

International Journal of Food Engineering

Volume 4, Issue 4

2008

Article 7

Composition Analysis of Litchi Juice and Litchi Wine

Xin An Zeng*
Frank G.F. Qin[‡]

Xiao Dong Chen[†]
Lu Zhang**

*South China University of Technology, xazeng@scut.edu.cn

[†]Monash University, dong.chen@eng.monash.edu.au

[‡]The University of Auckland, f.qin@uniservices.auckland.ac.nz

**The University of Auckland, bluehope1999@yahoo.com.cn

Composition Analysis of Litchi Juice and Litchi Wine*

Xin An Zeng, Xiao Dong Chen, Frank G.F. Qin, and Lu Zhang

Abstract

In this study the total components of the litchi juice and litchi wine in the brewing process were analyzed with both a low temperature (13-16 °C) and a long time (8-10 days) for fermentation. 161.4 g/l total sugar, 2.2 g/l to With today's advancement in nanotechnology, Polymer-Clay Nanocomposite has emerged as a novel food packaging material. Content of Vitamin C (354.12 mg/l) and K (1251.8 mg/l) were found in litchi extract (25.6 g/l), low volatile acid (0.36 g/l) and low higher alcohols (0.36 g/l) in the young litchi wine. The total free amino acid content in litchi wine (700-1300 mg/l). However the content of each compound in litchi wine was all at an average level (less than 50 mg/l). In total, 33 compounds were identified in litchi wine by liquid-liquid extraction and GC/MS. Esters, 5 alcohols, 4 acids, 2 ketones, 1 phenol and 4 other chemicals. Ethyl decylate (15.41%), acetic ester (11.36%), ethyl hexoate (7.14%), acetic isopentyl ester (4.72%) and citronel ethyl acetate (1.94%) were the main constituents, which covered 75.39% and 93.03% of the total compounds and ester compounds, respectively. Content of higher alcohols was only 7.85%. A very interesting phenomenon was found in that no matter how much sulfurous acid was added and how much total SO₂ content was observed in litchi juice or wine, the measured free state SO₂ content was always below 10 ppm, which was quite different from that of grape wine.

KEYWORDS: litchi, litchi wine, free state SO₂, aroma, GC/MS analysis

*The authors are grateful to Master Yue Qiang, Mr. Chen Yong and the first author's former colleagues at Kingsrich Winery Co. LTD, Guangdong Province, P. R.China, who conducted a lot of apparatus analysis. Authors are also grateful to the sensory group members who contributed with their time, skills and experiences. This research was supported by the Key Technologies R&D Program co-bided by the Guangdong Province (P. R. China) and the Hongkong Government (20054983207). Xin An Zeng, College of Light Chemistry and Food Science, South China University of Technology, Guangzhou 510640, Guangdong, China; tel.: 0086-2087113668; fax: 0086-2087112894. E-mail address: xazeng@scut.edu.cn. Xiao Dong Chen, Department of Chemical Engineering, Monash University, Building 36, Clayton Campus, VIC 3800, Australia. Frank G. F. Qin and Lu Zhang, Dept. of Chemical and Materials Engineering, The University of Auckland, Private Bag 92019, Auckland City, New Zealand.

1. INTRODUCTION

Litchi (*Litchi chinensis* Sonn.) is a nourishing and flavourful fruit that grows in tropical and subtropical areas. The major planting areas all over the world are China, India, Thailand, Vietnam, South Africa, Australia, USA and the Malagasy Republic, etc. (Menzel et al., 1988; Phunchaisri and Apichartsrangkoon, 2005). At present the total litchi planting area in China is up to 600,000 hectares, and yields about 1.4 million tons per year, covering over almost 70% of the world's total output. Guangdong, Guangxi, Fujian, Hainan and Taiwan are the main produce provinces in China. (Chen and Ni, 2006). But up to now, scientific research about litchi is only limited to a very narrow aspect for the reason of its restricted planting areas. Most of the research carried out on litchi focuses on planting techniques and fruit preservation methods to reduce postharvest pericarp browning and pulp decaying, etc. (Paull et al., 1998; Jiang et al., 2000, 2004; Dong et al., 2004; Olesen et al., 2004; Wu and Chen, 1999).

In the field of composition analysis about litchi juice and its products, Cheng FL et al. (2002) determined multi-trace elements such as Al, Ba, Cr, Mg, Mn, Sr, Mo, Pb, Se, Zn; Fe, Cu and Ca simultaneously in different parts and different growth periods of litchi fruit. Sarni-Manchado P et al. (2000) determined and quantified the polyphenolic composition of litchi pericarp by low-pressure chromatography, high-pressure liquid chromatography (HPLC), W-visible spectrum analysis, mass spectrometry (MS), and nuclear magnetic resonance. Gontier E et al. (2000) analyzed the variability in the relative levels of unusual fatty acids in the seeds of 28 different litchi varieties.

Analysis of aroma or volatile compounds is one of the most important steps in the evaluation of fruit juice or wine quality. Usually, the concentration of the volatile compounds in juice or fruit wine aroma are quite low (varying from $\mu\text{g/l}$ to mg/l), so it should be enriched before identification and quantification. Liquid-liquid extraction, simultaneous distillation/extraction, dynamic and static headspace sampling methods and solid phase micro extraction followed by gas chromatography and mass spectrometry (GC/MS) were widely used in all kinds of aroma compounds analysis in the recent years. (Zalacain et al., 2006; Comuzzo et al., 2006; Gómez-Míguez et al., 2007; Zea et al., 2001; Perestrelo et al., 2006; Selli et al., 2006; Bonino et al., 2003; Alves et al., 2005; Gil et al., 2006). Ong and Acree (1998) extracted volatile compounds from litchi juice using both Freon 113 and ethyl acetate solvents. Ong and Acree (1999) also detected the odor potent compounds in the canned litchi by GC/O analysis, found cis-rose oxide, beta-damascenone, linalool, furaneol, ethyl isobutyrate, (E)-2-nonenal, ethyl isohexanoate, geraniol, and delta-decalactone were determined to be the main contributors of canned litchi aroma. Chyau et al. (2003) isolated and separated free and glycosidically-bound volatile compounds from fresh clear litchi juice using an

Amberlite XAD-2 column. The volatile compounds from the bound fraction were released by hydrolysis with almond β -glucosidase. Volatile components of both free and bound fractions were then determined by GC and GC-MS. In total, 25 compounds were identified in both fractions, including one ester, 14 alcohols, two aldehydes, four acids, two ketones and two terpenes. Zhang et al. (2004) extracted, purified and identified litchi fruit peel anthocyanins by HPLC equipped with mass spectrometry.

The current post harvest processes of litchi are producing juice, concentrated juice, cans and dry fruit, etc. Grape wine is the most popular fruit wine in the world and has a history over thousands of years. Litchi juice just like grape juice, have large amounts of, and quite equilibrium, fermentable sugar and acid contents. For thoroughly matured litchi, its sugar content can reach up to 160 g/l and the organic acid is usually around 2-3 g/l. This is quite suitable for making wine. But there is very little literature that mentions making wine by litchi, and even fewer people who have analyzed the compositions of litchi wine. The main purpose of this work aims to develop a new process of making litchi wine and to determine its composition, detail data involved in the general composition, amino acids of both litchi juice and wine, aroma compounds of litchi wine, etc.

2. MATERIALS AND METHODS

2.1 MATERIALS

Mature litchi is a spherical fruit with a red color shell, white color meat and a dark brown hard core. The sample used in this research was *Litchi chinensis* Sonn. cv. Wuye, a major cultivar with bright red color skin and large size, which was purchased from a planting field in Huilai, Guangdong province, China. Fresh litchi fruits were peeled and extracted 24 hours after being harvested to avoid decaying. The juice was cooled to below 10 °C in 20 minutes by a heat-exchanger. 40 ppm liquid composite pectinase (Novozymes) for clarification and 60 ppm SO₂ were added for preservation.(O.I.V. 1990). 10~15 hours later, the upper clear juice was separated for the purpose of analysis and subsequent fermentation. Three 200 liters capacity stainless tanks with jacket cooling were used and 150 liters clear litchi juice was added into each tank for fermentation.

Some sulfurous acid was added to the clear juice to maintain the total SO₂ content at 100ppm. 3.0 g/l tartaric acid was added to make the total organic acid content reach 5.0 g/l and the pH lower than 3.8 to avoid microorganisms other than yeast growing. 150 ppm activated industrial yeast (R2, Mauri Co., Australia) was added to start the fermentation. According to Wondra and Berovic. (2001), when the fermentation temperature is between 10 to 15 °C, most of the main aromatic compounds that endow the products with wine character can be preserved. In this

research, the fermentation temperature was kept at 13-16 °C and the liquid was stirred twice a day by a pump during fermenting. The circulating ratio was 30% of the whole fluid. 3-4 days later, when the specific gravity of the fluid reached 1.040, 40 g/l sugar was added to increase the alcohol content. Finally, 8-10 days later when the specific gravity of the fluid reached 0.997, the fermentation process was over and the raw wine was obtained by separating the clear liquid with the sediment. About 2 months later when the wine was stabilized and clarified via a natural process, it was then filtered. The clear wine sample was ready for sensory evaluation and chemical analysis.

2.2 ANALYSIS METHODS

The general constituents of the litchi juice and wine were analyzed with routine methods carried out in the winery. The analysis method of each composition was shown in **Table 1**. Liquid-liquid extraction and GC-MS were also used to analyze the trace aroma compounds in litchi wine. Before entering the chromatography column, the samples were prepared using the following procedures: 350 ml wine was put in a 1 L volume separating funnel, 100 ml analytically pure methylene chloride was added and the separating funnel was shaken intensively for 3-4 min. 3-4 hours later when the liquid become clearly stratified, the lower part which was organic phase was released to a 1 L triangular flask. 60 ml methylene chloride was added to the remaining water phase part to extract for a second time following the same procedures as above. The water phase was extracted once more again by adding another 60 ml methylene chloride. Subsequently, the organic phases of the three times extracted sample were collected and concentrated to about 5 ml using a rotational-vacuum-evaporator at room temperature. This was the sample ready to be tested by GC/MS. (Selli et al., 2006; Perestrelo et al., 2006).

A Hewlett-Packard 6890 Series II gas chromatograph (Hewlett-Packard Co.) equipped with a fused silica capillary column (HP-INNOWAX 30 m×0.25 mm×0.17 µm) and a flame ionization detector (FID) was used to analyze the extracted aroma compounds in wine using the following operating conditions: injection temperature, 250 °C; detector temperature, 280 °C; nitrogen carrier flow rate, 1.2 ml/min; temperature programme, 80–240 °C at 6 °C /min and held at 240 °C for 30 min. A split ratio of 80:1 was used.

GC-MS analysis was carried out by using a Hewlett-Packard 6890 Series II gas chromatograph coupled directly to a Hewlett-Packard 5972A MSD mass spectrometer. An identical column and temperature programmes were used as the above-mentioned GC analysis. The temperature of transfer line was 265 °C. The helium carrier flow rate was 1.0 ml/min. Splitless injections were employed with a purge delay time of 1 min and the mass spectra were obtained with electron multiplier voltage and electron ionization energy of 1353 V and 70 eV,

respectively. The amount of each compound was determined by each peak area in the gas chromatogram.

Table 1. Methods for quantitative analysis of compositions in litchi juice and wine

Composition	Analysis method	Ref.	Composition	Analysis method	Ref.
Protein	Kjeldahl's	1	Vitamins A	HPLC	2,3,4
Total sugar	Fehling's Titration	5,6	Vitamin C	HPLC	2,3,4
Reductive sugar	Fehling's Titration	5,6	Vitamin B1	HPLC	2,3,4
Total acid	Potential titration	5	Vitamin B2	HPLC	2,3,4
Organic acid	HPLC	5	Total ester	O.I.V.	5,8
Lipid	Soxhlet Extraction	5	Dry extract	O.I.V.	8
Glucose,fructose	HPLC	5,6	Alcohol	O.I.V.	8
Amino acids	HPLC	1,5	Volatile acid	Titration	8
Total aldehyde	O.I.V.	5,8	Total SO ₂	Titration	8
Niacin	HPLC	2,3	Free SO ₂	Titration	8
β-carotene	Paper chromatography	4,5	Total ester	O.I.V.	8
Phosphorus	Spectrophotometry	5,7	Higher alcohol	O.I.V.	8
Methyl alcohol	O.I.V.	8	Total polyphenol	O.I.V.	8
Mineral elements	Atomic absorption spectrophotometry	5,7	Selenium	Fluorescence method	5,7

Ref: [1] Ötleş S. 2005; [2] Eitenmiller and Landen 1998; [3] Vanderslice and Higgs 1984; [4] Bates 2000; [5] A.O.A.C. 1995; [6] Cui 2005; [7] John K. 2003; [8] O.I.V. 1990.

2.3 SENSORY EVALUATION

Litchi wines were evaluated using triangle and preference tests by 12 qualified and experienced wine sensory tasters (Amerine and Roessler 1976; Roessler et al. 1978). The extermination report stated that the litchi wine came with clean, light amber color, a beautiful and plummy aroma of natural litchi fruit and a harmonious wine taste.

3. RESULTS AND DISCUSSION

3.1 GENERAL COMPOSITION OF LITCHI JUICE

The main compositions of clear litchi juice were quantified by routine methods and the results are shown in **Table.2**. Total sugar and acid contents are two main factors that should be taken into account before litchi juice was chosen to make wine. In this research there were 161.4 g total sugar and 2.2 g total acids in 1 liter litchi juice. Theoretically, it can produce 9 % (v/v) alcohol content according to the rule that 1 % (v/v) alcohol was produced per ~17 g reductive sugar. Usually the alcohol content of fruit wine is around 11 % (v/v) for its better taste, so 40 g/l sugar was added to increase the alcohol content from 9% to 11% in this experiment in order to simulate other kinds of fruit wine. The content of Vitamin C in litchi juice is up to 354.12 mg/l which is 8 times that of apple juice (40 mg/l) and 5 times that of peach (70 mg/l) (Yang et al. 2002). The minerals contents in litchi juice are shown in **Table. 3**, and much higher content of K (1251.8 mg/l) than in other fruits was found (Yang et al 2002). In contrast to those data obtained by Cheng et. al. (2002), there exists big differences, except for Cu and Mn.

Table 2. Content of main compositions in Litchi juice

Composition	Content	Composition	Content
Total sugar ^{*1} (g/l)	161.4	Tartaric acid (g/l)	
Reductive sugar (g/l)	158.5	Malic acid (g/l)	0.28
Glucose (g/l)	84.3	Lactic acid (g/l)	1.52
Fructose (g/l)	74.2	Acetic acid (g/l)	0.10
Sugar (g/l)	2.66	Vitamin A (mg/l)	0.04
Protein (g/l)	9.0	Vitamin B1 (mg/l)	0.02
Lipid (g/l)	0.5	Vitamin B2 (mg/l)	0.16
Total acid ^{*2} (g/l)	2.2	(mg/l)	354.12
Heat quantity (kcal/l)	586	Vitamin C (mg/l)	2.52
Citric acid (g/l)	0.36	Nisin (mg/l)	0.12
		β-carotene (mg/l)	

^{*1}calculated by glucose, ^{*2}calculated by tartaric acid.

Table 3. The content of minerals in Litchi juice

Element	Content (mg/l)	Element	Content (mg/l)
Ca	31.2	Fe	4.0
P	240.3	Zn	1.7
K	1251.8	Cu	1.6
Na	17.0	Mn	0.9
Mg	120.5	Se	1.4×10^{-3}

3.2 GENERAL COMPOSITION OF LITCHI WINE

Litchi wine was brewed following the procedures explained in section 2.1. The wine contents were also analyzed as shown in *Table 4*. The general dry extract content was up to 25.6 g/l, which was much higher than the criterion value of 17 g/l for grape wine suggested by the International Organization of Vine and Wine (O.I.V.,1990),.

Reductive sugar was almost consumed by the fermentation because of the low contents of total sugar and reductive sugar that remained in litchi wine, which were 2.1 g/l and 1.6 g/l, respectively. These met the dry wine standard (below 4.0 g/l for total sugar). The tartaric acid content was 4.0 g/l, as shown in Table 4. Actually 3.0 g/l of the tartaric acid was artificially added to the Litchi juice before the commencement of fermentation in order to increase total acid content and control the pH below 3.8. The original tartaric acid concentration in the juice was only 0.28 g/l and the balance, 0.72 g/l, was produced by fermentation. As for the volatile acid, it is usually a very important factor for grading fruit wine. Because up to now there is no authorized litchi wine criterion yet, we can only refer to the grape wine, which should be less than 1.1 g/l as suggested by O.I.V. criterion which specifies that the volatile acid which was calculated by acetic acid should be less than 1.1 g/l for quality products. Presumably, if this criterion was still valid in this case, the Litchi wine brewed in our experimental process was very satisfactory for its lower volatile acid level (only 0.56 g/l).

SO₂ is a necessary additive in many kinds of fruit wine making. Usually it is added by the forms of sulfurous acid or sulphite and functions as antioxidant, antibacterial agent and clarifying agent, etc. It exists in wine in two forms: bonding state and free state, but only the free state SO₂ has above functions. The measured total SO₂ content was the sum of both states. We found a very interesting and unusual phenomenon when we analyzed the free state SO₂ content in litchi wine. No matter how much sulfurous acid was added and how much total SO₂ content was measured, the measured free state SO₂ content was always below 10 ppm. This is quite different from the grape wine. The free state SO₂ content in grape wine can be accurately determined and controlled. But we can't do that in litchi

wine. Maybe there were a lot of compounds in litchi juice or wine which were easy to bond SO₂ and then reduced free state SO₂ content. Suppose this hypothesis is true, there should be a critical saturation value that exists. But we never found that even we added more than enough sulfurous acid. So the real function of the added SO₂ in fermentation process or the test method should be suspected. It is also a question whether we should use SO₂ in litchi wine making or not.

Table 4. The contents of main compositions in the Litchi wine

Composition	Content	Composition	Content
		Total acid ^{*4} (g/l)	
Total ester ^{*1} (g/l)	0.05675	Citric acid (g/l)	5.7
Total aldehyde ^{*2} (g/l)	0.1078	Tartaric acid (g/l)	0.2
Dry extract (g/l)	25.6	Malic acid (g/l)	4.0
Alcohol (%v/v)	11.2	Lactic acid (g/l)	1.0
Total sugar ^{*3} (g/l)	2.1	Volatile acid (g/l) ^{*5}	0.6
Reductive sugar (g/l)	1.6	Total SO ₂ (ppm)	0.56
Glucose (g/l)	0.5	Free SO ₂ (ppm)	125
Fructose (g/l)	0.3	Methyl alcohol (g/l)	10
Sugar (g/l)	0.6	Higher alcohols (g/l)	0.13
Protein (g/l)	5.6	Total polyphenol (g/l)	1.5
Lipid (g/l)	1.2		

*1calculated by acetic ester, *2calculated by acetic aldehyde, *3 calculated by glucose, *4calculated by tartaric acid. *5 calculated by acetic acid.

3.3 AMINO ACIDS

According to the research of Mauricio et. al.(2001), during wine fermentation and biological aging process, the yeasts can use amino acids not only as nitrogen sources but also as redox agents to balance the oxidation-reduction potential under conditions of restricted oxygen, when electron transport along the respiratory chain may be hindered or limited. On the basis of his research and the results of Jones and Fink (1982), most of arginine (Arg) but little proline (Pro) was consumed during fermentation while only Pro was used as nitrogen sources in wine aging process. So the kinds and contents of amino acids perform an important role in wine making. According to our previous research (Chen et al, 2004) , there was very high content of free proline in dry red wine (up to 1000 mg/l in some planting areas of China) and proline covered 60%-80% of the total free amino acids content. From **Table 5.**, we can find that the total free amino acid content in litchi wine was similar to that of grape wine (700-1300 mg/l). But the content of each

single kind of amino acid in litchi wine was all on an average level (less than 50 mg/l) contrast to those of grape wine. Not one of them was much higher than any others. This may reflect that there are different amino acids metabolic mechanisms other than that of grape wine in yeast fermentation process and results in different metabolic products such as glycerol, high alcohols, acetate, 2,3-butanediol, acetoin and succinate released to wine (Remize et. al., 1999; Romano and Suzzi, 1996). We have a long history of grape wine making, so we have already made out of the yeast metabolic mechanisms in grape wine making process.(Mauricio et al., 2001). But as for litchi wine making, we have just started relevant research in the past a few years and may not copy those technologies of grape wine making. Some deep research should be carried out and new technologies should be developed.

Table 5. The contents of amino acids in the Litchi juice and Litchi wine (mg/l)

Amino acid	Litchi juice		Litchi wine	
	Free amino acid	Total amino acid	Free amino acid	Total amino acid
Asp	106.0	124.1	10.0	64.0
Glu	60.8	155.2	13.5	97.9
Gly	45.2	98.6	5.2	45.3
Gly	16.5	49.5	6.3	41.3
His	19.0	64.3	14.2	30.5
Arg	89.6	165.3	17.5	88.1
Thr	400.9	1155.1	13.1	44.5
Ala	285.2	382.4	8.2	45.4
Pro	71.3	230.4	50.1	97.7
Tyr	61.9	159.1	19.4	51.0
Val	69.4	98.4	5.4	33.5
Met	100.6	163.6	5.2	19.6
Cys	7.1	33.6	1.9	3.0
Ile	2.5	69.0	0.8	17.6
Leu	3.8	57.6	29.9	30.9
Phe	13.7	28.1	3.8	15.8
Lys	10.9	23.6	7.2	28.0
Try	—	—	—	—
Total content	1364.4	3055.9	211.8	784.1

Total amino acid content reflects available nitrogen sources but has little contribution to mouth taste. Different kinds of free amino acids have different taste and help in forming litchi wine's special flavor. For example, Ser and Gly are sweet, Asp and Glu are sour, Pro, Thr and Lys are both sweet and bitter, and most

of the other amino acids are bitter. As for free amino acid in litchi wine, there were no prominent content amino acid that existed, so the flavor of the wine was balanced in the point of view of amino acid composition.

3.4 AROMA ANALYSIS OF WINE

Fruit wine aroma, widely considered to be a key aspect of quality, is the result of the interaction between components of the fruits themselves and those produced during processing, fermentation and aging, and the consumers sense of smell.(Sanchez Palomo et al., 2005). In this research, the litchi wine aroma compounds were subjected to gas chromatographic analysis, and the relative percentages of their contents were shown in Tab.6. The shape of the figure was not so good because of the dominant peak of ethyl caprelate. In total, 33 kinds of compounds were identified, including 16 esters, five alcohols, four acids, two ketones, one phenol and four other chemicals. These data were quite different from those of litchi juice. Chyau et.al (2003) found there were one ester, 14 alcohols, two aldehydes, four acids, two ketones and terpenes in clear litchi juice. Major compositions found in litchi wine were ester (totally 81.04 %). Ethyl octanoate (34.82 %), ethyl decylate (15.41 %), acetic ester(11.36 %), ethyl hexoate (7.14 %), acetic isopentyl ester (4.72 %) and citronel ethyl acetate (1.94 %) which covered 75.39 % and 93.03 % of the total compounds and ester compounds, respectively. This phenomenon was quite reasonable because there were a lot of biochemistry reactions such as oxidation, reduction, esterification, etc., that occurred during the fermentation process. (Mauricio et al., 2001). So we should compare those data with fruit wines, for example, white wine. Wondra and Berovic. (2001) found 14 esters, 4 higher alcohols and 4 fatty acids in young Chardonnay wine. Their results were quite similar to those from these experiments.

According to the research of Arctander (1969), ethyl octanoate has pleasant flower and fruit aroma, ethyl decylate has fatty acid smell and fruit fragrance. While acetic ester and ethyl hexoate are the most common aroma compositions in Chinese wine (Zeng et al., 2001.), the former has smells like pineapple and honey, while the latter has stronger pineapple and banana aroma. (Arctander,1969). Acetic isopentyl ester also has a strong fruit aroma, especially like that of pear and citronel ethyl acetate has a kind of comfortable fresh rose aroma. (Arctander,1969) . From the results of the sensory evaluation, the young litchi wine (less than three months after fermentation) had intense pleasant tropical and subtropical fruits aroma, just like many kinds of fruits mixed together. Obviously, these two conclusions (chemical and sensorial analysis) were coordinate.

According to Chyau et. al (2003), octanoic acid was the 4th highest content aroma compound (7.28 %) and the major acid tested by GC in litchi juice. Ethyl octanoate was produced from octanoic acid and ethanol by esterification. So it was

reasonable that ethyl octanoate became the predominant aroma compound (34.82 %) in litchi wine. Actually, ethyl octanoate was considered as one of the most important aromatic compounds in many other kinds of wine. (Alves et al., 2005; Wondra and Berovic, 2001; Zeng, et. al., 2001)

The percentage of higher alcohols (isobutanol, 2.82%; isoamyl alcohol, 4.44 %; isopulegol, 0.46 %; 1-deoxidate-D-mannitol, 0.13 %) was identified in **Table 6**. The total value (7.85%) of this was much less than those of rice wine (42.37 %, Zeng et. al., 2001) and young Chardonnay wine (33.44 %, Wondra and Berovic, 2001, No.1 sample data). Its quantity, measured by routine method, was 0.048 g/l. Usually, higher alcohols were produced from amino acids by yeast metabolic introversion. (Remize et. al, 1999), and they have important

Table 6. GC-MS analysis results of aroma compositions in *Litchi* wine

No	Hold time	Compounds	Molecular formula	Relative area Percentage(%)
1	1.7	methyl sulfide	C ₂ H ₆ S	2.64
2	1.91	acetic acid	C ₂ H ₄ O ₂	0.94
3	2.05	acetic ester	C ₄ H ₈ O ₂	11.36
4	2.14	Isobutanol	C ₄ H ₁₀ O	2.82
5	3.12	2,4,5-trimethyl-1,3-dioxide pentane	C ₆ H ₁₂ O ₂	0.52
6	3.21	isoamyl alcohol	C ₅ H ₁₂ O	4.44
7	4.34	ethyl butyrate	C ₆ H ₁₂ O ₂	0.51
8	6.48	acetic isopentyl ester	C ₇ H ₁₄ O ₂	4.72
9	8.87	1-deoxidate-D-mannitol	C ₆ H ₁₄ O ₅	0.13
10	9.24	ethyl hexoate	C ₈ H ₁₆ O ₂	7.14
11	11.17	Ethylphenol	C ₁₀ H ₁₈ O	0.72
12	11.39	rhodinol	C ₁₀ H ₁₈ O	2.91
13	11.57	9-oxo-methyl nonanote	C ₁₀ H ₁₈ O ₃	0.22
14	12.93	ethyl octanoate	C ₁₀ H ₂₀ O	34.82
15	13.81	pentadecanone	C ₁₅ H ₂₄ O	1.18

1	13.91	phenethyl acetate	C ₁₀ H ₁₂ O	0.62
6			2	
1	13.98	Isopulegol	C ₁₀ H ₁₈ O	0.46
7				
1	14.13	3,7-dimethyl-2-octylene-1- alcohol	C ₁₀ H ₂₀ O	0.72
8				
1	14.50	α-terpinyl ester acetate	C ₁₂ H ₂₀ O	0.74
9			2	
2	15.23	3,7-dimethyl-1,6-octyldiene- 3-alcohol	C ₁₀ H ₁₈ O	0.28
0				
2	15.44	ethyl laurate	C ₁₄ H ₂₈ O	0.36
1			2	
2	16.34	methyl caprate	C ₁₁ H ₂₂ O ₂	0.08
2				
2	17.36	citronellyl acetate	C ₁₂ H ₂₂ O	1.94
3			2	
2	19.43	ethyl decylate	C ₁₂ H ₂₄ O	15.41
4			2	
2	26.75	ethyl pentadecanate	C ₁₇ H ₃₄ O	0.98
5			2	
2	27.43	ethyl lauric acid	C ₁₅ H ₃₀ O	0.16
6			2	
2	27.80	dilauryl phthalic acid	C ₃₂ H ₅₄ O	0.96
7			4	
2	29.37	isopropyl myristic acid	C ₁₇ H ₃₄ O	0.90
8			2	
2	29.47	diethyl phthalate	C ₁₆ H ₂₂ O	0.72
9			4	
3	30.11	dilauryl phthalate	C ₃₂ H ₅₄ O	0.26
0			4	
3	30.65	6,10,14-trimethyl-2- phetadecanone	C ₁₈ H ₃₆ O	0.12
1				
3	32.50	hexadecylic acid	C ₁₆ H ₃₂ O	0.12
2			2	

role in wine. On one hand, they have a special attractive aroma and strong sharp alcohol taste (Arctander, 1969), so they enrich the wine's sensory characteristics

and make the wine taste more like wine. Consumers usually like proper amounts of higher alcohols in wine. But on the other hand, higher alcohols are harmful chemicals to the human body and may cause some side-effects such as the ease of which to get drunk, headaches and thirstiness, etc. So the amount of higher alcohols in wine should be limited in suitable content. The quite lower relative content of higher alcohols in litchi wine may reflect its high quality. The reason of such low higher alcohols content may due to low fermentation temperature and the amino acids constituents of litchi juice.

In total, high quality wines should have a characteristic bouquet and taste which depends on the cultivar, maturity and phytosanitary conditions of the litchi fruit, pedoclimatic conditions, and most importantly, on yeast fermentation physiology. All these will cause the differences of aroma fragrance composition and quantity among litchi wines or other kinds of fruit wine.

CONCLUSIONS

The general compositions of litchi juice and litchi wine were measured by O.I.V. routine analysis methods. 161.4 g/l total sugar, 2.2 g/l total acids and high content of Vitamin C (354.12 mg/l) and K (1251.8 mg/l) were found in litchi juice. This made it possible to choose litchi juice to make wine. The technology of low temperature (13-16°C) and long fermentation time (8-10 days) was used to make litchi wine in this research. High dry extract (25.6 g/l), low volatile acid (0.36 g/l) and low high alcohols (0.048 g/l) were identified in the young litchi wine. Sensory evaluation results showed that it was well-balanced and typical with strong body, complex content and intense tropical and subtropical fruity and floral aroma.

In contrast to that of grape wine, the content of free SO₂ in litchi wine was very difficult to quantify. No matter how much sulfurous acid was added and how much total SO₂ content was measured, the measured free state SO₂ content was always below 10 ppm. Deeper research should be carried to make out the reasons.

The total free amino acid content in litchi wine (784.1 mg/l) was similar to that of grape wine (700-1300 mg/l). But the content of each single kind of amino acid in litchi wine was all on an average level (less than 50mg/l) .Not one was prominently higher than any others.

In total, 33 kinds of aroma compounds were identified in litchi wine by liquid-liquid extraction and GC/MS analysis, including 16 esters, five alcohols, four acids, two ketones, one phenol and four other chemicals. Ethyl octanoate, ethyl decylate, acetic ester, ethyl hexoate, acetic isopentyl ester and citronel ethyl acetate were main compositions, which covered 75.39 % and 93.03 % of the total compounds and ester compounds, respectively. Higher alcohols occupied 7.85 % of the total aroma compounds which was much lower than some other rice wines

(42.37 %) and young Chardonnay wines (33.44 %) which may be due to the low fermentation temperature and the amino acids constituents of litchi juice.

REFERENCES

- Alves, R.F., Nascimento, A.M.D., Nogueira, J.M.F., 2005. Characterization of the aroma profile of Madeira wine by sorptive extraction techniques. *Analytica Chimica Acta* 546, 11–21
- A.O.A.C. 1995. *Official Methods of Analysis*. Washington D.C. USA: Association of Official Analytical Chemists.
- Arctander, S. (Ed.), 1969. *Perfume and flavor chemicals (aroma chemicals)*. Montclair, NJ: Steffen Arctander.
- Amerine, M. A., Roessler, E. B.(Eds.), 1976. *Wines: Their sensory evaluation*. San Francisco: W.H. Freeman and Co.
- Arévalo Villena, M., Díez Pérez, J., Úbeda, J.F., Navascués, E. A., Briones, I., 2006. A rapid method for quantifying aroma precursors: Application to grape extract, musts and wines made from several varieties. *Food Chemistry, Analytical, Nutritional and Clinical Methods* 99, 183-190.
- Bates, C.J. (Ed.), 2000. *Vitamins: Fat and Water Soluble: Analysis*, Encyclopedia of Analytical Chemistry, John Wiley & Sons Ltd, Chichester
- Bonino, M., Schellino, R., Rizzi, C., Aigotti, R., Delfini, C., Baiocchi, C., 2003. Aroma compounds of an Italian wine (Ruché) by HS–SPME analysis coupled with GC–ITMS. *Food Chemistry, Analytical, Nutritional and Clinical Methods Section* 80, 125–133.
- Cheng, F.L., Ning, M.X., Mo, J.Y., Liang, Y.C., 2002. Determination of trace elements in lychee of different growth periods. *Spectroscopy and Spectral Analysis* 22 (4), 676-678.
- Chen, H.B., Ni, Y.Y., 2006. A few points of view about the development of litchi industry. *China Tropical Agriculture* 1, 7-8.
- Chen, Y., Zeng, X.A., Dong, X.P., Xiao, L.M., 2004. Comparison of free amino acids content of domestic dry red wine. *Food and Fermentation Industries* 030(001), 107-110
- Chyau, C.C., Ko, P.T., Chang, C.H., Mau, J.L., 2003. Free and glycosidically bound aroma compounds in lychee. *Food Chemistry* 80, 387-392.
- Comuzzo, P., Tat, L., Tonizzo, A., Battistutta, F., 2006. Yeast derivatives (extracts and autolysates) in winemaking: Release of volatile compounds and effects on wine aroma volatility. *Food Chemistry* 99, 217-230
- Cui, S.W. (Ed.), 2005. *Food Carbohydrates*. Taylor & Francis Group.
- Dong, H.Q., Cheng, L.Y., Tan, J.H., Zheng, K.W., Jiang, Y.M., 2004. Effects of chitosan coating on quality and shelf life of peeled litchi fruit. *Journal of Food Engineering* 64, 355-358.

- Eitenmiller, R.R., Landen, W.(Eds), 1998. Vitamin Analysis for Health and Food Sciences, RC Press LLC, New York, 1.
- Garruti, D.S., Franco, M.R.B., Silva, M.A.A.P., Janzanti, N.S., Alves, G.L., 2006. Assessment of aroma impact compounds in a cashew apple-based alcoholic beverage by GC-MS and GC-olfactometry. *LWT* 39,372–377
- Gil, M., Cabellos, J.M., Arroyo, T., Prodanov, M., 2006. Characterization of the volatile fraction of young wines from the Denomination of Origin “Vinos de Madrid” (Spain). *Analytica Chimica Acta* 563, 145-153.
- Gómez-Míguez, M.J., Cacho, J.F., Ferreira, V., Vicario, I.M., Heredia, F.J., 2007. Volatile components of Zalema white wines. *Food Chemistry* 100, 1464-1473.
- Gontier, E., Boussouel, N., Terrasse, C., Jannoyer, M., Menard, M., Thomasset, B., Bourgaud, F., 2000. Litchi chinensis fatty acid diversity: occurrence of the unusual cyclopropanoic fatty acids. *Biochemical Society Transactions* 28, 578-580.
- Jiang, Y.M., 2000. Role of anthocyanins, polyphenol oxidase and phenols in lychee pericarp browning . *Journal of the Science of Food and Agriculture* 80(3), 305-310.
- Jiang, Y.M., Li, Y.B., Li, J.R., 2004. Browning control, shelf life extension and quality maintenance of frozen litchi fruit by hydrochloric acid. *Journal of Food Engineering* 63, 147-151
- Jones, E.W., Fink, G. R., 1982. Regulation of amino acid and nucleotide biosynthesis in yeast. In *The Molecular Biology of the Yeast Saccharomyces. Metabolism and Gene Expression*; Strathern, J.n., Jones, E.W., Broach. J.R., Eds.; NY, 181-299
- John, K., 2003. Analytical chemistry for technicians. 3rd edition, CRC Press LLC.
- Mauricio, J.C., Valero, E., Millian, C., Ortega, J.M., 2001. Changes in Nitrogen Compounds in Must and Wine during Fermentation and Biological Aging by Flor Yeasts. *Journal of Agricultural and Food Chemistry* 49, 3310-3315
- Menzel, C.M., Watson, B.J., Simpson, D.R.,1988. The lychee in Australia. *Queensland Agriculture Journal* 2,19-27.
- Ong, P.K.C., Acree, T.E., 1998. Gas chromatography olfactory analysis of lychee (Litchi chinensis Sonn.) .*Journal of Agricultural and Food Chemistry* 46 (6), 2282-2286.
- Ong, P.K.C., Acree, T.E., 1999. Similarities in the aroma chemistry of Gewurztraminer variety wines and lychee (Litchi chinensis Sonn.) fruit. *Journal of Agricultural and Food Chemistry* 47 (2), 665-670.
- Olesen, T., Nacey, L., Wiltshire, N., O'Brien, S., 2004. Hot water treatments for the control of rots on harvested litchi (Litchi chinensis Sonn.) fruit. *Postharvest Biology and Technology* 32, 135-146
- O.I.V., 1990. Recueil des methodes internationales d'analyse des vins et des mouts. Office International de la Vigne et du Vin, Paris.

- Ötleş, S. (Ed.), 2005. *Methods of analysis of food components and additives*, Taylor & Francis Group.
- Ough, C. S., Amerine, M. A. (Eds.), 1988. *Methods for analyses of musts and wines* (2nd ed.). New York: John Wiley and Sons.
- Paull, R.E., Reyes, M.E.Q., Reyes, M.U., 1998. Sulfite residues on litchi fruit treated with sulfur dioxide. *Postharvest Biology and Technology* 14, 229-233
- Perestrelo, R., Fernandes, A., Albuquerque, F.F., Marques, J.C., Câmara, J.S., 2006. Analytical characterization of the aroma of Tinta Negra Mole red wine: Identification of the main odorants compounds. *Analytica Chimica Acta* 563, 154–164
- Remize, F., Roustan, J.L., Sablayrolles, J.M., Barre, P., Dequin. S., 1999. Glycerol overproduction by engineered *Saccharomyces cerevisiae* wine yeast strains leads to substantial changes in by-product formation and to a stimulation of fermentation rate in stationary phase. *Appl. Environ. Microbiol.* 65, 143-149.
- Roessler, E.B., Pangborn, R.M., Sidel, J.L., Stone, H., 1978. Expanded statistical tables for estimating significance in paired preference, paired difference, duo-trio and triangle tests. *Journal of Food Science* 43, 940-943.
- Romano, P., Suzzi, G., 1996. Origin and production of acetoin during wine yeast fermentation. *Appl. Environ. Microbiol.* 62, 309-315.
- Sánchez Palomo, E., Díaz-Maroto Hidalgo, M.C., González-Viñas, M.Á., Pérez-Coello, M.S., 2005. Aroma enhancement in wines from different grape varieties using exogenous glycosidases. *Food Chemistry* 92, 627-635.
- Sarni-Manchado, Phunchaisri, C., Apichartsrangkoon, A., 2005. Effects of ultra-high pressure on biochemical and physical modification of lichee (*Litchi chinensis* Sonn.). *Food Chemistry* 93, 57-64
- Sarni-Manchado, P., Le, R.E., Le, G.C., Lozano, Y., Cheynier, V., 2000. Phenolic composition of litchi fruit pericarp. *Journal of Agricultural and Food Chemistry* 48 (12), 5995-6002.
- Selli S., Canbas A., Cabaroglu, T., Erten, H., Gunata, Z., 2006. Aroma components of cv. Muscat of Bornova wines and influence of skin contact treatment. *Food Chemistry* 94, 319-326.
- Vanderslice, J., Higgs, D., 1984. HPLC analysis with FD of vitamin C in food samples, *Journal of Chromatographic Science* 22, 485
- Wang, W.H., 1980. The techniques of wine sensory evaluation. *Techniques of wine produce—Taiwan Tobacco and Wine Monopoly Bureau* 2, 40-57.
- Wondra, M., Berovic, M., 2001. Analyses of Aroma Components of Chardonnay Wine Fermented by Different Yeast Strains. *Food Technology Biotechnology* 39(2), 141-148
- Wu, M.C., Chen, C.S., 1999. A research note: Effect of sugar types and citric acid content on the quality of canned lychee. *Journal of Food Quality* 22 (4), 461-469.

- Yang, Y.X., 2002. Composition of Chinese Food. Published by Medical Bureau of Beijing University, Beijing, 95
- Zalacain, A., Marín, J., Alonso, G.L., Salinas, M.R., 2007. Analysis of wine primary aroma compounds by stir bar sorptive extraction. *Talanta*, 71(4), 1610-1615.
- Zea, L., Moyano, L., Moreno, J., Cortes, B., Medina, M., 2001. Discrimination of the aroma fraction of Sherry wines obtained by oxidative and biological ageing. *Food Chemistry* 75,79–84
- Zeng, X.A., Zhang, B.S., Yu, S.J., 2001. A Study on the Typical Fragrance Components of CHI-XIANG Style Rice Wine. *Journal of South China University of Technology (Natural Science Edition)* 29(12), 93-95
- Zhang, Z.Q., Pang, X.Q., Yang, C., Ji, Z.L., Jiang, Y.M., 2004. Purification and structural analysis of anthocyanins from litchi pericarp. *Food Chemistry* 84, 601-604