARKHAM, K. (Eds.). . **Flavonoids: Chemistry, Biochemistry and Applications**. Boca Raton: CRC Press, 2005. p. 263–318.

CHIRA, K.; TEISSEDRE, P.-L. Chemical and sensory evaluation of wine matured in oak barrel : effect of oak species involved and toasting process. **European Food Research Technology**, v. 240, p. 533–547, 2015.

CORDONNIER, R. A bioquímica do vinho. **Scientific American Brasil**, v. 1, p. 22–35, 2007.

CVEJIĆ, J.; ATANACKOVIĆ, M. Effect of Wine Production Techniques on Wine Resveratrol and Total Phenolics. In: **Processing and Impact on Active Components in Food**. Elsevier ed. [s.l.] Elsevier, 2015. p. 501–508.

DANNER, L. et al. Context and wine quality effects on consumers' mood, emotions, liking and

willingness to pay for Australian Shiraz wines. **Food Research International**, v. 89, p. 254–265, nov. 2016.

DAY, M. P. et al. Use and impact of oxygen during winemaking. **Australian Journal of Grape and Wine Research**, v. 21, p. 693–704, 2015.

DE OLIVEIRA, W. P. et al. Phenolics from Winemaking By-Products Better Decrease VLDL-Cholesterol and Triacylglycerol Levels than Those of Red Wine in Wistar Rats. **Journal of Food Science**, v. 82, n. 10, p. 2432–2437, 2017.

DIAS, F. D. S. et al. Fast determination of phenolic compounds in brazilian wines from Vale do São Francisco region by ce. **Chromatographia**, v. 76, n. 9–10, p. 559–563, 2013.

DURNER, D. Improvement and Stabilization of Red Wine Color. In: **Handbook on Natural Pigments in Food and Beverages: Industrial Applications for Improving Food Color**. [s.l.] Elsevier Ltd, 2016. p. 240–264.

EIRIZ, N.; OLIVEIRA, J. F. S.; CLÍMACO, M. C. Fragmentos de carvalho no estágio de vinhos tintos. **Ciência Técnica Vitivinícola**, v. 22, n. 2, p. 63–71, 2007.

FEDERICO CASASSA, L. et al. Effect of extended maceration and ethanol concentration on the extraction and evolution of phenolics, colour components and sensory attributes of Merlot wines. **Australian Journal of Grape and Wine Research**, v. 19, n. 1, p. 25–39, 2013.

FERREIRA, P. Brasil inova na produção de vinhos tropicais. **Inovação em Pauta**, n. 2, p. 16–19, 2008.

FIALHO, V. Z. B. **Brasil é referência na produção de vinhos tropicais**. Disponível em: <http://www.cnpuv.embrapa.br/noticias/2004/2004-09-13.html>. Acesso em: 21 maio. 2014.

FIGUEIREDO, E. A. DE et al. Antioxidant and Antihypertensive Effects of a Chemically Defined Fraction of Syrah Red Wine on Spontaneously Hypertensive Rats. **Nutrients**, v. 9, n. 6, p. 574, 2017.

FLINT, D. J.; SIGNORI, P.; GOLICIC, S. L. A Global Overview of Wine. In: **Contemporary Wine Marketing and Supply Chain Management**. New York: Palgrave Macmillan, 2016. p. 1–14.

FOLMER, F. et al. Anticancer effects of bioactive berry compounds. **Phytochemistry Reviews**, v. 13, n. 1, p. 295–322, 2014.

FRANCIS, I. L.; WILLIAMSON, P. O. Application of consumer sensory science in wine research. **Australian Journal of Grape and Wine Research**, v. 21, p. 554–567, 2015.

GALLEGO, M. A. G. et al. Effect of oak chip addition at different winemaking stages on phenolic composition of Moravia Agria red wines. **South African Journal for Enology and Viticulture**, v. 36, n. 1, p. 21–31, 2015.

GARCÍA-CARPINTERO, E. G. et al. Impact of alternative technique to ageing using oak chips in alcoholic or in malolactic fermentation on volatile and sensory composition of red wines.

Food Chemistry, v. 134, p. 851–863, 2012.

GARRIDO, J.; BORGES, F. Wine and grape polyphenols — A chemical perspective. **Food Research International**, v. 54, n. 2, p. 1844–1858, 2013.

GEFFROY, O. et al. Changes in analytical and volatile compositions of red wines induced by pre-fermentation heat treatment of grapes. **Food Chemistry**, v. 187, p. 243–253, 2015.
GERMAN, J. B.; WALZEM, R. L. The health benefits of wine. **Annual review of nutrition**, v. 20, p. 561–593, 2000.

GÓMEZ GARCÍA-CARPINTERO, E.; SÁNCHEZ-PALOMO, E.; GONZÁLEZ VIÑAS, M. A. Volatile composition of Bobal red wines subjected to alcoholic/malolactic fermentation with oak chips. **LWT - Food Science and Technology**, v. 55, n. 2, p. 586–594, 2014.

GONZÁLEZ-CENTENO, M. R.; CHIRA, K.; TEISSEDRE, P. L. Ellagitannin content, volatile composition and sensory profile of wines from different countries matured in oak barrels subjected to different toasting methods. **Food Chemistry**, v. 210, p. 500–511, 2016.

GORDILLO, B. et al. Application of the differential colorimetry and polyphenolic profile to the evaluation of the chromatic quality of Tempranillo red wines elaborated in warm climate. Influence of the presence of oak wood chips during fermentation. **Food Chemistry**, v. 141, n. 3, p. 2184–2190, 2013.

GORDILLO, B. et al. Optimisation of an oak chips-grape mix maceration process . Influence of chip dose and maceration time. **Food Chemistry**, v. 206, p. 249–259, 2016.
GORTZI, O. et al. Effect of artificial ageing using different wood chips on the antioxidant activity, resveratrol and catechin concentration, sensory properties and colour of two Greek red wines. **Food Chemistry**, v. 141, n. 3, p. 2887–2895, 2013.

GRANATO, D.; KATAYAMA, F. C. U.; CASTRO, I. A. DE. Characterization of red wines from South America based on sensory properties and antioxidant activity. **Journal of science food and agriculture**, v. 92, n. May, p. 526–533, 2012.

GRIS, E. F. et al. Phenolic profile and effect of regular consumption of Brazilian red wines on in vivo antioxidant activity. **Journal of Food Composition and Analysis**, v. 31, n. 1, p. 31–40, 2013.

GUERRA, C. C. et al. Vinhos Tropicais: nova paradigma enológico e mercadológico. **Informe Agropecuário**, v. 27, n. 234, p. 100–104, 2006.

GUERRA, C. C.; ZANUS, M. C. Uvas viníferas para processamento em regiões de clima temperado. **Embrapa Uva e Vinho Sistema de Produção**, v. 4, 2003.

HARTMEIER, W.; REISS, M. Production of Beer and Wine. In: **Industrial Applications**. Berlin, Heidelberg: Springer Berlin Heidelberg, 2002. p. 49–65.

HERNÁNDEZ-ORTE, P. et al. Criteria to discriminate between wines aged in oak barrels and macerated with oak fragments. **Food Research International**, v. 57, p. 234–241, mar. 2014.

HORNSEY, I. S. Chapter 1. The History of Wine. In: **The Chemistry and Biology of**

Winemaking. Cambridge: Royal Society of Chemistry, 2007. p. 11–64.

HUI, Y. H. et al. **Handbook of Food and Beverage Fermentation Technology**. 1st Editio ed. Boca Raton: CRC Press, 2004.

IBRAVIN, I. B. DO V. **Regiões produtoras**. Disponível em: <http://www.ibravin.org.br/Regioes-Produtoras>. Acesso em: 21 nov. 2017.

IBRAVIN, I. B. DO V. **Vinho fino recuou 30,5%, com a comercialização de dois milhões de litros. Foto: Gilmar Gomes Venda de vinhos brasileiros tem recuo de 20% no trimestre**. Disponível em: <http://www.ibravin.org.br/Noticia/venda-de-vinhos-brasileiros-tem-recuo-de-20-no-trimestre/260>. Acesso em: 11 nov. 2017a.

IBRAVIN, I. B. DO V. **Comercialização e Importação**. Disponível em: <http://www.ibravin.org.br/Dados-Estatisticos>. Acesso em: 11 nov. 2017b.

INSEL, B. The evolving global wine market. **Business Economics**, v. 49, n. 1, p. 56–48, 2014.

INSTITUTO BRASILEIRO DE VINHO. **Panorama Geral**. Disponível em: <http://www.ibravin.org.br/Panorama-Geral>. Acesso em: 21 nov. 2017.

JACKSON, R. S. **Wine science: Principles and application**. Academic P ed. California: Elsevier, 2008.

JACKSON, R. S. Vineyard Practice. In: **Wine Science**. Elsevier, 2014. p. 143–306.

JEZIORMY, D. L.; ORTEGA, A. C. Inovação e performance competitiva na indústria vitivinícola brasileira. **Ensaios FEE**, v. 33, p. 864–886, 2013.

KALLAS, Z.; ESCOBAR, C.; GIL, J. M. Analysis of consumers' preferences for a special-occasion red wine: A dual response choice experiment approach. **Food Quality and Preference**, v. 30, n. 2, p. 156–168, 2013.

KILMISTER, R. L. et al. A role for anthocyanin in determining wine tannin concentration in Shiraz. **Food Chemistry**, v. 152, p. 475–482, 2014.

KOK, D. A Review on Grape Growing in Tropical Regions. **Türk Tarım ve Doğa Bilimleri**, p. 1236–1241, 2014.

KOUSSISSI, E. et al. Influence of toasting of oak chips on red wine maturation from sensory and gas chromatographic headspace analysis. **Food Chemistry**, v. 114, n. 4, p. 1503–1509, 2009.

KOUSSISSI, E.; PATERSON, A.; CRISTOVAM, E. Sensory Discrimination of Dry Red Wines from Greece. **Journal of Wine Research**, v. 13, n. 2, p. 165–179, 2002.

KYRALEOU, M. et al. Differentiation of Wines Treated with Wood Chips Based on Their Phenolic Content, Volatile Composition, and Sensory Parameters. **Journal of Food Science**, v. 80, n. 12, p. C2701–C2710, 2015.

KYRALEOU, M. et al. Effect of irrigation regime on perceived astringency and proanthocyanidin composition of skins and seeds of Vitis vinifera L. cv. Syrah grapes under semiarid conditions. **Food Chemistry**, v. 203, p. 292–300, 2016.

LAGO, L. O. et al. Influence of ripeness and maceration of the grapes on levels of furan and carbonyl compounds in wine – Simultaneous quantitative determination and assessment of the exposure risk to these compounds. **Food Chemistry**, v. 230, p. 594–603, 2017.

LATTEY, K. A.; BRAMLEY, B. R.; FRANCIS, I. L. Consumer acceptability, sensory properties and expert quality judgements of Australian Cabernet Sauvignon and Shiraz wines. **Australian Journal of Grape and Wine Research**, v. 16, n. 1, p. 189–202, 2010.

LEÃO, A. et al. Valores pessoais dos consumidores de vinhos do Vale do São Francisco (Brasil): Uma proposição tipológica de perfis. **Revista Portuguesa e Brasileira de Gestão**, p. 23–36, 2011.

LEÃO, P. C. DE S. Breve histórico da vitivinicultura e a sua evolução na região semiárida brasileira. **Anais da Academia Pernambucana de Ciência Agronômica**, v. 7, p. 81–85, 2010.

LICK, E. et al. Sensory expectations generated by colours of red wine labels. **Journal of Retailing and Consumer Services**, v. 37, n. April 2016, p. 146–158, 2017.

LIMA, M. DOS S. et al. Phenolic compounds , organic acids and antioxidant activity of grape juices produced in industrial scale by different processes of maceration. **Food Chemistry**, v. 188, p. 384–392, 2015.

LIU, S. et al. Effect of Oak Chips on Evolution of Phenolic Compounds and Color Attributes of Bog Bilberry Syrup Wine During Bottle-Aging. **Jounal of Food Science**, v. 81, n. 11, p. 2697–2707, 2016.

LLOBODANIN, L. G.; BARROSO, L. P.; CASTRO, I. A. Sensory characterization of young south american red wines classified by varietal and origin. **Journal of Food Science**, v. 79, n. 8, 2014.

LOCKSHIN, L.; CORSI, A. M. Consumer behavior for wine 2.0: A review since 2003 and future directions. **Wine Economics and Policy**, v. 1, p. 2–23, 2012.

LONA, A. A. **Vinho – Degustação, Elaboração e Serviço.** Porto Alegre: Age Ltda, 1996.

LUCENA, A. P. S. et al. Antioxidant activity and phenolics content of selected Brazilian wines. **Journal of Food Composition and Analysis**, v. 23, n. 1, p. 30–36, 2010.

MACKNEIL, K. Como se prepara o vinho. In: **A bíblia do vinho**. Rio de Janeiro: Ediouro, 2003. p. 39–52.

MACNEIL, K. **A Bíblia do Vinho**. Rio de Janeiro: Editouro, 2003.

MINISTÉRIO DA AGRICULTURA. **Saiba mais**. Disponível em: <http://www.agricultura.gov.br/vegetal/culturas/uva/saiba-mais>. Acesso em: 20 abr. 2014.

MORENO, J.; PEINADO, R. **Polyphenols**. EUA: Elsevier, 2012.

NATIVIDADE, M. M. P. et al. Simultaneous analysis of 25 phenolic compounds in grape juice for HPLC: Method validation and characterization of São Francisco Valley samples. **Microchemical Journal**, v. 110, p. 665–674, 2013.

NAVARRO, M. et al. Oxygen consumption by oak chips in a model wine solution ; Influence of the botanical origin , toast level and ellagitannin content. **Food Chemistry**, v. 199, p. 822–827, 2016.

NIERDELE, P. A.; VITROLLES, D. Indicações Geográficas e qualificação no setor vitivinícola brasileiro. **Estudos Sociedade e Agricultura**, v. 18, n. 1, p. 5–55, 2010.

NILE, S. H.; PARK, S. W. Edible berries: Bioactive components and their effect on human health. **Nutrition**, v. 30, n. 2, p. 134–144, 2014.

OIV. **Pieces of oak wood**. [s.l: s.n.].

OIV, O. I. DE LA V. ET DU V. **State of the Vitiviniculture World Market**. [s.l: s.n.].

OLIVEIRA, W. P. DE et al. Phenolics from Winemaking By-Products Better Decrease VLDL-Cholesterol and Triacylglycerol Levels than Those of Red Wine in Wistar Rats. **Journal od Food Science**, 2017.

OU BX, CHANG T, H. D. AND P. R. Phenolic compounds, organic acids and antioxidant activity of grape juices produced from new Brazilian varieties planted in the Northeast Region of Brazi. **J Aoac Int**, v. 96, p. 1372–1376, 2013.

PADILHA, C. V. DA S. et al. Phenolic compounds profile and antioxidant activity of commercial tropical red wines (Vitis vinifera L.) from São Francisco Valley, Brazil. **Journal of Food Biochemistry**, v. 41, n. 3, p. 1–9, 2017.

PAN-MONTOJO, J. Las vitiviniculturas europeas: de la primeira a la segunda glabalización. **Mundo Agrario**, v. 9, n. 18, 2009.

PEÑA-NEIRA, A. Grapes. In: YAHIA, E. M. (Ed.). . **Fruit and Vegetable Phytochemicals**. 2nd. ed. Chichester, UK: John Wiley & Sons, Ltd, 2017. v. II p. 1041–1054.

PEREIRA, G. E. **Notas Técnicas**. Disponível em: <www.vinhovasf.com.br/site/internas/valetecnico.php>. Acesso em: 10 abr. 2014.

PEREIRA, G. E. Os vinhos tropicais em desenvolvimento no Nordeste do Brasil. **Com Ciência**, n. 149, 2013.

PÉREZ-MAGARIÑO, S.; ORTEGA-HERAS, M.; GONZÁLEZ-SANJOSÉ, M. L. Wine consumption habits and consumer preferences between wines aged in barrels or with chips. **Journal of the Science of Food and Agriculture**, v. 91, n. 5, p. 943–949, 30 mar. 2011.

PROTAS, J. F. S. **Vitivinicultura brasileira: panorama setorial de 2010**. Brasilia: [s.n.].

RAY, J. **O vinho tinto**. Barcelona: Folio, 2001.

RENFREW, J. M. Archaeology and the origins of wine production. In: MERTON SANDLER, R. P. (Ed.). . **Wine**. 1st Edition ed. London: CRC Press, 2003. p. 56–69.

RIBEREAU-GAYON, P. et al. Phenolic Compounds. In: **Handbook of Enology: The Chemistry of Wine and Stabilization and Treatments.** [s.l: s.n.]. v. 2p. 141–203.

RIBÉREAU-GAYON, P. et al. Making Red Table Wines. In: **Handbook of Enology**. Chichester, UK: John Wiley & Sons, Ltd, 2006a. p. 327–395.

RIBÉREAU-GAYON, P. et al. Red Winemaking. In: **Handbook of Enology**. Chichester, UK: John Wiley & Sons, Ltd, 2006b. v. 1p. 327–395.

RODRÍGUEZ-BENCOMO, J. J.; POZO-BAYÓN, M. Á.; MORENO-ARRIBAS, M. V. Wine Fermentation and Production. In: HUI, Y. H.; EVRANUZ, E. Ö. (Eds.). . **Handbook of Plant-Based Fermented Food and Beverage Technology**. [s.l.] CRC Press, 2012. p. 179–200.

ROSA, S. E. S.; SIMÕES, P. M. Desafios da vitivinicultura brasileira. **BNDS Setorial**, n. 19, p. 67–90, 2004.

ROSSI, J. A.; SINGLETON, V. L. Colorimetry of total phenolics winth phosphomolybdic phosphotungstic acid reagents. **American Journal of Enology and Viticulture**, v. 16, p. 144–158, 1965.

SÁNCHEZ-PALOMO, E. et al. Improvement of Verdejo white wines by contact with oak chips at different winemaking stages. **LWT - Food Science and Technology**, v. 79, p. 111–118, 2017.

SARTOR, S. et al. Particularities of Syrah wines from different growing regions of Southern Brazil: grapevine phenology and bioactive compounds. **Journal of Food Science and Technology**, v. 54, n. 6, p. 1414–1424, 2017.

SCHUMACHER, R. et al. Evaluation of Oak Chips Treatment on Volatile Composition and Sensory Characteristics of Merlot Wine. **Jounal of Food Quality**, v. 36, p. 1–9, 2013.

SETFORD, P. C. et al. **Factors affecting extraction and evolution of phenolic compounds during red wine maceration and the role of process modellingTrends in Food Science and Technology.** Elsevier Ltd, , 2017. Disponível em: <https://doi.org/10.1016/j.tifs.2017.09.005>.

SHI, P. B. et al. Phenolic compound profiles in grape skins of Cabernet Sauvignon, Merlot, Syrah and Marselan Cultivated in the Shacheng Area (China). **South African Journal of Enology and Viticulture**, v. 37, n. 2, p. 132–138, 2016.

SILLANI, S.; MICCOLI, A.; NASSIVERA, F. Different preferences for wine communication. **Wine Economics and Policy**, v. 6, n. 1, p. 28–39, 2017.

SILVA, P. C. G. DA; COELHO, R. C. **A viticultura no Submédio do Vale São Francisco: Importância econômica e social da videira**. Disponível em:

<http://www.cpatsa.embrapa.br:8080/sistema_producao/spuva/Caracterizaca_social_da_videira.html>. Acesso em: 21 nov. 2017.

SMITH, P. A.; MCRAE, J. M.; BINDON, K. A. Impact of winemaking practices on the concentration and composition of tannins in red wine. **Australian Journal of Grape and Wine Research**, v. 21, p. 601–614, 2015.

SOUZA, M. L. V. **Rio São Francisco**. Disponível em: <geografiaeletrica.blogspot.com.br/2011/10/rio-sao-francisco.html>. Acesso em: 1 jan. 2014.

TAO, Y.; GARCÍA, J. F.; SUN, D. Advances in Wine Aging Technologies for Enhancing Wine Quality and Accelerating Wine Aging Process. **Critical Reviews in Food Science and Nutrition**, v. 54, p. 817–835, 2014.

THE AUSTRALIAN WINE RESEARCH INSTITUTE. **Progress in wine technology slowed down**. Disponível em: <http://www.winehistory.com.au/wiki/Wine_History/Progress_in_wine_technology_slowed_down>. Acesso em: 16 nov. 2017.

TONIETTO, J.; TEIXEIRA, A. H. DE C. **O clima vitícola do submédio são francisco e o zoneamento dos períodos de produção de uvas para elaboração de vinhos. I Workshop Internacional de Pesquisa**. 2007.

TORRI, L.; NOBLE, A. C.; HEYMANN, H. Exploring American and Italian consumer preferences for Californian and Italian red wines. **Journal of the Science of Food and Agriculture**, v. 93, n. 8, p. 1852–1857, 2013.

TOUNSI, M. S. et al. Valorization of three varieties of grape. **Industrial Crops and Products**, v. 30, n. 2, p. 292–296, 2009.

TOUSSAINT-SAMAT, M. From the Vine to Wine. In: **A History of Food**. [s.l: s.n.]. p. 223–262, 2009.

VECCHIO, R. et al. European consumers' perception of moderate wine consumption on health. **Wine Economics and Policy**, v. 6, n. 1, p. 14–22, 2017.

VITAL, T. Vitivinicultura no Nordeste Brasileiro: Situação Recente e Perspectivas. **Revista Econômica do Nordeste**, v. 40, n. 3, p. 500–524, 2009.

WATERHOUSE, A. L. The Phenolic wine antioxidants. In: CADENAS, E.; PACKER, L. (Eds.). . **Handbook of antioxidants**. New York: Marcel Dekker, 2005.

WATERHOUSE, A. L.; SACKS, G. L.; JEFFERY, D. W. Maceration and Extraction of Grape components. In: **Understanding Wine Chemistry**. England: John Wiley & Sons, 2016. p. 179–193.

WUCHERPFENNIG, K. WINES | Production of Table Wines. In: **Encyclopedia of Food Sciences and Nutrition**. [s.l.] Elsevier, 2003. p. 6195–6203.

WYLER, P. et al. Effect of oak wood on the quality of beer. **Journal of the Institute of**

Brewing, v. 121, n. 1, p. 62–69, 2015.

YOO, Y. J. et al. A cross-cultural study of wine consumers with respect to health benefits of wine. **Food Quality and Preference**, v. 28, n. 2, p. 531–538, 2013.

YOO, Y. J.; SALIBA, A. J.; PRENZLER, P. D. Should red wine be considered a functional food? **Comprehensive Reviews in Food Science and Food Safety**, v. 9, n. 5, p. 530–551, 2010.

ZANINI, T. V.; ROCHA, J. M. O enoturismo no Brasil: um estudo comparativo entre as regiões vinícolas do Vale dos Vinhedos (RS) e do Vale do São Francisco (BA/PE). **Turismo em Análise**, v. 21, n. 1, p. 68–88, 2010.

CAPÍTULO 2

Influência do tempo de maceração nos compostos fenólicos e atividade antioxidante de mosto e vinho Syrah.

Natália Manzatti Machado Alencar; Cinthia Baú Betim Cazarin; Luiz Cláudio Corrêa; Mário Roberto Maróstica Junior; Aline Camarão Telles Biasotos; Jorge Herman Behrens. Influence of maceration time on phenolic compounds and antioxidant activity of the Syrah must and wine. **Journal of Food Biochemistry,** v. 22, n. 2, 2018.

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INFLUENCE OF MACERATION TIME ON PHENOLIC COMPOUNDS AND ANTIOXIDANT ACTIVITY OF THE SYRAH MUST AND WINE

Running title: Influence of prolonged maceration time.

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ABSTRACT

The main objective of the present study was to evaluate the effect of the maceration time up to 30 days on the phenolic compounds, monomeric anthocyanins, color intensity and antioxidant activity (FRAP, DPPH, ORAC) of both must and Syrah wine produced in the São Francisco Valley, Brazil. Up to the 15th day, the maceration process promoted an increase in phenolic compounds, while the major extraction of anthocyanins occurred up to the 20th days. The results showed the concentration of total phenolic compounds stabilized in 20th days until the end of maceration and it was higher in comparison with the concentration found at the beginning. Consequently, prolonging the maceration time to 20 days not only improved the phenolic compounds profile but also the antioxidant activity of the tropical Syrah wine.

PRACTICAL APPLICATIONS

The maceration process in winemaking is an important step aimed at extracting and increasing the concentration of phenolic compounds and, as a consequence, the antioxidant activity of the wine improves as well. In practical terms, this process takes about 5 days to complete in some wineries. This paper demonstrates that higher maceration times, up to 20 days, have improve the concentration of phenolic compounds in tropical wines produced with Syrah grapes and such improvement has a great impact not only in the sensory properties but in antioxidant properties of the beverage, which may be positively seen by the consumer.

Keywords: color stability; tropical red wine; *trans*-resveratrol; bioactive compounds, malvidin-3-O-glycoside, prolonged maceration.

1. INTRODUCTION

Brazil is part of the new world of wine and the São Francisco Valley region in the Brazilian northeast is a distinctive area to produce wines. Located at 30 m altitude, between the evenly-matched 8° and 9° S, the region is characterized by high insolation, average temperature of 27°C and rainfall around 500 mm per year. These conditions provide scaled grape production in two and half harvests a year (Sá, Silva, & Bandeira, 2015). The Syrah grape variety is well adapted to the São Francisco Valley and it has been used to produce tropical young wines,

which has contributed to improve the enological potential of this region (Lima, Leite, Sampaio, Vianello, & Lima, 2015). Wines produced there show high concentration of bioactive compounds like malvidin-3-O-glycoside, (+) - catechin, procyanidin B2, gallic acid, syringic acid, kaempferol-3-O-glucoside and rutin . The anthocyanins content is also higher than the Chilean Syrah wine, and this difference could be linked to the northeastern environmental conditions (temperature, illumination and temperature range) that are favorable to the synthesis and accumulation of these pigments (Andrade, Nascimento, Pereira, Hallwass, & Paim, 2013).

The maceration process generally occurs simultaneously to the alcoholic fermentation, and it is characterized by the time the grape skins and seeds stay in contact with the must, under controlled temperature to avoid loss of aromatic compounds and to reduce the risk posed by spoilage organisms. Usually, this process happens in 5 days in some wineries, but research has pointed out the effects of prolonged soaking time (7, 10 or 30 days) in the quality of wine. Increasing the maceration time is useful to increase the concentration of anthocyanins, phenolic compounds and, consequently, the antioxidant activity of wine improves (Gomez-Mıguez, Gonzalez-Miret, & Heredia, 2007; Ivanova-Petropulos, Durakova, Ricci, Parpinello, & Versari, 2016; Lingua, Fabani, Wunderlin, & Baroni, 2016; Kelebek et al., 2006). Besides, maceration is applied in wines that will be submitted to oak-aging to improve the color stability (Heredia, Escudero-gilete, Hernanz, Gordillo, & Meléndez-martínez, 2010; Gordillo et al., 2016). Nonetheless, it is necessary to consider the phenolic compounds extractability, that may promote a high level astringency (Rinaldi, Iturmendi, Jourdes, Teissedre, & Moio, 2015). There are few studies evaluating the sensory profile and acceptance of red wines with prolonged maceration (30 days). The work of Cadot, Caillé, Samson, Barbeau, & Cheynier (2012) studied the impact of maturing stage and maceration time (15 days) of the Cabernet Franc grape, and observed that astringency and color intensity increased with maceration time. Federico Casassa, Beaver, Mireles, & Harbertson (2013) studied the effect of prolonged 30-day maceration on the wine sensory attributes of the Merlot varietal, concluding that the prolonged maceration duration increased the extraction of seed tannins which, in turn, resulted in higher astringency. For the foregoing, this study aimed to evaluate the phenolic compounds, antioxidant activity, color intensity and sensory profile of the wine produced with Syrah tropical grapes (*Vitis vinifera* L.) for 30 days of maceration cultivated in São Francisco Valley, Brazil.

2. MATERIALS AND METHODS

2.1 Grapes

Syrah grapes (*Vitis vinifera* L.), from a seven years old vineyard, were used in winemaking. The vines were grown in experimental field belonging to Embrapa Semi-Arid Station at Petrolina, State of Pernambuco, Brazil, situated at 09 ° 09 'S, 40 ° 22 'W, in an altitude of 365,5 m. The grapes were harvested in July 2015, transported to the Enology Laboratory. Physical chemical analyses were performed to characterize the grape must regarding to: soluble solids (ºBrix), reducing sugars, pH, total acidity, volatile acidity, and density were determined according to the Association of Official Analytical Chemists (2007) .

2.2 Vinification

At the start, the grapes were manually selected and stored for 10 hours in a cold chamber at 10°C to decrease the temperature.

The grapes were then destemmed with the aid of a commercial destemmer. Afterwards, the grapes were transferred to a 50 liters stainless steel vat. Maceration carried out in a one single 50 liters stainless steel tank with the addition of potassium metabisulfite (0.10 g L^{-1}). Pectinolytic enzyme Everum Thermp Everintec® (0.01 g L^{-1}), commercial yeast Maurivin PDM Coatec® *Saccharomyces cerevisiae* bayanus (0.20 g L^{-1}) and activating ammonium phosphate Gesferm plus Coatec® (0.20 g L^{-1}) were added to the must to start the alcoholic fermentation under controlled temperature (24 °C ± 2).

Maceration with the skins and seeds was carried out for 30 days, simultaneously to alcoholic and malolactic fermentation. During this period the environmental temperature was controlled in 24°C ± 2 and must aliquots (50 ml) were taken every 5 days for monitoring the process, as described in Table 1. The end of alcoholic fermentation (the alcoholic fermentation lasted 20 days) was determined by the wine density until constant; the alcoholic level of the wine was reducing the sugars that was below 2 g L^{-1}. The natural malolactic fermentation was performed in controlled temperature (18 °C ± 1).

Table 1 – Maceration time planning and samples.

Samples code	Time of maceration (days)
MD1	1st (must)
MD2	5th
MD3	10th
MD4	15th
MD5	20th
MD6	25th
MD7	30th
W	Syrah Wine

After the 30 days of maceration, skins and seeds were separated from the wine through pressing. The end of the malolactic fermentation was checked by paper chromatography. The wines were stabilized by cold stabilization (during 10 days at 0ºC) and using 0.40 g L^{-1} of Stabigum AEB Group® (mixture of gum arabic and metatartaric acid). Before bottling, the concentration of free SO_2 was corrected to 50 mg L^{-1}. The wine was bottled (750 mL) and stored in a cellar at 18 °C in horizontal position for 30 days until the analysis.

2.3 Color Intensity (CI)

The color intensity of the samples was evaluated through the absorbance reading at different wavelengths (420, 520 and 620 nm), using a spectrophotometer Genesys™ 10S (Thermo Fisher Scientific, Waltham, MA, USA) (P. Ribereau-Gayon, Maujean, & Dubourdieu, 2005).

2.4 Total phenolics and anthocyanins

The total polyphenol index was determined diluting 1 mL of the sample in 99 mL distilled water, followed by reading absorbance at 280 nm in a spectrophotometer in Q-4 quartz cuvettes (Harbertson & Spayd, 2006). Total phenolic compounds content was quantified using the Folin-Ciocalteu methodology (Swain & Hills, 1959). Aliquots of 50 μL of the maceration samples and wine were mixed with 800 uL of deionized water and 50 uL of Folin-Ciocalteu reagent (Sigma-Aldrich, St. Louis, MO, USA). This mixture was allowed to stand for 3 min to react and then 100-μL of 1N Na_2CO_3 solution was added. The solution was incubated in a dark place for 2 hours. The absorbance was measured using a UV–Vis multi-detection microplate

reader (Synergy HT, Biotek, Winooski, USA) and the results were expressed as gallic acid (Sigma-Aldrich, St, Louis, MO, USA) equivalents (GAE mg mL^{-1}). The standard curve was linear between 16 to 250 GAE mg mL^{-1}.

Anthocyanins were quantified according to the methodology described by Lee, Durst, & Wrolstad (2005). Samples were diluted in 0.025M KCl buffer (pH 1.0) and the absorbance was read at 520 nm and 700 nm using a UV–Vis multi-detection microplate reader (Synergy HT, Biotek, Winooski, USA). Then a second dilution was made in 0.4M $C_2H_3NaO_2$ buffer (pH 4.5) and the absorbance was read in the same wavelengths. It was performed the calculation presented in the equation 1. The quantification of total anthocyanins (malvidin-3-glucoside mg L^{-1}) was done using malvidin-3-glucoside (MW 493.2) as a reference using the equation 2.

Eq. (1) $A = [(A520\ nm - A700\ nm)\ pH = 1.0] - [(A520\ nm - A700\ nm)\ pH = 4.5]$

Eq. (2) $C\ (mg\ malvidin\text{-}3\text{-}glucoside\ L^{-1}) = A \times MW \times DF \div £ \times 1$

Where: MW = molecular weight; DF = dilution factor; £ = molar absorptivity (28 000 mol L^{-1}) and 1 = path length (cm).

2.5 Chromatographic analysis to identification and quantification of phenolic compounds

Phenolic compounds were analyzed by High Performance Liquid Chromatography (Waters 2695 Aliance system, Milford, MA, USA) equipped with a diode array detector (DAD) and a fluorescence detector (FD). Data acquisition and analysis were performed with Waters Empower™ 2 software (Milford, MA, USA). According to an in-house validated procedure published elsewhere (Natividade, Corrêa, Souza, Pereira, & Lima, 2013), the conditions were set at: pre-Gemini-NX C18 column (4.0 mm x 3.0 mm, Phenomenex®, Torrance, CA, USA), Gemini-NX C18 column (150 mm x 4,60 mm x 3 μm, Phenomenex®, Torrance, CA, USA), oven temperature at 40 °C and flow rate of 0.5 mL min^{-1} mobile phases consisting of (A) 0.85% phosphoric acid solution (Fluka Switzerland) and (B) acetonitrile (HPLC grade, JT Baker, Phillipsburg, NJ, USA). The elution gradient was used as follows; 0 minutes 100% A; 10 minutes, 93% A; 20 minutes, 90% A; 30 minutes, 88% A; 40 minutes, 77% A; 45 minutes, 65.0% A, and 100% B at 55 min. DAD was employed in the wavelengths 280, 320, 360 and 520 nm and fluorescence at 280 nm excitation and 320 nm emission) were used to identify and quantify the compounds.

The identification and quantification of phenolic compounds was performed according to Natividade et al. (2013) following the validation parameters. The linearity of the method

consisted of different concentration ranges in calibration curve of the standards, (0.625 to 15.00 μg ml^{-1}). The equations of regression coefficients (R^2) ranged from 0.9838 to 0.9999. The limits of detection (LOD) ranged between 0.001- 0.190 μg mL^{-1}, while the limits of quantification (LOQ) ranged between 0.003 and 0.370 μg mL^{-1}. The mean recovery value ranged from 98.27 to 102.01% (anthocyanins), 86.18 to 106.50% (flavonols), 83.97 to 100.93% (phenolic acids) and 86.86 to 97.10% (tannins). The precision of the method RSD_r ranged from 0.73 to 2.87% for unspiked samples and from 0.71 to 9.24% for spiked samples. For RSD_R were between 1.99 and 6.46% for unspiked samples and between 1.34 and 9.26% for spiked samples.

The samples were previously filtered in 0.45 μm nylon membrane (Phenomenex®, Torrance, CA, USA) and injected in triplicate (10 μL).

Calibration curves were prepared using standards for 25 phenolic compounds. The standards of ferulic, and quercetin acids were obtained from ChemService (West Chester, USA). Caffeic, *ρ*-coumaric, chlorogenic and gallic acids were obtained from Sigma-Aldrich (St. Louis, MO, USA). Kaempferol-3-O-glucoside, myricetin, isorhamnetin-3-O-glucoside, quercetin 3-β-D-glucoside, rutin, (+)-catechin, (-)-epicatechin, (-)-gallate epicatechin, (-)-epigallocatechin gallate, procyanidin A2, procyanidin B1, procyanidin B2, pelargonidin-3-O-glucoside, cyanidin-3-O-glucoside, malvidin-3-O-glucoside, delphinidin-3-O-glucoside, peonidin-3-O-glucoside, petunidin 3-O-glucoside and *trans*-resveratrol were obtained from Extrasynthese (Genay, France).

2.6 Antioxidant activity

The antioxidant activity of the samples was evaluated through the assays DPPH (1,1-diphenyl-2-picrylhydrazil) (Brand-Williams, Cuvelier, & Berset, 1995), FRAP (ferric reducing antioxidant power) (Rufino et al., 2010) and ORAC (hydrophilic oxygen radical absorbance capacity)(Ou, Chang, 2013).

Briefly, to determine the antioxidant activity against DPPH radical the samples were mixed with ethanolic DPPH solution (61 μM) and incubated in the dark for 30 minutes. The absorbance was read at 515 nm using a UV–Vis multi-detection microplate reader (Synergy HT, Biotek, Winooski, USA). The results are expressed in μmol Trolox (Sigma-Aldrich, St, Louis, MO, USA) equivalent (TE) L^{-1} of sample (Lee et al., 2005). The standard curve was linear between 100,000 to 1100,000 μmol of Trolox. The ability of the samples to reduce ferric to ferrous ion was evaluated reacting them with FRAP (Sigma-Aldrich, St, Louis, MO, USA) reagent (ferric-tripyridyltriazine complex). Samples were incubated for 30 minutes at 37 °C.

The absorbance was read at 595 nm using a UV–Vis multi-detection microplate reader (Synergy HT, Biotek, Winooski, USA) and the results are expressed in μmol Trolox equivalent (TE) L^{-1} of sample (Benzie & Strain, 1996). The standard curve was linear between 10,000 to 800,000 μmol of Trolox.

For ORAC assay, the samples were diluted in phosphate buffer (75 mmol L^{-1}, pH 7.4). The peroxil radical was generated through spontaneous decomposition of AAPH (Sigma-Aldrich, St, Louis, MO, USA) (2,2′-Azobis(2-methylpropionamidine) dihydrochloride at 37 °C and fluorescein was used as a fluorescent probe. The decay of the fluorescence was read in UV–Vis multi-detection microplate reader (Synergy HT, Biotek, Winooski, USA) using emission filter at 520 nm and excitation at 485 nm. The results are expressed μmol Trolox (Sigma-Aldrich, St, Louis, MO, USA) equivalent (TE) L^{-1} of sample. The standard curve was linear between 10,000 to 120,000 μmol of Trolox

2.7 Sensory Evaluation

The wine produced with 30 days of maceration was subjected to descriptive analysis and a consumer acceptance test. Approval for the study was obtained from the Ethics Committee of the University of Campinas (CAE: 34586014.9.0000.5404), and all volunteers gave a written consent.

2.7.1 Descriptive analysis

All individuals involved in the study (n = 12, 03 males and 09 females, ages ranging from 20 to 45 years old) were staff members of Embrapa Semi-Arid, with experience in descriptive analysis of wines and trained for wine aroma recognition using the Wine Aroma Wheel developed by Noble et al. (1987).

This panel assessed the wine and described it using 15 descriptors: two for appearance (red color and brightness), six for odor (aromatic intensity, alcohol, red fruits, sweet-like aroma, herbaceous and spicy), five for taste and flavor (taste persistence, sweetness, bitterness, sourness, alcohol/hot) and two for mouthfeel (astringency and body).

Panelists were trained over ten training sessions (1h) and during the training and evaluation sessions, a 9-cm line scale was used, labelled with terms ‘low’ and ‘high’, at the ends, respectively. The standards were prepared at ‘low’ and ‘high’ levels (Table 5), representing anchors and were available to panelists until the beginning of the formal session.

Three replicates were assessed in independent evaluation sessions in individual booths (T= 22 °C) under white light. Aliquots (30 mL) of wine at 18°C were poured into ISO 3591 wine glasses coded with three-digit and covered with aluminum lids to trap volatiles. Results were collected in ballots with responses decoded in cm. Individual performances were assessed by checking the repeatability (Anova, P rep > 0.05) of correlation of each panelist ratings with the panel mean (Damásio & Costelli, 1991). Based on these analyses, it was decided to remove data from two panelist (final n = 10).

2.7.2 Sensory Acceptance

A total of 129 regular wine consumers, aged between 21 and 50 (49% male and 51% female) participated in an acceptance test. The wine (30 ml) was served at 18 ° C in an ISO wine glass (3591). The evaluation was performed in individual cabin under white light. Wine was evaluated for overall liking using a 9-point structured hedonic scale (1 = "I disliked it extremely", 5 = “neither like, nor dislike”, and 9 = "I liked it extremely).

2.8 Data analysis

The results of the chemical analyses were expressed in terms of mean ± standard deviation (SD). Data were analyzed by ANOVA followed by Tukey’s means comparison test ($P<0.05$). Pearson correlation was used to correlate the results and Principal component analysis was separately performed on anthocyanins and phenolic compounds data using the Factominer package for the software R (Team, 2017) .

Descriptive and acceptance data was expressed in terms of mean ± standard deviation (SD).

3. RESULTS AND DISCUSSION

3.1 Characterization of must

The must presented 10.8 ± 0.45 g L^{-1} of tartaric of total acidity, 0.51 ± 0.09 g L^{-1} of acetic volatile acid, pH = 3.61 ± 0.07; 276.33 ± 25.63g L^{-1} of reducing sugar, 1.096±0.02 g/cm^3 density; 25.99 ± 0.52 °Brix. The quantity of sugar revealed the potential of Syrah must of Submiddle São Francisco Valley to produce wines with a high level of alcohol. These results

are similar to those found by Lima et al. (2015) using Syrah grape from this region and harvested after 119 days after pruning. The authors argued that a large concentration of soluble solids is an attribute that improve the wine quality, and these parameters usually characterize grapes cultivated in Submiddle São Francisco Valley due to the sun light and temperature of this region.

São Francisco Valley is located in the tropical semi-arid climate zone that confers environmental conditions peculiars to the region, such as high temperatures throughout the year, high incidence of solar radiation and the efficient irrigation of crops promoted by the São Francisco river. Some of these characteristics contribute to increase the amount of secondary metabolites, like polyphenols in the fruits as a protection to plant stress against the environmental conditions (Cazarin et al., 2013; da Silva et al., 2016).

3.2 Phenolics compounds and color intensity

The color intensity and the total polyphenols index are presented in Table 2. Higher color intensity was observed after five days of maceration and the total polyphenols index is in agreement with this result. The literature reports that during the maceration, the highest polyphenols extraction is observed until the 4th day and, afterwards, the extraction diminishes until the removal of the skins of the grapes (Gomez-Mıguez et al., 2007).

Table 2 - Determination of color intensity and total phenolic index of Syrah wine during the maceration time.

Samples	Color intensity	Total Phenolic Index
MD1	$27.1^{b} \pm 0.25$	$47.9^{f} \pm 0.64$
MD2	$29.5^{a} \pm 0.44$	$81.9^{a} \pm 0.35$
MD3	$20.1^{c} \pm 0.81$	$74.6^{b} \pm 0.99$
MD4	$16.2^{d} \pm 0.13$	$72.9^{c} \pm 0.78$
MD5	$11.8^{f} \pm 0.01$	$69.6^{d} \pm 0.14$
MD6	$12.4^{f} \pm 0.47$	$72.6^{c} \pm 0.35$
MD7	$12.1^{f} \pm 0.38$	$71.6^{c} \pm 0.14$
W	$13.75^{e} \pm 0.63$	$47.85^{e} \pm 0.05$

Data are expressed as mean ± standard deviation. Values followed by different letters in the same column are significantly different ($P < 0.05$) according to Tukey's test.

Phenolic compounds play an important role in wines, especially in flavor and color (Ribereau-Gayon et al., 2005). The concentration of total phenolic compounds increased until the fifteen days of maceration maintaining during the rest the winemaking process (Table 3). The extraction of phenolic compounds, in particular anthocyanins and tannins, are exponential during the maceration while the skins and seed are in the must (Waterhouse, Sacks, & Jeffery, 2016). In the present study, the wine produced after 30 days of maceration showed a higher concentration of monomeric anthocyanins (331.12 ± 23.53g malvidin-3-glucoside mg L^{-1}) (Table 3). The prolonged maceration time (30 days) maintained the amounts of monomeric anthocyanins observed in the Syrah wine produced in the present work. Padilha et al., (2017) observed in commercial Syrah wine a total monomeric concentration of anthocyanins (36.2 ± 1.6 mg cyanidin 3-O-glucoside L^{-1}) in agreement wines of various regions worldwide.

Table 3 - Total phenolic compounds, monomeric anthocyanins and antioxidant activity of Syrah wine during maceration time and in the wine.

Samples	Total phenolic compounds (GAE mg mL^{-1})	Monomeric anthocyanins (Malvidin 3-O-glucoside mg L^{-1})	DPPH (μmol TE L^{-1})	FRAP (μmol TE L^{-1})	ORAC (μmol TE L^{-1})
MD1	$531.6^{d} \pm 24.50$	$370.81^{b} \pm 8.43$	$4,200^{b} \pm 0.26$	$8,800^{d} \pm 0.24$	$14,100^{d} \pm 1.02$
MD2	$1556.6^{c} \pm 82.63$	$161.09^{d} \pm 29.79$	$6,400^{b} \pm 0.25$	$12,600^{c} \pm 0.36$	$32,300^{c} \pm 2.12$
MD3	$1741.1^{b} \pm 19.77$	$336.41^{bc} \pm 5.52$	$16,200^{a} \pm 2.33$	$21,600^{b} \pm 0.70$	$44,800^{b} \pm 2.46$
MD4	$2031.3^{a} \pm 37.92$	$359.89^{b} \pm 8.43$	$16,000^{a} \pm 0.84$	$25,200^{a} \pm 1.86$	$45,100^{b} \pm 5.11$
MD5	$1904.0^{ab} \pm 33.21$	$424.73^{a} \pm 5.52$	$17,100^{a} \pm 0.64$	$21,500^{b} \pm 2.64$	$48,700^{a.b} \pm 4.81$
MD6	$2091.7^{a} \pm 43.09$	$289.11^{c} \pm 29.03$	$14,600^{a} \pm 1.96$	$20,100^{b} \pm 0.84$	$56,600^{a} \pm 5.15$
MD7	$2024.8^{a} \pm 64.83$	$372.79^{b} \pm 4.47$	$16,800^{a} \pm 1.12$	$20,200^{b} \pm 1.11$	$54,200^{a} \pm 1.56$
W	$2102.4^{a} \pm 248.08$	$331.12^{bc} \pm 23.53$	$15,500^{a} \pm 1.74$	$20,300^{b} \pm 1.11$	$46,300^{b} \pm 7.70$

GAE = Gallic acid equivalent; malvidin-3-glucosideo; TE = Trolox equivalent. Data are expressed as mean ± standard deviation. Values followed by different letters in the same column are significantly different ($P < 0.05$) according to Tukey's test.

Twenty five phenolic compounds were identified in the samples collected in the different times of the maceration and in the wine, varying among phenolic acids, flavanols, procyanidins, flavonols anthocyanins and stilbenes, as shown in Table 4. Significant differences ($P<0.05$) among the compounds were observed during the maceration duration time and in the wine, with the exception of phenolic acids (*p*-cumaric and ferulic acid).

The phenolic compounds profile changed along the maceration period, however, the main phenolics acids found in the wine were gallic and chlorogenic acid. Prolonged maceration promoted increase in the amounts of gallic acid in the wine (70.7 ± 1.51 mg L^{-1}) compared to the wine produced with Syrah grapes from San Juan province, Argentina (62.85 ± 1.91 mg L^{-1}) (Lingua et al., 2016).

Caffeic acid decreased during the maceration duration time, but chlorogenic acid concentration increased (Table 4). This change could be explained by the combination of the caffeic acid with quinic, shikimic or tartaric acid to form phenolic acid esters like chlorogenic acid (Kosseva; Maria, Joshi; & Panesar; 2017). Chlorogenic acid is related to some health benefits mainly with the prevention of diseases such as diabetes mellitus type 2 and cardiovascular diseases. In addition, its antioxidant activity can reduce the oxidative stress, inhibiting the growth of cancer cells in liver and colon (Garambone & Rosa, 2007).

According to Lingua et al., (2016) the most abundant monomeric flavanol in the grape's skins and seeds are (+)-catechin and (-)-epicatechin, and their concentrations increased during the maceration process. The incremental amounts of (+)-catechin observed during the maceration can be related to the hydrolysis of its precursor (-)-epicatechin gallate, as shown in Table 4. In the same way, an increase on proanthocyanidins level was observed, which is the polymeric form of these two monomeric compounds (Kosseva; Maria et al., 2017). According to González-Manzano, Rivas-Gonzalo, & Santos-Buelga (2004) procyanidin B1 is the main flavanol present in the grape skins, while procyanidin B2 is present in the seeds. The results showed that the main compound in the flavanols group is the procyanidin B2.

Table 4 - Phenolic compounds (n =25) (mg L^{-1}) of Syrah wine during maceration time and in the wine.

Polyphenols (mg L^{-1})	MD1	MD2	MD3	MD4	MD5	MD6	MD7	W
Phenolic acid								
p- Coumaric acid	$0.2^{a} \pm 0.00$	$0.2^{a} \pm 0.00$	$0.2^{a} \pm 0.00$	$0.3^{a} \pm 0.00$	$0.2^{a} \pm 0.00$	$0.2^{a} \pm 0.00$	$0.2^{a} \pm 0.00$	$0.2^{a} \pm 0.00$
Ferulic acid	$0.1^{a} \pm 0.00$	$0.1^{a} \pm 0.00$	$0.3^{a} \pm 0.00$	$0.3^{a} \pm 0.00$	$0.0^{a} \pm 0.00$	$0.3^{a} \pm 0.00$	$0.2^{a} \pm 0.06$	$0.1^{a} \pm 0.00$
Caffeic acid	$5.9^{b} \pm 0.66$	$8.1^{a} \pm 0.06$	$6.1^{b} \pm 0.00$	$5.0^{c} \pm 0.00$	$3.7^{d} \pm 0.10$	$3.1^{de} \pm 0.00$	$2.5^{e} \pm 0.00$	$3.5^{de} \pm 0.06$
Gallic acid	$2.5^{g} \pm 0.19$	$15.3^{f} \pm 0.81$	$33.9^{e} \pm 0.21$	$45.4^{d} \pm 0.66$	$62.6^{b} \pm 0.40$	$58.4^{c} \pm 0.06$	$62.5^{b} \pm 0.12$	$70.7^{a} \pm 1.51$
Chlorogenic acid	$0.2^{g} \pm 0.05$	$2.6^{f} \pm 0.06$	$2.7^{f} \pm 0.00$	$2.0^{e} \pm 0.00$	$3.6^{c} \pm 0.12$	$3.3^{d} \pm 0.00$	$3.8^{b} \pm 0.06$	$5.3^{a} \pm 0.06$
Flavanols and Procyanidins								
(-)-Epicatechin	$0.7^{f} \pm 0.07$	$1.0^{f} \pm 0.06$	$2.9^{e} \pm 0.06$	$4.4^{d} \pm 0.29$	$5.4^{c} \pm 0.06$	$5.9^{b} \pm 0.29$	$5.6^{bc} \pm 0.12$	$7.1^{a} \pm 0.40$
(+)-Catechin	$0.5^{e} \pm 0.00$	$4.5^{d} \pm 0.06$	$9.6^{c} \pm 0.10$	$11.6^{b} \pm 0.06$	$13.7^{a} \pm 0.15$	$13.8^{a} \pm 0.26$	$14.5^{a} \pm 0.06$	$12.5^{b} \pm 1.10$
(-) – Epicatechin gallate	$2.0^{e} \pm 0.09$	$6.2^{a} \pm 0.10$	$3.3^{b} \pm 0.12$	$2.7^{c} \pm 0.06$	$2.4^{d} \pm 0.06$	$2.0^{e} \pm 0.31$	$1.8^{e} \pm 0.06$	$1.5^{f} \pm 0.00$
(-)-Epigalatocatechin gallate	$0.4^{d} \pm 0.03$	$5.6^{c} \pm 0.21$	$8.5^{a} \pm 0.10$	$7.3^{b} \pm 0.25$	$7.3^{b} \pm 0.06$	$6.2^{c} \pm 0.20$	$6.2^{c} \pm 0.50$	$6.0^{c} \pm 0.51$
Procyanidin A2	$0.5^{a} \pm 0.04$	$0.3^{b} \pm 0.00$	$0.4^{b} \pm 0.00$	$0.0^{b} \pm 0.00$	$0.4^{b} \pm 0.00$	$0.4^{b} \pm 0.06$	$0.4^{b} \pm 0.12$	$0.6^{a} \pm 0.00$
Procyanidin B1	$1.3^{f} \pm 0.04$	$3.4^{e} \pm 0.00$	$5.7^{d} \pm 0.00$	$6.7^{c} \pm 0.00$	$7.9^{b} \pm 0.00$	$7.8^{b} \pm 0.44$	$7.6^{b} \pm 0.14$	$8.6^{a} \pm 0.61$
Procyanidin B2	$0.5^{f} \pm 0.04$	$4.6^{e} \pm 0.12$	$11.3^{d} \pm 0.10$	$14.2^{c} \pm 0.40$	$17.1^{b} \pm 0.12$	$17.4^{b} \pm 0.46$	$18.0^{a.b} \pm 0.10$	$19.1^{a} \pm 0.55$
Flavonols								
Isorhamnetin-3-O-glucoside	$2.6^{d} \pm 0.10$	$19.7^{a} \pm 0.15$	$19.7^{a} \pm 0.21$	$14.9^{bc} \pm 5.53$	$20.3^{a} \pm 0.10$	$15.8^{abc} \pm 0.15$	$15.5^{abc} \pm 0.15$	$14.0^{c} \pm 0.23$
Quercetin 3-β-D-glucoside	$0.1^{f} \pm 0.00$	$0.6^{e} \pm 0.05$	$1.5^{a} \pm 0.00$	$1.0^{c} \pm 0.06$	$1.2^{b} \pm 0.00$	$0.8^{d} \pm 0.00$	$1.5^{a} \pm 0.06$	$1.1^{c} \pm 0.15$
Quercetin	$4.7^{e} \pm 0.19$	$31.8^{a} \pm 0.15$	$30.7^{b} \pm 0.32$	$32.0^{a} \pm 0.40$	$30.9^{b} \pm 0.20$	$23.7^{c} \pm 0.10$	$23.3^{c} \pm 0.15$	$20.7^{d} \pm 0.35$
Kaempferol-3-O-glucoside	$0.5^{f} \pm 0.00$	$3.8^{a} \pm 0.05$	$3.5^{b} \pm 0.06$	$3.4^{b} \pm 0.15$	$3.0^{c} \pm 0.00$	$2.3^{d} \pm 0.00$	$2.2^{d} \pm 0.06$	$2.1^{e} \pm 0.06$
Rutin	$0.5^{f} \pm 0.04$	$3.2^{a} \pm 0.05$	$2.8^{b} \pm 0.06$	$2.7^{b} \pm 0.00$	$2.4^{c} \pm 0.06$	$1.8^{d} \pm 0.00$	$1.7^{de} \pm 0.00$	$1.6^{e} \pm 0.10$
Myricetin	$0.7^{c} \pm 0.00$	$4.9^{a.b} \pm 0.10$	$5.3^{a} \pm 0.06$	$3.1^{b} \pm 2.63$	$6.1^{a} \pm 0.06$	$4.9^{a.b} \pm 0.06$	$4.9^{ab} \pm 0.06$	$4.6^{ab} \pm 0.12$
Anthocyanins								
Cyanidin 3-O-glucoside	$1.1^{d} \pm 0.09$	$1.8^{a} \pm 0.00$	$1.5^{b} \pm 0.06$	$1.3^{c} \pm 0.00$	$1.1^{d} \pm 0.00$	$0.8^{e} \pm 0.00$	$0.6^{f} \pm 0.00$	$0.4^{g} \pm 0.00$

Table 4- Continuation

Polyphenols (mg L^{-1})	MD1	MD2	MD3	MD4	MD5	MD6	MD7	W
Anthocyanins								
Peonidin 3-O-glucoside	$5.2^{d} \pm 0.44$	$13.9^{a} \pm 0.15$	$10.3^{b} \pm 0.10$	$9.8^{b} \pm 0.12$	$8.4^{c} \pm 0.10$	$6.3^{d} \pm 0.06$	$5.0^{e} \pm 0.10$	$4.4^{e} \pm 0.06$
Petunidin 3-O-glucoside	$0.0^{e} \pm 0.00$	$1.1^{bcd} \pm 0.00$	$0.9^{cd} \pm 0.06$	$1.3^{ab} \pm 0.46$	$1.5^{a} \pm 0.00$	$1.2^{abc} \pm 0.00$	$1.0^{bcd} \pm 0.00$	$0.8^{d} \pm 0.00$
Delphinidin 3-O-glucoside	$2.0^{g} \pm 0.21$	$8.1^{a} \pm 0.21$	$6.7^{b} \pm 0.15$	$6.8^{b} \pm 0.17$	$6.0^{c} \pm 0.00$	$4.6^{d} \pm 0.00$	$3.3^{e} \pm 0.06$	$3.2^{f} \pm 0.06$
Malvidin 3-O-glucoside	$28.4^{g} \pm 2.20$	$164.3^{a} \pm 1.62$	$137.3^{b} \pm 1.19$	$137.4^{b} \pm 1.36$	$124.7^{c} \pm 0.80$	$95.5^{d} \pm 0.61$	$77.3^{e} \pm 0.55$	$68.3^{f} \pm 1.44$
Pelargonidin 3-O-glucoside	$4.8^{g} \pm 0.42$	$22.4^{a} \pm 0.45$	$18.5^{b} \pm 0.32$	$18.8^{b} \pm 0.25$	$16.6^{c} \pm 0.10$	$12.6^{d} \pm 0.20$	$10.2^{e} \pm 0.10$	$8.7^{f} \pm 0.15$
Stilben								
Trans-resveratrol	$0.4^{g} \pm 0.00$	$0.4^{g} \pm 0.00$	$2.2^{a} \pm 0.00$	$1.7^{b} \pm 0.00$	$1.6^{c} \pm 0.00$	$1.3^{d} \pm 0.00$	$0.8^{e} \pm 0.06$	$0.6^{f} \pm 0.00$

Data are expressed as mean ± standard deviation. Values followed by different letters in the same line are significantly different ($P < 0.05$) according to Tukey's test.

Flavonols are the yellow pigments in the skin of red or white grapes. These compounds are present in red wine in the aglycone form which are released during the fermentation through the action of the glycosidase (Ribereau-Gayon, Maujean, & Dubourdieu, 2006). In the red grapes, flavonol 3-O-glycosides are characterized in three different series, according to the natural sugar connected in the carbon C-3. They are derivatives of six flavonols aglycones: kaempferol -3-O-glucoside, quercetin-3-β-D-glucoside, isorhamnetin-3-O-glucoside, myricetin, laricitrin, and syringetin (Garrido & Borges, 2013). In the present study, Isorhamnetin-3-O-glucoside, quercetin-3-β-D-glucoside, and kaempferol-3-O-glucoside decreased during the maceration process. Although, conjugated flavonol amounts appears to be higher than free flavonol (Tsanova-savova & Ribarova, 2002). However, high concentrations of free flavonols were determined in the wine, specially quercetin and myricetin. Samples from different times of maceration and wine showed quercetin concentration in the range of 4.71 - 20.73 mg L^{-1}, in which quercetin was the most concentrated compound in the flavonols group. Interestingly, these results are in agreement with those of wines produced in Serra Gaúcha and Planalto Catarinense (Arcari, Chaves, Vanderlinde, Rosier, & Bordignon-Luiz, 2013), Southern Brazil regions, that are characterized by temperate climate in contrast to the São Francisco Valley where higher temperatures are predominants. Quercetin is reported to have antioxidant activity and plays a role in neurodegenerative disorders, coronary heart disease, LDL-cholesterol oxidation prevention, and as a potent anticancer agent, it attenuates tumor progression, oxidative stress, apoptosis and metastasis proliferation (Suganthya, Devib, Nabavic, Braidyd, & Nabavi, 2016).

Anthocyanins are responsible for the color in red grapes and wines, being malvidins the dominant compound found mainly in the grape skins (Ribereau-Gayon et al., 2005). The concentration of malvidin-3-O-glucoside increased during the maceration period from 28.37 to 68.30 mg L^{-1}, which was the major compound in the anthocyanins group. This range is higher than those found by Andrade et al. (2013) for Syrah wine from the São Francisco Valley (21.5 mg L^{-1}) and Syrah wine from the Aconcagua Valley and Central Valley (22.7 mg L^{-1}) in Chile. Apparently, the environmental conditions of the São Francisco Valley promote greater synthesis and accumulation of anthocyanins in grapes, evidencing that Syrah cultivar is well adapted to the northeastern region of Brazil. The concentration of the compounds belonging to this group is higher than the results reported by Giacosa, Marengoa, Guidoni, Rolle, & Hunter (2015) that evaluated Syrah wines produced with grapes in two ripening times (25.6 and 27.4 ° Brix) from South Africa using seven days of maceration.

During the maceration, *trans*-resveratrol ranged from 0.4 to 2.2 mg L^{-1} (MD1 to MD3) reaching up to 0.6 mg L^{-1} in the wine (W). The maximum level observed for *trans*-resveratrol was reached with 10 days of maceration (2.2 mg L^{-1}). These amounts found in wine are in agreement with the study of Padilha et al. (2017) on Syrah wine from the São Francisco Valley (0.77 mg L^{-1}). In other regions of the world as Portugal and Italy the amounts of *trans*-resveratrol in Syrah wines ranged from 2.5 and 0.72-5.36 mg L^{-1} respectively (Preti, Vieri, & Vinci, 2016; Sun, Ribes, Leandro, Belchior, & Spranger, 2006). Wines with large amounts of resveratrol are recognized as functional beverages, given the benefits that this compound promotes to health (Barreiro-Hurle, Colomboa, & Cantos-Villar, 2008). The *trans*-resveratrol have been associated to some health promoting properties as antioxidant activity, cardioprotective capacity, anticancer activity and neuroprotective activity (Fernandez-Mar, Mateos, Garcia-Parrilla, Puertas, & Cantos-Villar, 2012).

3.3 Antioxidant Activity

Table 3 shows the results of the antioxidant activity evaluated during the maceration process and in the wine. According to the DPPH data the antioxidant power against this radical increased until 10 days in maceration (MD3), remaining stable during the rest of maceration duration time. The results obtained during the Syrah grape vinification ranged from 4,200-17,100 μmol TE L^{-1}. Nevertheless, commercial wine produced in São Francisco Valley shows 19.3l mM TEAC L^{-1} according to DPPH method Padilha et al. (2017). DPPH activity of the Syrah from São Francisco Valley wine was similar to some Australian Cabernet Sauvignon wines analyzed by Yoo, Prenzler, Saliba, & Ryan, (2011). Jordão, Simões, Correia, & Gonçalves, (2012) also observed stabilization on the DPPH antioxidant activity with nine days of maceration of Portuguese wines made with the varieties *Tinta Roriz* and *Touriga Nacional*.

The stabilization in the FRAP antioxidant assays was observed after 15 days in maceration process. While for ORAC antioxidant assay was observed the stabilization in 25 days of maceration. Villaño, Fernández-Pachón, Troncoso, & Garcıía-Parrilla, (2006) showed a lower antioxidant activity with the ORAC method in Syrah wines with 15 days of maceration (14,294 μM), less than the same period (MD4) in the present study (44,800 μmol TE L^{-1}). Granato, Katayama, & Castro, (2012) observed similar ORAC values in Syrah wines from Brazil (29,801 μmol TE L^{-1}), Argentina (28,966 μmol TE L^{-1}) and Chile (31,470 μmol TE L^{-1}) as compared to the wine obtained in the present study.

According to Gris et al. (2013) the ingestion of red wine contributes to increase the antioxidant activity *in vivo* and this effect can be attributed to the phenolic compounds present in the beverage. The quality of the grapes and the environmental conditions in which they were grown are important factors that influence the quantity of phenolic compounds whose health claims have been highly valued by the consumer (Samoticha, Wojdylo, & Golis, 2017).

3.4 Principal component analysis

Principal component analysis was performed to characterize the phenolic compounds profile, TPI, color intensity and antioxidant activity of the aliquots collected until 30 days of maceration (MD1, MD2, MD3, MD4, MD5, MD6, MD7) and the wine (W). The PCA was separately performed on anthocyanins and phenolic compounds. PCA revealed the evolution of the anthocyanins profile and color intensity in relation to the maceration duration time (Figure 1).

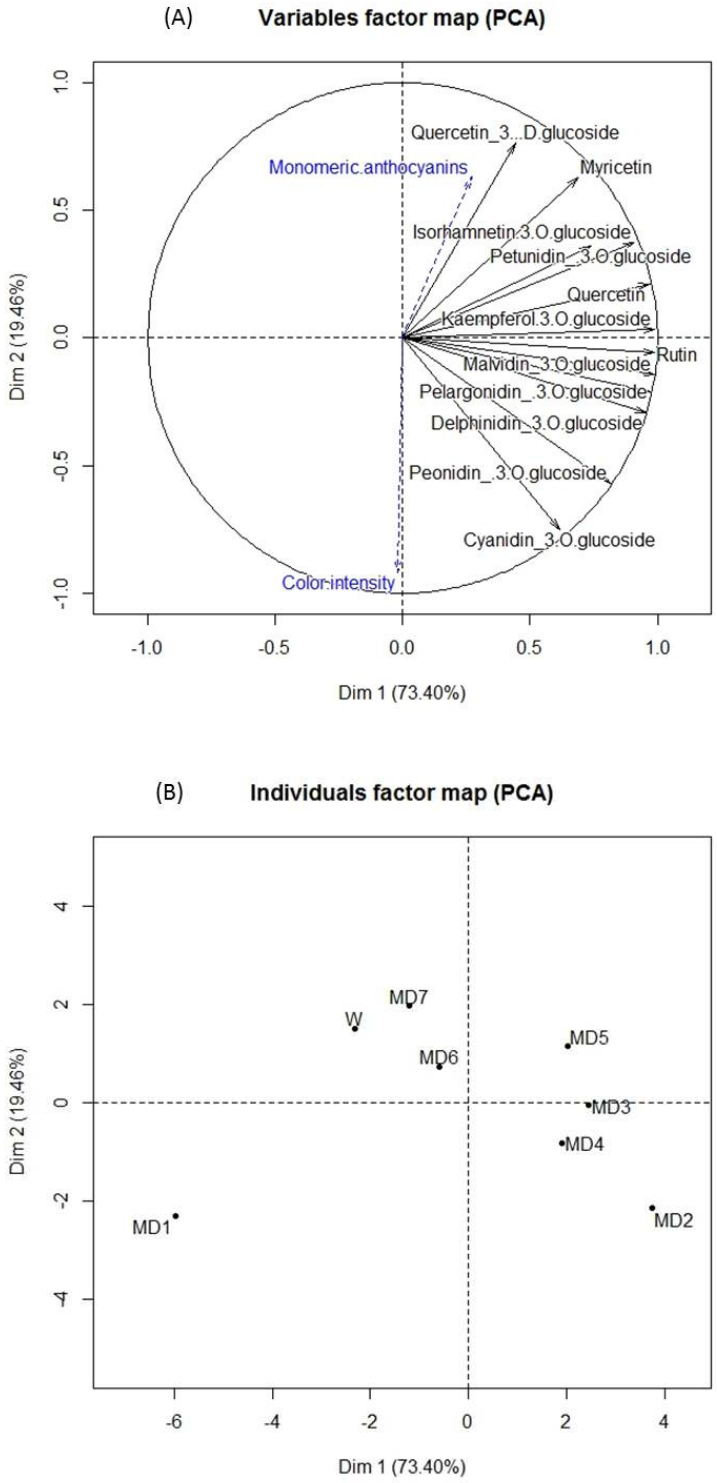

Figure 1. Principal component analysis (PCA) plot (PC1 x PC2) representating (A) the anthocianins, and the color intensity and monomeric anthocianins (in blue) as supplementary variables; (B) the special representation of the samples.

The first two dimensions derived from PCA (Fig.1) explain 92.86% of the original variance on data. PC1 (73.40%) is correlated with petunidin 3-O-glucoside, quercetin, kaempeferol-3-O-glucoside, rutin, malvidin-3-O-glucoside. On the other hand, PC2 (19.46%)

is correlated with monomeric anthocyanins, quercetin 3-β-D-glucoside, color intensity, cyanidin-3-O glucoside, peonidin-3-O glucoside. The distribution of the samples (Figure 1) shows a trend separating the MD1 (group 1), from the samples MD2, MD3, MD4 and MD5 (group 2), and MD6, MD7 and W (group 4). The must (MD1) not appears to be associated with any anthocyanins. MD2 collected from the beginning of the maceration period (five days) is characterized by a higher content of the anthocyanins pelargonidin 3-O-glucoside and delphinidin 3-O-glucoside (Table 4). MD3, MD4 and MD5 maceration points were mainly characterized by the highest contents of petunidin 3-O-glucoside, quercetin, isorhamnetin-3-O-glucoside and myricetin.

The influence of phenolic compounds, antioxidant activity, total phenolic compounds and total phenolic index in maceration days was demonstrated by PCA (Fig.2). The two dimensions derived from PCA represent 86.71% of the original data of variance. PC1 explains 66.31% of the data and is correlated with gallic acid, procyanidins B1 and B2, chlorogenic acid, (+)-catechin, DPPH, FRAP, ORAC and total phenolic compounds. Thus, this component comprises phenolic acids, procyanidins, and the antioxidant activity by the methods DPPH, FRAP and ORAC. In this way PC2 (20.40%) is characterized by total phenolic index, procyanidin A2. The MD2 (5 days of maceration) is correlated with a higher concentration of (-) – epicatechin gallate (Table 4). MD4, MD5, MD6, MD7 maceration points were mainly characterized by antioxidants activity by the methods (DPPH, FRAP, ORAC), (+)-catechin, while the sample W is characterized by (-)-Epicatechin.

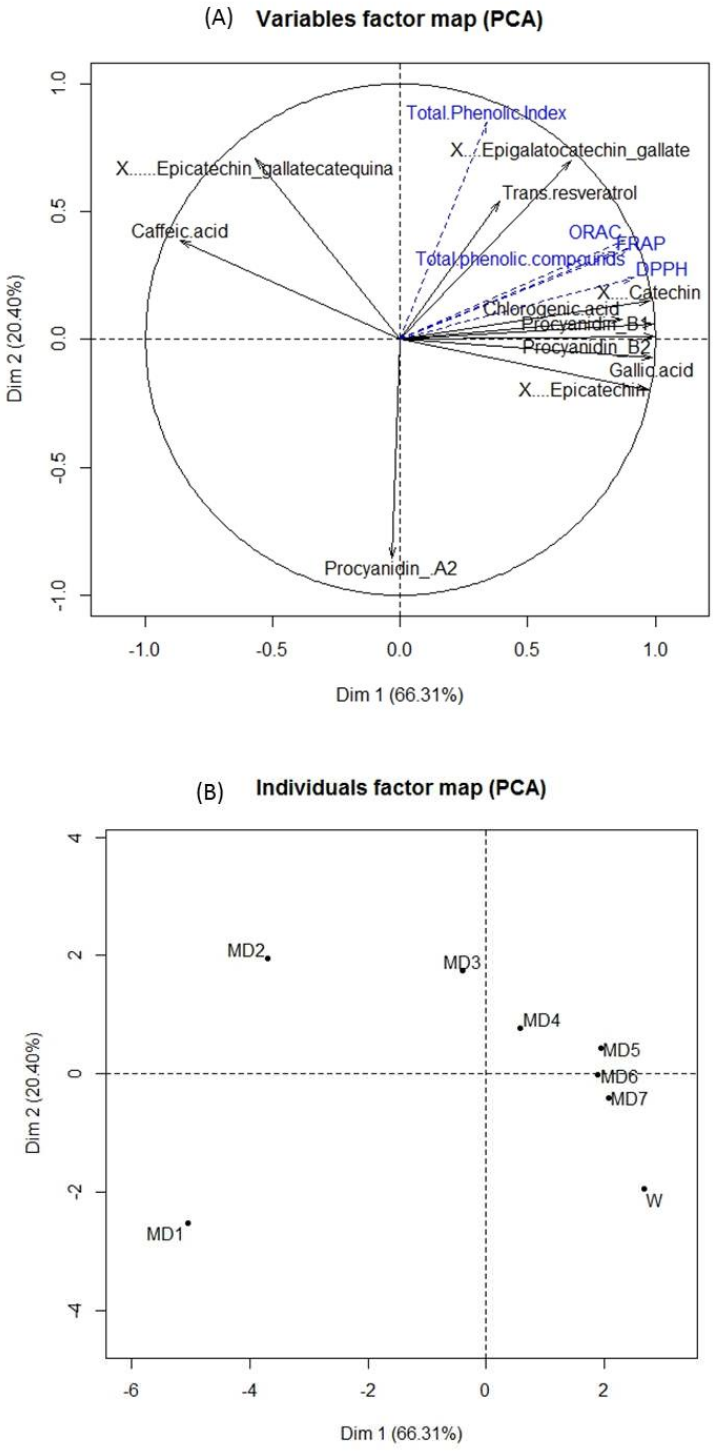

Figure 2. Principal component analysis (PCA) plot (PC1 x PC2) representating (a) the phenolic compounds, and FRAP, ORAC, DPPH, total phenolic compounds and total phenolic index (in blue) as supplementary variables; (b) the special representation of the samples.

3.5 Sensory Evaluation

The results obtained for the wine with 30 days of maceration in the descriptive analysis are presented in Table 5. The prolonged maceration yielded in high wine color (6.11) and brightness (5.66), moderate aromatic intensity (4.16) and body (4.39). On the other hand, the trained panel perceived low intensity of alcoholic aroma (3.17), red fruit aroma (3.28), taste persistence (3.69), sourness (3.43), alcoholic/hot sensation (3.32), bitterness (2.68) and astringency (1.5). According to Cadot et al., (2012) , both 9 and 15 days of maceration produce wines with very similar sensory profile, characterized by high color intensity, similarly to the present study. Nonetheless, this study reported higher astringency and bitterness, which were not perceived in high intensity in the wine of the present study. In another study by Federico Casassa et al. (2013) that identified the sensory profile of Merlot wine with 10 and 30 days of maceration and alcoholic degree of 12% and 13%. The wines with maceration of 30 days were perceived by the authors as having more pronounced the color brown, in which according to the authors justified by the oxidative polymerization of the anthocyanins and phenols, in addition, the same wines were perceived with greater astringency compared to maceration of 10 days, which was justified by the content of tannins from the grape seeds and by the time of the skins in contact with the must.

Regarding the sensory acceptance test, the Syrah wine produced in the present studied reach an overall liking mean rating of 5.59 in the hedonic scale, meaning that the wine was fairly accepted by the Brazilian wine consumers. From the 129 consumers 25.58% (17 males and 16 females) scored ≤ 4 for wine, while 74.42% (46 males and 50 females) gave a ≥ 6 score, evidencing that the majority of the consumers appreciated the wine.

Table 5 - Descriptors, specifications and mean ratings of the 15 sensory attributes evaluated in the Syrah wine produced with 30 days of maceration.

Descriptor	Reference materials	Mean rating
Red Color	**Low:** Munsell – Book of Color: 5 R 8/6.H **High:** Munsell - Book of Color: 5 R P 3/2**.**	6.11±1.48
Brightness	**Low:** 100mL of Syrah wine (13% alcohol) **High:** 200mL Syrah (13% alcohol) added with 10g of grape gelatin (Royal®) in 200mL of water.	5.66±1.46
Aromatic intensity	**Low:** Winemarker's Selection Touriga Nacional wine (Rio Sol, Lagoa Grande-PE) diluted in water 1:5 **High:** Winemarker's Selection Touriga Nacional wine (Rio Sol, Lagoa Grande-PE)	4.16±1.97
Alcohol	**Low:** 11.5% wine alcohol (63.43%) in distiled water **High:** 14.5% wine alcohol (63.43%) in distiled water	3.17±2.23
Red fruits	**None:** water **High:** 100mL of Syrah wine (13% alcohol) added with 1 drop of Le Nez Du Vin cherry essence, 1 drop of Le Nez Du Vin mulberry essence and 4 drops Le Nez Du Vin raspeberry essence.	3.28±1.96
Sweet-like aroma	**None:** water **High:** 50mL of Syrah wine (13% alcohol) added with 10g honey.	2.1±2.15
Herbaceous	**None: water** **High:** 100mL of Syrah wine (13% alcohol) added with 10g baked green beans.	2.07±1.86
Spicy	**Low:** 100mL of Syrah wine (13% alcohol) added with 1g grains of black pepper. **High:** 100mL of Syrah wine (13% alcohol) added with de 4g grains of black pepper	1.18±1.34
Taste persistence	**Low:** Winemarker's Selection Touriga Nacional (Rio Sol, Lagoa Grande- PE) diluted in water 1:5 **High:** Vinho Winemarker's Selection Touriga Nacional (Rio Sol, Lagoa Grande- PE).	3.69±1.99
Sweetness	**None:** water **High:** 0.8% sucrose aqueous solution.	1.2±1.86
Bitterness	**Low:** 0.06% caffeine aqueous solution. **High:** 0.1% caffeine aqueous solution.	2.68±1.87
Sourness	**Low:** 0.05% tartaric acid aqueous solution. **High:** 0.10% tartaric acid aqueous solution.	3.43±1.93
Alcohol/hot	**Low:** 1:1 Syrah wine (13% alcohol) in water **High:** Wine spirit (36% alcohol) brand Imperial (Miolo Wine Group- Casa Nova -BA).	3.32±1.96
Astringency	**Low:** 0.1 % tannic acid aqueous solution. **High:** 0.3 % tannic acid aqueous solution.	1.50±1.81
Body	**Low:** 1:2 Syrah wine (13% alcohol) in water **High:** Splendor chocolate liqueur (Bento Gonçalves, Brazil).	4.39±1.64

4. CONCLUSION

Prolonged maceration time increased the concentration of phenolic compounds and antioxidant activity in the wine. However, the results showed that from the 15th day on, the concentration of total phenolic compounds stabilized until the end of maceration, though it was higher in comparison to the concentration found in the initial must. This influenced in the wine antioxidant activity positively. The environmental conditions prior to the harvest may have favored the synthesis and accumulation of these compounds in the grapes that, in turn, contributed to the improvement of the wine. From the sensory point of view, the prolonged maceration of 30 days yielded in a wine with moderate color intensity, lower bitterness and astringency, differently from the expected since prolonged extraction of tannins is related to astringency and some degree of bitterness. The overall acceptance of the 30-day maceration wine was fairly high, which demonstrate the potential of this wine, especially for ageing in wood. As a final conclusion, the authors recommended the prolonged maceration process until the 15th of maceration which could be useful for making tropical red wines with enhanced concentration of antioxidants and potential for aging in wood, either in oak barrels or using oak chips, to improve the color stability and the sensory characteristics of the beverage.

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REFERENCES

Andrade, R. H. S. de, Nascimento, L. S. do, Pereira, G. E., Hallwass, F., & Paim, A. P. S. (2013). Anthocyanic composition of Brazilian red wines and use of HPLC-UV – Vis associated to chemometrics to distinguish wines from different regions. *Microchemical Journal*, *110*, 256–262. http://doi.org/10.1016/j.microc.2013.04.003

Arcari, S. G., Chaves, E. S., Vanderlinde, R., Rosier, J. P., & Bordignon-Luiz, M. T. (2013). Brazilian forti fi ed wines : Chemical composition , chromatic properties and antioxidant activity. *Food Rearch International*, *53*(1), 164–173. http://doi.org/10.1016/j.foodres.2013.04.025

Association of Official Analytical Chemists. (2007). *Official methods of analysis.*

Barreiro-Hurle, J., Colomboa, S., & Cantos-Villar, E. (2008). s there a market for functional wines? Consumer preferences and willingness to pay for resveratrol-enriched red wine. *Food Quality and Preference*, *19*, 360–371. http://doi.org/10.1016/j.foodqual.2007.11.004

Benzie, I., & Strain, J. (1996). The ferric reducing ability of plasma (FRAP) as a measure of "antioxidant power": the FRAP assay. *Analytical Biochemistry*, *239*(1), 70–6. http://doi.org/10.1006/abio.1996.0292

Brand-Williams, W., Cuvelier, M. E., & Berset, C. (1995). Use of a Free Radical Method to Evaluate Antioxidant Activity. *Lwt- Food Science and Technology*, *28*, 25–30.

Cadot, Y., Caillé, S., Samson, A., Barbeau, G., & Cheynier, V. (2012). Sensory representation of typicality of Cabernet franc wines related to phenolic composition: Impact of ripening stage and maceration time. *Analytica Chimica Acta*, *732*, 91–99. http://doi.org/10.1016/j.aca.2012.02.013

Cazarin, C. B. B., Correa, L. C., Silva, J. K., Batista, A. G., Furlan, C. P. B., Biasoto, A. C., Pereira, Giuliano Elias; Rybka, Ana Cecília Poloni; Junior, M. R. M. (2013). Tropical Isabella grape juices: bioactive compounds and antioxidant power depends on harvest season. *Journal of Food Science and Engineering*, *3*(2013), 64–70.

da Silva, J. K., Cazarin, C. B. B., Correa, L. C., Batista, Â. G., Furlan, C. P. B., Biasoto, A. C. T., … Maróstica Junior, M. R. (2016). Bioactive compounds of juices from two Brazilian grape cultivars. *Journal of the Science of Food and Agriculture*, *96*(6), 1990–1996. http://doi.org/10.1002/jsfa.7309

Damásio, H. M., & Costelli, E. (1991). Análisis sensorial descriptivo: generación de descriptores y selección de catadores. *Revista de Agroquıca Y Tecnologia de Alímentos*, *31*(2), 165–178.

Federico Casassa, L., Beaver, C. W., Mireles, M. S., & Harbertson, J. F. (2013). Effect of extended maceration and ethanol concentration on the extraction and evolution of phenolics, colour components and sensory attributes of Merlot wines. *Australian Journal of Grape and Wine Research*, *19*(1), 25–39. http://doi.org/10.1111/ajgw.12009

Fernandez-Mar, M. I., Mateos, R., Garcia-Parrilla, M. C., Puertas, B., & Cantos-Villar, E. (2012). Bioactive compounds in wine: Resveratrol, hydroxytyrosol and melatonin: A review. *Food Chemistry*, *130*(4), 797–813. http://doi.org/10.1016/j.foodchem.2011.08.023

Garambone, E., & Rosa, G. (2007). Possíveis benefícios do ácido clorogênico à saúde. *Alimentos E Nutrição*, *18*(2), 229–235.

Garrido, J., & Borges, F. (2013). Wine and grape polyphenols — A chemical perspective. *Food Research International*, *54*(2), 1844–1858. http://doi.org/10.1016/j.foodres.2013.08.002

Giacosa, S., Marengoa, F., Guidoni, S., Rolle, L., & Hunter, J. J. (2015). Anthocyanin yield and skin softening during maceration, as affected by vineyard row orientation and grape ripeness of Vitis vinifera L. cv. Shiraz. *Food Chemistry*, *174*, 8–15. http://doi.org/10.1016/j.foodchem.2014.10.155

Gomez-Mıguez, M., Gonzalez-Miret, M. L., & Heredia, F. J. (2007). Evolution of colour and anthocyanin composition of Syrah wines elaborated with pre-fermentative cold maceration. *Journal of Food Engineering*, *79*, 271–278. http://doi.org/10.1016/j.jfoodeng.2006.01.054

González-Manzano, S., Rivas-Gonzalo, J. C., & Santos-Buelga, C. (2004). Extraction of flavan-3-ols from grape seed and skin into wine using simulated maceration. *Analytica Chimica Acta*, *513*, 283–289. http://doi.org/10.1016/j.aca.2003.10.019

Gordillo, B., Baca-bocanegra, B., Rodriguez-Pulído, F. J., González-Miret, M. L., García, I. E., Quijada-Morín, N., … Escribano-Bailón, M. T. (2016). Optimisation of an oak chips-grape mix maceration process . Influence of chip dose and maceration time. *Food*

Chemistry, *206*, 249–259. http://doi.org/10.1016/j.foodchem.2016.03.041

Granato, D., Katayama, F. C. U., & Castro, I. A. De. (2012). Characterization of red wines from South America based on sensory properties and antioxidant activity. *Journal of Science Food and Agriculture*, *92*(May), 526–533. http://doi.org/10.1002/jsfa.4602

Gris, E. F., Mattivi, F., Ferreira, E. A., Vrhovsek, U., Filho, D. W., Pedrosa, R. C., & Bordignon-luiz, M. T. (2013). Phenolic profile and effect of regular consumption of Brazilian red wines on in vivo antioxidant activity. *Journal of Food Composition and Analysis*, *31*(1), 31–40. http://doi.org/10.1016/j.jfca.2013.03.002

Harbertson, J. F., & Spayd, S. (2006). Measuring phenolics in the Winery. *American Journal Enology and Viticulture*, *57*(3), 280–288.

Heredia, F. J., Escudero-gilete, M. L., Hernanz, D., Gordillo, B., & Meléndez-martínez, A. J. (2010). Influence of the refrigeration technique on the colour and phenolic composition of syrah red wines obtained by pre-fermentative cold maceration. *Food Chemistry*, *118*, 377–383. http://doi.org/10.1016/j.foodchem.2009.04.132

Ivanova-Petropulos, V., Durakova, S., Ricci, A., Parpinello, G. P., & Versari, A. (2016). Extraction and evaluation of natural occurring bioactive compounds and change in antioxidant activity during red winemaking. *Journal of Food Science and Technology*, *53*, 2634–2643. http://doi.org/10.1007/s13197-016-2235-7

Jordão, A. M., Simões, S., Correia, A. C., & Gonçalves, F. J. (2012). Antioxidant Activity Evolution During Portuguese Red Wine Vinification and their Relation with the Proanthocyanidin and Anthocyanin Composition. *Journal of Food Processing and Preservation*, *36*, 298–309. http://doi.org/10.1111/j.1745-4549.2011.00588.x

Kelebek, H., Canbas, A., Selli, S., Saucier, C., Jourdes, M., & Glories, Y. (2006). Influence of different maceration times on the anthocyanin composition of wines made from Vitis vinifera L. cvs. Bogazkere and Okuzgozu. *Journal of Food Engineering*, *77*, 1012–1017. http://doi.org/10.1016/j.jfoodeng.2005.08.032

Kosseva;Maria R., Joshi;, V. K., & Panesar;, P. S. (2017). *Science and Technology of Fruit Wine Production*. (M. R. Kosseva, P. V.K. Joshi, & S. Panesar, Eds.). San Diego: Academic Press Limited.

Lee, J., Durst, R., & R E Wrolstad. (2005). Determination of total monomeric anthocyanin pigment content of fruit juices, beverages, natural colorants, and wines by the pH differential method: Collaborative Study. *Journal of AOAC International*, *88*(5), 1269–1278.

Lima, M. dos S., Leite, A. P. da S., Sampaio, Y. C., Vianello, F., & Lima, G. P. P. (2015). Influences of the Harvest Season on Analytical Characteristics of Syrah Grapes and Wines Produced in the Northeast Region of Brazil. *International Journal of Agriculture and Forestry*, *5*, 151–159. http://doi.org/10.5923/j.ijaf.20150502.09

Lingua, M. S., Fabani, M. P., Wunderlin, D. A., & Baroni, M. V. (2016). From grape to wine : Changes in phenolic composition and its influence on antioxidant activity. *Food Chemistry*, *208*, 228–238. http://doi.org/10.1016/j.foodchem.2016.04.009

Natividade, M. M. P., Corrêa, L. C., Souza, S. V. C. de, Pereira, G. E., & Lima, L. C. de O. (2013). Simultaneos analysis of 25 phenolic compounds in grape juice for HPLC: Method validation and characterization of São Francisco Valley samples. *Microchemical Journal*, *110*, 665–674.

Noble, A. C., Arnold, R. A., Buechsenstein, J., Leach, E. J., Schmidt, J. O., & Stern, P. M. (1987). Modification of a standardized system of wine aroma terminology. *American Journal of Enology and Viticulture*, *38*, 143–146.

Ou BX, Chang T, H. D. and P. R. (2013). Phenolic compounds, organic acids and antioxidant activity of grape juices produced from new Brazilian varieties planted in the Northeast Region of Brazi. *J Aoac Int*, *96*, 1372–1376.

Padilha, C. V. da S., Camarão Telles Biasoto, A., Corrêa, L. C., dos Santos Lima, M., & Pereira, G. E. (2017). Phenolic compounds profile and antioxidant activity of commercial tropical red wines (Vitis vinifera L.) from São Francisco Valley, Brazil. *Journal of Food Biochemistry*, *41*(3), 1–9. http://doi.org/10.1111/jfbc.12346

Preti, R., Vieri, S., & Vinci, G. (2016). Biogenic amine profiles and antioxidant properties of Italian red wines from different price categories. *Journal of Food Composition and Analysis*, *46*, 7–14. http://doi.org/10.1016/j.jfca.2015.09.014

Ribereau-Gayon, Y. G., Maujean, A., & Dubourdieu, D. (2005). *The Chemistry of Wine Stabilization and Treatments* (2nd Editio, Vol. 2). England: John Wiley & Sons.

Ribereau-Gayon, P., Glories, Y., Maujean, A., & Dubourdieu, D. (2006). Phenolic Compounds. In *Handbook of Enology: The Chemistry of Wine and Stabilization and Treatments.* (Vol. 2, pp. 141–203).

Rinaldi, A., Iturmendi, N., Jourdes, M., Teissedre, P.-L., & Moio, L. (2015). Transfer of tannin characteristics from grape skins or seeds to wine-like solutions and their impact on potential astringency Alessandra. *LWT - Food Science and Technology*, *63*(July), 667–676. http://doi.org/10.1016/j.lwt.2015.02.037

Rufino, M. do S. M., Alves, R. E., Brito, E. S. de, Pérez-Jiménez, J., Saura-Calixto, F., & Mancini-Filho, J. (2010). Bioactive compounds and antioxidant capacities of 18 non-traditional tropical fruits from Brazil. *Food Chemistry*, *12*, 996–1002.

Sá, N. C. de, Silva, E. M. S., & Bandeira, A. da S. (2015). A cultura da uva e do vinho no Vale do São Francisco. *Revista de Desenvolvimento Econômico*, 461–491.

Samoticha, J., Wojdylo, A., & Golis, T. (2017). Phenolic composition, physicochemical properties and antioxidant activity of interspecific hybrids of grapes growing in Poland. *Food Chemistry*, *215*, 263–273. http://doi.org/10.1016/j.foodchem.2016.07.147

Suganthya, N., Devib, K. P., Nabavic, S. F., Braidyd, N., & Nabavi, S. M. (2016). Bioactive effects of quercetin in the central nervous system: Focusing on the mechanisms of actions. *Biomedicine et Pharmacotherapy*, *84*, 892–908. http://doi.org/10.1016/j.biopha.2016.10.011

Sun, B., Ribes, A. M., Leandro, M. C., Belchior, A. P., & Spranger, M. I. (2006). Stilbenes: Quantitative extraction from grape skins, contribution of grape solids to wine and variation during wine maturation. *Analytica Chimica Acta*, *563*(1–2 SPEC. ISS.), 382–390. http://doi.org/10.1016/j.aca.2005.12.002

Swain, T., & Hills, W. E. (1959). The phenolic constituents of Prunus domestica I. - The quantitative analysis of phenolic constituents. *Journal of the Science of Agriculture*, *10*(1), 63–68.

Team, R. C. (2017). R: A language and environment for statistical computing. Vienna: R Foundation for Statistical Computing. Retrieved from https://www.r-project.org/

Tsanova-savova, S., & Ribarova, F. (2002). Free and Conjugated Myricetin , Quercetin , and Kaempferol in Bulgarian Red Wines. *Jounal of Food Composition and Analysis*, *15*, 639–645. http://doi.org/10.1006/jfca.2002.1099

Villaño, D., Fernández-Pachón, M. S., Troncoso, A. M., & Garcıía-Parrilla, M. C. (2006). Food Chemistry Influence of enological practices on the antioxidant activity of wines. *Food Chemistry*, *95*, 394–404. http://doi.org/10.1016/j.foodchem.2005.01.005

Waterhouse, A. L., Sacks, G. L., & Jeffery, D. W. (2016). Maceration and Extraction of Grape components. In *Understanding Wine Chemistry* (pp. 179–193). England: John Wiley & Sons.

Yoo, Y. J., Prenzler, P. D., Saliba, A. J., & Ryan, D. (2011). Assessment of Some Australian Red Wines for Price , Phenolic Content , Antioxidant Activity , and Vintage in Relation to Functional Food Prospects. *Jounal of Food Science*, *76*(9), 1355–1364. http://doi.org/10.1111/j.1750-3841.2011.02429.x

CAPÍTULO 3

Influência do envelhecimento artificial utilizando diferentes chips de carvalho em vinhos jovens Syrah: mudanças na cor, perfil de compostos fenólicos e atividade antioxidante.

Natália Manzatti Machado Alencar; Cinthia Baú Betim Cazarin; Luiz Cláudio Corrêa; Mário Roberto Maróstica Junior; Davi José Silva; Aline Camarão Telles Biasoto; Jorge Herman Behrens. Influence of oak chips on Syrah wine: effects on phenolic compounds and antioxidant activity. Submetido à revista internacional **Food Research International,** Elsevier, ISSN: 0963-9969.

INFLUENCE OF THE ARTIFICIAL AGEING USING DIFFERENTS OAK CHIPS IN SYRAH YOUNG WINES: CHANGES ON THE COLOR, PHENOLIC COMPOUNDS PROFILE AND ANTIOXIDANT ACTIVITY

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ABSTRACT

American (*Quercus Alba*) and French (*Quercus Petreae*) oak chip were added to wines in 4g L^{-1} doses in the winemaking process, during alcoholic fermentation and malolactic fermentation or solemnly in the malolactic fermentation, substituting the traditional aging method in oak barrels. The results showed that Syrah tropical wines from San Francisco Valley presented higher levels of phenolic compounds, with greater concentrations of gallic acid (70.67 mg L^{-1}), chlorogenic acid (5.27 mg L^{-1}), (-) - epicatechin (7.10 mg L^{-1}), epigallocatechin gallate (1.50 mg L^{-1}), procyanidin A2 (0.60 mg L^{-1}), B1 (8.63 mg L^{-1}), and B2 (19.07 mg L^{-1}), (+) - catechin (12.50 mg L^{-1}) isorhamnetin-3-O-glucoside (13.97 mg L^{-1}), quercetin 3-β-D-glucoside (1.05 mg L^{-1}) , cyanidin 3-O-glucoside (0.40 mg L^{-1}), rutin (1.60 mg L^{-1}), myricetin (4.57 mg L^{-1}), rutin (1.60 mg L^{-1}), kaempferol-3-O- glucoside (20.7 mg L^{-1}), peginidin 3-O-glucoside (4.37 mg L^{-1}), petunidin 3-O-glucoside (0.80 mg L^{-1}), delphinidin 3-O-glucoside (3.17 mg L^{-1}), malvidin 3-O-glucoside (68.30 mg L^{-1}) and pelargonidin 3-O-glucoside (8.67 mg L^{-1}) Emphasizing the contribution of these polyphenols to the antioxidant potential of the wines. Aging the wines caused a decrease in the concentration of procyanidins as compared to control wine. In addition, the wine with French oak chip added in malolactic fermentation showed a higher antioxidant activity for ORAC. As a conclusion, oak chips are an alternative for aging tropical wines with low cost to the wineries and improved quality of wines.

Keywords: *Vitis vinifera* L.; bioactive compounds; HPLC-DAD-FD; *in vitro* antioxidant capacity; tropical red wine.

1. INTRODUCTION

The São Francisco Valley, located between the states of Pernambuco and Bahia in the northeastern region of Brazil, is a region characterized by dry and hot climate and currently emerging in the production of wines in the country, with current production of 5 million liters of wine per year (Instituto Brasileiro de Vinho, n.d.). There, each vine produces two harvests per year, in cycles of 120 to 130 days, and are irrigated with water from the São Francisco River.

In the region of São Francisco the Valley, the grape Syrah stands out for being the one that best adapted to the Brazilian semiarid region (Ferreira, 2008). The period from May to September is the most favorable for its production because of lower risk of rain and milder temperatures, with the possibility of controlling the water availability of the soil by irrigation (Tonietto & Teixeira, 2007). Thus, the climate and the soil of the region provide young, fruity and aromatic wines (Fialho, 2004). This makes the region good potential for production of quality Syrah wines (Sartor, Malinovski, Caliari, da Silva, & Bordignon-Luiz, 2017).

Syrah grape, originated from France, have been successfully inserted in tropical climate countries like Brazil, and the factors favoring cultivation in the tropical area are resistance to fungal disease, adaptation to extreme climatic conditions and high yield of the grape (Kok, 2014). Moreover, the higher concentration of phenolic compounds in the Syrah grape is influenced by the climate of the producing region, which has a great solar radiation that makes wines suitable for aging in wood (Lucena et al., 2010; Oliveira et al., 2017; Padilha, Camarão Telles Biasoto, Corrêa, dos Santos Lima, & Pereira, 2017; Viviani, Moreno, & Peinado, 2007).

The use of oak chip in winemaking is an alternative to the traditional barrels in order to improve the sensory quality of wines mainly through extraction of volatiles compounds with odoriferous potential from the wood (Arapitsas, Antonopoulos, Stefanou, & Dourtoglou, 2004; Frangipane, Santis, & Ceccarelli, 2007). According to specifications of the International Oenological Codex, it is an authorized practice, since 2005 as an alternative for barrels from the *Quercus* genus (OIV, 2007). Using oak chips instead of barrels is cheaper for winemakers in regions where oak is unavailable; and demands less time to promote the similar characteristics as those of wines aged in barrels (Cejudo-bastante, Hermosín-gutiérrez, & Pérez-coello, 2011a, 2011b), that is to say, impact on mouthfell, taste, and color, besides protecting the beverage against the oxidation due to the release of ellagitannins (Canas et al., 2016; Navarro et al., 2016).

Aging wines in wood significantly improves the content of phenolic compounds and promotes fewer color changes during the storage in the bottle (Liu et al., 2016). Both American (*Q. alba)* and French (*Q. petraea*) oak chip added during fermentation increases the concentration of total phenolic compounds, improves color intensity, and may contribute to enhance the concentration of anthocyanins (petunidin 3-glucoside; petunidin 3-acetyl-glucoside; peonidin 3-acetyl-glucoside; malvidin 3 *p*-coumaroil-glucoside) in young wines (Rodriguez-Solana, Rodriguez-Freigedo, Salgado, Domínguez, & Cortés-Diéguez, 2017).

For the foregoing, Syrah grapes produced in tropical regions is rich in antioxidant phenolic compounds and the regular consumption of these compounds helps to prevent various chronic degenerative diseases, including cancer and inflammatory diseases, as a function of the neuroprotective, antimicrobial and anti-mutagenic properties of these compounds (Cheynier, 2005; Figueiredo et al., 2017; Folmer et al., 2014; German & Walzem, 2000; Nile & Park, 2014; Peña-Neira, 2017). Moreover, the use for oak chips for ageing has potential for producing quality Syrah wines in the São Francisco Valley. Nonetheless, it seems that there are no reports in the literature on ageing of tropical wines with addition of American or French (or mixtures) oak chips during alcoholic or malolactic fermentation.

For the foregoing, the objective of this study was to characterize the phenolic compounds and the antioxidant activity of Syrah wines produced in São Francisco Valley using French and American oak chips added in different stages of the winemaking process.

2. MATERIALS AND METHODS

2.1 Feedstock

Syrah grapes (*Vitis vinifera* L.) cultivated in the experimental field of the Embrapa Semi-Arid, Petrolina, Brazil, situated at 09 ° 09 'S, 40 ° 22 'W, 365,5 m, were harvested in July 2015. The grape must presented Brix = 25.99 ± 0.52 º of soluble solids, 276.33 ± 25.63 g / L reducing sugars, pH = 3.61 ± 0.07, 10.8±0.45 g / L of tartaric acidity , 0.51 ± 0.09 g / L of acid acetic, and 1.096±0.02 g / cm^3density, 531.6 ± 24.50 GAE mg mL^{-1} of phenolic compounds 47.9 ± 0.64 total polyphenols index.

French oak chips (France / AEB-group), in pieces of 2.5 x 5.0 x 0.5 cm, 100% of *Quercus Petreae*, with higher roasting, and American oak chips (Italy / Everintec), in pieces of 2.5 x 2.0 x 1.0 cm; 100% *Quercus Alba*, medium roasting, were added in different stages of the fermentative process.

2.2Vinification

After the grapes were destemmed in a commercial destemmer (Ricefer®, Garibaldi, RS, Brazil), they were transferred to a 25 L stainless steel vat with the addition of potassium metabisulfite (0.10 g L^{-1}) (Monte Belo do Sul, RS, Brazil). The alcoholic fermentation was started under controlled temperature (24 °C ± 2ºC) with addition of pectinolytic enzyme (0.01 g L^{-1}) (Everum Thermp Everintec®,Garibaldi, RS, Brazil), commercial yeast *Saccharomyces cerevisiae* bayanus (0.20 g L^{-1}) (Maurivin PDM Coatec® , Monte Belo do Sul, RS, Brazil), and ammonium phosphate (0.20 g L^{-1}) (Gesferm plus Coatec®, Monte Belo do Sul, RS, Brazil) to the musts to start the alcoholic fermentation. The maceration period with the skins and seeds was conducted in 30 days along with alcoholic and malolactic fermentations (Alencar et al., 2017). The alcoholic fermentation lasted 20 days and the end was determined by measuring the density in precision analytical balance (Alfa Instrumentos®, model 3110), until it remained constant and the reducing sugars content was bellow 2 g L^{-1} (Ribereau-Gayon & Peynaud, 1980). In the end of the alcoholic fermentation, the skins and seeds were separated from the wine through pressing at 50 bar (Control Tech Automação). The wines were placed in carboy containers (20 liter). The spontaneous malolactic fermentation was performed in controlled temperature (18 °C ± 1) and the end was verified by paper chromatography to check the presence of the malic acid. At the end of this stage, the wine was filtered to retain yeasts and suspended particles.

Cold stabilization in cold chamber during 10 days at 0 ºC was performed using Stabigum (0.40 g L^{-1}). (AEB Group®,São José dos Pinhais, PR, Brazil). Afterwards, the wine was bottled (750 mL) and stored in a cellar at 18°C in the horizontal position for 30 days until analysis. Before bottling, the concentration of free SO_2 was corrected to 50 mg L^{-1} (Ribereau-Gayon & Peynaud, 1980).

American and/or French oak chips (4 g L^{-1}) were added at the start of the alcoholic or malolactic fermentations as described in Table 1. The quantities of the chips and degree of toast were determined according to the manufacturer's specifications and preliminary studies in the literature (García-Carpintero, Sánchez-Palomo, & González Viñas, 2014; Gordillo, Cejudo-Bastante, Rodríguez-Pulido, González-Miret, & Heredia, 2013). For the wines that receive the oak chip in the alcoholic and malolatic fermentations, chips were substituted for new ones prior to the malolactic fermentation. The oak chips were placed in nylon bags positioned at the center of the tank.

Table 1: The winemaking experimental design.

Samples	Description
WC	Control wine without wood chip
WAAMF	Wine added with American oak chip in both alcoholic and malolactic fermentation
WAMF	Wine added with American oak chip during malolactic fermentation
WFAMF	Wine added with French oak chip in both alcoholic and malolactic fermentation
WFMF	Wine added with French oak chip during malolactic fermentation
WAFAMF	Wine added with American and French oak chips in both alcoholic and malolactic fermentation

2.3 Physical and chemical analyses

The color of the wines was determined using the CIELab system were the parameters L* (luminosity), a* (green-red) and b* (blue-yellow) were measured in a Hunter Lab model Color Quest II spectrophotometer (Hunter Associates Laboratory, Reston, VA, USA). The equipment was calibrated with the illuminant D65, 10° hue angle and the RSIN calibration mode (Minolta, 1994).

Color intensity was measured in terms of absorbance at different wavelengths (420nm, 520nm and 620nm), using a spectrophotometer Genesys™ 10S (Thermo Fisher Scientific, Waltham, MA, USA). The color intensity value was obtained by summing the absorbance measurements of the wine in the violet, green and red regions of the visible spectrum, according to Ribereau-Gayon, Maujean, & Dubourdieu (2005), Equation (1).

$$\text{Wine color intensity} = A420 + A520 + A620 \quad (1)$$

Where: A = absorbance

The total polyphenol index was performed diluting 1 mL of the wine in 99 mL distilled water, followed by reading absorbance at 280 nm in the spectrophotometer in Q-4 quartz cuvettes (Harbertson & Apayd, 2006).

The oenological quality parameters of the wines, namely pH, total acidity, volatile acidity, alcoholic content, free and total sulfur dioxide and reducing sugars were carried out according to the standard method of the Association of Official Analytical Chemists (2007). Triplicates were taken and the results are shown in Table S1 (ANEXO 3).

The reducing capability of phenolic compounds were quantified using the Folin-Ciocalteu methodology (Swain & Hills, 1959). Aliquots of 800 uL of deionized water and 50uL of Folin-Ciocalteu reagent (Sigma-Aldrich, St. Louis, MO, USA) were mixed using a vortex with 50 μL of the wine. The mixture was allowed to stand for 3 min to react and sequentially 100-μL of 1N Na_2CO_3 solution was added. The solution was incubated in a dark place (24 °C) for 2h. Absorbance was measured at 725 nm using a UV–Vis multi-detection microplate reader (Synergy HT, Biotek, Winooski, USA) and the results were expressed as gallic acid equivalents (GAE mg mL^{-1}). The calibration curve ($R^2 = 0.998$) was obtained using concentrations ranging from 16 to 250 GAE mg mL^{-1}.

Total monomeric anthocyanins were determined by the pH differential method (Lee, Durst, & Wrolstad, 2005). Wine aliquots were diluted in 0.025M KCl buffer (pH 1.0). Then the absorbance was measured at 520 nm and 700 nm using a UV–Vis multi-detection microplate reader (Synergy HT, Biotek, Winooski, USA). Another dilution was made in 0.4 M $C_2H_3NaO_2$ buffer (pH 4.5) and the absorbance was read in the same wavelengths. The calculation was performed using the equation 2. The quantification of total anthocyanins was done using malvidin-3-glucoside (MW 493.2) as a reference using the equation 3.

$$A = [(A520\ nm - A700\ nm)\ pH = 1.0] - [(A520\ nm - A700\ nm)\ pH = 4.5] \quad \text{Eq. (2)}$$

$$C\ (mg\ malvidin\ 3\text{-}glucoside\ L^{-1}) = A \times MW \times DF \div £ \times 1 \quad \text{Eq. (3)}$$

Where: MW = molecular weight; DF = dilution factor; £ = molar absorptivity (28 000 mol L^{-1}) and 1 = path length (cm).

Total condensed tannins were determined by protein precipitation following the methodology described by Harbertson, Spayd (2006).

Chromatographic identification and quantification of phenolic compounds was performed by HPLC-DAD-FD (Waters 2695 Aliance system,Milford, MA) using the wavelengths 280 nm, 320 nm, 360 nm and 520 nm for the diode array detector (DAD) and 280 nm excitation and 360 nm emission for the fluorescence detector (FD), according to an in-house validated procedure published elsewhere (Natividade, Corrêa, Souza, Pereira, & Lima, 2013) following the validation parameters (Table S2 – ANEXO 4). Data acquisition and processing were carried out in a Waters Empower™ 2 software (Milford, MA, USA). Briefly, pre-Gemini-NX C18 column (4.0 mm x 3.0 mm, Phenomenex®, Torrance, CA, USA) and Gemini-NX C18 column (150 mm x 4.60 mm x 3.0 μm, Phenomenex®, Torrance, CA, USA) were used to separate the compounds. The mobile phases used were 0.85% phosphoric acid solution (Fluka

Switzerland) (A) and acetonitrile (HPLC grade, JT Baker, Phillipsburg, NJ, USA) (B). The running conditions were: 0.5 mL min^{-1} flow rate, 40°C oven temperature, 10 uL injection volume; using a gradient elution as follows: 0 minutes 100% A; 10 minutes, 93% A; 20 minutes, 90% A; 30 minutes, 88% A; 40 minutes, 77% A; 45 minutes, 65.0% A, and 100% B at 55 min (totaling 60 minutes of running).

The polyphenols identification and quantification were based on relative retention time, peak area percentage, spectrum (280nm, 320nm and 360nm) and fluorescence data (360nm emission) compared to the results obtained using 25 standards to do the calibration curves. The samples were filtered in 0.45μm nylon membrane (Phenomenex®, Torrance, CA, USA).

Twenty five standards of phenolic compounds were used to prepare the calibration curves: ferulic acid and quercetin were obtained from ChemService (West Chester, USA); caffeic, ρ-coumaric, chlorogenic and gallic acids were obtained from Sigma-Aldrich (St. Louis, MO, USA); kaempferol-3-O-glucoside, myricetin, isorhamnetin-3-O-glucoside, quercetin 3-β-D-glucoside, rutin, (+)-catechin, (-)-epicatechin, (-)-gallate epicatechin, (-)- epigallocatechin gallate, procyanidin A2, procyanidin B1, procyanidin B2, pelargonidin-3-O-glucoside, cyanidin-3-O-glucoside, malvidin-3-O-glucoside, delphinidin-3-O-glucoside, peonidin-3-O-glucoside, petunidin 3-O-glucoside and *trans*-resveratrol were obtained from Extrasynthese (Genay, France). Methanol (Vetec Química, Rio de Janeiro, Brazil) was used to clean the chromatographic system.

FRAP (ferric reducing antioxidant power) was evaluated reacting the wines with FRAP (ferric-tripyridyltriazine complex) (Sigma-Aldrich, St, Louis, MO, USA) reagent (Rufino et al., 2010; Benzie & Strain, 1996). Then, the samples were incubated for 30 min at 37 °C. The absorbance was measured using a UV–Vis multi-detection microplate reader (Synergy HT, Biotek, Winooski, USA) at 595 nm. The results were expressed in μmol Trolox equivalent (TE) L^{-1} of sample (Benzie & Strain, 1996). The standard curve (R^2 = 0.999) was linear between 10 to 800 mol of Trolox.

ORAC (hydrophilic oxygen radical absorbance capacity) (Ou & Chang, 2013) were used to evaluate the antioxidant activity of the wines. The wines were diluted in phosphate buffer (75 mmol L^{-1}, pH 7.4). Sequentially the peroxil radical was generated through spontaneous decomposition of AAPH (2,2′-Azobis(2-methylpropionamidine dihydrochloride) (Sigma-Aldrich, St, Louis, MO, USA) at 37 °C and fluorescein was used as a fluorescent probe. The decay of the fluorescence was read in a UV–Vis multi-detection microplate reader (Synergy HT, Biotek, Winooski, USA) using emission filter at 520 nm and excitation at 485 nm. The results were expressed as μmol Trolox (Sigma-Aldrich, St, Louis, MO, USA)

equivalent (TE) L^{-1} of sample. The standard curve (R^2 = 0.998) was linear between 10 to 120 mol of Trolox.

2.4 Statistical analysis

The results were expressed in triplicate (means ± standard deviation); for each parameter analyzed samples of 3 bottles of each wine were collected. Analysis of variance and Tukey's means comparison test were performed to check for significant differences ($P < 0.05$). The statistical analyses were carried out using the software XLStat 2013 (Addinsoft, New York, USA).

3. RESULTS AND DISCUSSION

3.1 Must of Syrah grape quality for winemaking and oenological quality parameters of wines

Typical semi-arid temperature and luminosity have an impact in the quality of grapes cultivated in this region, especially through the increasing soluble solids concentration and decreasing the acidity. The semi-arid region presents average annual temperatures around 26ºC and approximately 3000 hours of sun per year (Souza, 2008).

The analysis of the oenological quality parameters in the wines showed that the oak chips did not influence in the wine pH, total acidity, volatile acidity, alcoholic content, free sulfur dioxide content e reducing sugars content (Table S1- ANEXO 3). These parameters are in accordance with the European Union legislation (European Union, 2011).

3.2 Oak chips effect on color parameters, phenolic composition, condensed tannins and antioxidant activity of the wines

Table 2 shows the color parameters of the wines. The addition of the oak chip during the fermentation process did not affect the color of the wines as no significant difference ($P > 0.05$) were observed in the parameters *L, a** and *b**. Previous research had reported that the use of the oak chip in the fermentation process increased the concentration of phenolic compounds affecting the wine color. According to the authors, the amount of 5 g / L chip used causes a significant increase in the red color of the wines compared to the amount of 2 g / L (Liu et al., 2016). On the other hand, the color intensity analysis, according to Equation 1, showed that 4 g / L of oak chip significantly affected ($P < 0.05$) this parameter. In this study, the wines WC

and WAMF were higher in color intensity as compared to the other samples. Table 3 shows in the control wine the concentration of anthocyanins was significantly higher compared to the other wine samples aged with wood chip. The color of the wines is due to the anthocyanins that are unstable during aging, as they may react with other phenolic compounds present in the wine, especially the tannins, besides the clivage of the molecules (Ribereau-Gayon, Glories, Maujean, & Dubourdieu, 2006). In another study on the effect of micro-oxygenation and oak chip in Petit Verdot and Merlot, no significant differences (P <0.05) in color intensity were found compared to the control wine (without micro-oxygenation and oak chips). In these studies, the explanation according to the authors would be the fact that the oak chips cause the adsorption and condensation of the monomeric anthocyanins (Cejudo-Bastante et al., 2011a, 2011b).

Table 2: Color parameters and color intensity of Syrah wines aged with American and French oak chips.

Samples	Color parameters			Color Intensity (420+520+620nm)
	L	a*	b*	
WC	3.01[a]±0.68	18.89 [a] ±0.98	3.23 [a] ±0.85	13,75[a]±0,64
WAAMF	4.33 [a] ±0.48	27.01 [a] ±0.70	5.47 [a] ±0.24	9,93[c]±0,34
WAMF	3.04 [a] ±0.29	18.88 [a] ±0.93	3.08 [a] ±0.38	13,64[a]±0,11
WFAMF	2.79 [a] ±0.73	14.69 [a] ±0.23	3.11 [a] ±0.76	11,08[b]±0,15
WFMF	3.98 [a] ±0.66	23.03 [a] ±0.77	4.32 [a] ±0.85	10,71[bc]±0,24
WAFAMF	3.37 [a] ±0.28	20.49 [a] ±0.37	3.39 [a] ±0.29	11,16[b]±0,14

Data are expressed as triplicate means ± standard deviation. Values followed by different letters in the same column are significantly different (P < 0.05) according to Tukey's test. L, a*, b* of CIELab (1994).
WC: Control wine without wood chip; WAAMF: Wine with added American oak chip in both alcoholic and malolactic; WAMF: Wine with added American oak chip in malolactic fermentation; WFAMF: Wine with added French oak chip in both alcoholic and malolactic; WFMF: Wine with added French oak chip in malolactic fermentation; WAFAMF: Wine with added American and French oak chips in both alcoholic and malolactic fermentation.

González-Sáiz et al., (2014) observed a decrease in anthocyanin concentration in wine produced with Tempranillo grapes after prolonged maceration time and addition of medium or highly toasted chip infused in the wine for four weeks. The authors observed that the use of French oak caused a slight decrease in the color parameters *L, a*, b**, while the American oak promoted a decreased in brightness according to the toasting degree and the amount of chips

added. In contrast to these authors, in the present research the oak chips addition did not affect monomeric anthocyanins concentration (Figure 1b); the same performance was observed for the color parameters *L, a*, b** and polyphenol index (Table 2 and Table S1), for no significant differences ($P > 0.05$). All the wines presented similar content of anthocyanins (297.71 - 350.63 malvidin-3-glucoside mg L^{-1}) (Figure 1b). The São Francisco Valley Syrah wine presented a higher concentration of monomeric anthocyanin (36.2 mg cyanidin 3-O-glucoside L-1), which could be explained by the harvest date, grape ripeness level, environmental conditions, such as temperature and rainfall, as well as time and temperature of wine maceration during the winemaking (Padilha et al., 2017). Another factor that may affect the amount of anthocyanins in wine is the maceration time, which is mainly responsible for extracting phenolic compounds (anthocyanins and tannins) present in the skin and seed of the grape (Ribéreau-Gayon, Dubourdieu, Donèche, & Lonvaud, 2006). Thus, during maceration the extraction of phenolic compounds varies according to time, with anthocyanin extraction being highest during the first 8-10 days of maceration (Ribéreau-Gayon et al., 2006). However, the present study shows that on the 20th day of maceration there was higher extraction of monomeric anthocyanins (424.73 malvidin-3-glucoside mg L^{-1}) (Alencar et al., 2017), suggesting that prolonged maceration was important to extract monomeric anthocyanins and intensify the color of the wine.

Previous studies had concluded that maceration the proportion of chips added during the alcoholic fermentation can improve the extraction of phenolic compounds in young wines (Gordillo et al., 2016).However, in the present study, wines added with oak chip and the control wine presented similar reducing ability of the phenolic compounds ($P > 0.05$), suggesting that the addition of different oak chips and the time spent by the oak chip in wine did not affect this characteristic (Figure 1a). On the other hand, the reducing ability of the phenolic compounds found in the present study was similar to the study of Padilha et al. (2017), that observed 1992 mg L^{-1} GAE in commercial Syrah wine produced from the São Francisco Valley.

According to Liu et al., (2016), the toast degree of oak chip has an impact in the amount of phenolic compounds in the wine. Nonetheless, this was not observed neither with American oak chips (medium toast) nor French oak (high toast).

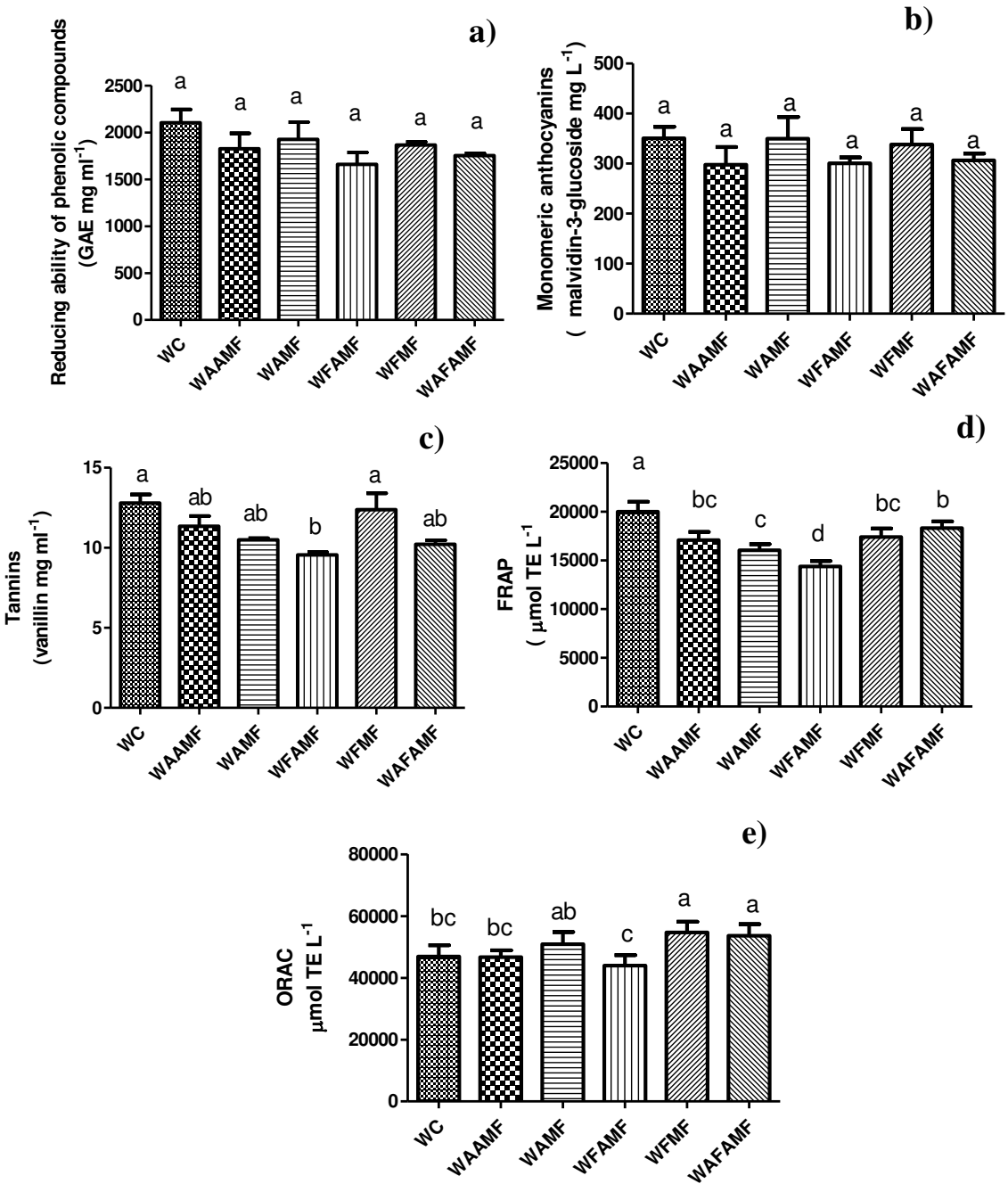

Figura 1. Total phenolics compounds in Syrah wines aged with American and French oak chips (a), monomeric anthocyanins (b), condensed tannins (c), antioxidant activity by the FRAP (d) and ORAC (e) in Syrah wines. Results expressed as mean ± SD. Bars sharing different superscript are significantly different by the Tukey test ($P < 0.05$). WC: Control wine without wood chip; WAAMF: Wine with added American oak chip in both alcoholic and malolactic; WAMF: Wine with added American oak chip in malolactic fermentation; WFAMF: Wine with added French oak chip in both alcoholic and malolactic; WFMF: Wine with added French oak chip in malolactic fermentation; WAFAMF: Wine with added American and French oak chips in both alcoholic and malolactic fermentation.

Tannins (Figure 1c) are polyphenolic compounds present in the wine which are able to produce stable combinations with proteins and other vegetable polymers causing enzymatic inhibition and astringency. Tannins in the hydrolyzable form (ellagitannin) are present in greater quantity in French oak species when compared to American oaks (Ribereau-Gayon, Maujean, & Dubourdieu, 2006). It is expected that the toasting process decreases the amounts of ellagitanins in the wood because of the thermolytic degradation. In this way, wines aged in barrels of toasted wood show less tannins (Chira & Teissedre, 2015). The decline in tannins amounts in WFAMF could be related to the ellagitannin oxidation and the formation of hemiketal and ketal ellagitannin structures (Puech, Feuillat, & Mosedale, 1999).

The oak chips seemed not to affect the total phenolic composition (Figure 1a), however, when we analyze the individual compounds by HPLC-DAD-FD, some differences appeared (Table 3).

Table 3: Phenolic compounds (mg L^{-1}) of Syrah wine fermented with American and French oak chips of the São Francisco Valley, Brazil.

Polyphenols	**WC**	**WAAMF**	**WAMF**	**WFAMF**	**WFMF**	**WAFAMF**
Phenolic acid						
ρ- Coumaric acid	$0.20^{a} \pm 0.00$	$0.10^{a} \pm 0.00$	$0.20^{a} \pm 0.00$	$0.10^{a} \pm 0.00$	$0.10^{a} \pm 0.00$	$0.20^{a} \pm 0.00$
Ferulic acid	$0.10^{a} \pm 0.00$	$0.10^{a} \pm 0.00$	$0.10^{a} \pm 0.00$	$0.10^{a} \pm 0.00$	$0.10^{a} \pm 0.00$	$0.10^{a} \pm 0.00$
Caffeic acid	$3.47^{e} \pm 0.06$	$6.93^{ab} \pm 0.46$	$5.87^{cd} \pm 0.15$	$7.83^{a} \pm 0.50$	$5.47^{d} \pm 0.15$	$6.43^{bc} \pm 0.38$
Gallic acid	$70.67^{a} \pm 1.50$	$63.63^{c} \pm 0.72$	$68.80^{ab} \pm 0.90$	$65.20^{bc} \pm 0.90$	$68.83^{ab} \pm 1.87$	$68.37^{ab} \pm 1.64$
Chlorogenic acid	$5.27^{a} \pm 0.06$	$2.03^{c} \pm 0.06$	$1.63^{d} \pm 0.06$	$1.53^{d} \pm 0.06$	$2.27^{b} \pm 0.06$	$2.13^{bc} \pm 0.06$
Total phenolic acid	79.71 ± 1.62	72.79 ± 1.24	76.6 ±1.1	74.76 ± 1.46	76.77 ± 2.08	77.23 ±2.08
Flavanols and Procyanidins						
(-)-Epicatechin	$7.10^{a} \pm 0.40$	$4.87^{d} \pm 0.42$	$5.97^{bc} \pm 0.06$	$5.20^{cd} \pm 0.30$	$6.90^{ab} \pm 0.53$	$5.33^{cd} \pm 0.40$
(+)-Catechin	$12.50^{a} \pm 1.10$	$11.10^{a} \pm 1.01$	$12.05^{a} \pm 0.55$	$12.65^{a} \pm 1.05$	$12.30^{a} \pm 0.90$	$12.50^{a} \pm 1.23$
(-)- epigallocatechin gallate	$1.50^{a} \pm 0.00$	$1.03^{c} \pm 0.06$	$1.17^{bc} \pm 0.06$	$1.20^{b} \pm 0.10$	$1.20^{b} \pm 0.10$	$1.17^{bc} \pm 0.06$
(-)-gallate epicatechin	$6.03^{c} \pm 0.51$	$5.53^{c} \pm 0.47$	$5.90^{c} \pm 0.53$	$7.97^{a} \pm 0.15$	$7.30^{ab} \pm 0.53$	$6.67^{bc} \pm 0.38$
Procyanidin A2	$0.60^{a} \pm 0.00$	$0.40^{b} \pm 0.10$	$0.40^{b} \pm 0.10$	$0.25^{bc} \pm 0.05$	$0.28^{bc} \pm 0.08$	$0.20^{c} \pm 0.00$
Procyanidin B1	$8.63^{a} \pm 0.61$	$6.17^{c} \pm 0.49$	$7.80^{ab} \pm 0.35$	$6.40^{c} \pm 0.44$	$8.30^{a} \pm 0.44$	$6.77^{bc} \pm 0.25$
Procyanidin B2	$19.07^{a} \pm 0.55$	$14.03^{d} \pm 1.40$	$16.07^{bc} \pm 0.32$	$14.90^{cd} \pm 0.60$	$17.10^{b} \pm 0.32$	$14.40^{cd} \pm 0.26$
Total *Flavanols and Procyanidins*	55.43 ± 3.17	43.13 ± 3.95	49.36 ± 1.97	48.57 ± 2.69	53.38 ± 2.9	47.04 ± 2.58
Flavonols						
Isorhamnetin-3-O-glucoside	$13.97^{a} \pm 0.23$	$11.67^{d} \pm 0.51$	$12.67^{bc} \pm 0.38$	$12.10^{cd} \pm 0.26$	$13.43^{ab} \pm 0.31$	$12.63^{bc} \pm 0.31$
Quercetin 3-β-D-glucoside	$1.05^{a} \pm 0.15$	$0.60^{bc} \pm 0.10$	$0.70^{b} \pm 0.10$	$0.40^{c} \pm 0.00$	$0.55^{bc} \pm 0.05$	$0.40^{c} \pm 0.00$
Quercetin	$20.73^{a} \pm 0.35$	$17.43^{c} \pm 0.74$	$18.77^{bc} \pm 0.57$	$17.90^{c} \pm 0.53$	$19.90^{ab} \pm 0.44$	$18.83^{bc} \pm 0.50$
Kaempferol-3-O-glucoside	$2.07^{a} \pm 0.06$	$1.67^{d} \pm 0.06$	$1.83^{bc} \pm 0.06$	$1.77^{cd} \pm 0.06$	$1.97^{ab} \pm 0.06$	$1.83^{bc} \pm 0.06$
Rutin	$1.60^{a} \pm 0.10$	$1.27^{c} \pm 0.06$	$1.40^{bc} \pm 0.00$	$1.33^{c} \pm 0.06$	$1.50^{ab} \pm 0.00$	$1.40^{bc} \pm 0.00$

Table 3 – continuation

Polyphenols	**WC**	**WAAMF**	**WAMF**	**WFAMF**	**WFMF**	**WAFAMF**
Myricetin	$4.57^{a} \pm 0.12$	$3.73^{c} \pm 0.15$	$4.00^{bc} \pm 0.10$	$3.87^{c} \pm 0.12$	$4.23^{ab} \pm 0.15$	$4.00^{bc} \pm 0.10$
Total Flavonols	43.99 ± 1.01	36.37 ± 1.62	39.37 ± 1.21	37.37 ± 1.03	41.58 ± 1.01	39.09 ± 0,97
Anthocyanins						
Cyanidin 3-O-glucoside	$0.40^{a} \pm 0.00$	$0.20^{a} \pm 0.00$	$0.20^{a} \pm 0.00$	$0.20^{a} \pm 0.00$	$0.30^{a} \pm 0.00$	$0.20^{a} \pm 0.00$
Peonidin 3-O-glucoside	$4.37^{a} \pm 0.06$	$3.23^{c} \pm 0.06$	$3.03^{d} \pm 0.06$	$2.87^{d} \pm 0.06$	$3.83^{b} \pm 0.06$	$3.00^{d} \pm 0.10$
Petunidin 3-O-glucoside	$0.80^{a} \pm 0.00$	$0.70^{ab} \pm 0.00$	$0.63^{c} \pm 0.06$	$0.63^{c} \pm 0.06$	$0.77^{ab} \pm 0.06$	$0.67^{bc} \pm 0.06$
Delphinidin 3-O-glucoside	$3.17^{a} \pm 0.06$	$2.33^{c} \pm 0.06$	$2.10^{d} \pm 0.00$	$2.17^{d} \pm 0.06$	$2.63^{b} \pm 0.06$	$2.20^{d} \pm 0.00$
Malvidin 3-O-glucoside	$68.30^{a} \pm 1.44$	$56.63^{b} \pm 0.42$	$54.50^{b} \pm 0.78$	$55.10^{b} \pm 0.82$	$66.80^{a} \pm 1.41$	$51.20^{c} \pm 0.46$
Pelargonidin 3-O-glucoside	$8.67^{a} \pm 0.15$	$6.83^{c} \pm 0.06$	$6.23^{d} \pm 0.06$	$6.43^{d} \pm 0.12$	$7.80^{b} \pm 0.20$	$6.17^{d} \pm 0.12$
Total Anthocyanins	85.71 ± 1.71	69.92 ± 0.66	66.69 ± 0.96	67.4 ± 1.12	82.13 ± 1.79	63.44 ± 0.74
Stilbenes						
Trans-resveratrol	$0.60^{a} \pm 0.00$	$0.50^{a} \pm 0.00$	$0.50^{a} \pm 0.00$	$0.50^{a} \pm 0.00$	$0.50^{a} \pm 0.00$	$0.50^{a} \pm 0.00$

Data are expressed as triplicate mean ± standard deviation. Values followed by different letters in the same line are significantly different according to Tukey's test ($P < 0.05$). WC: Control wine without wood chip; WAAMF: Wine with added American oak chip in both alcoholic and malolactic; WAMF: Wine with added American oak chip in malolactic fermentation; WFAMF: Wine with added French oak chip in both alcoholic and malolactic; WFMF: Wine with added French oak chip in malolactic fermentation; WAFAMF: Wine with added American and French oak chips in both alcoholic and malolactic fermentation.

Twenty-five phenolic compounds were studied and identified in the Syrah wines, including phenolic acids, flavonoids, procyanidins, flavanols, anthocyanins and stilbenes (Table 3). Among them, *p*-coumaric acid, ferulic acid and (+)-catechin did not show differences ($P < 0.05$) among the treatments.

Two phenolic acids identified in the control wine showed the highest concentration of these compounds as compared to the samples fermented with oak chips. Gallic acid is the major phenolic compounds present in the wines, especially in the control (70.67 mg L^{-1}) sample. The amount was higher than those reported by Padilha et al. (2017) in Syrah commercial wine from the São Francisco Valley (31.47 mg L^{-1}) and Oliveira et al. (2017), in Syrah wine produced with 7 days of maceration in the same region (19.77mg L^{-1}). The high concentration of gallic acid in wine samples may have occurred due to the tannins that under acidic hydrolysis liberate gallic and ellagic acids (Ribereau-Gayon et al., 2006). Control and WFMF samples showed highest concentrations of chlorogenic acid (5.27 mg L^{-1} and 2.27 mg L^{-1}), respectively. Liu et al. (2016) observed an increase in phenolic acids (chlorogenic acid, gallic acid, caffeic acid) was content after the addition of 5 g L^{-1} of oak chip. The addition of French oak chip in the alcoholic and malolactic fermentation promoted an increase in caffeic acid concentration (7.83 mg L^{-1}); however, it is possible that the amount of oak chips (4g L^{-1}) used in the present study was not sufficient to promote an increase in the chlorogenic acid, gallic acid, caffeic acid in the wine.

Four flavanoids and three procyanidins were detected in the wines. It was clear that the use of the oak chip during the fermentation process made changes in the flavonols and procyanidins concentrations. During aging, before or after bottling, the tannins undergo transformations that alter the color and decrease the astringency of the wine. Procyanidins can combine with flavanoids, resulting in decreased astringency of the wine (Ribereau-Gayon et al., 2006). The combination of procyanidins with flavanols may have occurred in chip-fermented wines. It is observed that wine control (WC) higher concentrations for the compounds (-) - epigallocatechin gallate; procyanidin A2 and B2. On the other hand, the use of the French oak chip promoted an increase in the content concentration of (-)-gallate epicatechin compound in the wine (WAFAMF and WFAMF). High amounts of (+)-catechin were found in all the wines independently of the treatment adopted during the fermentation process; however, the use of both chips decreased the amounts of its more common isomer, that is, (-) - epicatechin. Padilha et al. (2017) and Oliveira et al. (2017) found (+)- catechin as a predominant compound among the

flavanols in the Syrah wine also from São Francisco Valley, though in lower concentration in comparison with the present study. However, when comparing the results of the present study with wines produced in other countries it is observed that the concentration of (+) -catechin observed is lower. In the study by Gortzi, Metaxa, Mantanis, & Lalas, (2013) Syrah wine from Greece aged with wood presented the catechin concentration of 13.5 - 23.4 mg / L, whereas in Gallego, Sánchez-Palomo, Hermosín-Gutiérrez, And Viñas, (2015) Syrah wines from the "Condado de Huelva" region (Spain) aged with 3 g / L and 6 g / L of American oak chip for 10 days of maceration showed 19.7 and 14.9 mg / L catechin, respectively. Liu et al. (2016) argued that the use of oak chip of medium toasting degree significantly increased the flavanol content in wine. The present results suggest that the oak toasting degree had less influence in the content of flavanols in the wine than the fermentation stage when chips were added. Procyanidin B2 was the predominant procyanidin in all wines, and although the control wine has shown a higher concentration of procyanidins as compared to the wines added with oak chips. All the treatments had more procyanidins than the results reported in the literature to Syrah wines from São Francisco Valley and Colchagua Valley (Barrio-galán, Medel-marabolí, & Peña-neira, 2015; Oliveira et al., 2017; Padilha et al., 2017). The sensation of astringency is mainly influenced by the source of procyanidins (oligomeric or polymeric) (Chira et al., 2015). According to a previous study by Coninck, Jordão, Ricardo-Da-Silva, & Laureano, (2006), the concentration of 4 g / L of oak chip did not significantly affect the concentration of protocyanidins fraction in aged wines and in the test of sensorial profile of the wines it was observed that the fermentation with chip affected the perception of astringency compared to the control wine (without chip).

Flavonols are yellow pigment present in high quantities in the grape skin and that have an important function in the pigmentation of wine (Ribereau-Gayon et al., 2005). Six flavonols (isorhamnetin-3-O-glucoside, quercetin-3-β-D-glucoside, quercetin, kaempferol-3-O-glucoside, rutin and myricetin) were determined in the wines, being myricetin the main flavonol found in all the wines, in line with the study of Gris et al. (2013) with Syrah wine from São Joaquim region, Brazil.

According to Table 3, six anthocyanins were identified and quantified in the wines, being malvidin 3-O-glucoside the major one. The use of the French chips during the malolactic fermentation increased the contend of petunidin 3-O-glucoside (0.77 mg L^{-1}) and malvidin 3-O-glucoside (66.80 mg L^{-1}) in the wine, comparing only the aged treatments, since the control wine stood out with high concentration of petunidin 3-O-

glucoside (0.80 mg L^{-1}) and malvidin 3-O-glucoside (68.30 mg L^{-1}) compounds (Table 3). During aging in wood the decrease of anthocyanin concentration occurs due to degradation reactions, being broken by heat in phenolic acids or reacting with double bond compounds resulting in a new compound and also in the stabilization reactions several mechanisms lead to the formation of tannin-anthocyanin combination, tannin-dependent and tannin / anthocyanin ratio (Ribereau-Gayon et al., 2006). Independently of the treatment, the amounts of malvidin 3-O-glucoside found in the wines in the present study were higher than in commercial Syrah wine produced in São Francisco Valley reported by Padilha et al. (2017) (34.07 mg L^{-1}). However, the amount of malvidin 3-O-glucoside found in this study is lower than the content present in Syrah wine from "Condado de Huelva" (Spain), in which wines aged 3g / L and 6g / L with an American oak chip for 10 days presented respectively 583.8 mg / L and 544.3 mg / L malvidin 3-O-glucoside.

The present study showed that the addition of the oak chips may contribute to the reduction in the antioxidant potential (Figure 1d e 1e). Phenolic compounds exhibit antioxidant activity due to reducing properties and chemical structure, in which they are able to sequester free radicals or as metal chelants (Soares, 2002). The FRAP assay is based on the presence of 2,4,6-tripyridyl triazine promoting Fe3 + reduction in Fe2 + (Benzie & Strain, 1996). It is observed in the FRAP assay (Figure 1e) higher antioxidant activity was observed in the control wine than in the samples with addition of oak chip. In agreement with the results, Alañón, Castro-Vázquez, Díaz-Maroto, Gordon, & Pérez-Coello, (2011) showed decreased antioxidant activity when American and French oak wood were used. The FRAP antioxidant activity observed in this study was higher as compared to Syrah wines from San Juan province, Argentina, that is, 17,300 µmol Trolox L^{-1}.

The antioxidant activity could be modulated during winemaking process as maceration or wood aging, which affect the extraction of the phenolic into the must. The ORAC methodology uses peroxyl radicals that consider the better model of antioxidant reaction for oxidizing lipids and oxygen (Schaich, Tian, & Xie, 2015). This method is based on the ability of antioxidants to protect the fluorescence probe from damage caused by free radicals. Thus, 2,2'-azobis (2-amidinopropane) dihydrochloride (AAPH) is used as the source for the peroxyl radical, which is generated as a result of the decomposition of AAPH at 37 ± 1 ° C. Fluorescein is the probe whose loss of fluorescence is an indication of the extent of the damage of its reaction with the peroxyl radical (Ou, Chang,

Huang, & Prior, 2013). According to Figure 1e , the addition of the French and American oak chips during the alcoholic and malolactic fermentations (WAFAMF), as well as when each the chips were added only in the malolactic fermentation (WAMF and WFMF) improved the ability of the antioxidants present in the wine to scavenge the peroxyl radical derived from the thermal decomposition of 2,2′-azobis(2-methylpropionamidine) dihydrochloride (AAPH). The antioxidant activity of the wines evaluated, measured by the ORAC assay showed to be higher than wines from other Brazilian regions (29801 μmol TE L^{-1}), Argentine (28966 μmol TE L^{-1}) and Chile (31470 μmol TE $^{L-1}$) (Granato, Katayama, & Castro, 2012).

To a better understanding of the influence of French and/or American oak chips during the winemaking more studies are necessary, especially in order to understand the influence of the amounts of oak and the time when those are added in the wine.

4. CONCLUSIONS

The oak chips (4g / L) did not increase the amount of monomeric anthocyanins, the reducing ability, the concentration of some phenolic compounds and the antioxidant activity; however, the amounts of the phenolic compounds found in wine without fermentation with chip showed to be higher compared to other studies with Syrah wine. It was demonstrated that the fermentation with oak chip decreases the contraction of procyanidin A2 and B2, which may be favorable to decrease the perception of astringency. However, it would be interesting to conduct sensory tests to identify whether the fermentation of Syrah wine with oak chips may be an alternative to tropical wine aging and study of the stability of these wines must be conducted to understand the aging of these wines in the bottle.

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AUTHORS' CONTRIBUTIONS

N.M.M.A., A.T. B.M., J.H.B. conceived and planned the study. N.M.M.A participated of the winemaking process. L.C.C.; M.R.M.J.; D.J.S. contributed to data collection and analysis. All authors contributed to this article.

CONFLICTS OF INTEREST

The authors declare no conflicts of interest.

REFERENCES

Alañón, M. E., Castro-Vázquez, L., Díaz-Maroto, M. C., Gordon, M. H., & Pérez-Coello, M. S. (2011). A study of the antioxidant capacity of oak wood used in wine ageing and the correlation with polyphenol composition. *Food Chemistry*, *128*, 997–1002. http://doi.org/10.1016/j.foodchem.2011.04.005

Alencar, N. M. M., Cazarin, C. B. B., Corrêa, L. C., Junior, M. R. M., Biasoto, A. C., & Behrens, J. H. (2017). Influence of maceration time on phenolic compounds and antioxidant activity of the Syrah must and wine. *Journal of Food Biochemistry*, *in press*, 1–11. http://doi.org/10.1111/jfbc.12471

Arapitsas, P., Antonopoulos, A., Stefanou, E., & Dourtoglou, V. G. (2004). Artificial aging of wines using oak chips. *Food Chemistry*, *86*, 563–570. http://doi.org/10.1016/j.foodchem.2003.10.003

Association of Official Analytical Chemists. (2007). *Official methods of analysis.*

Barrio-galán, R., Medel-marabolí, M., & Peña-neira, Á. (2015). Effect of different aging techniques on the polysaccharide and phenolic composition and sensory characteristics of Syrah red wines fermented using different yeast strains. *Food Chemistry*, *179*, 116–126. http://doi.org/10.1016/j.foodchem.2015.01.075

Benzie, I. ., & Strain, J. . (1996). The ferric reducing ability of plasma (FRAP) as a measure of "antioxidant power": the FRAP assay. *Analytical Biochemistry*, *239*(1), 70–6. http://doi.org/10.1006/abio.1996.0292

Canas, S., Caldeira, I., Anjos, O., Lino, J., Soares, A., & Antonio Pedro Belchior. (2016). Physicochemical and sensory evaluation of wine brandies aged using oak and chestnut wood simultaneously in wooden barrels and in stainless steel tanks with staves. *International Journal of Food Science and Technology*, *51*, 2537–2545. http://doi.org/10.1111/ijfs.13235

Cejudo-bastante, M. J., Hermosín-gutiérrez, I., & Pérez-coello, M. S. (2011a). Micro-oxygenation and oak chip treatments of red wines : Effects on colour-related phenolics , volatile composition and sensory characteristics . Part I : Petit Verdot wines. *Food Chemistry*, *124*, 727–737. http://doi.org/10.1016/j.foodchem.2010.06.067

Cejudo-bastante, M. J., Hermosín-gutiérrez, I., & Pérez-coello, M. S. (2011b). Micro-oxygenation and oak chip treatments of red wines : Effects on colour-related phenolics , volatile composition and sensory characteristics . Part II : Merlot wines. *Food Chemistry*, *124*, 738–748. http://doi.org/10.1016/j.foodchem.2010.07.064

Cheynier, V. (2005). Flavonoids. In K. R. M. Oyvind M. Andersen (Ed.), *Flavonoids in Wine* (pp. 263–318). Boca Raton: CRC Press.

Chira, K., & Teissedre, P.-L. (2015). Chemical and sensory evaluation of wine matured

in oak barrel : effect of oak species involved and toasting process. *European Food Research Technology*, *240*, 533–547. http://doi.org/10.1007/s00217-014-2352-3

Chira, K., Zeng, L., Floch, A. Le, Péchamata, L., Jourdes, M., & Teissedre, P.-L. (2015). Compositional and sensory characterization of grape proanthocyanidins and oak wood ellagitannin. *Tetrahedron*, *71*, 2999–3006. http://doi.org/10.1016/j.tet.2015.02.018

Coninck, G. De, Jordão, A. M., Ricardo-Da-Silva, J. M., & Laureano, O. (2006). Evolution of Phenolic Composition And Sensory Properties in Red Wine Aged in Contact With Portuguese and French Oak Wood Chips. *Journal International Des Sciences de La Vigne et Du Vin*, *40*(1), 25–34.

European Union. (2011). *The Wine Regulations 2011*.

Ferreira, P. (2008). Brasil inova na produção de vinhos tropicais. *Inovação Em Pauta*, (2), 16–19.

Fialho, V. Z. B. (2004). Brasil é referência na produção de vinhos tropicais. Retrieved May 21, 2014, from http://www.cnpuv.embrapa.br/noticias/2004/2004-09-13.html

Figueiredo, E. A. de, Alves, N. F. B., Monteiro, M. M. de O., Cavalcanti, C. de O., Silva, T. M. S. da, Silva, T. M. G. da, … Oliveira, E. de J. (2017). Antioxidant and Antihypertensive Effects of a Chemically Defined Fraction of Syrah Red Wine on Spontaneously Hypertensive Rats. *Nutrients*, *9*(6), 574. http://doi.org/10.3390/nu9060574

Folmer, F., Basavaraju, U., Jaspars, M., Hold, G., El-Omar, E., Dicato, M., & Diederich, M. (2014). Anticancer effects of bioactive berry compounds. *Phytochemistry Reviews*, *13*(1), 295–322. http://doi.org/10.1007/s11101-013-9319-z

Frangipane, M. T., Santis, D. De, & Ceccarelli, A. (2007). Influence of oak woods of different geographical origins on quality of wines aged in barriques and using oak chips. *Food Chemistry*, *103*, 46–54. http://doi.org/10.1016/j.foodchem.2006.07.070

Gallego, M. A. G., Sánchez-Palomo, E., Hermosín-Gutiérrez, I., & Viñas, M. A. G. (2015). Effect of oak chip addition at different winemaking stages on phenolic composition of Moravia Agria red wines. *South African Journal for Enology and Viticulture*, *36*(1), 21–31.

García-Carpintero, E. G., Sánchez-Palomo, E., & González Viñas, M. A. (2014). Volatile composition of Bobal red wines subjected to alcoholic/malolactic fermentation with oak chips. *LWT - Food Science and Technology*, *55*(2), 586–594. http://doi.org/10.1016/j.lwt.2013.10.024

German, J. B., & Walzem, R. L. (2000). The health benefits of wine. *Annual Review of Nutrition*, *20*, 561–593.

González-Sáiz, J. M., Esteban-Díez, I., Rodríguez-Tecedor, S., Pérez-Del-Notario, N., Arenzana-Rámila, I., & Pizarro, C. (2014). Modulation of the phenolic composition and colour of red wines subjected to accelerated ageing by controlling process variables. *Food Chemistry*, *165*, 271–281. http://doi.org/10.1016/j.foodchem.2014.05.016

Gordillo, B., Baca-bocanegra, B., Rodriguez-Pulído, F. J., González-Miret, M. L., García, I. E., Quijada-Morín, N., … Escribano-Bailón, M. T. (2016). Optimisation of an oak chips-grape mix maceration process . Influence of chip dose and maceration time. *Food Chemistry*, *206*, 249–259. http://doi.org/10.1016/j.foodchem.2016.03.041

Gordillo, B., Cejudo-Bastante, M. J., Rodríguez-Pulido, F. J., González-Miret, M. L., & Heredia, F. J. (2013). Application of the differential colorimetry and polyphenolic profile to the evaluation of the chromatic quality of Tempranillo red wines elaborated in warm climate. Influence of the presence of oak wood chips during fermentation. *Food Chemistry*, *141*(3), 2184–2190.

http://doi.org/10.1016/j.foodchem.2013.05.014

Gortzi, O., Metaxa, X., Mantanis, G., & Lalas, S. (2013). Effect of artificial ageing using different wood chips on the antioxidant activity , resveratrol and catechin concentration , sensory properties and colour of two Greek red wines. *Food Chemistry*, *141*(3), 2887–2895. http://doi.org/10.1016/j.foodchem.2013.05.051

Granato, D., Katayama, F. C. U., & Castro, I. A. De. (2012). Characterization of red wines from South America based on sensory properties and antioxidant activity. *Journal of Science Food and Agriculture*, *92*(May), 526–533. http://doi.org/10.1002/jsfa.4602

Gris, E. F., Mattivi, F., Ferreira, E. A., Vrhovsek, U., Filho, D. W., Pedrosa, R. C., & Bordignon-luiz, M. T. (2013). Phenolic profile and effect of regular consumption of Brazilian red wines on in vivo antioxidant activity. *Journal of Food Composition and Analysis*, *31*(1), 31–40. http://doi.org/10.1016/j.jfca.2013.03.002

Harbertson, J. Spayd, S. (2006). Measuring phenolics in the winery. *American Journal Enological and Viticultural*, *57*, 280–288.

Harbertson, J. F., & Apayd, S. (2006). Measuring Phenolics in the Winery. *American Journal Enological and Viticultural*, *57*(3), 280–288.

Instituto Brasileiro de Vinho. (n.d.). Panorama Geral. Retrieved November 21, 2017, from http://www.ibravin.org.br/Panorama-Geral

Kok, D. (2014). A Review on Grape Growing in Tropical Regions. *Türk Tarım ve Doğa Bilimleri*, 1236–1241.

Lee, J., Durst, R. W., & Wrolstad, R. E. (2005). Determination of total monomeric anthocyanin pigment content of fruit juices, beverages, natural colorants, and wines by the pH differential method: Collaborative study. *Journal of AOAC International*, *88*(5), 1269–1278. http://doi.org/10.5555/jaoi.2005.88.5.1269

Liu, S., Wang, S., Yuan, G., Ouyang, X., Liu, Y., Zhu, B., & Zhang, B. (2016). Effect of Oak Chips on Evolution of Phenolic Compounds and Color Attributes of Bog Bilberry Syrup Wine During Bottle-Aging. *Jounal of Food Science*, *81*(11), 2697–2707. http://doi.org/10.1111/1750-3841.13532

Lucena, A. P. S., Nascimento, R. J. B., Maciel, J. A. C., Tavares, J. X., Barbosa-filho, J. M., & Oliveira, E. J. (2010). Antioxidant activity and phenolics content of selected Brazilian wines. *Journal of Food Composition and Analysis*, *23*(1), 30–36. http://doi.org/10.1016/j.jfca.2009.08.004

Minolta. (1994). *Precise color commmunication: Color control from feeding to instrumentantion*.

Natividade, M. M. P., Corrêa, L. C., Souza, S. V. C. de, Pereira, G. E., & Lima, L. C. de O. (2013). Simultaneous analysis of 25 phenolic compounds in grape juice for HPLC: Method validation and characterization of São Francisco Valley samples. *Microchemical Journal*, *110*, 665–674. http://doi.org/10.1016/j.microc.2013.08.010

Navarro, M., Kontoudakis, N., Giordanengo, T., Gómez-alonso, S., García-romero, E., Fort, F., … Zamora, F. (2016). Oxygen consumption by oak chips in a model wine solution ; Influence of the botanical origin , toast level and ellagitannin content. *Food Chemistry*, *199*, 822–827. http://doi.org/10.1016/j.foodchem.2015.12.081

Nile, S. H., & Park, S. W. (2014). Edible berries: Bioactive components and their effect on human health. *Nutrition*, *30*(2), 134–144. http://doi.org/10.1016/j.nut.2013.04.007

OIV. (2007). *Pieces of oak wood*.

Oliveira, W. P. de, Biasoto, A. C. T., Marques, V. F., Santos, I. M. dos, Magalhães, K., Correa, L. C., … Shahidi, F. (2017). Phenolics from Winemaking By-Products Better Decrease VLDL-Cholesterol and Triacylglycerol Levels than Those of Red

Wine in Wistar Rats. *Journal Od Food Science*.

Ou, B., Chang, T., Huang, D., & Prior, R. L. (2013). Determination of total antioxidant capacity by oxygen radical absorbance capacity (ORAC) using fluorescein as the fluorescence probe: First action 2012.23. *Journal of AOAC International*, *96*(6), 1372–1376. http://doi.org/10.5740/jaoacint.13-175

Padilha, C. V. da S., Camarão Telles Biasoto, A., Corrêa, L. C., dos Santos Lima, M., & Pereira, G. E. (2017). Phenolic compounds profile and antioxidant activity of commercial tropical red wines (Vitis vinifera L.) from São Francisco Valley, Brazil. *Journal of Food Biochemistry*, *41*(3), 1–9. http://doi.org/10.1111/jfbc.12346

Peña-Neira, A. (2017). Grapes. In E. M. Yahia (Ed.), *Fruit and Vegetable Phytochemicals* (2nd ed., Vol. II, pp. 1041–1054). Chichester, UK: John Wiley & Sons, Ltd. http://doi.org/10.1002/9781119158042.ch51

Puech, J. L., Feuillat, F., & Mosedale, J. R. (1999). The Tannins of oak heartwood: Structure, properties, and Their influence on Wine Flavor. *American Journal Enology and Viticulture*, *50*(4), 469–478.

Ribereau-Gayon, P. Y. G., Maujean, A., & Dubourdieu, D. (2005). *The Chemistry of Wine Stabilization and Treatments* (2nd Editio, Vol. 2). England: John Wiley & Sons.

Ribereau-Gayon, J., & Peynaud, E. Y. O. (1980). *Ciências e Técnicas del vino*. Editorial Hemisfério Sur.

Ribéreau-Gayon, P., Dubourdieu, D., Donèche, B., & Lonvaud, A. (2006). Making Red Table Wines. In *Handbook of Enology* (pp. 327–395). Chichester, UK: John Wiley & Sons, Ltd. http://doi.org/10.1002/0470010363.ch12

Ribereau-Gayon, P., Glories, Y., Maujean, A., & Dubourdieu, D. (2006). Phenolic Compounds. In *Handbook of Enology: The Chemistry of Wine and Stabilization and Treatments.* (Vol. 2, pp. 141–203).

Rodriguez-Solana, R., Rodriguez-Freigedo, S., Salgado, J. M., Domínguez, J. M., & Cortés-Diéguez, S. (2017). Optimisation of accelerated ageing of grape marc distillate on amicro-scale process using a Box–Benhken design: influence of oak origin, fragment size and toast level on the composition of the final product. *Australian Journal of Grape and Wine Research*, *23*, 5–14. http://doi.org/10.1111/ajgw.12249

Rufino, M. do S. M., Alves, R. E., Brito, E. S. de, Pérez-Jiménez, J., Saura-Calixto, F., & Mancini-Filho, J. (2010). Bioactive compounds and antioxidant capacities of 18 non-traditional tropical fruits from Brazil. *Food Chemistry*, *12*, 996–1002.

Sartor, S., Malinovski, L. I., Caliari, V., da Silva, A. L., & Bordignon-Luiz, M. T. (2017). Particularities of Syrah wines from different growing regions of Southern Brazil: grapevine phenology and bioactive compounds. *Journal of Food Science and Technology*, *54*(6), 1414–1424. http://doi.org/10.1007/s13197-017-2557-0

Schaich, K. M., Tian, X., & Xie, J. (2015). Reprint of "Hurdles and pitfalls in measuring antioxidant efficacy: A critical evaluation of ABTS, DPPH, and ORAC assays." *Journal of Functional Foods*, *18*, 782–796. http://doi.org/10.1016/j.jff.2015.05.024

Soares, S. E. (2002). Ácidos Fenólicos Como Antioxidantes. *Revista de Nutricao*. http://doi.org/10.1590/S1415-52732002000100008

Souza, L. C. (2008). *Desafios da implementação da Política Nacional de Recursos Hídricos no semiárido nordestino: o caso da Bacia Hidrográfica do Rio São Francisco.* Universidade de Brasília.

Swain, T., & Hills, W. E. (1959). The phenolic constituents of Prunus domestica I. - The quantitative analysis of phenolic constituents. *Journal of the Science of Agriculture*, *10*(1), 63–68.

Tao, Y., García, J. F., & Sun, D. (2014). Advances in Wine Aging Technologies for

Enhancing Wine Quality and Accelerating Wine Aging Process. *Critical Reviews in Food Science and Nutrition*, *54*, 817–835. http://doi.org/10.1080/10408398.2011.609949

Tonietto, J., & Teixeira, A. H. de C. (2007). *O clima vitícola do submédio são francisco e o zoneamento dos períodos de produção de uvas para elaboração de vinhos*. *I Workshop Internacional de Pesquisa*.

Viviani, A., Moreno, J., & Peinado, R. A. (2007). Differentiation of young red wines obtained in a warm climate region. *International Journal of Food Science and Technology*, *42*, 523–527. http://doi.org/10.1111/j.1365-2621.2006.01240.x

CAPÍTULO 4

Vinho Syrah do Vale do São Francisco, Brazil: Caracterização sensorial e percepção dos consumidores.

Natália Manzatti Machado Alencar; Tatiane Godoy Ribeiro; Bruna Barone; Ana Paula André Barros; Aline Telles Biasoto Marques; Jorge Herman Behrens. **Syrah Wine from the São Francisco Valley, Brazil: Sensory Characterization and Consumers' Perception.** Submetido em 26 de março de 2018 à revista internacional **Food Research International,** Elsevier, ISSN: 0963-9969.

Vide autorização do parecer consubstanciado do CEP (ANEXO 2).

Vide submissão do artigo (ANEXO 5).

SYRAH WINE FROM THE SÃO FRANCISCO VALLEY, BRAZIL: SENSORY CHARACTERIZATION AND CONSUMERS' PERCEPTION

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ABSTRACT

Aging wine in oak barrels provides more complexity to the beverage, but due to the high cost of wood barrels, alternative aging methodologies such as the use of oak chips gain interest of winemakers in the new world of wines, particularly in the tropical regions. This paper reports the sensory profile and consumer perception of Syrah wine produced in the São Francisco Valley, Brazil, with the addition of American and French oak chips at different stages of the fermentation process. Higher intensity of color, coffee, woody, and sweet/caramelized aromas, sweet and woody taste were observed in the age wines as compared to a control wine (no chip addition). An affective sensory test (N=129) revealed two segments of consumers exhibiting different attitudes to the wines: one group (N=60) rejected all samples, whereas the other (N=69) liked them moderately. In both segments the woody character appeared to be the most important driver of liking and this characteristic was related to the use of American oak chips in wine making. In short, the findings provide relevant information for stakeholders in the new world of wine where oak barrels impact negatively on the cost of production of wines, as is the case in Brazil.

Keywords: Syrah, oak chip, descriptive analysis, sensory profile, CATA.

1. INTRODUCTION

Wine represents a category of products capable of activating a variety of meanings and representations (Amine & Lacœuilhe, 2007; Parr, Mouret, Blackmore, Pelquest-Hunt, & Urdapilleta, 2011), mentioned that wines one of the most described beverages in the literature (Brochet & Dubourdieu, 2001). Directly related to the western culture (Mouret, Lo Monaco, Urdapilleta, & Parr, 2013) numerous countries make and sell wines being part of the so-called "old world of wine" – Europe - in comparison with the more recent producers or the "new world of wine" – Americas, South Africa and Oceania (Le Parisien, 2016).

Brazil is part of the new world of wine and has been making efforts not only to improve the quality and the internal consumption of Brazilian wines, but also to promote the product into the global market (Vieira, Watanabe, & Bruch, 2012). In this sense, the producers seek to consolidate regional identities, for example *Vale dos Vinhedos* and *Campanha Gaúcha* in southern Brazil, and invest in emerging locations such as the *São Francisco Valley*, in the states of *Bahia* and *Pernambuco* in the northeast part of the country (Niederle & Vitrolles, 2010). This is a semi-arid region characterized by high temperatures, high insolation rates, absence of winter and water supplied in abundance from the São Francisco River (Pereira, 2013).

Syrah, is among the cultivars of *Vitis vinifera,* the varietal that better adapted to the edafoclimatic conditions of the region (Camargo, Amorim, Guerra, & Lima, 2004), accounting for about 30% of the vineyards. Syrah of the São Francisco Valley produces wine with intense red color and fruity (plum, raspberry and gooseberry), violet and pepper aromatic notes. Syrah wines from the São Francisco Valley have also shown high phenolic compounds content, especially tannins which making them suitable for aging in wood (Andrade, Nascimento, Pereira, Hallwass, & Paim, 2013; Lucena et al., 2010).

The influence of wood on wine aging is an interesting and controversial subject. Traditionally, oak wood is used because it is flexible enough to mount barrels, with little porosity, an acceptable level of tannins and mild aromatic impact. The oak barrels not only add aromas and tannins to the wine making it more complex, but also, because of their porosity, they allow the drink to breathe, develop and mature.

Among the best species suited to the manufacture of barrels are the *Quercus alba*, also called American oak, *Quercus robur* and *Quercus petraea*, found in most of the French forests (Lona, 1996). While the American oak gives an aromatic note reminiscent

of coconut and vanilla to the wine, the French oak, which is considered more sophisticated, passes to the wine different aromas that resemble coffee, spices and butter (Amerine & Singleton, 1984; Jackson, 2008). The geographic origins and species of wood (American: *Quercus alba* or French: *Quercus petrae*) improve the volatile profile and implied in different sensory characteristics; it could be more accentuated when the toasting chips are used (Schumacher, Alañón, Castro-Vázquez, Pérez-Coello, & Díaz-Maroto, 2013). The degree of toast of the wood may also contribute with roasted and smoked aromas (Koussissi et al., 2009). Wines aged in medium-toasted oak have richer vanilla and wood aromas, while wines aged in oak with light toast are characterized by vegetative aroma and have less intense sweet taste and higher astringency when compared to medium-toasted oak (Chira & Teissedre, 2015; González-centeno, Chira, & Teissedre, 2016).

Brazilian wineries and most of new world need to import oak barrels from North America and Europe, which has an impact on the cost of producing wine. An alternative is the use of oak chips, an economically more viable alternative for wines to gain or increased the complexity and aromas of the wood without suffering great impact of cost (Eiriz, Oliveira, & Clímaco, 2007). The chips can be put in contact with the wine in several stages, from the fermentation of the must until the wine is ready. In addition, they are either used in place of the aging period in barrels, or to supplement it, in cases when more intense influence of wood is desired. Although somewhat controversial, it is a valid artifice and, when used properly and consciously, satisfactory results may be obtained (Koussissi et al., 2009; Pizarro, Rodríguez-Tecedor, Esteban-Díez, Pérez-Del-Notario, & González-Sáiz, 2014; Tao, García, & Sun, 2014).

Notwithstanding investments in viticulture and technology, the *per capita* consumption of wine in Brazil remain low, reaching 2 liters, as compared to other South-American neighbors such as Chile (14. 7 liters) and Argentina (31.6) (Almeida, Bragagnolo, & Chagas, 2015). Moreover, data from the *Instituto Brasileiro do Vinho* showed a decrease of 7.94% in the consumption of wines produced locally between 2016 and 2017, while wine imports rose by 39.1% (IBRAVIN, 2017). This raises a question: do Brazilian consumers actually reject national wine and for what reasons ? (e.g., sensory quality, product image, stereotypes?).

From the sensory point of view, traditional sensory methods such as Quantitative Descriptive Analysis (QDA) and acceptability testing (e.g, hedonic and JAR scales) have been used to reveal and quantify sensory characteristics as well as consumer attitudes to

wine (Behrens & Silva, 2000; Behrens, Silva, & Wakeling, 1999; Villanueva & Da Silva, 2009; Biasoto, Netto, Marques, & Silva, 2014) . Though affective methods are the most suitable way of assessing consumers' preference, the traditional descriptive methods, despite their validity and robustness for describing and quantifying sensory characteristics by means of trained or expert panels, demand a lot of time and effort to produce results (Cadena et al., 2014; Dehlholm, Brockhoff, Bredie, 2012; Meinert, Aaslyng, & Antúnez, Vidal, Saldamando, Giménez, & Ares, 2017). In this sense, novel and "quick" methods have gained interest among sensory and consumer scientists in the recent years like flash profile, napping and CATA (Check-all-that-apply) (Valentin, Chollet, Lelièvre, & Abdi, 2012).

CATA has shown as prominent tool to describe and differentiate samples in relation to their specific characteristics, helping in the identification of consumers' perceptions and preferences for food products and beverages (Ares, Deliza, Barreiro, Giménez, & Gámbaro, 2010), for its simplicity and ease to be applied with trained or untrained assessors (Meyners & Castura, 2014; Ares, 2015). CATA questions consist of a preselected list of terms from which respondents select all that they consider appropriate to describe a product. The data can be further analyzed by non-parametric tests based on the Chi-square distribution or correspondence analysis (CA) to obtain a perceptual map (Meyners, Castura, & Carr, 2013).

Vidal, Giménez, Medina, Boido, & Ares, (2015) used CATA list comprising 44 terms to assess how consumers describe astringency in red wine. Results showed that, although consumers' vocabulary seemed limited, they could accurately describe wine astringency being this sensation mainly related to dry and roughness sensations. In another research involving wine consumers in China, Corsi, Cohen, Lockshin, & Williamson (2017) performed an acceptance test along with a CATA to test the equivalence of flavor descriptors typically used for wine in Western countries with local descriptors for Chinese consumers. Generic descriptors (e.g., oaky, astringent, fruity, etc.) terms were used approximately three times more often than specific (e.g. vanilla, plum, green bell pepper) terms, and local specific descriptors were not selected consistently more often than the non-local (western) specific descriptors. These studies demonstrate the importance of assessing how the consumer perceives and describe wine instead of relying only on answers from expert panels, whose way of evaluating and even assigning quality to the wine does not match with that of the consumer.

Therefore, the present work aimed, in a first step, to determine the descriptive sensory profile of Syrah wine from the São Francisco Valley elaborated with and without (control) addition of American and French oak chips. Afterwards, consumers evaluated the acceptability and characterized their perceptions of the beverages.

2. MATERIALS AND METHODS

2.1 Winemaking

Syrah grapes from Embrapa Semi-Arid vines situated at 09 ° 09 'S, 40 ° 22 'W, 365.5 m in the São Francisco Valley, Pernambuco, Brazil were harvested manually in the vineyard on July 2015 in their optimal ripening stage (25.99 º Brix). After the harvest, the grapes were kept in a cold chamber for 10 hours at 10 °C to decrease and stabilize the temperature. Sequentially, the grapes were destemmed with the aid of a commercial destemmer and were transferred to a stainless-steel tank. Six batches of grapes were processed in a semi-industrial scale (Table 1); maceration started with the addition of potassium metabisulfite (0.10 g L^{-1}). Pectinolytic enzyme Everinetec® (0.008 mL L^{-1}), commercial yeast Maurivin Consistent Quality® *Saccharomyces cerevisiae* (0.20 g L^{-1}) and activating Coatec® ammonium phosphate (0.20 g L^{-1}) were added to the must to start the alcoholic fermentation under controlled temperature (24 °C ± 2 ºC) and 4 g L^{-1} of American and/or French oak chips were added in the must. For the wines that receive the oak chip in the alcoholic and malolatic fermentations, chips were substituted for new ones prior to the malolactic fermentation. The oak chips were placed in nylon bags positioned at the center of the tank.

The oak chips tested were French oak chips (France / AEB-group) and it was 2.5 x 5.0 x 0.5 cm, 100% wood *Quercus Petreae*, with higher toasted. American oak chips (Italy / Everintec) dimensions was 2.5 x 2.0 x 1.0 cm; 100% wood *Quercus Alba*, with medium toast. The quantities of the chips and degree of toast were determined according to the manufacturer's specifications and preliminary studies in the literature (Bélen Gordillo, Cejudo-Bastante, Rodríguez-Pulido, González-Miret, & Heredia, 2013; Gómez García-Carpintero, Sánchez-Palomo, & González Viñas, 2014).

The maceration processes with the skins and seeds were carried for all up to a maxi mean of 30 days. At this stage, the open pumping of 40% of the total volume of the tank was daily made, in order to promote homogenization. In the end of the alcoholic

fermentation (20 days) was determined by measuring the density in precision analytical balance (Alfa Instrumentos®, model 3110), the skins and seeds were separated from the wine and pressed through pressing at 50 bar (Control Tech Automação). The wines were placed in a carboy container (20 liter) and the malolactic fermentation started naturally and it was temperature-controlled (18 °C ± 1 ºC). Then, the wines received the oak chips; the treatments that received oak chips in the alcoholic fermentation had the chips replaced by new ones. The free concentration of sulphur dioxide levels in red wines were adjusted in 50 mg L^{-1} during overall experiment (Ribereau-Gayon & Peynaud, 1980). The end of the malolactic fermentation was checked by paper chromatography (to determine the presence of malic acid) and the wines were further stabilized using stabigum (0.4 g L^{-1}), in cold chamber, to induce tartaric stabilization. At the end of this stage, the wine was subjected filtered to retain yeasts and suspended particles. The wine was bottled (750 mL), in bottles previously filled with nitrogen to prevent oxidation, and stored in a cellar at 18 °C in horizontal position for 30 days until the analyses.

The color of the wines was determined using the CIELab system were the parameters L* (luminosity), a* (green-red) and b* (blue-yellow) were measured in a Hunter Lab model Color Quest II spectrophotometer (Hunter Associates Laboratory, Reston, VA, USA). The equipment was calibrated with the illuminant D65, 10° hue angle and the RSIN calibration mode (Minolta, 1994). Finally, pH, total acidity, volatile acidity, alcoholic content, free and total sulfur dioxide, and reducing sugars were determined (triplicates) for all the samples according to the standard methods Association of Official Analytical Chemists, (2007).

Table 1 summarized the winemaking and presents the physicochemical characteristics of the wines.

Table 1: Wines and their physicochemical characterization.

Samples	Color			pH	Total Acidity (tartaric acid g L^{-1})	Volatile acidity (acetic acid g L^{-1})	Alcoholic content (% v/v)	Free sulfur dioxide (g)	Total sulfur dioxide (g)	Reducing sugars (g)
	L	a*	b*							
WC	3.01±0.68	18.89 ±0.98	3.23 ±0.85	4.04	6.50	0.56	13.43	34.30	74.24	1.42
WAAMF	4.33 ±0.48	27.01 ±0.70	5.47 ±0.24	4.05	6.60	0.76	12.61	33.28	66.22	1.66
WAMF	3.04 ±0.29	18.88 ±0.93	3.08 ±0.38	4.01	6.65	0.74	12.63	30.55	88.41	1.59
WFAMF	2.79 ±0.73	14.69 ±0.23	3.11 ±0.76	4.06	6.50	0.80	12.90	28.50	70.48	1.92
WFMF	3.98 ±0.66	23.03 ±0.77	4.32 ±0.85	4.03	6.65	0.58	13.35	31.48	62.63	2.00
WAFAMF	3.37 ±0.28	20.49 ±0.37	3.39 ±0.29	4.02	6.95	0.68	13.15	30.80	80.90	2.02

WC: Control wine without wood chip; WAAMF: Wine with added American oak chip in both alcoholic and malolactic; WAMF: Wine with added American oak chip in malolactic fermentation; WFAMF: Wine with added French oak chip in both alcoholic and malolactic; WFMF: Wine with added French oak chip in malolactic fermentation; WAFAMF: Wine with added American and French oak chips in both alcoholic and malolactic fermentation.

2.2 Sensory evaluation

The protocols of the sensory studies were approved by the Ethics Committee of the University of Campinas, under the protocol (CAAE nº 34586014.9.0000.5404). All the subjects were properly informed of the objectives of the research and gave signed consent for their participation.

2.2.1 Descriptive Sensory profiling

The sensory profile of the wines was performed in the Sensory Analysis Laboratory of Embrapa Semi-arid. Candidates for assessors were preselected based on recognition of basic tastes and aromas (Biasoto, Netto, Marques, & Silva 2014; Sánchez-Palomo, Trujillo, Ruiz, & Viñas, 2017). Afterwards, they were trained using the terms in the in Wine Aroma Wheel® to memorize the types of aromas regularly found in red wines (Noble et al., 1987). The panelists were asked to identify each reference and find the identical pattern in a second subset containing the same scents, but coded with different three-digit numbers. Thus, the Wine Aroma Wheel® standards were evaluated in several sessions until panelists correctly identified 100% of the standards presented. Finally the 12 assessors were chosen according to their ability of correctly identified the aromas.

The Kelly's Repertory Grid Method was applied to generate a list of descriptors (Moskowitz, 1983). For each session, the samples were presented in pairs and the assessors had to describe similarities and differences regarding appearance, aroma, taste, and mouthfeel. In subsequent sessions, the panel discussed the descriptors and consensually defined the 17 attributes that characterized the wines, their verbal definitions and references as shown in Supplementary Table S1 (Anexo 6).

Ten training sessions (1hour each) were performed for the assessors to learn the attributes having references of maximum and minimal intensity for each established attribute (Table S1 – Anexo 6). Also during the training, a 9-cm linear unstructured scale was presented to the panel as an instrument for measuring the intensity of each sensory attribute (Associação Brasileira de Normas Técnicas, 1998; Behrens & Silva, 2000). After the training, the panel evaluated all the samples in three replicates, using a ballot comprising the 17 attributes and the respective rating scales.

For the sensory profiling 30 ml of wine were served at 18 ºC, in ISO (3591) wine glasses, coded with three-digit numbers, and covered with watch glass to prevent the loss of volatile aroma. The evaluations were carried out in individual booths under white

illumination and the samples were presented monadically in a completely randomized blocks design. The assessors were oriented to rinse the palate with water and crackers between the samples.

Analysis of Variance (Anova) was performed on the whole panel data for selecting the assessors based on discrimination of wines ($p \leq 0.30$ of F) and repeatability of the judgements ($pF > 0.05$ of repetition). Anova with interaction (assessors x wines) for each attribute was performed to evaluate the panel (Damásio & Costell, 1991). On this basis, 10 assessors were chosen to integrate the final panel to evaluate the wines.

Principal component analysis (PCA) was used to summarize the sensory data and to uncover underlying dimensions that help to explain the relationship between the processes variables and the resulting sensory profile of the wines. The procedure was performed using the package FactoMiner for R (Lê, Josse, & Husson, 2008).

2.2.2 Consumer testing

A total of 129 Brazilian consumers (aged between 21 and 50 years old, 49% male and 51% female) evaluated the samples in four different settings: two wine stores, a wine-lovers group and the Sensory and Consumer Science Laboratory at the School of Food Engineering of the University of Campinas, Brazil. About 80% of the participants stated to consume at least one glass of wine on a weekly basis.

Wines (Table 1) were monadically served in 30 mL samples, in wine glasses coded with three - digit random numbers following a balanced presentation order to mimimize first-order and carry over effects (Macfie, 1989). Water and unsalted crackers were available to clean the palate.

Liking was assessed using a 9-point structured scale anchored at the extremes by "disliked very much" and "liked very much" (Stone & Sidel, 2004). Then, they completed the CATA question comprising 21 terms related to sensory attributes (*persistence of flavor, alcoholic, equilibrate, dryness, sourness, sweetness, bitterness, light red color, deep red color, young, body, watery, bouquet, herbaceous, red fruit, vanilla, woody, spicy, vegetative, aromatic, grape*). These terms were chosen considering the descriptors elicited by the trained accessors and other terms selected from literature (Schumacher et al., 2013; Biasoto et al., 2014; Izquierdo-Cañas, Mena-Morales, & García-Romero, 2016). The order in which the CATA terms appeared in the list were balanced for each

consumer (Antúnez et al., 2017; Ares, Barreiro, Deliza, Giménez, & Gámbaro, 2010; Ares & Jaeger, 2013).

Hierarchical Cluster on Principal Components (HCPC) was used to identify groups of consumers with similar acceptance patterns (Husson, Josse, & Pagès, 2010). The procedure basically consisted on a PCA performed on the acceptance data (matrix of the 129 consumers on the six wines) followed by hierarchical clustering (Ward's method) performed on the principal components of the PCA (i.e. the scores scaled to the associated eigenvalues). Two clusters of consumers were then identified and characterized by means of demographics.

Cochran's Q test, Correspondence analysis (CA) and Principal Coordinate Analysis (PcoA) were used to treat CATA data within each cluster in order to verify how the two groups of consumers described their perceptions of the wines.

The FactoMiner package for R (Lê et al., 2008) was used to perform HCPC whereas Cochran's Q, CA and PCoA were performed using the feature CATA Analysis on XLStat, Version 2017.4 (Addinsoft, New York, USA).

3. RESULTS AND DISCUSSION

3.1 Sensory profile

According to Table 3, significant differences ($P \leq 0.05$) were found with respect to 7 out of 17 attributes describing the wines.

Table 2: Attribute mean scores for appearance, aroma, taste and mouth sensations for each sample of Syrah wine by the trained sensory panel (n_1 = 10 assessors, n_2 = 3 repetitions/sample).

	Control Wine	***American oak chip***		***French oak chip***		***American and French oak chip***	
Descriptors	WC	WAAMF	WAMF	WFAMF	WFMF	WAFAMF	*LSD**
Appearance							
Wine Color	6.06^{ab}	5.39^{b}	6.27^{a}	5.74^{ab}	5.94^{ab}	6.32^{a}	0.81
Brightness	5.70^{a}	5.60^{a}	5.16^{a}	5.21^{a}	5.47^{a}	5.14^{a}	0.91
Aroma							
Aromatic intensity	4.07^{b}	5.35^{a}	4.63^{ab}	4.70^{ab}	4.64^{ab}	5.62^{a}	1.00
Alcoholic	3.38^{a}	3.27^{a}	3.18^{a}	3.14^{a}	3.34^{a}	3.30^{a}	0.80
Coffee	0.73^{ab}	1.17^{a,b}	1.22^{a}	0.64^{a,b}	0.44^{b}	0.78^{ab}	0.76
Woody	1.35^{d}	5.20^{a}	4.14^{a,b}	2.58^{c}	2.68^{c}	3.43^{bc}	1.10
Sweet/caramelized	1.92^{a}	2.22^{a}	1.71^{a}	1.73^{a}	1.60^{a}	1.99^{a}	0.78
Vegetative	1.48^{a}	1.03^{a}	1.24^{a}	1.38^{a}	1.46^{a}	0.77^{a}	0.74
Spicy	1.23^{a}	1.14^{a}	1.29^{a}	1.21^{a}	1.30^{a}	1.48^{a}	0.64
Taste / Flavor							
Taste persistence	3.98^{a}	3.67^{a}	3.64^{a}	4.23^{a}	4.11^{a}	3.78^{a}	0.99
Sweetness	0.78^{b}	0.96^{b}	1.01^{b}	1.14^{b}	1.84^{a}	1.12^{b}	0.51
Bitterness	2.30^{a}	1.71^{a}	2.03^{a}	1.97^{a}	1.75^{a}	1.74^{a}	1.02
Sourness	2.59^{a}	2.12^{a}	2.29^{a}	2.25^{a}	2.23^{a}	2.07^{a}	0.90
Alcoholic	3.76^{a}	3.31^{a}	3.49^{a}	3.73^{a}	3.83^{a}	3.18^{a}	0.91
Woody	1.35^{c}	3.15^{ab}	3.5^{a}	2.63^{ab}	2.11^{bc}	2.91^{a,b}	1.21
Mouth sensations							
Astringency	4.05^{a}	3.62^{ab}	3.74^{ab}	3.82^{ab}	3.02^{b}	3.42^{ab}	1.00
Full-bodied	3.36^{a}	3.98^{a}	3.56^{a}	3.53^{a}	3.31^{a}	3.58^{a}	0.97

Means in the same line showing common letters are not significantly different ($P \leq 0.05$) according to Tukey's test. *Least significant difference. WC: Control wine without wood chip; WAAMF: Wine with added American oak chip in both alcoholic and malolactic; WAMF: Wine with added American oak chip in malolactic fermentation; WFAMF: Wine with added French oak chip in both alcoholic and malolactic; WFMF: Wine with added French oak chip in malolactic fermentation; WAFAMF: Wine with added American and French oak chips in both alcoholic and malolactic fermentation.

Wine color is influenced by fruit maturity because ripe grapes impart higher concentration of monomeric anthocyanins which, in turn, increase the intensity of red color (Sherman, Greenwood, Villas-Boâs, Heymann, & Harbertson, 2017). A recent study reported that wines from the São Francisco Valley, due to the climatic conditions favoring maturation, are rich in anthocyanins, being the violet-colored malvidin-3-O-glucoside the major compound present (Padilha, Biasoto, Corrêa, Lima, & Pereira, 2016). In the present research, high intensity of color was observed in wines, which could be explained by the Syrah grape maturity harvested with 26 °Brix, suggesting a higher content of monomeric anthocyanins that was extracted during the prolonged maceration time (30 days).

Despite the significant differences ($P<0.05$) in color intensity as measured by the trained panel, a short variation in the mean ratings is observed (less than one point in the scale) suggesting that the differences may have been aleatory. As a conclusion, the combined effect of the kind of chip and fermentation can be hardly addressed to the color of the wines. In addition, no significant difference ($P \geq 0.05$) was observed in the brightness of the wines; thus, the chip addition might not be an important factor to this attribute as well.

Wines added with oak chips, as expected, presented significantly higher aromatic intensity ($P>0.05$) as compared to the control wine. However, medium-toast American oak chips added higher coffee and woody notes to the wine, differently from the wines aged with high-toasted French oak which were characterized by higher perception of sweetness, especially after upon malolactic fermentation ($P < 0.05$).

Toasted oak chips improve wine aroma by adding components such as phenols (e.g., vanillin, eugenol and guaiacol), aldehydes (e.g., furfural and syringaldehyde), lactones, furanic compounds (Arapitsas, Antonopoulos, Stefanou, & Dourtoglou, 2004; Gómez García-Carpintero, Sánchez-Palomo, & González Viñas, 2014), and these compounds impart aromas of wood, nuts, vanilla and clove (Cano-López, Bautista-Ortín, Pardo-Mínguez, López-Roca, & Gómez-Plaza, 2008; Schumacher, Alañón, Castro-Vázquez, et al., 2013) On the other hand, wines elaborated with oak chips during alcoholic fermentation may suffer a decrease in some aromas associated with floral, fruity, citric and spicy notes (García-Carpintero et al., 2012). In this research a higher intensity of aroma, along with woody aromas was found in samples elaborated with American oak chips, and it is possible that the intensity of these attributes have decreased the perception of alcoholic, sweet/caramelized, vegetative and spicy aromas in the wines.

According to González-Centeno et al. (2016) the toasting of the oak chip used for wine aging does not impact the sweet aroma differentiation. As expected, the WC wine showed the lowest intensity of all aromas descriptors used compared to aged wines, in agreement with Cano-López et al. (2008) who found that oak chip addition improve the aroma profile and modify the sensory descriptors as compared to the control wine (without chips).

Astringency was more intense in the control wine (WC), and significantly less perceived in the sample WFMF (P <0.05) added with French oak chips during the malolactic fermentation, which suggests that both the chips and the time that they remained in contact with the must have diminished the perception of astringency. In general, both American and French chips had little impact on astringency, differently from García-Carpintero et al., (2012) report that oak chips promoted higher astringency in wines because the extraction of phenolic compounds. The main phenolic compound responsible for astringency in wine is procyanidin B3; however, the aging process in wood causes modification in the phenol structure, resulting in softening of the wine tannins (Ribereau-Gayon, Glories, Maujean, & Dubourdieu, 2006). Moreover, oak chips may also impart a sensation of sweetness resulting from the triterpenoids from the oak wood (Marchal, Cretin, Sindt, Waffo-Téguo, & Dubourdieu, 2015). In the present study, the wine added with French chip during malolactic fermentation presented a sweeter taste as compared to the other samples. This result was expected, since sweetness in the increase during maturation, which could be explained by the extraction of sweet compounds from oak; this phenomenon depends on the nature of the aging (Marchal, Pons, Lavigne, & Dubourdieu, 2013). No significant differences (P >0.05) were observed regarding the attributes, sweet/caramelized, vegetative and spicy aroma, taste persistence, bitterness, acidity, and alcoholic flavor. In fact, as observed in Table 1 the alcohol content was similar among all the wines as they were produced with the same musts and under similar processes.

The aroma and flavor descriptors that showed significant differences between the samples (P > 0.05) were, respectively, coffee and woody aromas and sweetness. Table 3 show that the wood species influenced in the perception these descriptors. Summarizing the descriptive data, the first two principal components obtained from the PCA on the attributes' mean ratings accounted for 76.8% of the original variance and the plot on Figure 1 depict the similarities and differences among the wines as a function of the fermentative processes and chips additions.

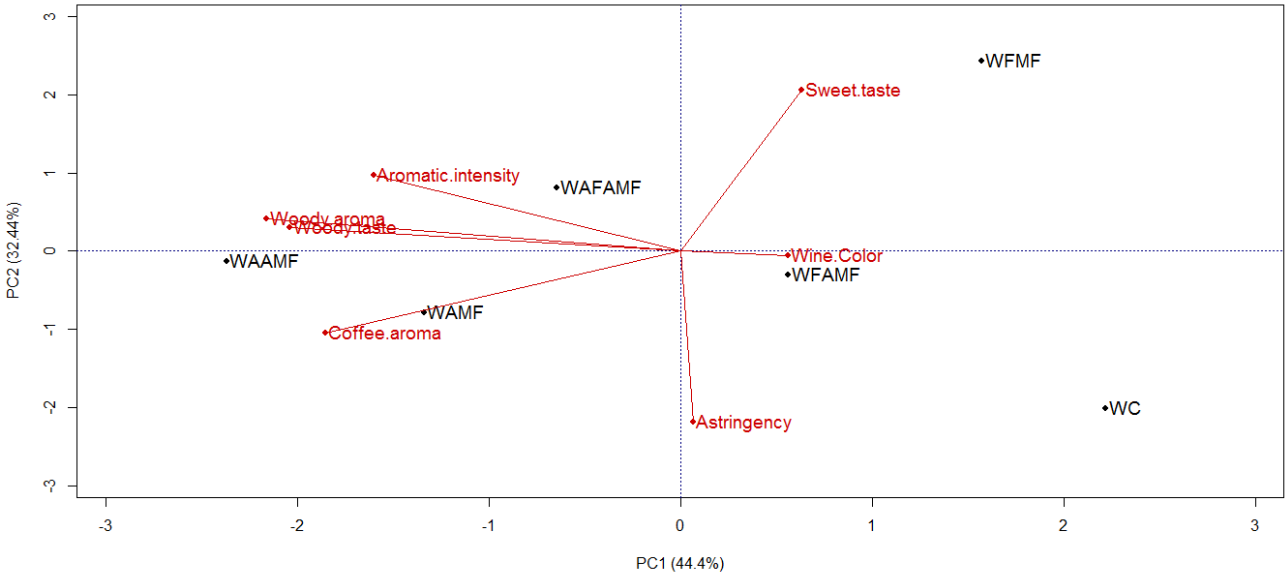


Figure 1. PCA plot depicting the relationship of attributes and wines in the Descriptive Analysis performed by the trained panel (n_1 = 10 assessors, n_2 = 3 repetitions/sample). WC: Control wine without wood chip; WAAMF: Wine with added American oak chip in both alcoholic and malolactic; WAMF: Wine with added American oak chip in malolactic fermentation; WFAMF: Wine with added French oak chip in both alcoholic and malolactic; WFMF: Wine with added French oak chip in malolactic fermentation; WAFAMF: Wine with added American and French oak chips in both alcoholic and malolactic fermentation.

PCA was used to summarize data from the sensory descriptive profile (Table 3). PC 1 (44.4%) had greater contribution of coffee aroma and woody aroma/taste (correlations varying from 0.75 to 0.88) which, in turn, were also related to the aromatic intensity. Wines added with American oak chips (WAAMF, WAMF and WAFAMF) appeared highly associated with these attributes. Plotted in opposition to these wines on the other side of PC1 is the sample WC, which was not submitted any addition. PC2 (32.4%) was related to sweetness, a characteristic of the wine WFMF, added with French oak during the malolactic fermentation, in opposition to the stronger astringency of the control wine WC.

Some works in the literature report that the insertion stage of the oak chips – during the alcoholic or the malolactic fermentation (García-Carpintero et al., 2012; Sánchez-Palomo, Alonso-Villegas, Delgado, & González-Viñas, 2017) and the toasting

degree (Koussissi et al., 2009; Schumacher, Alañón, Castro-vázquez, Pérez-Coello, & Díaz-Maroto, 2013) may promote different sensory characters in wine. In this way, the addition of the chip increases the aromatic complexity of the wine with aromas of clove, vanilla and wood, while the wood flavor is perceived more intensely in wines fermented with American oak chip.

3.2 Consumer testing

HCPC on the liking data revealed two clusters of consumers, as shown in Table 4. It is interesting to note that the most of the consumers participating in the sensory test stated consuming wine regularly (1 x a week or more) and 61.2% were between 26 and 50 years old, that is to say, pertaining to the Millenials and Generation-X consumers. As these individuals were recruited in liquor shops and wine-lovers groups, the sample somewhat reflects a trend verifying in traditional wine markets like the USA and Australia, where wine intake is in increase among the Young, especially the female consumers (Bruwer, Saliba, & Miller, 2011).

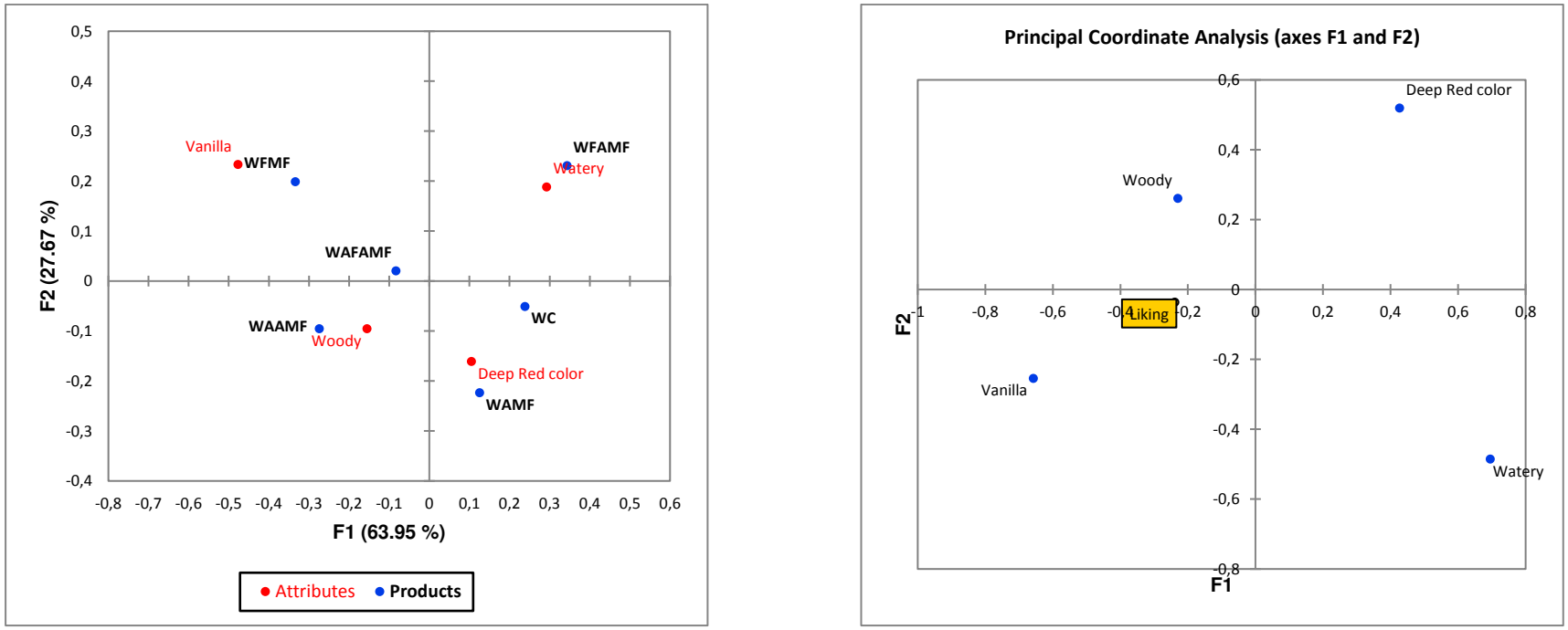
Table 4: Liking of the wines and profile of the two consumer segments identified in the HCPC analysis.

		Cluster 1 (N=60)	Cluster 2 (N=69)
Liking	WC	4.4	6.6
	WAAMF	5.1	6.4
	WAMF	4.4	6.9
	WFAMF	4.4	6.5
	WFMF	4.6	6.6
	WAFAMF	4.8	6.5
	pF	0.072	0.2490
Gender	Male	35	28
	Female	25	41
	Chi-square value (df=1)	4.04*	
Age	18-25 YO	5	8
	26-35 YO	19	23
	35-50 YO	20	17
	>50 YO	16	21
	Chi-square value (df=3)	1.34	
Wine intake	Daily	5	12
	Up to 3x a week	26	28
	1x a week	16	18
	Occasionally	13	11
	Chi-square value (df=3)	2.66	

* Significant (P<0.05); HCPC: Hierarchical Cluster on Principal Components; WC: Control wine without wood chip; WAAMF: Wine with added American oak chip in both alcoholic and malolactic; WAMF: Wine with added American oak chip in malolactic fermentation; WFAMF: Wine with added French oak chip in both alcoholic and malolactic; WFMF: Wine with added French oak chip in malolactic fermentation; WAFAMF: Wine with added American and French oak chips in both alcoholic and malolactic fermentation.

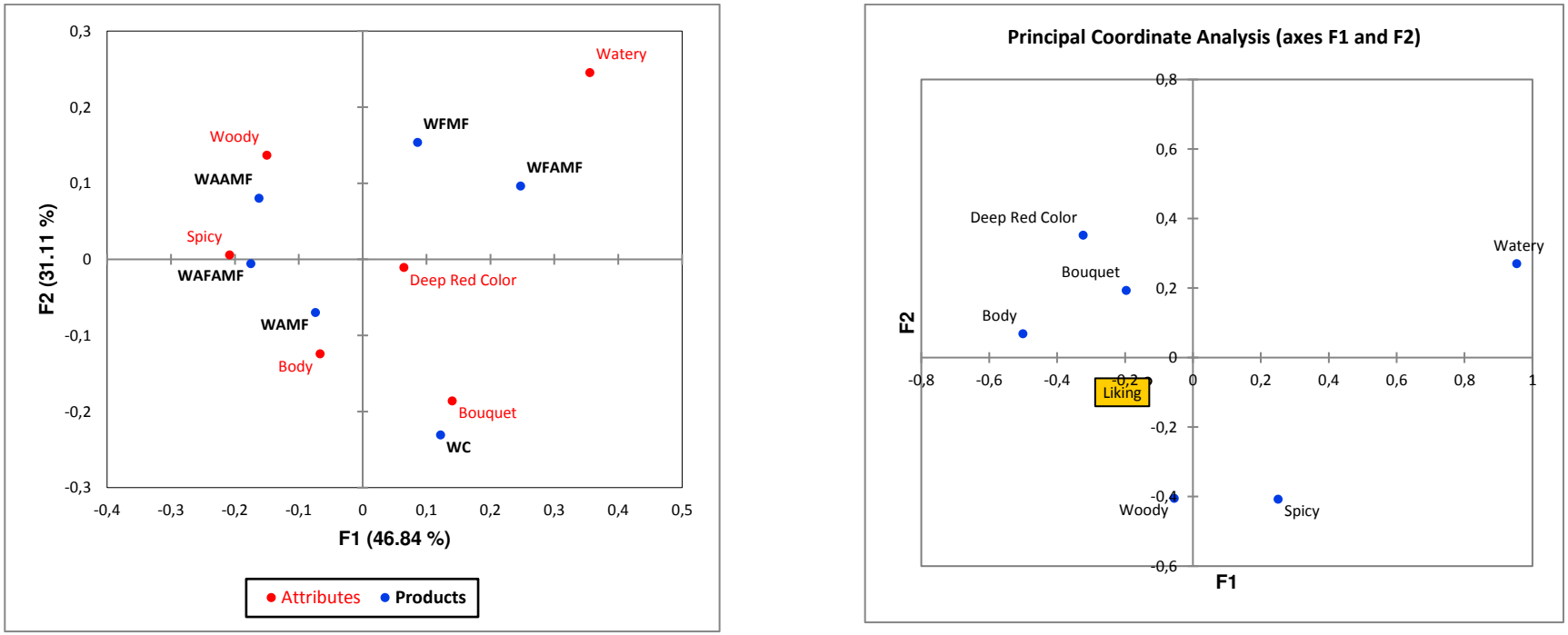
Cluster 1 (N-60) comprised consumers who disliked the wines, given that the mean acceptance scores ranged between 4.4 and 5.1 ($P > 0.05$). On the other hand, the consumers of cluster 2 (N = 69) liked the wines, since the mean acceptance scores were between 6.4 and 6.9 ($P > 0.05$). However, gender was a factor of differentiation between the clusters: there were significantly more male consumers in cluster 1, while in cluster 2 women were a significant majority ($P < 0.05$).

Analyzing the CATA data within each cluster helps to understand how wines were perceived. In cluster 1, only four of the 21 CATA terms showed association with the wine samples ($P < 0.05$) according to the Cochran's Q test and then only these terms, namely vanilla, woody, deep red color and watery, were then used in CA (Figure 2A).



(A) (B)

Figure 2: Cluster 1: (A) Perceptual map resulting from correspondence analysis on significant CATA terms. (B) Principal Coordinate Analysis plot showing the association between liking and CATA terms. WC: control wine without wood chip; WAAMF: Wine with added American oak chip in both alcoholic and malolactic; WAMF: Wine with added American oak chip in malolactic fermentation; WFAMF: Wine with added French oak chip in both alcoholic and malolactic; WFMF: Wine with added French oak chip in malolactic fermentation; WAFAMF: Wine with added American and French oak chips in both alcoholic and malolactic fermentation.



(A) (B)

Figure 3: Cluster 2: (A) Perceptual map resulting from correspondence analysis on significant CATA terms. (B) Principal Coordinate Analysis plot showing the association between liking and CATA terms. WC: control wine without wood chip; WAAMF: Wine with added American oak chip in both alcoholic and malolactic; WAMF: Wine with added American oak chip in malolactic fermentation; WFAMF: Wine with added French oak chip in both alcoholic and malolactic; WFMF: Wine with added French oak chip in malolactic fermentation; WAFAMF: Wine with added American and French oak chips in both alcoholic and malolactic fermentation

The wines added with American oak chips were more associated with woody (especially WAFAMF and WAAMF), whereas WFMF was associated with vanilla and WFAMF was associated with watery. WC and WAMF were associated with deep red color. PCoA revealed that liking was more associated with woody (WAAMF and WAFAMF) and vanilla character (WFMF) (Figure 2B), and it is interesting to note that the trained panel actually rated WAAMF as the most intense woody wine.

Cluster 2, on the other hand, also associated the wines with the terms woody, watery and deep red color, but also with other terms like spicy, body and bouquet. CA (Figure 3A) shows that the wines added with American oak chips (WAAMF, WAFAMF and WAMF) stand out from the rest because they are similarly perceived as woody, spicy and bodied. On the other hand, wines with French oak (WFMF and WFAAMF) were associated with watery, while WC (control) was perceived as having bouquet. Finally, PCoA (Figure 3B) shows liking in cluster 2 associated with woody, body, deep red color, bouquet and spicy characters.

For the foregoing, results suggest that a woody character is an important driver of liking for Syrah wine and this was achieved in the present study by means of addition of American oak chip as it was demonstrated in the descriptive analysis and in the consumer test. Other characteristics appeared to be important as well like bouquet (an aromatic sensation), body and the red color. The study of Bruwer et al., (2011) reports that, from a sensory viewpoint, fruit tastes and aromas are the most important characteristics of a wine valued by consumers. However, while males prefer aged characters of wine, females value oaky and woody aromas these are aged characters also, which is in line with the results presented herein.

The sensory descriptive profile produces a greater detail of the characteristics of the products, however it is a methodology that on average is developed in 2 months or more and requires trained assessors to analyze the samples (Cadena et al., 2014). On the other hand, the CATA method, which has been widely used in sensory and consumer science, the same attributes obtained in the sensorial profiles can be used in a way that consumers can mark the terms that best characterize the product according to their perception (Santos et al., 2015). These methodologies are considered different in their ability to detect differences between samples; however, when comparing the results, it may be noted that the techniques provide similar information. Thus, we observed similarities between the PCA (Fig 1) and the perceptual maps generated from the CATA analysis (Fig 2 and 3). In the PCA (Fig. 1) and perceptual maps of clusters 1 and 2 the WAAMF sample was better related to the woody descriptor and, on the

other hand, the WFMF sample was characterized by sweet taste (Fig 1) and vanilla aroma (Fig 2). Thus, the consumers presented the ability to describe the wines in a similar way to the trained assessors, showing the similarity between the methodologies.

Finally, a major limitation of this study is that it was intended to assess the effect of chip addition as a substitute for the traditional oak barrels in Syrah wine obtained from just one harvest. Nonetheless, other vines are cultivated in the São Francisco valley having potential for wine production. Alternatives should also be further investigated such as the combined use of oak chips (with different degrees of toasting) and micro-oxigenation. Another limitation is that only Brazilian consumer assessed the wines and, therefore, subsequent studies should focus on international markets as long as Brazilian wineries intend to promote their products abroad. Anyway, the findings presented herein are of value to researchers, wine industry and other wine distribution channel members, as it provides insights into the use of oak chip to produce quality wines in the emerging tropical regions and how consumers make sense of them through a sensory viewpoint.

4. CONCLUSIONS

The main objective of this study was to investigate the impact of American and French oak chips in the sensory profile and acceptance of Shiraz wine produced in the São Francisco Valley. Results from sensory descriptive profile revealed that American oak chips imparted higher woody and coffee aroma to the wine, while French oak slightly decreased astringency and increased the perception of sweetness. These differences impacted on the acceptance of the wines and two segments of consumers showed different attitudes to the wine: one group (N=60) rejected all samples, while the other one (N=69) liked them. Through the CATA test it was possible to verify that characters like vanilla bouquet, body, spicy, color, and woody, in special, seem to drive the consumer's preference and in this line. The consumer study revealed that the American oak chips added during the malolactic fermentation produced a wine more appreciated by consumers and, therefore, medium toasted American oak chips seems to be more promising. The treatments tested in the study provide important information for winemakers regarding the potential use of oak chips rather than traditional (and more expensive) barrels, producing quality wines well accepted by consumers. Further study with larger amounts of the chips in different grades of toast would be of interest, since the wood contribute to a greater

transfer of aromatic compounds and enhancing flavor complexity and affecting the consumer preference.

AUTHORS' CONTRIBUTIONS

N.M.M.A., A.T. B.M., J.H.B. conceived and planned the study. N.M.M.A and A.P.A.B. participated of the winemaking process. N.M.M.A., T.R.G. and B.B. contributed to data collection and analysis. All authors contributed to this article.

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CONFLICTS OF INTEREST

The authors declare no conflicts of interest.

REFERENCES

Almeida, A. N., Bragagnolo, C., & Chagas, A. L. S. (2015). A Demanda por Vinho no Brasil : elasticidades no consumo das famílias e. *Revista de Economia E Sociologia Rural*, *53*(3), 433–454. http://doi.org/http://dx.doi.org/10.1590/1234-56781806-9479005303004

Amerine, M. A., & Singleton, V. L. (1984). *Wine an introduction* (2nd.). Berkeley: University of California Press.

Amine, A., & Lacœuilhe, J. (2007). Les pratiques de consommation du vin : rôle des représentations et des situations de consommation. In *XXIIIème Congres International de l'AFM, Aix-les-Bains.* (pp. 1–24).

Andrade, R. H. S. de, Nascimento, L. S. do, Pereira, G. E., Hallwass, F., & Paim, A. P. S. (2013). Anthocyanic composition of Brazilian red wines and use of HPLC-UV – Vis associated to chemometrics to distinguish wines from different regions. *Microchemical Journal*, *110*, 256–262. http://doi.org/10.1016/j.microc.2013.04.003

Antúnez, L., Vidal, L., Saldamando, L. de, Giménez, A., & Ares, G. (2017). Comparison of consumer-based methodologies for sensory characterization: Case study with four sample sets of powdered drinks. *Food Quality and Preference*, *56*, 149–163. http://doi.org/10.1016/j.foodqual.2016.09.013

Arapitsas, P., Antonopoulos, A., Stefanou, E., & Dourtoglou, V. G. (2004). Artificial aging of wines using oak chips. *Food Chemistry*, *86*, 563–570.

http://doi.org/10.1016/j.foodchem.2003.10.003

Ares, G. (2015). Methodological challenges in sensory characterization. *Current Opinion in Food Science*, *3*, 1–5. http://doi.org/10.1016/j.cofs.2014.09.001

Ares, G., Barreiro, C., Deliza, R., Giménez, A., & Gámbaro, A. (2010). Application of a check-all-that-apply question to the development of chocolate milk desserts. *Journal of Sensory Studies*, *25*, 67–86. http://doi.org/10.1111/j.1745-459X.2010.00290.x

Ares, G., Deliza, R., Barreiro, C., Giménez, A., & Gámbaro, A. (2010). Comparison of two sensory profiling techniques based on consumer perception. *Food Quality and Preference*, *21*(4), 417–426. http://doi.org/10.1016/j.foodqual.2009.10.006

Ares, G., & Jaeger, S. R. (2013). Check-all-that-apply questions : Influence of attribute order on sensory product characterization. *Food Quality and Preference*, *28*, 141–153. http://doi.org/10.1016/j.foodqual.2012.08.016

Associação Brasileira de Normas Técnicas. (1998). *Escalas utilizadas em análise sensorial de alimentos e bebidas – NBR 14141*. Rio de Janeiro.

Association Of Official Analytical Chemists. (2007). *Official methods of analysis*.

Behrens, J. H., & Silva, M. A. P. (2000). Perfil sensorial de vinhos brancos varietais brasileiros através de Análise Descritiva Quantitativa. *Ciência E Tecnologia de Alimentos*, *20*(1), 60–67.

Behrens, J. H., Silva, M., & Wakeling, I. N. (1999). Avaliação da aceitação de vinhos brancos varietais brasileiros através de testes sensoriais afetivos e técnica multivariada de mapa de preferência interno. *Ciência E Tecnologia de Alimentos*, *19*(2), 214–220.

Behrens, J. H., Vedovato, G. M., Cervato-Mancuso, A. M., & Bastos, D. H. M. (2015). Social representations of safety in food services. *Food Research International*, *74*, 324–328. http://doi.org/10.1016/j.foodres.2015.05.024

Biasoto, A. C. T., Netto, F. M., Marques, E. J. N., & Silva, M. A. A. P. da. (2014). Acceptability and preference drivers of red wines produced from Vitis labrusca and hybrid grapes. *Food Research International*, *62*, 456–466. http://doi.org/10.1016/j.foodres.2014.03.052

Brochet, F., & Dubourdieu, D. (2001). Wine descriptive language supports cognitive specificity of chemical senses. *Brain and Language*, *77*(2), 187–196. http://doi.org/10.1006/brln.2000.2428

Bruwer, J., Saliba, A., & Miller, B. (2011). Consumer behaviour and sensory preference differences: implications for wine product marketing. *Journal of Consumer Marketing*, *28*(1), 5–18. http://doi.org/10.1108/07363761111101903

Cadena, R. S., Caimi, D., Jaunarena, I., Lorenzo, I., Vidal, L., Ares, G., … Giménez, A. (2014). Comparison of rapid sensory characterization methodologies for the development of functional yogurts. *Food Research International*, *64*, 446–455. http://doi.org/10.1016/j.foodres.2014.07.027

Camargo, U. A., Amorim, F. M. de, Guerra, C. C., & Lima, M. V. D. O. (2004). Introdução e Avaliação de Novas Cultivares para vinho no Vale do São Francisco. In *I Workshop Internacional de Pesquisa* (pp. 103–109).

Cano-López, M., Bautista-Ortín, A. B., Pardo-Mínguez, F., López-Roca, J. M., & Gómez-Plaza, E. (2008). Sensory Descriptive Analysis of a Red Wine Aged with Oak Chips in Stainless Steel Tanks or Used Barrels: Effect of the Contact Time and Size of the Oak Chips. *Journal of Food Quality*, *31*(5), 645–660. http://doi.org/10.1111/j.1745-4557.2008.00226.x

Chira, K., & Teissedre, P. L. (2015). Chemical and sensory evaluation of wine matured in oak barrel: effect of oak species involved and toasting process. *European Food Research and*

Technology, (240), 533–547. http://doi.org/10.1007/s00217-014-2352-3

Corsi, A. M., Cohen, J., Lockshin, L., & Williamson, P. (2017). Testing lexical equivalences for wine flavours in emerging markets: Do hawthorns taste like blackberries? *Food Quality and Preference*, *62*, 296–306. http://doi.org/10.1016/j.foodqual.2017.03.011

Damásio, H. M., & Costelli, E. (1991). Análisis sensorial descriptivo: generación de descriptores y selección de catadores. *Revista de Agroquíca Y Tecnologia de Alímentos*, *31*(2), 165–178.

Dehlholm, C., Brockhoff, P. B., Meinert, L., Aaslyng, M. D., & Bredie, W. L. P. (2012). Rapid descriptive sensory methods - Comparison of Free Multiple Sorting, Partial Napping, Napping, Flash Profiling and conventional profiling. *Food Quality and Preference*, *26*(2), 267–277. http://doi.org/10.1016/j.foodqual.2012.02.012

Eiriz, N., Oliveira, J. F. S., & Clímaco, M. C. (2007). Fragmentos de carvalho no estágio de vinhos tintos. *Ciência Técnica Vitivinícola*, *22*(2), 63–71.

García-Carpintero, E. G., Gallego, M. A. G., Sánchez-Palomo, E., & Viñas, M. A. G. (2012). Impact of alternative technique to ageing using oak chips in alcoholic or in malolactic fermentation on volatile and sensory composition of red wines. *Food Chemistry*, *134*, 851–863. http://doi.org/10.1016/j.foodchem.2012.02.194

Gómez García-Carpintero, E., Sánchez-Palomo, E., & González Viñas, M. A. (2014). Volatile composition of Bobal red wines subjected to alcoholic/malolactic fermentation with oak chips. *LWT - Food Science and Technology*, *55*(2), 586–594. http://doi.org/10.1016/j.lwt.2013.10.024

González-Centeno, M. R., Chira, K., & Teissedre, P. (2016). Ellagitannin content , volatile composition and sensory profile of wines from different countries matured in oak barrels subjected to different toasting methods. *Food Chemistry*, *210*, 500–511. http://doi.org/10.1016/j.foodchem.2016.04.139

Gordillo, B., Cejudo-Bastante, M. J., Rodríguez-Pulido, F. J., González-Miret, M. L., & Heredia, F. J. (2013). Application of the differential colorimetry and polyphenolic profile to the evaluation of the chromatic quality of Tempranillo red wines elaborated in warm climate. Influence of the presence of oak wood chips during fermentation. *Food Chemistry*, *141*(3), 2184–2190. http://doi.org/10.1016/j.foodchem.2013.05.014

Husson, F., Josse, J., & Pagès, J. (2010). *Principal component methods - hierarchical clustering - partitional clustering: why would we need to choose for visualizing data*. *Technical report*. Retrieved from http://factominer.free.fr/more/HCPC_husson_josse.pdf

IBRAVIN, I. B. do V. (2017). Comercialização e Importação. Retrieved November 11, 2017, from http://www.ibravin.org.br/Dados-Estatisticos

Izquierdo-Cañas, P. M., Mena-Morales, A., & García-Romero, E. (2016). Malolactic fermentation before or during wine aging in barrels. *LWT - Food Science and Technology*, *66*, 468–474. http://doi.org/10.1016/j.lwt.2015.11.003

Jackson, R. S. (2008). *Wine science: Principles and application*. *Elsevier* (Academic P). California: Elsevier.

Koussissi, E., Dourtoglou, V. G., Ageloussis, G., Paraskevopoulos, Y., Dourtoglou, T., Paterson, A., & Chatzilazarou, A. (2009). Influence of toasting of oak chips on red wine maturation from sensory and gas chromatographic headspace analysis. *Food Chemistry*, *114*(4), 1503–1509. http://doi.org/10.1016/j.foodchem.2008.11.003

Lê, S., Josse, J., & Husson, F. (2008). FactoMineR : An R Package for Multivariate Analysis. *Journal of Statistical Software*, *25*(1). http://doi.org/10.18637/jss.v025.i01

Le Parisien. (2016). Les Vins du Nouveau Monde, une Menace Pour les Vins Français ?

Retrieved December 12, 2017, from http://lesgrappes.leparisien.fr/les-vins-du-nouveau-monde-tendance/

Lona, A. A. (1996). *Vinhos: Degustação, Elaboração e Serviço*. Age.

Lucena, A. P. S., Nascimento, R. J. B., Maciel, J. A. C., Tavares, J. X., Barbosa-filho, J. M., & Oliveira, E. J. (2010). Antioxidant activity and phenolics content of selected Brazilian wines. *Journal of Food Composition and Analysis*, *23*(1), 30–36. http://doi.org/10.1016/j.jfca.2009.08.004

Macfie, H. J. H. (1989). Assessment of the sensory properties of food. *Journal of Sensory Studies*, *4*, 129–148.

Marchal, A., Cretin, B. N., Sindt, L., Waffo-Téguo, P., & Dubourdieu, D. (2015). Contribution of oak lignans to wine taste: Chemical identification, sensory characterization and quantification. *Tetrahedron*, *71*(20), 3148–3156. http://doi.org/10.1016/j.tet.2014.07.090

Meyners, M., & Castura, J. C. (2014). Novel Techniques sensory. In Gáston Ares & Paula Varela (Ed.), *Novel Techniques in Sensory Characterization and Consumer Profiling* (CRC Press, p. 408). Taylor & Francis.

Meyners, M., Castura, J. C., & Carr, B. T. (2013). Existing and new approaches for the analysis of CATA data. *Food Quality and Preference*, *30*, 309–319. http://doi.org/10.1016/j.foodqual.2013.06.010

Minolta. (1994). *Precise color commmunication: Color control from feeding to instrumentantion*.

Moskowitz, H. R. (1983). Product Testing and Sensory Evaluation of Foods. *Food & Nutrition Press*, 605.

Mouret, M., Lo Monaco, G., Urdapilleta, I., & Parr, W. V. (2013). Social representations of wine and culture: A comparison between France and New Zealand. *Food Quality and Preference*, *30*(2), 102–107. http://doi.org/10.1016/j.foodqual.2013.04.014

Niederle, P. A., & Vitrolles, D. (2010). Indicações Geográficas e qualificação no setor vitivinícola brasileiro. *Estudos Sociedade E Agricultura*, *18*(1), 5–55.

Noble, A. C., Arnold, R. A., Buechsenstein, J., Leach, E. J., Schmidt, J. O., & Stern, P. M. (1987). Modification of a standardized system of wine aroma terminology. *American Journal of Enology and Viticulture*, *38*, 143–146.

Padilha, C. V. da S., Biasoto, A. C. T., Corrêa, L. C., Lima, M. dos S., & Pereira, G. E. (2016). Phenolic compounds profile and antioxidant activity of commercial tropical red wines (Vitis vinifera L.) from Sao Francisco Valley, Brazil Carla. *Journal of Food Biochemistry*, *in press*(October). http://doi.org/10.1111/jfbc.12346

Parr, W. V., Mouret, M., Blackmore, S., Pelquest-Hunt, T., & Urdapilleta, I. (2011). Representation of complexity in wine: Influence of expertise. *Food Quality and Preference*, *22*(7), 647–660. http://doi.org/10.1016/j.foodqual.2011.04.005

Pereira, G. E. (2013). Os vinhos tropicais em desenvolvimento no Nordeste do Brasil. *Com Ciência*, *149*, 13–15.

Pizarro, C., Rodríguez-Tecedor, S., Esteban-Díez, I., Pérez-Del-Notario, N., & González-Sáiz, J. M. (2014). Experimental design approach to evaluate the impact of oak chips and micro-oxygenation on the volatile profile of red wines. *Food Chemistry*, *148*, 357–366. http://doi.org/10.1016/j.foodchem.2013.10.039

Ribereau-Gayon, J., & Peynaud, E. Y. O. (1980). *Ciências e Técnicas del vino*. Editorial Hemisfério Sur.

Ribereau-Gayon, P., Glories, Y., Maujean, A., & Dubourdieu, D. (2006). Phenolic Compounds. In *Handbook of Enology: The Chemistry of Wine and Stabilization and Treatments.* (Vol.

2, pp. 141–203).

Sánchez-Palomo, E., Alonso-Villegas, R., Delgado, J. A., & González-Viñas, M. A. (2017). Improvement of Verdejo white wines by contact with oak chips at different winemaking stages. *LWT - Food Science and Technology*, *79*, 111–118. http://doi.org/10.1016/j.lwt.2016.12.045

Sánchez-Palomo, E., Trujillo, M., Ruiz, A. G., & Viñas, M. A. G. (2017). Aroma pro fi le of malbec red wines from La Mancha region : Chemical and sensory characterization. *Food Research International*. http://doi.org/10.1016/j.foodres.2017.06.036

Santos, B. A. Dos, Campagnol, P. C. B., Cruz, A. G., Galvão, M. T. E. L., Monteiro, R. A., Wagner, R., & Pollonio, M. A. R. (2015). Check all that apply and free listing to describe the sensory characteristics of low sodium dry fermented sausages : Comparison with trained panel. *Food Research International*, *76*, 725–734. http://doi.org/10.1016/j.foodres.2015.06.035

Schumacher, R., Alañón, M. E., Castro-Vázquez, L., Pérez-Coello, M. S., & Díaz-Maroto, M. C. (2013). Evaluation of Oak Chips Treatment on Volatile Composition And Sensory Characteristics Of Merlot Wine. *Jounal of Food Quality*, *36*, 1–9.

Schumacher, R., Alañón, M. E., Castro-vázquez, L., Pérez-Coello, M. S., & Díaz-Maroto, M. C. (2013). Evaluation of Oak Chips Treatment on Volatile Composition and Sensory Characteristics of Merlot Wine. *Journal of Food Quality*, *36*, 1–9.

Sherman, E., Greenwood, D. R., Villas-Boâs, S. G., Heymann, H., & Harbertson, J. F. (2017). Impact of Grape Maturity and Ethanol Concentration on Sensory Properties of Washington State Merlot Wines. *American Journal of Enology and Viticulture*, ajev.2017.16076. http://doi.org/10.5344/ajev.2017.16076

Stone, H., & Sidel, J. L. (2004). *Sensory evaluation practices* (3 ed.). California: Elsevier.

Tao, Y., García, J. F., & Sun, D. (2014). Advances in Wine Aging Technologies for Enhancing Wine Quality and Accelerating Wine Aging Process. *Critical Reviews in Food Science and Nutrition*, *54*, 817–835. http://doi.org/10.1080/10408398.2011.609949

Valentin, D., Chollet, S., Lelièvre, M., & Abdi, H. (2012). Quick and dirty but still pretty good: a review of new descriptive methods in food science. *International Journal of Food Science & Technology*, *47*(8), 1563–1578. http://doi.org/10.1111/j.1365-2621.2012.03022.x

Vidal, L., Giménez, A., Medina, K., Boido, E., & Ares, G. (2015). How do consumers describe wine astringency ? *Food Research International*, *78*, 321–326. http://doi.org/10.1016/j.foodres.2015.09.025

Vieira, A. C. P., Watanabe, M., & Bruch, K. L. (2012). Perspectivas de Desenvolvimento da Vitivinicultura em Face do Reconhecimento da Indicação de Procedência Vales da Uva Goethe. *Revista Gestão, Inovação E Tecnologia*, *2*(4), 327–343. http://doi.org/10.7198/S2237-07222012000040001

Villanueva, N. D. M., & Da Silva, M. A. A. P. (2009). Comparative performance of the nine-point hedonic, hybrid and self-adjusting scales in the generation of internal preference maps. *Food Quality and Preference*, *20*(1), 1–12. http://doi.org/10.1016/j.foodqual.2008.06.003

DISCUSSÃO GERAL

O clima semiárido do Vale do São Francisco, caracterizado por elevadas temperaturas e luminosidade durante todo ano, favorece a síntese de compostos fenólicos na uva e, portanto, eleva atividade antioxidante, em especial da variedade Syrah (PADILHA et al., 2017).

No presente estudo comprovou-se que a maceração prolongada no processo de vinificação, combinada com a utilização de chips de carvalho americano e/ou francês, produziram bebidas de boa aceitação pelos consumidores brasileiros, o que torna viável sua produção na região do semi-árido brasileiro. A Figura 1 resume todas as etapas e resultados obtidos nesta pesquisa.

O nível de maturação da uva, além do teor de açúcar necessário à fermentação, determina a composição das substâncias de cor e aroma que, por sua vez, terão impacto direto na qualidade do vinho. Uma etapa crítica na extração dessas substâncias é a maceração ou o tempo de contato das cascas e sementes da uva com o mosto.

A extração das antocianinas ocorre em maior intensidade entre três e seis dias de maceração, sendo a malvidina-3-glicosídeo a antocianina predominante (GIACOSA et al., 2015; KELEBEK et al., 2006b). Durante a maceração ocorre um aumento constante de antocianinas até o 5º dia, quando ocorre uma ligeira estabilização na extração desse composto, diminuindo assim a concentração de antocianinas livres no mosto até a remoção das cascas (GOMEZ-MIGUEZ; GONZALEZ-MIRET; HEREDIA, 2007).

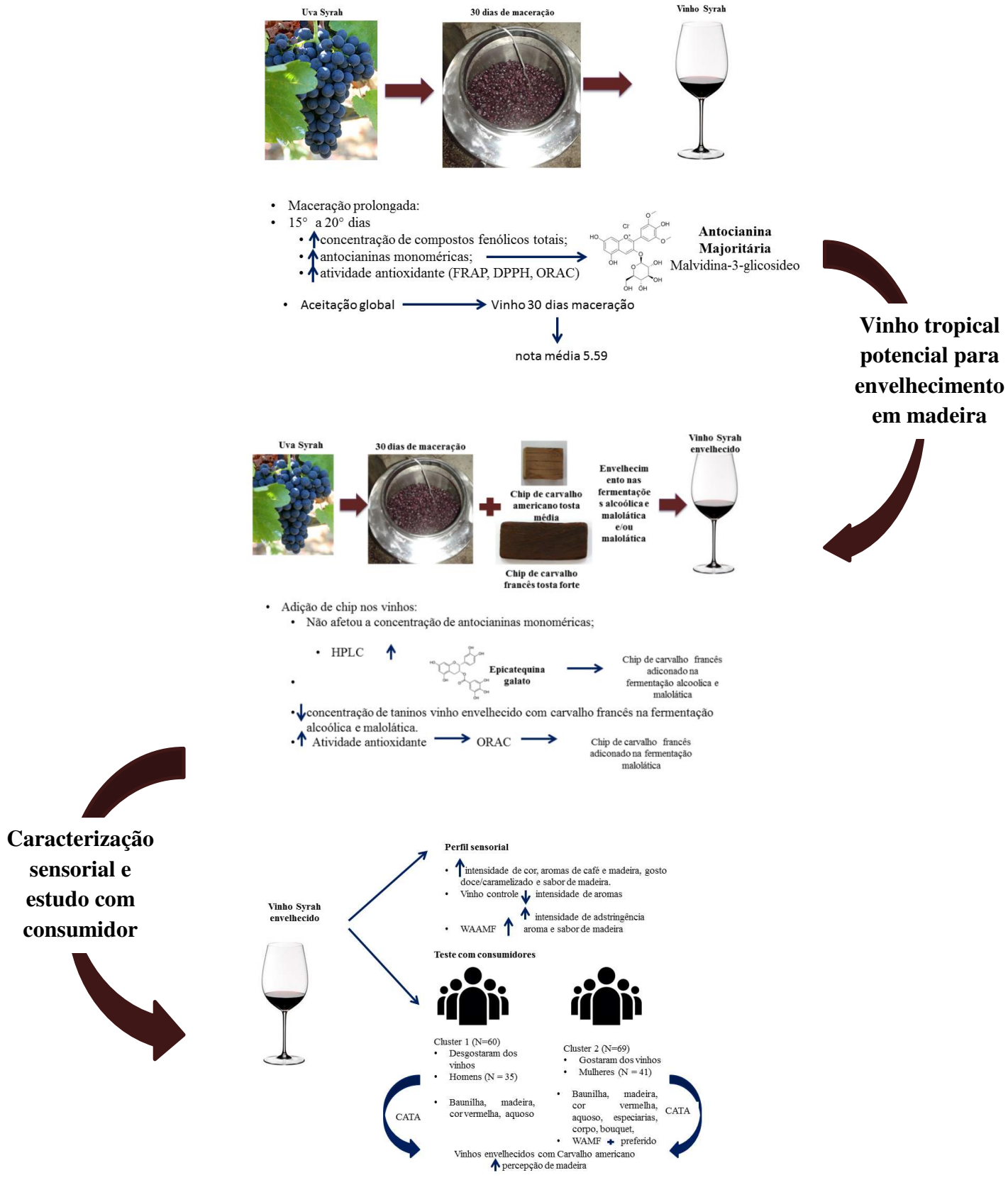

FIGURA 1. Produção e caracterização do vinho Syrah produzido no Vale do São Francisco.

Verificou-se que a maceração prolongada foi efetiva na extração de antocianinas principalmente no período do 15º ao 20º dia, sendo a malvidina-3-glicosídio a antocianina predominante. Além de antocianinas, ocorre também a extração de taninos e flavonóis presentes na semente e na casca da uva (RIBÉREAU-GAYON et al., 2006). Os taninos são responsáveis pela adstringência, enquanto os flavonóis podem contribuir para percepção de amargor da bebida (PREYS et al., 2006).

As práticas enológicas durante a vinificação podem implicar na maior extração de taninos, isso ocorre quando é realizada a maceração prolongada, em 30 dias, resultando em elevadas concentrações de taninos no vinho (WATERHOUSE; SACKS; JEFFERY, 2016) e, portanto, maior adstringência e amargor (FEDERICO CASASSA et al., 2013). Entretanto, no presente trabalho a avaliação sensorial descritiva realizada no vinho controle (sem adição de chip), obtido com 30 dias de maceração, revelou baixas intensidades de adstringência e amargor. Além disso, o vinho obteve boa aceitação, com nota 6,6 na escala hedônica de 9 pontos. Desta forma, maceração no período de 30 dias promoveu benefícios na vinificação de vinho Syrah.

A evolução de extração de compostos fenólicos e sua retenção no vinho que ocorre na maceração prolongada (MULERO et al., 2011) torna o vinho potencial para o envelhecimento em madeira, com impacto positivo no aroma e sabor, além da estabilidade da cor vermelha (CASASSA; HARBERTSON, 2014).

O envelhecimento permite a transferência de compostos de aroma da madeira ao vinho e a utilização de chip de carvalho em substituição ao tradicional barril é mais barata, rápida e efetiva no melhoramento do perfil sensorial através de modificações na composição fenólica e atividade antioxidante dos vinhos (GALLEGO et al., 2015).

A adição de chips não produziu aumento na capacidade redutora de compostos fenólicos e na atividades antioxidante do ensaio FRAP; no entanto, no ensaio ORAC observou-se um aumento da atividade antioxidante nos vinhos produzidos com chips de carvalho americano (*Quercus Alba*) e francês (*Quercus Petrea*) adicionados na fermentação malolática (WAMF e WFMF) e no vinho adicionado com a mistura dos chips nas duas fermentações (WAFAMF).

Em trabalhos recentes os chips também não aumentaram a concentração de compostos fenólicos totais resultando em um efeito irrelevante na capacidade antioxidante (GALLEGO et al., 2015; GORDILLO et al., 2016). Apesar dos chip de carvalho não mostrarem efeito

significativo de aumento dos compostos fenólicos e atividade antioxidante, os resultados encontrados no presente estudo foram superiores em comparação a vinhos de outras regiões ao redor do mundo e corroboram os resultados de outros estudos com vinhos produzidos no Vale do São Francisco (BELMIRO; PEREIRA; PAIM, (2017); DE OLIVEIRA et al., (2017); PADILHA et al., (2017).

O chip de carvalho é considerado um método alternativo de envelhecimento que melhora principalmente as características sensoriais de vinhos tintos, produzindo na bebida notas aromáticas que lembram madeira com resultado similar ao envelhecimento em barril novo (OBERHOLSTER et al., 2015). Os principais atributos sensoriais afetados pela madeira são a cor, o sabor e o aroma. A origem geográfica, qual seja, americana (*Quercus alba)* ou francesa (*Quercus petrae)* utilizadas isoladamente ou suas misturas, produzem na bebida características sensoriais de aroma e sabor especificas que podem ser acentuadas com o grau de tosta da madeira (CALDEIRA et al., 2010; KYRALEOU et al., 2015; PICARD et al., 2015; SCHUMACHER et al., 2013a). Dessa forma, o vinho torna-se mais refinado e complexo para ser comercializado (MARCHAL et al., 2015).

Com relação ao perfil sensorial dos vinhos, observou-se que os chips de carvalho americano produziram aromas de café e madeira mais intensos, enquanto o chip francês uma maior percepção de doçura. De fato, os vinhos envelhecidos com chip de carvalho são geralmente descritos por aromas tais como doce, especiarias, baunilha, coco e madeira (CALDEIRA et al., 2010; GARCÍA-CARPINTERO et al., 2012; PIZARRO et al., 2013; SÁNCHEZ-PALOMO et al., 2017c).

Estudos apontam que a aplicação de chip de carvalho em vinho Syrah promove amargor mais intenso a bebida devido às ligninas presentes na madeira (MARCHAL et al., 2015), além de adstringência dos compostos fenólicos (GARCÍA-CARPINTERO et al., 2012). Entretanto, os resultados encontrados no presente estudo mostram que não houve aumento da quantidade de taninos condensados nos vinhos com chips (capitulo 3), inclusive quanto no vinho WFAMF (carvalho francês adicionado nas fermentações alcoólica e malolática) houve um declínio do conteúdo de taninos quando comparado aos demais vinhos. Assim, na análise descritiva, apesar de diferença significativa (P >0,05) a adstringência dos vinhos mostrou-se relativamente baixa, principalmente na amostra WFMF (carvalho francês adicionado na fermentação malolática), percebida como a menos adstringente.

O estudo do consumidor revelou que os chips de carvalho americano adicionados durante as fermentação malolática produziram um vinho mais apreciado pelos consumidores e, portanto, o uso de chips de carvalho americano com tosta médias parece ser mais promissor. Os tratamentos testados no estudo fornecem informações importantes para os vinicultores quanto ao uso potencial de chips em vez de barris tradicionais (e mais caros), produzindo vinhos de qualidade bem aceitos pelos consumidores.

Por fim, esta pesquisa teve algumas limitações. Como a vinificação foi realizada com apenas uma safra, a repetição desse estudo avaliando diferentes safras e as características químicas e sensoriais devem ser realizados. Desta forma, além de entender de forma mais profunda os efeitos do clima e do solo sobre a uva, também teria uma melhor ideia de tipicidade e do *terroir* do vinho Syrah do Vale do São Francisco. Deve-se considerar também que a região produz duas safras anuais (Junho / Julho e Novembro / Dezembro) e possíveis diferenças devem ser avaliadas.

Sugere-se, ainda, um estudo do efeito da informação sobre a expectativa e a aceitação dos vinhos do Vale do São Francisco, visto que a informação pode influenciar as escolhas dos consumidores, uma vez que essa região muitas vezes é ainda desconhecida como produtora de vinhos por boa parte dos consumidores brasileiros da bebida.

CONCLUSÃO GERAL

A maceração prolongada avaliada nesse estudo foi efetiva para extrair maior quantidade de compostos fenólicos até o 20º dia, e após esse período houve uma estabilização da extração até o final do período estudado (30 dias). A técnica enológica foi efetiva para o acúmulo de compostos fenólicos que resultou em elevada atividade antioxidante contribuindo para a diferenciação dos vinhos sem afetar a aceitação sensorial da bebida. A maior extração de compostos fenólicos, notadamente antocianinas e taninos, tornou favorável o envelhecimento do vinho em madeira, além de ter proporcionado o densenvolvimento de compostos aromáticos, cor vermelha intensa e corpo a bebida, descritores identificados pelos assessores na análise descritiva.

O processo de envelhecimento rápido utilizando chip de carvalho americano e/ou francês em substituição ao barril, não afetou significativamente a concentração de compostos fenólicos. Entretanto, observou-se que pelo ensaio ORAC o vinho adicionado de carvalho francês na fermentação malolática apresentou maior atividade antioxidante.

Na avaliação sensorial dos vinhos, as espécies de madeira adicionadas foram favoráveis para diferenciar as bebidas mostrando que o carvalho americano proporcionou ao vinho aromas de café e madeira, enquanto o carvalho francês diminui a percepção de adstringência e aumenta a percepção de doçura na bebida. No teste afetivo, o vinho envelhecido com carvalho americano e tosta média foi o mais aceito pelos consumidores devido aos direcionadores de preferência identificados no estudo (buquê, corpo, aromas de madeira, baunilha e especiarias, além da cor).

Em suma, os resultados deste trabalho mostram o potencial da região semiárida brasileira para produção de vinhos tropicais com boa aceitação pelos consumidores e desenvolvimento de aromas intensos de café e madeira nos vinhos, diversificando o mercado enológico brasileiro.

REFERENCIAS

ALAÑÓN, M. E. et al. A study of the antioxidant capacity of oak wood used in wine ageing and the correlation with polyphenol composition. Food Chemistry, v. 128, p. 997–1002, 2011.

ALAÑÓN, M. E. et al. Enological potential of chestnut wood for aging Tempranillo wines Part II: Phenolic compounds and chromatic characteristics. Food Research International, v. 51, n. 2, p. 536–543, 2013.

ALMEIDA, A. N.; BRAGAGNOLO, C.; CHAGAS, A. L. S. A Demanda por Vinho no Brasil : elasticidades no consumo das famílias e. Revista de Economia e Sociologia Rural, v. 53, n. 3, p. 433–454, 2015.

AMERINE, M. A.; SINGLETON, V. L. Wine an introduction. 2nd. ed. Berkeley: University of California Press, 1984.

AMINE, A.; LACŒUILHE, J. Les pratiques de consommation du vin : rôle des représentations et des situations de consommation. XXIIIème Congres International de l'AFM, Aix-les-Bains. Anais...2007.

ANDRADE, R. H. S. DE et al. Anthocyanic composition of Brazilian red wines and use of HPLC-UV – Vis associated to chemometrics to distinguish wines from different regions. Microchemical Journal, v. 110, p. 256–262, 2013.

ANTÚNEZ, L. et al. Comparison of consumer-based methodologies for sensory characterization: Case study with four sample sets of powdered drinks. Food Quality and Preference, v. 56, p. 149–163, 2017.

ARAPITSAS, P. et al. Artificial aging of wines using oak chips. Food Chemistry, v. 86, p. 563–570, 2004.

ARCARI, S. G. et al. Brazilian forti fi ed wines : Chemical composition , chromatic properties and antioxidant activity. Food rearch international, v. 53, n. 1, p. 164–173, 2013.

ARES, G. et al. Application of a check-all-that-apply question to the development of chocolate milk desserts. Journal of Sensory Studies, v. 25, p. 67–86, 2010.

ARES, G. et al. Comparison of two sensory profiling techniques based on consumer perception. Food Quality and Preference, v. 21, n. 4, p. 417–426, 2010.

ARES, G. Methodological challenges in sensory characterization. Current Opinion in Food Science, v. 3, p. 1–5, 2015.

ARES, G.; JAEGER, S. R. Check-all-that-apply questions : Influence of attribute order on sensory product characterization. Food Quality and Preference, v. 28, p. 141–153, 2013.

ASSOCIAÇÃO BRASILEIRA DE NORMAS TÉCNICAS. Escalas utilizadas em análise sensorial de alimentos e bebidas – NBR 14141. Rio de Janeiro: [s.n.].

ASSOCIATION OF OFFICIAL ANALYTICAL CHEMISTS. Official methods of analysis. [s.l: s.n.].

ATHIA, F.; COSTA, A. D. Empresas e tecnologias na nova conjuntura vinícola brasileira do início do século XXI. Economia & Tecnologia, v. 18, p. 147–156, 2009.

AVILA, L. D. DE; DAUDT, C. E. Indução da fermentação maloláctica em vinho tinto com a cultura láctica viniflora oenos. Ciencia Rural, v. 27, n. 2, p. 325–330, 1997.

BAIANO, A. et al. Wine Processing: A Critical Review of Physical, Chemical, and Sensory Implications of Innovative Vinification Procedures. Critical Reviews in Food Science and Nutrition, v. 56, n. 14, p. 2391–2407, 2016.

BAKER, A. K.; CASTURA, J. C.; ROSS, C. F. Temporal Check-All-That-Apply Characterization of Syrah Wine. Journal of Food Science, v. 81, n. 6, p. 1521–1529, 2016.
BARBA, L. M.; LIZARRITURRY, M. Elaboração do vinho. Barcelona: Folio, 2007.

BARREIRO-HURLE, J.; COLOMBOA, S.; CANTOS-VILLAR, E. s there a market for functional wines? Consumer preferences and willingness to pay for resveratrol-enriched red wine. Food Quality and Preference, v. 19, p. 360–371, 2008.

BEHRENS, J. H. et al. Social representations of safety in food services. Food Research International, v. 74, p. 324–328, 2015.

BEHRENS, J. H.; SILVA, M. A. P. Perfil sensorial de vinhos brancos varietais brasileiros através de Análise Descritiva Quantitativa. Ciência e Tecnologia de Alimentos, v. 20, n. 1, p. 60–67, 2000.

BEHRENS, J. H.; SILVA, M.; WAKELING, I. N. Avaliação da aceitação de vinhos brancos varietais brasileiros através de testes sensoriais afetivos e técnica multivariada de mapa de preferência interno. Ciência e Tecnologia de Alimentos, v. 19, n. 2, p. 214–220, 1999.

BELMIRO, T. M. C.; PEREIRA, C. F.; PAIM, A. P. S. Red wines from South America: Content of phenolic compounds and chemometric distinction by origin. Microchemical Journal, v. 133, p. 114–120, 2017.

BELMIRO, T. M. C.; PEREIRA, C. F.; PAIM, A. P. S. Red wines from South America: Content of phenolic compounds and chemometric distinction by origin. Microchemical Journal, v. 133, p. 114–120, 2017.

BENZIE, I. .; STRAIN, J. . The ferric reducing ability of plasma (FRAP) as a measure of "antioxidant power": the FRAP assay. Analytical biochemistry, v. 239, n. 1, p. 70–6, 1996.

BIASOTO, A. C. T. et al. Acceptability and preference drivers of red wines produced from Vitis labrusca and hybrid grapes. Food Research International, v. 62, p. 456–466, 2014.

BISSON, L. F. et al. The present and future of the international wine industry. Nature, v. 418, n. 6898, p. 696–699, 8 ago. 2002.

BORTOLETTO, A. M.; ALCARDE, A. R. Aging marker profile in cachaça is influenced by toasted oak chips. Journal of the Institute of Brewing, v. 121, n. 1, p. 70–77, 2015.

BRAND-WILLIAMS, W.; CUVELIER, M. E.; BERSET, C. Use of a Free Radical Method to Evaluate Antioxidant Activity. Lwt- Food Science and Technology, v. 28, p. 25–30, 1995.

BRASIL. DECRETO No 8.198, DE 20 DE FEVEREIRO DE 2014. Regulamenta a Lei no 7.678, de 8 de novembro de 1988, que dispõe sobre a produção, circulação e comercialização do vinho e derivados da uva e do vinho. Brasilia: [s.n.].

BRASIL. Lei no 10970, de 12 de novembro de 2004. Dispõe sobre a produção, circulação e comercialização do vinho e derivados da uva e do vinho, e dá outras providências. Brasilia: [s.n.].

BRASIL. Instrução Normativa n° 14, de 08 de Fevereiro de 2018. Complementação dos Padrões de Identidade e Qualidade do Vinho e Derivados da Uva e do Vinho. Brasilia: [s.n.].

BROCHET, F.; DUBOURDIEU, D. Wine descriptive language supports cognitive specificity of chemical senses. Brain and Language, v. 77, n. 2, p. 187–196, 2001.

BRUWER, J.; SALIBA, A.; MILLER, B. Consumer behaviour and sensory preference differences: implications for wine product marketing. Journal of Consumer Marketing, v. 28, n. 1, p. 5–18, 2011.

CADENA, R. S. et al. Comparison of rapid sensory characterization methodologies for the development of functional yogurts. Food Research International, v. 64, p. 446–455, 2014.

CADOT, Y. et al. Sensory representation of typicality of Cabernet franc wines related to phenolic composition: Impact of ripening stage and maceration time. Analytica Chimica Acta, v. 732, p. 91–99, 2012.

CALDEIRA, I. et al. Sensory and chemical modifications of wine-brandy aged with chestnut and oak wood fragments in comparison to wooden barrels. Analytica Chimica Acta, v. 660, p. 43–52, 2010.

CAMARGO, U. A. et al. Introdução e Avaliação de Novas Cultivares para Vinho no Vale Do São Francisco. I Workshop Internacional de Pesquisa. Anais...2004.

CAMARGO, U. A.; TONIETTO, J.; HOFFMAN, A. Progressos na viticultura brasileira. Revista Brasileira de Fruticultura, v. 33, 2011.

CANO-LÓPEZ, M. et al. Sensory Descriptive Analysis of A Red Wine Aged With Oak Chips in Stainless Steel Tanks or Used Barrels: Effect of the Contact Time And Size of The Oak Chips. Journal of Food Quality, v. 31, n. 5, p. 645–660, 2008.

CASASSA, L. F.; HARBERTSON, J. F. Extraction, Evolution, and Sensory Impact of Phenolic Compounds During Red Wine Maceration. Annual Review of Food Science and Technology, v. 5, n. 1, p. 83–109, 2014.

CASSI, L.; MORRISON, A.; RABELLOTTI, R. Proximity and Scientific Collaboration: Evidence from the global wine industry. Wine Economics, v. 149, p. 1–28, 2014.

CAZARIN, C. B. B. et al. Tropical Isabella grape juices: bioactive compounds and antioxidant power depends on harvest season. Journal of Food Science and Engineering, v. 3, n. 2013, p. 64–70, 2013.

CEJUDO-BASTANTE, M. J.; HERMOSÍN-GUTIÉRREZ, I.; PÉREZ-COELLO, M. S. Micro-oxygenation and oak chip treatments of red wines : Effects on colour-related phenolics , volatile composition and sensory characteristics . Part II : Merlot wines. Food Chemistry, v. 124, p. 738–748, 2011.

CEJUDO-BASTANTE, M. J.; RIVERO-GRANADOS, F. J.; HEREDIA, F. J. Improving the color and aging aptitude of Syrah wines in warm climate by wood-grape mix maceration. European Food Research and Technology, v. 243, n. 4, p. 575–582, 2017.

CHANG, K. J.; LIZ THACH, M.; OLSEN, J. Wine and health perceptions: Exploring the impact of gender, age and ethnicity on consumer perceptions of wine and health. Wine Economics and Policy, v. 5, n. 2, p. 105–113, 2016.

CHEYNIER, V. Flavonoids in Wine. In: ANDERSEN, O.; MARKHAM, K. (Eds.). . Flavonoids: Chemistry, Biochemistry and Applications. Boca Raton: CRC Press, 2005. p. 263–318.

CHIRA, K.; TEISSEDRE, P. L. Chemical and sensory evaluation of wine matured in oak barrel: effect of oak species involved and toasting process. European Food Research and Technology, n. 240, p. 533–547, 2015.

CORDONNIER, R. A bioquímica do vinho. Scientific American Brasil, v. 1, p. 22–35, 2007.
CORSI, A. M. et al. Testing lexical equivalences for wine flavours in emerging markets: Do hawthorns taste like blackberries? Food Quality and Preference, v. 62, p. 296–306, 2017.

CVEJIĆ, J.; ATANACKOVIĆ, M. Effect of Wine Production Techniques on Wine Resveratrol and Total Phenolics. In: Processing and Impact on Active Components in Food. Elsevier ed. [s.l.] Elsevier, 2015. p. 501–508.

DA SILVA, J. K. et al. Bioactive compounds of juices from two Brazilian grape cultivars. Journal of the Science of Food and Agriculture, v. 96, n. 6, p. 1990–1996, abr. 2016.

DAMÁSIO, H. M.; COSTELLI, E. Análisis sensorial descriptivo: generación de descriptores y selección de catadores. Revista de Agroquıíca y Tecnologia de Alímentos, v. 31, n. 2, p. 165–178, 1991.

DANNER, L. et al. Context and wine quality effects on consumers' mood, emotions, liking and willingness to pay for Australian Shiraz wines. Food Research International, v. 89, p. 254–265, nov. 2016.

DAY, M. P. et al. Use and impact of oxygen during winemaking. Australian Journal of Grape and Wine Research, v. 21, p. 693–704, 2015.

DE OLIVEIRA, W. P. et al. Phenolics from Winemaking By-Products Better Decrease VLDL-Cholesterol and Triacylglycerol Levels than Those of Red Wine in Wistar Rats. Journal of Food Science, v. 82, n. 10, p. 2432–2437, 2017.

DEHLHOLM, C. et al. Rapid descriptive sensory methods - Comparison of Free Multiple Sorting, Partial Napping, Napping, Flash Profiling and conventional profiling. Food Quality and Preference, v. 26, n. 2, p. 267–277, 2012.

DIAS, F. D. S. et al. Fast determination of phenolic compounds in brazilian wines from Vale do São Francisco region by ce. Chromatographia, v. 76, n. 9–10, p. 559–563, 2013.

DURNER, D. Improvement and Stabilization of Red Wine Color. In: Handbook on Natural Pigments in Food and Beverages: Industrial Applications for Improving Food Color. [s.l.] Elsevier Ltd, 2016. p. 240–264.

EIRIZ, N.; OLIVEIRA, J. F. S.; CLÍMACO, M. C. Fragmentos de carvalho no estágio de vinhos tintos. Ciência Técnica Vitivinícola, v. 22, n. 2, p. 63–71, 2007.

FEDERICO CASASSA, L. et al. Effect of extended maceration and ethanol concentration on the extraction and evolution of phenolics, colour components and sensory attributes of Merlot wines. Australian Journal of Grape and Wine Research, v. 19, n. 1, p. 25–39, 2013.

FERNANDEZ-MAR, M. I. et al. Bioactive compounds in wine: Resveratrol, hydroxytyrosol and melatonin: A review. Food Chemistry, v. 130, n. 4, p. 797–813, 2012.

FERREIRA, P. Brasil inova na produção de vinhos tropicais. Inovação em Pauta, n. 2, p. 16–19, 2008.

FIALHO, V. Z. B. Brasil é referência na produção de vinhos tropicais. Disponível em: <http://www.cnpuv.embrapa.br/noticias/2004/2004-09-13.html>. Acesso em: 21 maio. 2014.

FIGUEIREDO, E. A. DE et al. Antioxidant and Antihypertensive Effects of a Chemically Defined Fraction of Syrah Red Wine on Spontaneously Hypertensive Rats. Nutrients, v. 9, n. 6, p. 574, 2017.

FLINT, D. J.; SIGNORI, P.; GOLICIC, S. L. A Global Overview of Wine. In: Contemporary Wine Marketing and Supply Chain Management. New York: Palgrave Macmillan, 2016. p. 1–14.

FOLMER, F. et al. Anticancer effects of bioactive berry compounds. Phytochemistry Reviews, v. 13, n. 1, p. 295–322, 2014.

FRANCIS, I. L.; WILLIAMSON, P. O. Application of consumer sensory science in wine research. Australian Journal of Grape and Wine Research, v. 21, p. 554–567, 2015.

GALLEGO, M. A. G. et al. Effect of oak chip addition at different winemaking stages on phenolic composition of Moravia Agria red wines. South African Journal for Enology and Viticulture, v. 36, n. 1, p. 21–31, 2015.

GARAMBONE, E.; ROSA, G. Possíveis benefícios do ácido clorogênico à saúde. Alimentos e Nutrição, v. 18, n. 2, p. 229–235, 2007.

GARCÍA-CARPINTERO, E. G. et al. Impact of alternative technique to ageing using oak chips in alcoholic or in malolactic fermentation on volatile and sensory composition of red wines. Food Chemistry, v. 134, p. 851–863, 2012.

GARCÍA-CARPINTERO, E. G.; SÁNCHEZ-PALOMO, E.; GONZÁLEZ VIÑAS, M. A. Volatile composition of Bobal red wines subjected to alcoholic/malolactic fermentation with oak chips. LWT - Food Science and Technology, v. 55, n. 2, p. 586–594, 2014.

GARRIDO, J.; BORGES, F. Wine and grape polyphenols — A chemical perspective. Food Research International, v. 54, n. 2, p. 1844–1858, 2013.

GEFFROY, O. et al. Changes in analytical and volatile compositions of red wines induced by pre-fermentation heat treatment of grapes. Food Chemistry, v. 187, p. 243–253, 2015.

GERMAN, J. B.; WALZEM, R. L. The health benefits of wine. Annual review of nutrition, v. 20, p. 561–593, 2000.

GIACOSA, S. et al. Anthocyanin yield and skin softening during maceration, as affected by vineyard row orientation and grape ripeness of Vitis vinifera L. cv. Shiraz. Food Chemistry, v. 174, p. 8–15, 2015.

GÓMEZ GARCÍA-CARPINTERO, E.; SÁNCHEZ-PALOMO, E.; GONZÁLEZ VIÑAS, M. A. Volatile composition of Bobal red wines subjected to alcoholic/malolactic fermentation with oak chips. LWT - Food Science and Technology, v. 55, n. 2, p. 586–594, 2014.

GOMEZ-MIGUEZ, M.; GONZALEZ-MIRET, M. L.; HEREDIA, F. J. Evolution of colour and anthocyanin composition of Syrah wines elaborated with pre-fermentative cold maceration. Journal of Food Engineering, v. 79, p. 271–278, 2007.

GONZÁLEZ-CENTENO, M. R.; CHIRA, K.; TEISSEDRE, P. Ellagitannin content , volatile composition and sensory profile of wines from different countries matured in oak barrels subjected to different toasting methods. Food Chemistry, v. 210, p. 500–511, 2016.

GONZÁLEZ-MANZANO, S.; RIVAS-GONZALO, J. C.; SANTOS-BUELGA, C. Extraction of flavan-3-ols from grape seed and skin into wine using simulated maceration. Analytica Chimica Acta, v. 513, p. 283–289, 2004.

GORDILLO, B. et al. Application of the differential colorimetry and polyphenolic profile to the evaluation of the chromatic quality of Tempranillo red wines elaborated in warm climate. Influence of the presence of oak wood chips during fermentation. Food Chemistry, v. 141, n. 3, p. 2184–2190, 2013.

GORDILLO, B. et al. Optimisation of an oak chips-grape mix maceration process . Influence of chip dose and maceration time. Food Chemistry, v. 206, p. 249–259, 2016.

GORTZI, O. et al. Effect of artificial ageing using different wood chips on the antioxidant activity, resveratrol and catechin concentration, sensory properties and colour of two Greek red wines. Food Chemistry, v. 141, n. 3, p. 2887–2895, 2013.

GRANATO, D.; KATAYAMA, F. C. U.; CASTRO, I. A. DE. Characterization of red wines from South America based on sensory properties and antioxidant activity. Journal of science food and agriculture, v. 92, n. May, p. 526–533, 2012.

GRIS, E. F. et al. Phenolic profile and effect of regular consumption of Brazilian red wines on in vivo antioxidant activity. Journal of Food Composition and Analysis, v. 31, n. 1, p. 31–40, 2013.

GUERRA, C. C. et al. Vinhos Tropicais: nova paradigma enológico e mercadológico. Informe Agropecuário, v. 27, n. 234, p. 100–104, 2006.

GUERRA, C. C.; ZANUS, M. C. Uvas viníferas para processamento em regiões de clima temperado. Embrapa Uva e Vinho Sistema de Produção, v. 4, 2003.

HARBERTSON, J. F.; SPAYD, S. Measuring phenolics in the Winery. American Journal Enology and Viticulture, v. 57, n. 3, p. 280–288, 2006.

HARTMEIER, W.; REISS, M. Production of Beer and Wine. In: Industrial Applications. Berlin, Heidelberg: Springer Berlin Heidelberg, 2002. p. 49–65.

HEREDIA, F. J. et al. Influence of the refrigeration technique on the colour and phenolic composition of syrah red wines obtained by pre-fermentative cold maceration. Food Chemistry, v. 118, p. 377–383, 2010.

HERNÁNDEZ-ORTE, P. et al. Criteria to discriminate between wines aged in oak barrels and macerated with oak fragments. Food Research International, v. 57, p. 234–241, mar. 2014.

HORNSEY, I. S. Chapter 1. The History of Wine. In: The Chemistry and Biology of Winemaking. Cambridge: Royal Society of Chemistry, 2007. p. 11–64.

HUI, Y. H. et al. Handbook of Food and Beverage Fermentation Technology. 1st Editio ed. Boca Raton: CRC Press, 2004.

HUSSON, F.; JOSSE, J.; PAGÈS, J. Principal component methods - hierarchical clustering - partitional clustering: why would we need to choose for visualizing dataTechnical report. [s.l: s.n.]. Disponível em: <http://factominer.free.fr/more/HCPC_husson_josse.pdf>.

IBRAVIN, I. B. DO V. Comercialização e Importação. Disponível em: <http://www.ibravin.org.br/Dados-Estatisticos>. Acesso em: 11 nov. 2017.

IBRAVIN, I. B. DO V. Regiões produtoras. Disponível em: <http://www.ibravin.org.br/Regioes-Produtoras>. Acesso em: 21 nov. 2017.

IBRAVIN, I. B. DO V. Vinho fino recuou 30,5%, com a comercialização de dois milhões de litros. Foto: Gilmar Gomes Venda de vinhos brasileiros tem recuo de 20% no trimestre. Disponível em: <http://www.ibravin.org.br/Noticia/venda-de-vinhos-brasileiros-tem-recuo-de-20-no-trimestre/260>. Acesso em: 11 nov. 2017a.

INSEL, B. The evolving global wine market. Business Economics, v. 49, n. 1, p. 56–48, 2014.
INSTITUTO BRASILEIRO DE VINHO. Panorama Geral. Disponível em: <http://www.ibravin.org.br/Panorama-Geral>. Acesso em: 21 nov. 2017.

IVANOVA-PETROPULOS, V. et al. Extraction and evaluation of natural occurring bioactive compounds and change in antioxidant activity during red winemaking. Journal of Food Science and Technology, v. 53, p. 2634–2643, 2016.

IZQUIERDO-CAÑAS, P. M.; MENA-MORALES, A.; GARCÍA-ROMERO, E. Malolactic fermentation before or during wine aging in barrels. LWT - Food Science and Technology, v. 66, p. 468–474, 2016.

JACKSON, R. S. Wine science: Principles and application. Academic P ed. California: Elsevier, 2008.

JEZIORMY, D. L.; ORTEGA, A. C. Inovação e performance competitiva na indústria vitivinícola brasileira. Ensaios FEE, v. 33, p. 864–886, 2013.

JORDÃO, A. M. et al. Antioxidant Activity Evolution During Portuguese Red Wine Vinification And Their Relation with the Proanthocyanidin and Anthocyanin Composition. Journal of Food Processing and Preservation, v. 36, p. 298–309, 2012.

KALLAS, Z.; ESCOBAR, C.; GIL, J. M. Analysis of consumers' preferences for a special-occasion red wine: A dual response choice experiment approach. Food Quality and Preference, v. 30, n. 2, p. 156–168, 2013.

KELEBEK, H. et al. Influence of different maceration times on the anthocyanin composition of wines made from Vitis vinifera L. cvs. Bogazkere and Okuzgozu. Journal of food engineering, v. 77, p. 1012–1017, 2006.

KILMISTER, R. L. et al. A role for anthocyanin in determining wine tannin concentration in Shiraz. Food Chemistry, v. 152, p. 475–482, 2014.

KOK, D. A Review on Grape Growing in Tropical Regions. Türk Tarım ve Doğa Bilimleri, p. 1236–1241, 2014.

KOSSEVA;MARIA R.; JOSHI;, V. K.; PANESAR;, P. S. Science and Technology of Fruit Wine Production. San Diego: Academic Press Limited, 2017.

KOUSSISSI, E. et al. Influence of toasting of oak chips on red wine maturation from sensory and gas chromatographic headspace analysis. Food Chemistry, v. 114, n. 4, p. 1503–1509, 2009.

KOUSSISSI, E.; PATERSON, A.; CRISTOVAM, E. Sensory Discrimination of Dry Red Wines from Greece. Journal of Wine Research, v. 13, n. 2, p. 165–179, 2002.

KYRALEOU, M. et al. Differentiation of Wines Treated with Wood Chips Based on Their Phenolic Content, Volatile Composition, and Sensory Parameters. Journal of Food Science, v. 80, n. 12, p. C2701–C2710, 2015.

KYRALEOU, M. et al. Differentiation of Wines Treated with Wood Chips Based on Their KYRALEOU, M. et al. Effect of irrigation regime on perceived astringency and proanthocyanidin composition of skins and seeds of Vitis vinifera L. cv. Syrah grapes under semiarid conditions. Food Chemistry, v. 203, p. 292–300, 2016.

LAGO, L. O. et al. Influence of ripeness and maceration of the grapes on levels of furan and carbonyl compounds in wine – Simultaneous quantitative determination and assessment of the exposure risk to these compounds. Food Chemistry, v. 230, p. 594–603, 2017.

LATTEY, K. A.; BRAMLEY, B. R.; FRANCIS, I. L. Consumer acceptability, sensory properties and expert quality judgements of Australian Cabernet Sauvignon and Shiraz wines. Australian Journal of Grape and Wine Research, v. 16, n. 1, p. 189–202, 2010.

LE PARISIEN. LES VINS DU NOUVEAU MONDE, UNE MENACE POUR LES VINS FRANÇAIS ? Disponível em: <http://lesgrappes.leparisien.fr/les-vins-du-nouveau-monde-tendance/>. Acesso em: 12 dez. 2017.

LÊ, S.; JOSSE, J.; HUSSON, F. FactoMineR : An R Package for Multivariate Analysis. Journal of Statistical Software, v. 25, n. 1, 2008.

LEÃO, A. et al. Valores pessoais dos consumidores de vinhos do Vale do São Francisco (Brasil): Uma proposição tipológica de perfis. Revista Portuguesa e Brasileira de Gestão, p. 23–36, 2011.

LEÃO, P. C. DE S. Breve histórico da vitivinicultura e a sua evolução na região semiárida brasileira. Anais da Academia Pernambucana de Ciência Agronômica, v. 7, p. 81–85, 2010.

LEE, J.; DURST, R.; R E WROLSTAD. Determination of total monomeric anthocyanin pigment content of fruit juices, beverages, natural colorants, and wines by the pH differential method: Collaborative Study. Journal of AOAC International, v. 88, n. 5, p. 1269–1278, 2005.

LICK, E. et al. Sensory expectations generated by colours of red wine labels. Journal of Retailing and Consumer Services, v. 37, n. April 2016, p. 146–158, 2017.

LIMA, M. DOS S. et al. Influences of the Harvest Season on Analytical Characteristics of Syrah Grapes and Wines Produced in the Northeast Region of Brazil. International Journal of Agriculture and Forestry, v. 5, p. 151–159, 2015.

LIMA, M. DOS S. et al. Phenolic compounds , organic acids and antioxidant activity of grape juices produced in industrial scale by different processes of maceration. Food Chemistry, v. 188, p. 384–392, 2015.

LINGUA, M. S. et al. From grape to wine : Changes in phenolic composition and its influence on antioxidant activity. Food Chemistry, v. 208, p. 228–238, 2016.

LIU, S. et al. Effect of Oak Chips on Evolution of Phenolic Compounds and Color Attributes of Bog Bilberry Syrup Wine During Bottle-Aging. Jounal of Food Science, v. 81, n. 11, p. 2697–2707, 2016.

LLOBODANIN, L. G.; BARROSO, L. P.; CASTRO, I. A. Sensory characterization of young south american red wines classified by varietal and origin. Journal of Food Science, v. 79, n. 8, 2014.

LOCKSHIN, L.; CORSI, A. M. Consumer behavior for wine 2.0: A review since 2003 and future directions. Wine Economics and Policy, v. 1, p. 2–23, 2012.

LONA, A. A. Vinho – Degustação, Elaboração e Serviço. Porto Alegre: Age Ltda, 1996.

LUCENA, A. P. S. et al. Antioxidant activity and phenolics content of selected Brazilian wines. Journal of Food Composition and Analysis, v. 23, n. 1, p. 30–36, 2010.

MACFIE, H. J. H. Assessment of the sensory properties of food. Journal of Sensory Studies, v. 4, p. 129–148., 1989.

MACKNEIL, K. Como se prepara o vinho. In: A bíblia do vinho. Rio de Janeiro: Ediouro, 2003. p. 39–52.

MACNEIL, K. A Bíblia do Vinho. Rio de Janeiro: Editouro, 2003.

MARCHAL, A. et al. Contribution of oak lignans to wine taste: Chemical identification, sensory characterization and quantification. Tetrahedron, v. 71, n. 20, p. 3148–3156, 2015.
MEYNERS, M.; CASTURA, J. C. Novel Techniques sensory. In: GÁSTON ARES & PAULA VARELA (Ed.). . Novel Techniques in Sensory Characterization and Consumer Profiling. CRC Press ed. [s.l.] Taylor & Francis, 2014. p. 408.
MEYNERS, M.; CASTURA, J. C.; CARR, B. T. Existing and new approaches for the analysis of CATA data. Food Quality and Preference, v. 30, p. 309–319, 2013.

MINISTÉRIO DA AGRICULTURA. Saiba mais. Disponível em: <http://www.agricultura.gov.br/vegetal/culturas/uva/saiba-mais>. Acesso em: 20 abr. 2014.

MINOLTA. Precise color commmunication: Color control from feeding to instrumentantion. [s.l: s.n.].

MORENO, J.; PEINADO, R. Polyphenols. EUA: Elsevier, 2012.
MOSKOWITZ, H. R. Product Testing and Sensory Evaluation of Foods. Food & Nutrition Press, p. 605, 1983.

MOURET, M. et al. Social representations of wine and culture: A comparison between France and New Zealand. Food Quality and Preference, v. 30, n. 2, p. 102–107, 2013.

MULERO, J. et al. Antioxidant Activity and Phenolic Compounds in Organic Red Wine Using Different Winemaking Techniques. Journal of Food Science, v. 76, n. 3, p. 436–440, 2011.

NATIVIDADE, M. M. P. et al. Simultaneos analysis of 25 phenolic compounds in grape juice for HPLC: Method validation and characterization of São Francisco Valley samples. Microchemical Journal, v. 110, p. 665–674, 2013.

NAVARRO, M. et al. Oxygen consumption by oak chips in a model wine solution ; Influence of the botanical origin , toast level and ellagitannin content. Food Chemistry, v. 199, p. 822–827, 2016.

NIEDERLE, P. A.; VITROLLES, D. Indicações Geográficas e qualificação no setor vitivinícola brasileiro. Estudos Sociedade e Agricultura, v. 18, n. 1, p. 5–55, 2010.

NILE, S. H.; PARK, S. W. Edible berries: Bioactive components and their effect on human health. Nutrition, v. 30, n. 2, p. 134–144, 2014.

NOBLE, A. C. et al. Modification of a standardized system of wine aroma terminology. American Journal of Enology and Viticulture, v. 38, p. 143–146, 1987.

OBERHOLSTER, A. et al. Barrel maturation , oak alternatives and micro-oxygenation : Influence on red wine aging and quality. Food Chemistry, v. 173, p. 1250–1258, 2015.

OIV, O. I. DE LA V. ET DU V. State of the Vitiviniculture World Market. [s.l: s.n.].

OIV. Pieces of oak wood. [s.l: s.n.].

OLIVEIRA, W. P. DE et al. Phenolics from Winemaking By-Products Better Decrease VLDL-Cholesterol and Triacylglycerol Levels than Those of Red Wine in Wistar Rats. Journal od Food Science, 2017.

OU BX, CHANG T, H. D. AND P. R. Phenolic compounds, organic acids and antioxidant activity of grape juices produced from new Brazilian varieties planted in the Northeast Region of Brazi. J Aoac Int, v. 96, p. 1372–1376, 2013.

PADILHA, C. V. DA S. et al. Phenolic compounds profile and antioxidant activity of commercial tropical red wines (Vitis vinifera L.) from São Francisco Valley, Brazil. Journal of Food Biochemistry, v. 41, n. 3, p. 1–9, 2017.

PADILHA, C. V. DA S. et al. Phenolic compounds profile and antioxidant activity of commercial tropical red wines (Vitis vinifera L.) from São Francisco Valley, Brazil. Journal of Food Biochemistry, v. 41, n. 3, p. 1–9, 2017.

PAN-MONTOJO, J. Las vitiviniculturas europeas: de la primeira a la segunda glabalización. Mundo Agrario, v. 9, n. 18, 2009.

PARR, W. V. et al. Representation of complexity in wine: Influence of expertise. Food Quality and Preference, v. 22, n. 7, p. 647–660, 2011.

PEÑA-NEIRA, A. Grapes. In: YAHIA, E. M. (Ed.). . Fruit and Vegetable Phytochemicals. 2nd. ed. Chichester, UK: John Wiley & Sons, Ltd, 2017. v. II p. 1041–1054.

PEREIRA, G. E. Notas Técnicas. Disponível em: <www.vinhovasf.com.br/site/internas/valetecnico.php>. Acesso em: 10 abr. 2014.

PEREIRA, G. E. Os vinhos tropicais em desenvolvimento no Nordeste do Brasil. Com Ciência, n. 149, 2013.

PÉREZ-MAGARIÑO, S.; ORTEGA-HERAS, M.; GONZÁLEZ-SANJOSÉ, M. L. Wine consumption habits and consumer preferences between wines aged in barrels or with chips. Journal of the Science of Food and Agriculture, v. 91, n. 5, p. 943–949, 30 mar. 2011.

PICARD, M. et al. A sensory study of the ageing bouquet of red Bordeaux wines: A three-step approach for exploring a complex olfactory concept. Food Quality and Preference, v. 42, p. 110–122, 2015.

PIZARRO, C. et al. A sensory approach for the monitoring of accelerated red wine aging processes using multi-block methods. Food Quality and Preference, v. 28, n. 2, p. 519–530, 2013.

PRETI, R.; VIERI, S.; VINCI, G. Biogenic amine profiles and antioxidant properties of Italian red wines from different price categories. Journal of Food Composition and Analysis, v. 46, p. 7–14, 2016.

PREYS, S. et al. Relationship between polyphenolic composition and some sensory properties in red wines using multiway analyses. Analytica Chimica Acta, v. 563, n. 1–2, p. 126–136, mar. 2006.

PROTAS, J. F. S. Vitivinicultura brasileira: panorama setorial de 2010. Brasilia: [s.n.].

RAY, J. O vinho tinto. Barcelona: Folio, 2001.

RENFREW, J. M. Archaeology and the origins of wine production. In: MERTON SANDLER, R. P. (Ed.). . Wine. 1st Edition ed. London: CRC Press, 2003. p. 56–69.

RIBEREAU-GAYON, Y. G.; MAUJEAN, A.; DUBOURDIEU, D. The Chemistry of Wine Stabilization and Treatments. 2nd Editio ed. England: John Wiley & Sons, 2005. v. 2.

RIBEREAU-GAYON, J.; PEYNAUD, E. Y. O. Ciências e Técnicas del vino. [s.l.] Editorial Hemisfério Sur, 1980.

RIBÉREAU-GAYON, P. et al. Making Red Table Wines. In: Handbook of Enology. Chichester, UK: John Wiley & Sons, Ltd, 2006a. p. 327–395.

RIBEREAU-GAYON, P. et al. Phenolic Compounds. In: Handbook of Enology: The Chemistry of Wine and Stabilization and Treatments. [s.l: s.n.]. v. 2p. 141–203.

RIBÉREAU-GAYON, P. et al. Red Winemaking. In: Handbook of Enology. Chichester, UK: John Wiley & Sons, Ltd, 2006b. v. 1p. 327–395.

RINALDI, A. et al. Transfer of tannin characteristics from grape skins or seeds to wine-like solutions and their impact on potential astringency Alessandra. LWT - Food Science and Technology, v. 63, n. July, p. 667–676, 2015.

RODRÍGUEZ-BENCOMO, J. J.; POZO-BAYÓN, M. Á.; MORENO-ARRIBAS, M. V. Wine Fermentation and Production. In: HUI, Y. H.; EVRANUZ, E. Ö. (Eds.). . Handbook of Plant-Based Fermented Food and Beverage Technology. [s.l.] CRC Press, 2012. p. 179–200.

ROSA, S. E. S.; SIMÕES, P. M. Desafios da vitivinicultura brasileira. BNDS Setorial, n. 19, p. 67–90, 2004.

ROSSI, J. A.; SINGLETON, V. L. Colorimetry of total phenolics winth phosphomolybdic phosphotungstic acid reagents. American Journal of Enology and Viticulture, v. 16, p. 144–158, 1965.

RUFINO, M. DO S. M. et al. Bioactive compounds and antioxidant capacities of 18 non-traditional tropical fruits from Brazil. Food Chemistry, v. 12, p. 996–1002, 2010.

SÁ, N. C. DE; SILVA, E. M. S.; BANDEIRA, A. DA S. A cultura da uva e do vinho no Vale do São Francisco. Revista de Desenvolvimento Econômico, p. 461–491, 2015.

SAMOTICHA, J.; WOJDYLO, A.; GOLIS, T. Phenolic composition, physicochemical properties and antioxidant activity of interspecific hybrids of grapes growing in Poland. Food Chemistry, v. 215, p. 263–273, 2017.

SÁNCHEZ-PALOMO, E. et al. Improvement of Verdejo white wines by contact with oak chips at different winemaking stages. LWT - Food Science and Technology, v. 79, p. 111–118, 2017.
SANTOS, B. A. DOS et al. Check all that apply and free listing to describe the sensory characteristics of low sodium dry fermented sausages : Comparison with trained panel. Food Research International, v. 76, p. 725–734, 2015.

SARTOR, S. et al. Particularities of Syrah wines from different growing regions of Southern Brazil: grapevine phenology and bioactive compounds. Journal of Food Science and Technology, v. 54, n. 6, p. 1414–1424, 2017.

SCHUMACHER, R. et al. Evaluation of Oak Chips Treatment on Volatile Composition and Sensory Characteristics of Merlot Wine. Jounal of Food Quality, v. 36, p. 1–9, 2013.

SETFORD, P. C. et al. Factors affecting extraction and evolution of phenolic compounds during red wine maceration and the role of process modellingTrends in Food Science and Technology. Elsevier Ltd, , 2017. Disponível em: <https://doi.org/10.1016/j.tifs.2017.09.005>.

SHERMAN, E. et al. Impact of Grape Maturity and Ethanol Concentration on Sensory Properties of Washington State Merlot Wines. American Journal of Enology and Viticulture, p. ajev.2017.16076, 2017.

SHI, P. B. et al. Phenolic compound profiles in grape skins of Cabernet Sauvignon, Merlot, Syrah and Marselan Cultivated in the Shacheng Area (China). South African Journal of Enology and Viticulture, v. 37, n. 2, p. 132–138, 2016.

SILLANI, S.; MICCOLI, A.; NASSIVERA, F. Different preferences for wine communication. Wine Economics and Policy, v. 6, n. 1, p. 28–39, 2017.

SILVA, P. C. G. DA; COELHO, R. C. A viticultura no Submédio do Vale São Francisco: Importância econômica e social da videira. Disponível em: <http://www.cpatsa.embrapa.br:8080/sistema_producao/spuva/Caracterizaca_social_da_videira.html>. Acesso em: 21 nov. 2017.

SMITH, P. A.; MCRAE, J. M.; BINDON, K. A. Impact of winemaking practices on the concentration and composition of tannins in red wine. Australian Journal of Grape and Wine Research, v. 21, p. 601–614, 2015.

SOUZA, M. L. V. Rio São Francisco. Disponível em: <geografiaeletrica.blogspot.com.br/2011/10/rio-sao-francisco.html>. Acesso em: 1 jan. 2014.
STONE, H.; SIDEL, J. L. Sensory evaluation practices. 3 ed. ed. California: Elsevier, 2004.

SUGANTHYA, N. et al. Bioactive effects of quercetin in the central nervous system: Focusing on the mechanisms of actions. Biomedicine et Pharmacotherapy, v. 84, p. 892–908, 2016.

SUN, B. et al. Stilbenes: Quantitative extraction from grape skins, contribution of grape solids to wine and variation during wine maturation. Analytica Chimica Acta, v. 563, n. 1–2 SPEC. ISS., p. 382–390, 2006.

SWAIN, T.; HILLS, W. E. The phenolic constituents of Prunus domestica I. - The quantitative analysis of phenolic constituents. Journal of the Science of Agriculture, v. 10, n. 1, p. 63–68, 1959.

TAO, Y.; GARCÍA, J. F.; SUN, D. Advances in Wine Aging Technologies for Enhancing Wine Quality and Accelerating Wine Aging Process. Critical Reviews in Food Science and Nutrition, v. 54, p. 817–835, 2014.

TEAM, R. C. R: A language and environment for statistical computing.ViennaR Foundation for Statistical Computing, , 2017. Disponível em: <https://www.r-project.org/>.

THE AUSTRALIAN WINE RESEARCH INSTITUTE. Progress in wine technology slowed down. Disponível em: <http://www.winehistory.com.au/wiki/Wine_History/Progress_in_wine_technology_slowed_down>. Acesso em: 16 nov. 2017.

TONIETTO, J.; TEIXEIRA, A. H. DE C. O clima vitícola do submédio são francisco e o zoneamento dos períodos de produção de uvas para elaboração de vinhos. I Workshop Internacional de Pesquisa. 2007.

TORRI, L.; NOBLE, A. C.; HEYMANN, H. Exploring American and Italian consumer preferences for Californian and Italian red wines. Journal of the Science of Food and Agriculture, v. 93, n. 8, p. 1852–1857, 2013.

TOUNSI, M. S. et al. Valorization of three varieties of grape. Industrial Crops and Products, v. 30, n. 2, p. 292–296, 2009.

TOUSSAINT-SAMAT, M. From the Vine to Wine. In: A History of Food. [s.l: s.n.]. p. 223–262, 2009.

TSANOVA-SAVOVA, S.; RIBAROVA, F. Free and Conjugated Myricetin , Quercetin , and Kaempferol in Bulgarian Red Wines. Jounal of food composition and analysis, v. 15, p. 639–645, 2002.

VALENTIN, D. et al. Quick and dirty but still pretty good: a review of new descriptive methods in food science. International Journal of Food Science & Technology, v. 47, n. 8, p. 1563–1578, ago. 2012.

VECCHIO, R. et al. European consumers' perception of moderate wine consumption on health. Wine Economics and Policy, v. 6, n. 1, p. 14–22, 2017.

VIDAL, L. et al. How do consumers describe wine astringency ? Food Research International, v. 78, p. 321–326, 2015.

VIEIRA, A. C. P.; WATANABE, M.; BRUCH, K. L. Perspectivas De Desenvolvimento da Vitivinicultura em Face Do Reconhecimento da Indicação de Procedência Vales da Uva Goethe. Revista Gestão, Inovação e Tecnologia, v. 2, n. 4, p. 327–343, 5 out. 2012.

VILLAÑO, D. et al. Food Chemistry Influence of enological practices on the antioxidant activity of wines. Food Chemistry, v. 95, p. 394–404, 2006.

VILLANUEVA, N. D. M.; DA SILVA, M. A. A. P. Comparative performance of the nine-point hedonic, hybrid and self-adjusting scales in the generation of internal preference maps. Food Quality and Preference, v. 20, n. 1, p. 1–12, 2009.

VITAL, T. Vitivinicultura no Nordeste Brasileiro: Situação Recente e Perspectivas. Revista Econômica do Nordeste, v. 40, n. 3, p. 500–524, 2009.

WATERHOUSE, A. L. The Phenolic wine antioxidants. In: CADENAS, E.; PACKER, L. (Eds.). . Handbook of antioxidants. New York: Marcel Dekker, 2005.

WATERHOUSE, A. L.; SACKS, G. L.; JEFFERY, D. W. Maceration and Extraction of Grape components. In: Understanding Wine Chemistry. England: John Wiley & Sons, 2016. p. 179–193.

WUCHERPFENNIG, K. WINES | Production of Table Wines. In: Encyclopedia of Food Sciences and Nutrition. [s.l.] Elsevier, 2003. p. 6195–6203.

WYLER, P. et al. Effect of oak wood on the quality of beer. Journal of the Institute of Brewing, v. 121, n. 1, p. 62–69, 2015.

YOO, Y. J. et al. A cross-cultural study of wine consumers with respect to health benefits of wine. Food Quality and Preference, v. 28, n. 2, p. 531–538, 2013.

YOO, Y. J. et al. Assessment of Some Australian Red Wines for Price , Phenolic Content , Antioxidant Activity , and Vintage in Relation to Functional Food Prospects. Jounal of Food Science, v. 76, n. 9, p. 1355–1364, 2011.

YOO, Y. J.; SALIBA, A. J.; PRENZLER, P. D. Should red wine be considered a functional food? Comprehensive Reviews in Food Science and Food Safety, v. 9, n. 5, p. 530–551, 2010.

ZANINI, T. V.; ROCHA, J. M. O enoturismo no Brasil: um estudo comparativo entre as regiões vinícolas do Vale dos Vinhedos (RS) e do Vale do São Francisco (BA/PE). Turismo em Análise, v. 21, n. 1, p. 68–88, 2010.

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ANEXO 2

FACULDADE DE CIENCIAS MEDICAS - UNICAMP (CAMPUS CAMPINAS)

PARECER CONSUBSTANCIADO DO CEP

DADOS DO PROJETO DE PESQUISA

Título da Pesquisa: VINHO SYRAH DO VALE DO SÃO FRANCISCO: CARACTERIZAÇÃO QUÍMICA, SENSORIAL E ESTUDO DE CONSUMIDOR

Pesquisador: Natália Manzatti Machado Alencar

Área Temática:

Versão: 2

CAAE: 34586014.9.0000.5404

Instituição Proponente: Faculdade de Engenharia de Alimentos

Patrocinador Principal: MINISTERIO DA EDUCACAO

DADOS DO PARECER

Número do Parecer: 822.379

Data da Relatoria: 02/10/2014

Apresentação do Projeto:

Esse projeto prevê a caraterização do perfil sensorial, através de Análise Descritiva Quantitativa do produto "Vinho Syrah do Submédio Vale do São Francisco". Serão também feitos testes de aceitação ('análise sensorial') com consumidores e sobre parâmetros que influenciariam a intenção de compra de consumidores.

Objetivo da Pesquisa:

São escassos os estudos sobre análises sensorial em vinho tinto da Vitis Vinifera Syrah produzidos na região do Vale do São Francisco, no qual hoje em dia, é considerado o segundo maior produtor de vinho do Brasil.

Novas técnicas vêm sendo aplicadas com objetivo de melhorar as características químicas e sensoriais de vinhos. Duas técnicas bem sucedidas na vinicultura, aplicadas com o intuito de reduzir o tempo do envelhecimento do vinhos tintos em barris de carvalho, são 1) a micro-oxigenação e 2) o uso de 'chips' de carvalho.

A micro-oxigenação é uma técnica que consiste na utilização de um equipamento especifico capaz de regular as doses de oxigênio aplicadas no barril. A técnica atua principalmente na intensificação e estabilização da cor de modo semelhante à envelhecimento de barril, além de

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melhorar o sabor e aroma do vinho.
Já a utilização de fragmentos de madeira, como chip de carvalho, é um método alternativo para envelhecimento de vinho. A utilização dessa técnica possibilita, em curto prazo, que os vinhos desenvolvam aromas típicos da madeira advindos de compostos fenólicos e cores semelhantes à de vinhos maturados em barril de carvalho.

O estudo contribuirá para caracterizar sensorialmente e quimicamente vinhos da uva Syrah produzido no Vale do São Francisco e verificar a aceitação das novas técnicas empregadas.

Avaliação dos Riscos e Benefícios:
Riscos:
Não deverão participar dos testes pessoas que: 1) tenham qualquer tipo de restrição ao consumo de bebidas alcoólicas, incluindo alcoolismo, problemas gástricos, uso de medicamentos que interajam com álcool ou ainda apresentam alergia a sulfito, pois esse é adicionado ao vinho para sua preservação.
Benefícios:
O Estudo não prevê benefícios diretos decorrente da sua participação na pesquisa, porém a participação dos voluntário auxiliará um projeto de pesquisa que visa o desenvolvimento de vinhos tintos da uva Syrah na região do Submédio São Francisco e esse novo produto poderá beneficiar a economia da região.

Comentários e Considerações sobre a Pesquisa:
Foi providenciada a autorização, por parte da direção da Faculdade de Engenharia de Alimentos, para a realização da pesquisa em suas depedências - ok

No item 'orçamento', foi esclarecido também que a ADQ será realizada sem pagamento pelos serviços da equipe de degustadores treinados da Embrapa Semi-Árido - ok

Considerações sobre os Termos de apresentação obrigatória:
Os termos foram readequados.

Recomendações:
No campo 'benefícios', do TCLE destinado aos testes de aceitação, lê-se 'No entanto a sua, participação dos voluntário auxiliará um projeto de pesquisa ...". Por favor, corrigir o parágrafo

Conclusões ou Pendências e Lista de Inadequações:
(nenhuma)

Situação do Parecer:
Aprovado

Necessita Apreciação da CONEP:
Não

Considerações Finais a critério do CEP:
- O sujeito de pesquisa deve receber uma cópia do Termo de Consentimento Livre e Esclarecido, na íntegra, por ele assinado.

- O sujeito da pesquisa tem a liberdade de recusar-se a participar ou de retirar seu consentimento em qualquer fase da pesquisa, sem penalização alguma e sem prejuízo ao seu cuidado.

- O pesquisador deve desenvolver a pesquisa conforme delineada no protocolo aprovado. Se o pesquisador considerar a descontinuação do estudo, esta deve ser justificada e somente ser realizada após análise das razões da descontinuidade pelo CEP que o aprovou. O pesquisador deve aguardar o parecer do CEP quanto à descontinuação, exceto quando perceber risco ou dano não previsto ao sujeito participante ou quando constatar a superioridade de uma estratégia diagnóstica ou terapêutica oferecida a um dos grupos da pesquisa, isto é, somente em caso de necessidade de ação imediata com intuito de proteger os participantes.

- O CEP deve ser informado de todos os efeitos adversos ou fatos relevantes que alterem o curso normal do estudo. É papel do pesquisador assegurar medidas imediatas adequadas frente a evento adverso grave ocorrido (mesmo que tenha sido em outro centro) e enviar notificação ao CEP e à Agência Nacional de Vigilância Sanitária – ANVISA – junto com seu posicionamento.

- Eventuais modificações ou emendas ao protocolo devem ser apresentadas ao CEP de forma clara e sucinta, identificando a parte do protocolo a ser modificada e suas justificativas. Em caso de projetos do Grupo I ou II apresentados anteriormente à ANVISA, o pesquisador ou patrocinador deve enviá-las também à mesma, junto com o parecer aprovatório do CEP, para serem juntadas ao protocolo inicial.

- Relatórios parciais e final devem ser apresentados ao CEP, inicialmente seis meses após a data deste parecer de aprovação e ao término do estudo.

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Continuação do Parecer: 822.379

CAMPINAS, 07 de Outubro de 2014

Assinado por:
Renata Maria dos Santos Celeghini
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ANEXO 3

Table S1: Quality oenological parameters (total polyphenol index, pH, total acidity, volatile acidity, alcoholic content, free and total sulfur dioxide, reducing sugars) of Syrah aged wines.

Samples	Total polyphenol index	pH	Total acidity (tartaric acid g/L)	Volatile acidity (acetic acid g/L)	Alcoholic content (% v/v)	Free sulfur dioxide (g)	Total sulfur dioxide (g)	Reducing sugars (g)
WC	67.23±0.15	4.04±0.01	6.50± 0.09	0.56± 0.07	13.43± 0.07	34.30±0.51	74.24±0.51	1.42 ±0.57
WAAMF	55.6±0.17	4.05±0.01	6.60± 0.00	0.76± 0.02	12.61± 0.06	33.28±0.51	66.22±0.59	1.66±0.6
WAMF	61.36±0.06	4.01±0.01	6.65± 0.17	0.74[a]± 0.01	12.63±0.23	30.55±0.30	88.41±0.59	1.59±0.98
WFAMF	58.40 ±0.20	4.06± 0.02	6.50± 0.09	0.80± 0.04	12.90±0.52	28.50±0.59	70.48±0.59	1.92±0.46
WFMF	61.40±0.30	4.03± 0.01	6.65±0.17	0.58± 0.11	13.35±0.19	31.48±0.26	62.63±0.59	2.00±0.76
WAFAMF	58.40±0.30	4.02± 0.02	6.95± 0.09	0.68± 0.02	13.15±0.15	30.80± 0.30	80.90±0.51	2.02±0.32

ANEXO 4

Table S2 – Validation parameters of chromatographic identification and quantification of phenolic compounds performed by HPLC-DAD-FD

Validation Parameters	
Linearity	0.625 – 15.00 mg/mL
Equations of regression coefficients (R^2)	0.9838- 0.9999
Limits of detection	0.001 - 0.190 µg/mL
Limits of quantification (LOQ)	0.003 -0.370 µg/mL
Mean recovery value	anthocyanins - 98.27- 102.01 %
	flavonols - 86.18–106.50 %
	phenolic acids - 83.97–100.93 %
	tannins - 86.86–97.10 %
RSD_r (precision of the method)	0.73 - 2.87 % - unspiked samples
	0.71 to 9.24 % - spiked samples
RSD_r	1.99 - 6.46 % - unspiked samples
	1.34 - 9.26 % for spiked samples

ANEXO 5

Submission Confirmation

1 mensagem

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Dear Miss Natalia Manzatti Machado Alencar,

Your submission entitled "SYRAH WINE FROM THE SÃO FRANCISCO VALLEY, BRAZIL: SENSORY CHARACTERIZATION AND CONSUMERS' PERCEPTION" of SI: SLACA 2017 has been received by Food Research International

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Thank you for submitting your work to this journal.

Kind regards,

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ANEXO 6

Table S2- Descriptors used for sensory profiling of Syrah aged wine.

Descriptors	*Definition*	*Reference*
Wine Color	Pigment content of the sample	*Weak:* Munsell Book: 5 R 8/6. / *Strong:* Munsell Book: 5 R P 3/2.
Brightness	Light reflection that could be more or less intense.	*Weak:* 100 ml of Syrah wine in the alcoholic fermentation. *Strong:* 200 ml of Syrah wine (control sample with 13% of alcohol content) added with 10g of grape jelly (Royal®) diluted in 200 ml of water.
Aromatic intensity	Concentration of volatiles compounds in the sample (An example in a red wine could be esters, aldehydes, higher alcohols; the effect is captured by olfactory organ of taster. Excluded of this concept undesirable volatiles compounds result of raw- material problems or technologies.	*Weak:* Diluted in water Winemarker's Selection wine (Rio Sol®, Lagoa Grande, PE) 1:5./*Strong:*100 ml of Winemarker's Selection wine (Rio Sol®, Lagoa Grande, PE).
Alcoholic aroma	Characteristic aroma ethanol.	*Weak:* Solution 11.5% of alcohol in distilled water. *Strong:* Solution with 14.5% of alcohol in water distilled.
Coffee aroma	Coffee-like aroma	*None***:** Distilled water. *Strong:* 10 mL of wine Syrah (control sample with 13% alcohol content) added of 5 g soluble coffee (Nescafé®).
Woody aroma	aroma that resembles ok	*None***:** Distilled water. *Strong:* 100ml of distilled wine soaked in 5 g of American oak chip.
Sweet/caramelized Aroma	Sweet aroma which ressembles caramel, molasses, honey or chocolate in the wine.	*None***:** Distilled water. *Strong:* 50 ml of Syrah wine (sample control with 13% alcohol content) added 10g of honey.
Vegetative aroma	Characteristic of cooked green beans.	*None***:** Distilled water. *Strong:* 100ml of Syrah (sample control with 13% alcohol content) added 10g of cooked green beans.
Spicy aroma	Characteristic of black pepper.	*Weak:* 100 ml of Syrah wine (sample control with 13% alcohol content) added 1g of black pepper grains. *Strong:* 100 ml of Syrah wine (sample control with 13% alcohol content) added 4g of black pepper grains.

Table S2 - continuation

Taste persistence	It should be evaluated immediately after the wine has passed into the mouth, when the taster swallows a small portion of the sample, rejects the rest, and exhales through the mouth and nose simultaneously. It is the time when the retronasal sensation (is felt until it dissipates completely. It lasts few seconds and can range from simple to fivefold.	*Weak:*Wine dilution of Winemarker's Selection, Touriga Nacional grape (Rio Sol®, Lagoa Grande- PE) 1:5. *Strong:* 100ml of Winemarker's Selection wine, Touriga Nacional grape (Rio Sol®, Lagoa Grande- PE).
Sweetness	Sweet taste characteristic of sucrose solution	*None:* Distilled water. Strong: 0.8%.Sucrose (União®) solution
Bitterness	Bitter taste characteristic of caffeine solution.	*Weak:* Caffeine solution 0.06%. *Strong*: Caffeine solution 0.1%.
Sourness	The sour taste associated with tartaric, malic, lactic and / or citric acids.	*Weak:* 0.05%. Tartaric acid solution *Strong:* 0.10%.Tartaric acid solution
Alcoholic mouthfeel	Taste characteristic of alcoholic beverage, which causes warmth due to ethanol.	*Weak:* Diluted of 1:1 of Syrah wine (control treatment with 13% alcohol content) in water. *Strong:* wine distillate with 36%of alcohol content brand Imperial (Miolo Wine Group- Casa Nova -BA).
Woody	Characteristic flavor of oak-aged beverage	*None:* Distilled water. *Strong:* wine distillate with 36% of alcohol content brand Imperial (Miolo Wine Group- Casa Nova -BA).
Astringency	Oral sensation of "dryness" in the mouth.	*Weak:* 0.1%. Tannic acid solution with *Strong:* 0.3%.Tannic acid solution with
Full-bodied	Sensation of volume in the mouth.	*Weak:* Red wine Syrah (control treatment with 13% alcohol content) diluted in mineral water in proportion 1:2. *Strong:* Creamy chocolate liqueur Splendor, Bento Gonsalves- RS.