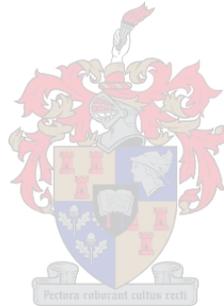


Evaluating the effect of pot still design on the resultant distillate

by

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DECLARATION

I, the undersigned, hereby declare that the work contained in this thesis is my own original work and that I have not previously in its entirety or in part submitted it at any university for a degree.

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SUMMARY

The total sale of brandy for 2007 in South Africa was R 7 300 000 000 and local statistics indicate that brandy is by far the most purchased spirit beverage. Sales of brandy even outweigh the total sales for whisky and the forecast for the estimated sales of brandy in the next five years is said to increase by 25%. It is therefore crucial to investigate those factors that influence the production of brandy as better understanding and control of these processes leads to the production of a brandy that is consistent and of premium quality.

Many factors influence the final outcome of distillates. Of these factors, the distillation technique, the apparatus used for the purpose of distillation together with the low wine is of utmost importance as they influence the sensory profile and the chemical composition of the distillate. The effect of different variations of pot still designs on the chemical composition and the sensory profile of the resultant distillate was investigated. Five different Pot still variations were used and varied with regards to the design of their pot still head and swans neck apparatus. Two low wines were used for the purpose of distillations and were both from 2007 vintage. GC-FID was used to identify the volatile compounds found in the distillates and together with Quantitative Descriptive Analysis (QDA) a profile of the distillates was produced which was used to differentiate between the different pot still variations and their effect on the final product. The data generated from the QDA sessions was subjected to Principal Component Analysis (PCA) and together with the chemical analysis a correlation between certain compounds and sensory attributes were found in the distillates. Distillate samples were also subjected to a sensory style classification system and were classified accordingly.

The chemical composition of the two low wines prior to distillations differed significantly from each other with low wine one containing a larger amount of total esters and carbonyl compounds whilst low wine two contained a larger amount of total higher alcohols and acids. The distillates of low wine one also contained over all larger amounts of total esters and in the case of the distillates of low wine two, they contained larger amounts of higher alcohols and acids than low wine one.

Variation one was based on the *Alambic Charentais* method of pot still design and it was found that only variation one influenced the chemical composition and the sensory profile of the distillates. This variation produced a distillate with a lower amount of total esters and more specifically ethyl acetate as well containing a lower intensity of the fruit and sweet associated caramel aromas and flavours. The esters, ethyl acetate and the ethyl esters of the long chained fatty acids were found to correlate with the sensory attributes known as fruit associated aroma, soapy aroma, and spicy aroma and therefore indicated that these compounds are responsible for these attributes. There were no correlations found between the chemical compounds, sensory attributes and sensory style classifications in the distillates of both low wine one and two. It was shown that the addition of certain esters, carbonyl compounds, higher alcohols and acids in specific ratios could alter the sensory classification of the distillates. Therefore the chemical composition and the sensory characteristics of distillates are largely dependent on the chemical composition of the low wine prior to distillation rather than the pot still design. Therefore, with further research it could be possible to predict the outcome of the chemical composition of the distillates by analyzing the chemical compounds found in the low wine prior to distillation.

OPSOMMING

Die totale verkope aan brandewyn vir 2007 in Suid Afrika beloop R7 300 000 000 en statistiek wys dat brandewyn by verre die mees gesogte spiritus drank is. Verkope van brandewyn is selfs meer as die verkope van whisky en die voorspelling is dat die verkope van brandewyn met 25% gaan vermeerder in die volgende vyf jaar. Dit is dus belangrik om die faktore te ondersoek wat die produksie van brandewyn beïnvloed om sodoende die verstokingsproses te verstaan en te kontroleer om 'n konsekwente kwaliteitsproduk op die mark te plaas.

Baie faktore beïnvloed die finale produk. Faktore soos die distillasie tegnieke, die apperate wat gebruik word vir distillasie tesame met die rabatspiritus is van die uiterste belang aangesien dit die sensoriese profiel en die chemiese samestelling van die distillaat beïnvloed. Die effek van die verskillende variasies van potketelhelms op die chemiese samestelling van die distillate word ondersoek. Vyf verskillende helms met variasies in die swaannek ontwerp was gebruik. Twee verskillende rabatspiritus, van die 2007 oesjaar, was gebruik vir distillasie. GC-FID was gebruik om die vlugtige komponente van die distillate mee vas te stel. Kwantitatiewe Beskrywende Analise (QDA) is gebruik om 'n profiel van die distillate op te stel wat weer gebruik is om te differensieer tussen die verskillende potketelhelm variasies en hulle effek op die finale produk. Die data wat deur die QDA sessies gegenereer was, is in die Vernaamste Komponent Analise (PCA) ingevoer en tesame met die chemiese analise is 'n korrelasie tussen sekere komponente en die sensoriese analise van die distillate gevind. Distillaat monsters was ook aan sensoriese styl van klassifikasie onderwerp en is as volg daarvan geklassifiseer.

Die chemiese samestelling van die twee rabatspiritus voor finale distillasie het betekenisvol van mekaar verskil ten opsigte daarvan dat die eerste rabatspiritus het hoë konsentrasies esters en karboniel verbindings gehad terwyl die tweede rabatspiritus meer hoë konsentrasies van sure en hoër alkohole gehad het. Die distillaat van die eerste rabatspiritus het ook hoë konsentrasies esters en karboniel verbindings gehad terwyl die distillaat van die tweede rabatspiritus weer hoë konsentrasies van sure en hoër alkohole gehad het.

Variasie een is gebaseer op die *Alambic Charentais* van potketel ontwerp en daar is ook gevind dat hierdie variasie die enigste een was wat die chemiese samestelling en die sensoriese profiel van die distillate beïnvloed het. Hierdie variasie het 'n distillaat geproduseer wat lae konsentrasies van totale esters, veral etielasetaat, sowel as laer intensiteit van vrugtige en soet geassosieerde karamel aromas en geure. Die esters, etielasetaat en etiel esters van die lang ketting vetsure, is gevind dat dit goed korreleer met die sensoriese eienskappe wat geassosieer word met vrugtige aromas, spesery-agtige aromas en seperige aromas. Daar is geen korrelasie gevind tussen die chemiese verbindings, sensoriese eienskappe en sensoriese styl van klassifikasie van distillate een en twee nie. Dit was ook bewys dat byvoeging van esters, karboniel verbindings, sure en hoër alkohole, in spesifieke verhoudings, die sensoriese eienskappe kan verander. Dus is die chemiese samestelling en sensoriese eienskappe van die distillate grootliks afhanklik van die chemiese samestelling van die rabatspiritus, voor die tweede distillasie, as wat dit afhanklik is van die potketelhelm ontwerp. Gevolglik, met verdere

navorsing, is dit moontlik om die uitkoms van die chemiese samestelling van die distillaat te voorspel deur die analise van die chemiese verbindings van die rabatspiritus te ontleed.

This thesis is dedicated to my parents for all their love, support and continuous guidance.

BIOGRAPHICAL SKETCH

Nina Bougas was born in Cape Town, South Africa on the 7th of March 1984. She matriculated at Parel Vallei High School, Somerset West in 2001 and enrolled at Stellenbosch University in 2002. She obtained a BSc Agric degree in Viticulture and Oenology in 2005.

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PREFACE

This thesis is presented as a compilation of 4 chapters. Each chapter is introduced separately and is written according to the style of the journal American journal of Enology and Viticulture.

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Chapter 2 **Literature review**

FACTORS AFFECTING THE CHEMICAL AND SENSORY
COMPOSITION OF UN-MATURED POT STILL BRANDY.

Chapter 3 **Research results**

THE INFLUENCE OF POT STILL DESIGN ON THE SENSORY
CHARACTERISTICS OF UN-MATURED BRANDY.

Chapter 4 **General discussion and conclusions**

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Chapter 1

INTRODUCTION AND PROJECT AIMS

1. INTRODUCTION AND PROJECT AIMS

1.1 INTRODUCTION

Brandy is one of the most important spirits consumed by the South African population (South African Wine Industry Information and Systems, 2007). South Africa is one of the largest brandy producing countries in the world and falls 6th in the global market. The total sale of brandy for 2007 in South Africa was R 7 300 000 000 and local statistics indicate that brandy is by far the most purchased spirit beverage. Sales of brandy even out-weigh the total sales for whisky and the forecast for the estimated sales of brandy in the next five years is said to increase by 25%.

Many different types and styles of brandy are available on the market today, to mention a few, Richelieu, Klipdrift, Flight of the Fish Eagle, Viceroy, OudeMeester, Mellowood and Van Ryn's brandy. Due to the fact that this liquid is so widely consumed, it is important as a company to be able to produce and replicate the desired product and ensure that it is authentic and of good quality (Jack 2003).

The main styles of brandy include blended, vintage, estate and pot still brandy. Each of these brandies varies greatly with regards to their sensory profile and is firstly dependent on legal classification. In the case of blended brandy, it consists of a minimum of 30% pot still brandy which has been matured for three years together with a maximum of 70% un-matured wine spirit (le Roux 1997). This brandy is not overly flavoured and is used together with a mixer. The alcohol concentration is 43% alcohol per volume. Vintage brandy has a distinct wood character when compared to pot still and blended brandy. It consists of a minimum of 30% pot still brandy matured for 8 years, 60% column still brandy matured for at least 8 years and a maximum of 10% un-matured wine spirits (Wine and Spirits Control Act No 47 of 1970). The alcohol concentration of this brandy is 38% alcohol per volume. For estate brandy, the only recommendation, is that this brandy must be produced and bottled on the estate.

Of the different styles of brandy available, pot still brandy is considered the richest, fruitiest and most layered brandy and has a vanilla flavour due to the wood maturation. One of the most premium styles of brandy that is produced by Distell is Van Ryn's pot still brandy. This brandy is made up of 90% pot still brandy and a maximum of 10% wine spirits and has alcohol concentration of 38% alcohol per volume (le Roux 1997). Van Ryn's pot still brandy is consumed neat or over ice, as this brandy is very complex and aromatic due to its extended maturation period. Van Ryn's reserve brandy collection consists of 12, 15 or 20 year pot still brandy each with its unique characteristics.

There are many factors that will influence the production and the quality of brandy. These include the type of vintage, geographical origin, cultivar, vinification techniques, malolactic fermentation, maturation and distillation. Of these factors, the fermentation and yeast type is of great importance as studies by Steger and Lambrechts (2000) indicate that the yeast strain together with the initial substrate has a large effect on the type and amount of compounds found in the product which will ultimately influence the sensory perception of the product. However, the actual process of distillation is one of the most important factors to consider (Leaute 1950).

At Distell, most of the brandy is distilled in copper pot stills and the initial substrate used for the distillation is grapes. This type of distillation is known as double or batch distillation. Brandy that is made from pot stills is normally found to be more aromatic than brandy made from continuous

stills, as this distillation technique enhances the aromatic qualities (Carnacini 1989). The first stage of batch distillation involves wine that is distilled and collected in one fraction and has an alcohol concentration of 28-30% a/v. This liquid is known as low wine. The second stage is to distil the low wine and collect it in three fractions, namely the heads hearts and tails (Leaute 1950). The heart fraction is the most important to the distiller and used for maturation. The heart fraction will have an alcohol strength of 65-75% a/v.

Pot stills vary in their capacities and shapes. Those that have a larger still head or a swan's neck that are orientated in such a way that it slopes up towards the condenser will have a greater degree of reflux (Leaute 1950). Reflux is the term used to describe the amount of vapour that condenses and runs back into the pot still to be reboiled (Hampel and Hawley 1982). Brandy produced from these pot stills will be purer and less aromatic as the denser heavier compounds will not distil easily and will remain behind in the pot still. There is little or no research regarding this topic of the influence of different shapes and sizes of pot stills, therefore making it an important concept to investigate.

Freshly distilled brandies are generally unacceptable with regards to sensory characteristics and are matured in oak barrels to produce a product of premium quality. By law in South Africa, brandy must be matured for a minimum of three years (South African Liquor Products Act No. 60 of 1989). The maturation is long and the gamble that is taken to ensure the brandy is of good quality and the correct style is large due to the extended time needed for maturation. Maturation is complex and the character of brandies can be related to the concentrations of volatile and non-volatile compounds. It is possible to predict the sensory scores of characteristics associated with maturation from the quantifications of non-volatile compounds, however with the volatile compounds it is more difficult to predict what role they play within the final product (Conner et al. 1994a). Studies done by Guymon (1972) show that a need arose for information on brandy distillate as most of the information available is on aged products, which makes it difficult to assess un-matured brandy.

In the industry, spirit products and more specifically brandy is evaluated using descriptive testing such as profiling or Quantitative Descriptive Analysis (QDA), where the product is assessed in order to gauge their aroma, flavour and mouthfeel and accordingly their style classification. A trained panel is used to evaluate the spirit products and to produce attributes that best describe the product. For example in the whisky industry, attributes mainly associated with maturation are used (Shortreed et al. 1979). As shown by Guymon (1972), there is only a small amount of information available on un-matured spirit products and especially un-matured pot still brandy, thereby making it crucial to produce a trained panel that can evaluate this product.

There are many factors that contribute towards the final brandy product, making the production of brandy a complex process. The way in which these factors influence the aroma and characteristics of the brandy, either as a whole or individually are important to understand and being able to manipulate them to such an extent could lead to an overall better control of the production process and therefore better quality products.

1.2 PROJECT AIMS

This study forms an integral part of an extensive research program aimed at understanding the factors that influence the quality and style of brandies to ensure a consistent product for the consumer and to be able to develop new styles. Due to the many shapes of still heads and swan's necks, the aim of this project was to investigate the influence of the different pot still heads and

swan's necks on the volatile compound composition of the un-matured pot still brandy and to establish the effect on the sensory profile and classification style of these products.

Pot stills that are used in the commercial industry are similar shape and size and vary little. They have normally the capacity of 2000 L and represent the *Alambic Charentais* style of pot still that originated in France for the production of Cognac. Studies conducted by Leaute (1950) shows that the "onion" shaped pot still head used for the production of Cognac produces a brandy that is more complex and richer due to the greater degree of reflux as a result of the larger surface area. Various shapes of the pot still head and swan's neck are used at Distell; however variations of pot stills other than the "onion" shape and how they influence the final sensory outcome of the brandy have not been previously investigated. The specific aims of the project were:

1. To determine if the different pot still designs have an effect on the chemical composition, sensory profile and sensory style classification of un-matured pot still brandy.
2. To apply GC-FID analysis to qualify and quantify the specific volatile compounds found in the un-matured Pot still brandy.
3. To determine the effect of the two different low wines on the chemical composition, sensory profile and sensory style classification of un-matured pot still brandy.
4. To develop reference standards for un-matured pot still brandy which can be used for future sensory profiling of un-matured pot still brandy.
5. To train a sensory panel using Quantitative Descriptive Analysis (QDA) to evaluate the un-matured pot still brandy and therefore to produce a sensory profile of the product.

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Chapter 2

LITERATURE REVIEW

Factors affecting the chemical and sensory composition of un-matured pot still brandy

2. FACTORS AFFECTING THE CHEMICAL AND SENSORY COMPOSITION OF UN-MATURED POT STILL BRANDY

“Claret is the liquor for boys; port for men; but he who aspires to be a hero must drink brandy”
Samuel Johnson.

2.1 INTRODUCTION

Brandy is a spirit that is made from fruit juice or fruit pulp and skin. There are many different fruit brandies available on the market, however for the purpose of this study, brandy is made from grapes.

Brandy can be said to have originated from the Moslem Mediterranean states in the 7th and 8th centuries. The Arab alchemists used the distillation technique to produce medicinal spirits; to them it was known as “aqua vitae”, meaning the water of life. To the Dutch the word for brandy was Brandewijn and literally means “burnt wine”. They described it as “burnt” as the wine had been boiled in order to distil it (Gold 1972).

The first time brandy was produced in South Africa was in 1659 and a total volume of 43,305.000 L of brandy was produced in South Africa in 2007 (South African wine industry information and systems, 2007). Statistics indicate that brandy is the most widely consumed spirit beverage on the market today. South Africans like to drink brandy with their favourite mixer or neat over ice, depending on the style of the brandy and the consumer preference. It is important for a company to be able to produce the desired product and to remain consistent and reliable and it is also in the company’s best interest to invest time and money into research and new product development as this will ensure that they are producing products that meet consumer demands (Jack 2003). This spirit product is then aged and matured in oak barrels, which adds colour, and additional aromas and flavours (Gold 1972).

The distillation technique has not changed a great deal over the years, and the same process and concept that was used then is being employed today in the alcohol industry (Leaute 1950). There are many techniques used for the process of distillation, the main one’s being batch distillation (discontinuous distillation) and column distillation (continuous distillation). However for the purpose of this literature review, focus will be given on batch distillation as this is the main technique used at Distell for the production of brandy.

2.1.1 Influence of cultivars on the final distillate

The body of flavour compounds are formed during the fermentation, but the flavour composition is strongly influenced by the precursors found in grapes prior to fermentation (Nykanen 1986). Studies by Ferrari et al. (2004) shows that the raw material used for the production of distilled beverages give these products their specific character. There is still a great deal of debate as to what the most desirable characteristics are in grapes specifically for the production of brandy distillates. However, studies conducted by Guymon (1969) show that the optimal grape variety for the production for brandy distillates is a white variety that displays a pleasing aroma and is also resistant to rot and oxidation. Further studies conducted by Quady and Guymon (1973) indicate that there is a good correlation between quality of brandy and grapes that are fruity and aromatic versus grapes that are overripe and oxidized. The main types of South African cultivars that are used in the production of brandy include Chenin blanc, Colombard, Cinsaut, Ugni blanc and Palomino (le Roux 1997).

2.1.2 Influence of yeast strain on the final distillate

The majority of the flavour compounds are formed during fermentation by the yeast (Nykanen 1986). These compounds include volatile organic acids, alcohols, aldehydes and esters (Fundira et al. 2002). The production and the amount of these compounds found in the wine are yeast strain dependent. Therefore the yeast strain used during the fermentation will ultimately influence the quality of the wine or distillate. Studies by Nykanen (1986) show that the aldehyde content increases when the action of the yeasts is most vigorous. This stage is found to be related to the activity of its pyruvate decarboxylase and when the nutrient content in the must is insufficient.

During the production of cognac the most used yeast strain is *Saccharomyces cerevisiae*, however it is important to remember that the effect of indigenous yeast strains can also be beneficial as studies by Fundira et al. (2002) show that indigenous yeast strains can produce desirable sensory characteristics. It is recommended that the evaluation of the yeast strain for the production of distillates should only be analysed after the distillation procedure.

Many authors have commented on the influence of fermentation temperature on the volatile compounds and have found that the amount of higher alcohols and aldehydes increase with an increase of temperature and that the esters and volatile organic acids increase with a decrease in temperature. If there are problems during the fermentation procedure then the amount of propanol will be higher than that of iso-butanol and acetoin (Cantagrel 1988). It is however difficult to predict the amount of volatile compounds that will land up in the distillate as the distillation technique along with the performance of the yeast in the wine plays an important role in the production of the volatile compounds.

Studies also show that if a higher percentage of lees content is used during the distillation process for brandy it can be highly correlated to even-numbered fatty acid ester content of the distillate (Watts et al. 2003). Therefore the amount of lees used is an important factor to consider as even-numbered fatty acids have a huge impact on the organoleptic properties of the distillate.

2.1.3 Influence of malolactic fermentation on the final distillate

Malolactic fermentation is the fermentation caused by lactic acid bacteria whereby malic acid is converted into lactic acid (Du Plessis et al. 2002). This reaction can contribute positively towards the flavour and aroma of the wine with increasing the “buttery aroma” flavour whilst decreasing the “green” characteristic in the wine. However if there is a large amount of lactic acid in the wine, this acid can combine with ethanol present and produce ethyl lactate thus making the wine undesirable. This reaction is accentuated if the wine is stored for a long time.

Wine that is destined for distillation contains no sulphur; this is a strong antimicrobial agent which can prevent contamination of the wine. Storage of the base wine prior to distillation can lead to an increase in ethyl lactate which can contribute negatively to the organoleptic properties of the distillate. Studies conducted by Du Plessis et al. (2002) found that with spontaneous malolactic fermentation during prolonged storage of the base wine leads to an increase in ethyl lactate and diethyl succinate. Compounds such as methyl alcohol and 2-butanol can also play a role, and be detrimental to the quality of the distillate (Dieguez et al. 2005).

2.2 DISTILLATION TECHNIQUES AND APPARATUS

2.2.1 Distillation

Distillation is the most important separation process in the chemical industry and entails heating of a solution, and condensing the resulting vapour into a different vessel (Leaute 1950). As the vapour and original substrate will have different compositions; the distillate at any given temperature will have a higher proportion of the original components and lower proportion of others. The way in which these volatile compounds will distil is governed by the distillation method and their volatility characteristics. This in turn is dependent entirely on the laws of vapour-liquid equilibrium thermo dynamics (Saco et al. 2006). Therefore distillation is a means of partial separation of the volatile components of the mixture.

This partial separation is based on the fact that the vapour phase is richer in the more volatile components than the liquid and this enrichment is determined by the vapour-liquid phase equilibrium (Hilmen 2000). Phases occur in equilibrium with each other, and because of this phase equilibria, a vapour phase can occur at a specific temperature and composition and can therefore occur in equilibrium with a liquid phase. Therefore the boiling point of a liquid mixture is the temperature at which the total vapour pressure is equal to the external pressure.

Figure 1.1 is a graph that describes a vapour liquid equilibrium curve. The lower curve of the graph gives the temperature at which different compositions of the liquid mixture reach a vapour pressure that is equal to atmospheric, therefore boiling point. The upper part of the curve represents the composition of the vapour that is in equilibrium with the liquid at its boiling point. The boiling point of the mixture will increase as B increases. And finally the horizontal line known as MN is the equilibrium line. This graph is typical of an ideal solution.

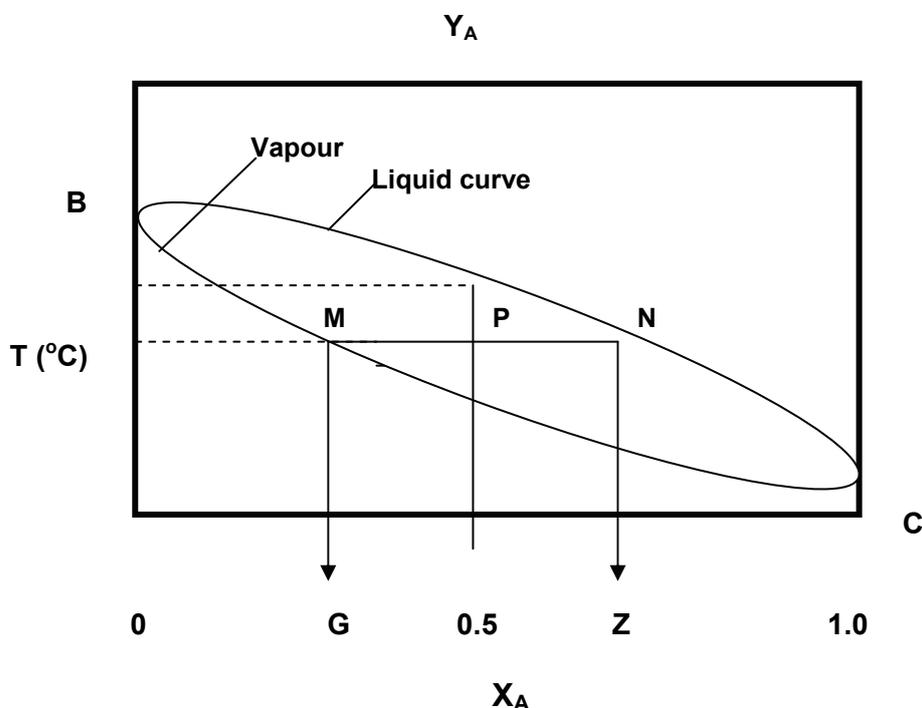


Figure 1.1 Vapour liquid equilibrium curve. X_A -Molar fraction of A in the vapour phase; Y_A -Molar fraction of A in the liquid phase; T ($^{\circ}\text{C}$) - Temperature in degrees Celsius; M-N (The temperature at which the total vapour pressure is equal to the external pressure; B (Boiling point of vapour); C (Boiling point of liquid); M (Temperature ($^{\circ}\text{C}$) at which the molar fraction of the vapour phase is equal to the molar fraction of the liquid phase); N (Temperature ($^{\circ}\text{C}$) at which the molar fraction of the liquid phase is equal to the molar fraction of the vapour phase); P (Temperature ($^{\circ}\text{C}$) at which molar fraction of vapour phase is equal to 0.5); G (Corresponding molar fraction at which the vapour phase is equal to the molar fraction of the liquid phase); Z

(Corresponding molar fraction at which the liquid phase is equal to the molar fraction of the vapour phase); (Snyman, 2005).

2.2.2 Pot stills

Studies conducted by Carnacini (1989) show that discontinuous distillation (batch or pot still distillation) enhance the aromatic quality of the original wine, while continuous distillation (column still distillation) results in a less aromatic end product.

Pot stills are used for what is known as double or batch distillation. Batch distillation is a term used for a distillation that entails distilling a mixture to obtain different component fractions. This is done before the distillation still is charged again with more mixture and the process is repeated again (Bernot et al. 1990). These stills are composed of copper, and the reason for this is that this metal is a good conductor of heat and it is capable of reacting with any sulphur that is present in the wine to ensure that it is removed effectively.

There are two stages of distillation when batch distillation is used. The first stage entails taking wine and distilling it until the alcohol strength is 28-30% alcohol per volume. This is now known as low wine. Low wine can be stored for a long period of time, as it is protected against microbial spoilage. The second stage is distilling the low wine and collecting it in three fractions. These are known as the heads, hearts and the tails. Each of these fractions contains different amounts and types of compounds. However, it is the heart fraction that is of importance as this is the fraction that is matured. The alcohol strength of the heart fraction ranges from 65-75% a/v. The heads and the tails are carried back into another batch of low wine and redistilled to ensure that all the alcohol is recovered (Gold 1972).

Not all the heads and tails are carried back to be redistilled, this will ultimately depend on the distiller as too much of these fractions can lead to a build up of undesirable aromas associated with cereal-like flavours. It has been found that an excess in tail fraction in the heart leads to an increase in ethyl lactate and 2-phenyl ethanol, and increase in the head section inclusion in the heart leads to increased short chain ethyl esters, aldehydes and higher alcohols (Cantagrel 1988). Figure 1.2 is a diagram representing a pot still which is used to produce Cognac and brandy in the distillation technique known as *Alambic Charentais*. This technique is also batch distillation (Leaute 1950).

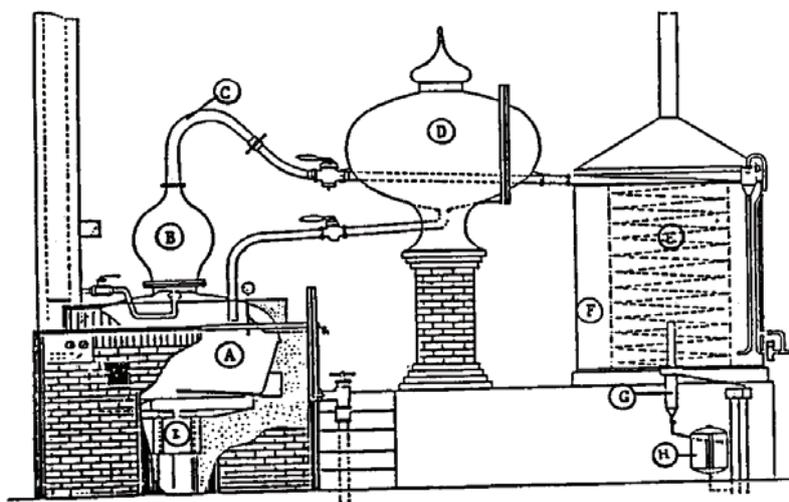


Figure 1.2 *Alambic Charentais* style Pot still. A -Boiler; B - Pot still head; C -Swan's neck; D -Reboiler; E-Copper coils; F -Condenser; G -Collector; H -Distillation safe (Leaute 1950).

Differences in shapes of the pot still head and swan's neck will alter the composition of the final distillate and is a crucial factor to consider when deciding to distil (Leaute 1950; Carnacini, 1989).

The choice of the distillation technique using either pot still or column still distillation is dependent of the style of final product. Changes of the distillation system greatly alter the volatile compounds found in distilled beverages. In the case of cider brandies, higher molecular weight alcohols were recovered better when using a rectification column than in pot still (batch) system, this was opposite for the esters produced (Ferrari et al. 2004). Therefore it is important to have a good understanding of the production process, as the distillation technique is of fundamental importance in influencing the organoleptic properties of the end product.

2.1.5 Influence of maturation on the final distillate

A large number of flavour compounds found in distilled beverages are a result of the slow chemical reactions that occur in the aging process during the maturation in barrels (Nykanen, 1986). Distillates are normally matured in oak barrels either American or French oak, of which the most employed species are *Quercus rubor* and *Quercus petraea* (Madrera et al. 2003). During the maturation process the oak imparts a specific flavour and colour and is a crucial element in the production of brandy (Robinson 1994). Studies conducted by Madrera et al. (2003) show that distillates aged in French oak compared to American oak, have a higher complexity. Phenolic acids are said to increase during the aging process, but the furanic compounds show no change.

Further studies conducted by Panosyan et al. (2001) where the composition of different ages Cognac's was determined showed that there was an increase in compounds such as diethylacetal and carboxylic acid esters, whilst the concentration of alcohols decreased. The explanation of the formation of these compounds can be explained by the nonenzymatic oxidation of alcohols and aldehydes to acids, which is then followed by their esterification in ethanol with the formation of ethylates and acetals from aldehydes. Also there is an increase in isoamyl acetate, ethyl acetate and butanal.

Very old Cognacs are said to develop a distinct "rancio" character (Watts et al. 2003). Depending on the cognac age, this character can be considered either negative or positive. Studies conducted by Watts et al. (2003) show that methylketones such as 2-heptanone and 2-nonanone are responsible for this character and can be used as a quality indicator for brandy. Methylketones develop as a result from the free fatty acid esters present in the distillate which are also said to increase during aging. It appears that ketone concentration is a reasonably reliable indicator of age and therefore value of cognac.

Therefore it shows that aging and long storage can lead to an improved chemical composition of cognacs, by reducing the concentration of negative compounds and increasing the amount of positive compounds that characterize its flavour.

2.3. FACTORS AFFECTING THE DISTILLATION OF VOLATILE COMPOUNDS

Wine is made up of mainly water and alcohol along with certain volatile compounds (Leaute 1950). It is not only their vapour-phase equilibrium that will determine the way in which these volatile compounds will ultimately distil but also their boiling point, their relationship with alcohol or water, and lastly, the variation of alcohol content in the vapour during the distillation.

These volatile compounds are mainly polar in nature and therefore they are more soluble in water. There are a number of possibilities that are present with regards to the relationship that the volatile compound has with alcohol of water namely:

Classification no 1, the compound is completely or partly soluble in alcohol and will distil when the vapour is rich in alcohol.

Classification no 2, the compound is soluble in water and will distil over when the vapour is low in alcohol.

Classification no 3, the compound is soluble in both and will distil over the entire distillation.

Classification no 4, the compound is not soluble in water, but the water vapour will carry it through to the final distillate (Leaute 1950).

Compounds that are completely or partially soluble in alcohol have low boiling points and will be the first to distil over as the concentration of alcohol is high at the beginning of the process. As the distillation continues, the compounds that are more soluble in water will start to be recovered as they have a higher boiling point and are more polar. The mixture contains less and less alcohol as the distillation continues and as time goes on more of the alcohol is recovered (Faundez et al. 2004). The Boiling point of a certain compound together with the solubility in both water and alcohol has a significant effect on the way in which these compounds distil over into the final distillate, which will influence the sensory outcome and profile of the unmaturred Pot still brandy.

Knowledge on how each compound reacts in the distillation process is valuable as this ensures the correct timing involved in the separation of unwanted compounds in the final product, thereby enabling the distiller to have control over the process and to ensure the production of optimum quality brandy (Saco et al. 2006).

2.3.1 Azeotropes and phase equilibrium

The Greeks defined the term azeotrope as “non-boiling by any means” (Greek: *a-non, zeo-boil, tropos-way/mean*) and represents a mixture of which two or more components where the equilibrium vapour and liquid composition are equal at a given pressure and temperature (Hilmen 2000). Azeotropy is characteristic of the nonlinear phase equilibria of mixtures that have strong molecular interactions and are formed due to the differences in the intermolecular forces of attractions among the mixture of components. Azeotropes form a non-ideal system and deviate from the norm which is Raoult’s law.

Raoult’s law states that the vapour pressures of an ideal mixture, is a function of the composition of the ratios of the constituents (Snyman 2005). Knowledge of this law and how mixtures behave is important when considering distillation, as azeotropes are not ideal mixtures and tend to deviate from the norm of Raoult’s law.

This deviation from the norm can either be positive or negative depending on the attractions between the components. For the mixture to form a positive azeotrope the components “dislike” each other and the attraction is stronger between identical molecules compared to between different molecules. This will cause the mixture to form a minimum-boiling azeotrope and heterogeneity. With the case of a negative deviation, the component “like” each other and form a stronger bond between different molecules. This may cause the formation of a maximum-boiling azeotrope. Even though the above mentioned explanation is used to explain binary models, (Moore et al. 1962; Hilmen 2000) there are many mixtures that are ternary models, but due to the

fact that in the case of alcohol distillation the main azeotrope is a binary one, this is what will be focused on.

An example of a positive azeotrope is a mixture that contains ethanol and water. This azeotrope is also known as a minimum boiling mixture (Hilmen 2000) and is known as homogenous as only one liquid phase is present. Due to the fact that water boils at 100°C and alcohol at 78.4°C, this mixture is a binary azeotrope and will therefore boil at the minimum boiling point of the combined temperatures i.e. 78.1°C. It is important to note that distillation cannot separate the constituents of azeotrope mixtures, thus making it an important concept to understand. When a mixture of two solvents is boiled and the vapour condensed, it changes the state of the compounds. If in this system the pressure is kept constant, then the only variables that can change are the temperature and the composition.

Figure 1.3 shows an example of a positive azeotrope of compounds X and Y. The bottom line shows some boiling points of the various compounds, and below this line is where the mixture is entirely in the liquid phase. Above this line the mixture is in a vapour phase. Between these two lines the mixture is both in the liquid and the vapour phase. At the point where these two lines cross each other is the azeotrope of the mixture. Note that repeated distillation can never produce a distillate that is richer in constituent X than the azeotrope.

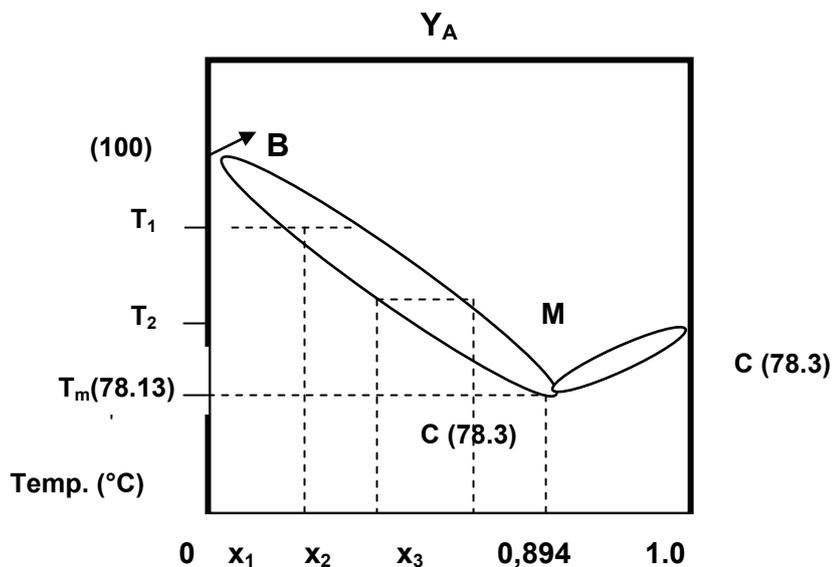


Figure 1.3 Diagram of a positive azeotrope. X_A (Molar fraction of A in the liquid phase); Y_A (Molar fraction of A in the vapour phase); T (°C) (Temperature in degrees Celsius); T_m (Temperature at which the positive azeotrope forms); B (Boiling point of water); C (Boiling point of alcohol); M (The temperature at which the azeotrope is formed). (Snyman 2005).

2.3.2 Reflux

Reflux is the term used to describe the amount of vapour that condenses and runs back into the pot still to be reboiled. If the still head has a surface area that is either too large or long, then the vapours will cool and condense and run back down into the original liquid inside the pot still (Kister 1992).

This is important as the vapours that have condensed and run back down will be boiled again. This reflux in the system ultimately influences the amount and types of compounds that will distil over into the distillate or un-matured Pot still brandy (Hampel and Hawley 1982).

Studies by Leaute (1950) show that the shape and the volume of the pot still head that is used will influence the separation, selection and concentration of the different volatile compounds found in the final product. A brandy that is made from a pot still that has a longer still head will be less flavoursome and contain less of the more full-bodied compounds, such as the longer chain fatty acids.

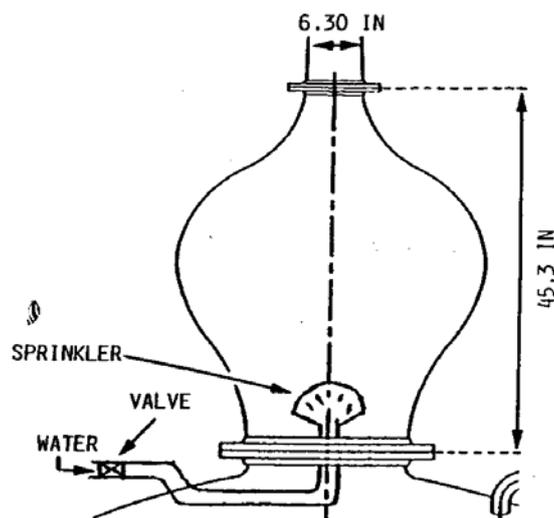


Figure 1.4 represents the original Pot still head of the Prulho Pot still which is used to produce Cognac. This Pot still is “onion” shape and has a larger surface area and therefore has more reflux. Brandies produced by this Still head are more aromatic and contain larger amounts of flavour compounds and is consequently more aromatic (Leaute 1950).

Further studies by Madrera (2003) show that pot stills with a small surface area generate poorer reflux during the distillation process as they do not allow for the recondensation of water into the pot and therefore the enrichment of volatile fraction in ethanol. This results in the distillate having an alcoholic content that was not as high as that obtained with other distillation systems.

In the production of Cognac, a still known as *Alambic Charentais* is used. This still consists of a boiler (*cucurbite*), still head (*chapiteau*), swan’s neck (*bec*) and a condenser. The height of the swan’s neck and the larger the still head in relation to the boiler will inevitably increase the rectification and therefore contribute to a smoother brandy with less character. Distillation technique is the same as in a normal pot still with the heart fraction being between 65-75% a/v (Faith 1992).

One can therefore see that the shape and the size of the components of the pot still, and specifically the still head and the swan’s neck will definitely influence the outcome of the distillate due to the fact that the reflux will change in the system

2.4 VOLATILE COMPOUNDS IN DISTILLATES

In earlier studies, it was believed that the flavours of alcoholic beverages were only made up of small amounts of compounds. However over 1300 different volatile compounds have been identified and if the non-volatile components are also included, then the amount would probably double (Nykanen 1986).

Both the major and minor components found in brandy are responsible and are essential for the total brandy aroma. However, fusel alcohols, fatty acids and their esters usually are more dominant than carbonyl phenolic, sulphur and nitrogen compounds (Jounela-Eriksson 1981) and play an important role in the overall aroma profile and quality of brandy.

2.4.1 Esters

Esters are abundant volatile constituents of different foods and beverages such as fruits and fruit juices, olive oil, beer, wine or distilled alcoholic beverage and were thought to be produced due to the esterification between alcohol and free acids in a fermentation medium. It was however shown that esters are formed as a part of the biosynthetic process, and their formation requires the activation of the fatty acid moiety of acyl-CoA compounds, which then combine with alcohols of the medium, of which ethyl alcohol predominates. There is strong evidence that suggests that the main source of ester formation is yeast growth (Guymon 1969).

Their presence strongly influences the bouquet of the wine and distillate and are said to increase in concentration during aging. Therefore their final amount found in brandies, are not a good estimation of the amounts originally found in the distillate. This makes them an important chemical group to investigate (de Villiers 2005).

Studies conducted by Von Adam et al. (1996) show that the amounts of esters vary between different distillates. French distillates contain 385 mg/L and Italian distillates contain higher amounts of total esters of approximately 406 mg/L, whilst German distillates contain the lowest with the concentration of esters in the distillates being only 10%. Of the esters found in distillates, the main ones that influence the total ester concentration are ethyl acetate and ethyl lactate. These two compounds make up approximately 90% of the total ester. Due to their low threshold values, low boiling esters from acetic and butanoic acids contribute to the main odour evaluation of the spirit together with ethyl esters from other acids as well as carbonyl compounds (Ferrari et al. 2004).

Figures 1.5 and 1.6 show the expected amount of certain esters during the first and second distillation (Piggott 1983).

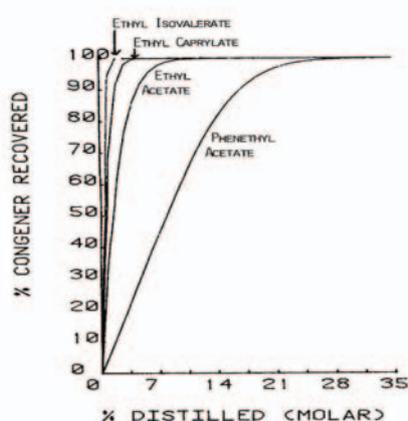


Figure 1.5 Recovery of some esters during the first distillation (Piggott 1983).

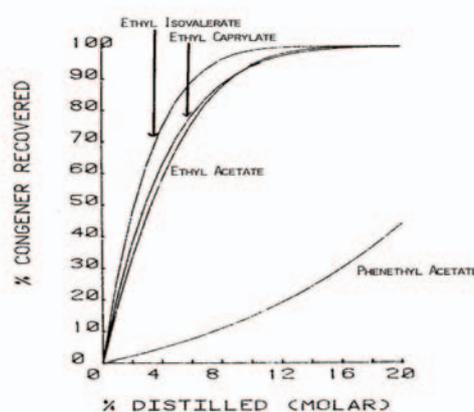


Figure 1.6 Recovery of some esters during the second distillation (Piggott 1983).

2.4.1.1 Ethyl acetate

Ethyl acetate forms the most important and main ester in wine and un-matured pot still brandy. This compound in small amounts can impart a fruity floral aroma, while in higher concentrations such as 150-200 mg/L in wine usually indicate microbial spoilage and infection from acetic acid bacteria but can also be influenced by the distillation process (Steger and Lambrechts 2000). Studies by Ferrari et al. (2004) also show that ethyl acetate is responsible for solvent, alcohol odour notes. Ribereau-Gayon et al. (2000) reported the threshold value for ethyl acetate is 160 mg/L. Since acetic acid and ethanol are the dominating acid and alcohol in the wine, ethyl acetate is produced in large amounts due to the reaction between these two compounds and normally constitutes 50% of all the esters (Satora and Tuszynski 2008).

This compound is found mainly in the heads fraction of the distillate when using the *Alambic Charentais* method for Pot still distillation, and so if the time taken for this fraction is increased it also limits the amount found in the distillate (Von Adam et al. 1996). The amount of ethyl acetate in the un-matured Pot still brandy can be decreased by controlling and maintaining good storage of the base wine prior to distillation to prevent spoilage or contamination. Postel and Adam (1980) mention that there should be a minimum of 175 mg/L and a maximum of 595 mg/L ethyl acetate present in wine distillates.

2.4.1.2 Ethyl lactate

Ethyl lactate is said to be a compound normally associated with the tail fraction of the distillate and is formed mainly when base wine is stored for long periods and is subjected to malolactic fermentation which is considered spoiled (Steger and Lambrechts 2000). The levels will vary within distillates, with concentrations lower than 154 mg/L being favourable whilst concentrations reaching above 455 mg/L will impart a negative aroma and flavour in the distillate (Cantagrel et al. 1992).

2.4.1.3 Ethyl esters of caproic, caprylic, capric and lauric acid.

These ethyl esters are formed from their corresponding fatty acids, and are quantitatively dominant and are generated through fermentation. Studies conducted by Guymon (1969) show that if the wine is distilled together with the yeast lees it will result in a brandy distillate with more ethyl esters and their fatty acids, yeast growth is the primary source of ester formation. The type of distillation technique employed will also influence the amount of ethyl esters found in the product, it is shown that continuous distillation leads to an increase in ethyl esters compared to those distillates produced by pot still distillation due to the fact that during pot still distillation the alcohol concentration may be too low at any given time to permit significant ester formation. It is recommended that fresh healthy lees is used together with the wine for the distillation purposes to ensure the distillates do not contain any organoleptic defects. These ethyl esters are amphiphilic and are more soluble in ethanol than in water and may form agglomerates in aqueous ethanol solutions if diluted (Conner et al. 1994).

Salo et al. (1972) identified ethyl esters of fatty acids, those with even carbons between 6 and 12, to be major contributors to whisky flavour. Jounela-Eriksson (1981) reported that if ethyl esters are added or removed from the spirits it results in a negative effect on overall odour intensity. Postel and Adam (1980) and Schreier et al. (1978) also show that ethyl esters can be used to analytically differentiate between Cognacs and other groups of grape brandies. For example Cognacs contain

less short-chained fatty acids (C3-C5) compared to the maximal values of esters of long-chain fatty acids (C10-C14).

Despite high boiling points, fatty acids/esters appear early on in the distillate obtained and contribute immensely to the aroma and flavour of the distillate (Simpson 1971). These ethyl esters of caproic, caprylic, capric and lauric acid exhibit characteristic fruity and flowery odour notes and form the largest group of flavour compounds (Ferrari et al. 2004). Caproate is fragrant and has an odour similar to that of banana oil; caprylate is more pungent and less fragrant and resembles crude grape fusel oil; caprate is less intense and milder with fatty tones and finally laurate is the least aromatic and had a waxy candle like odour (Guymon 1969).

The amount of ethyl esters found in distillates varies and can range from 2.1 to 70 mg/L, although it is recommended that the total concentration of the long chain ester (C6:C16) should be in the range of 2.0 mg/ mL A (14 mg/L) (Von Adam et al. 1996). It is important to use these quantitative measurements together with a sensory evaluation. Studies conducted by Ferrari et al. (2004) where the association between the chemical analysis and sensory analysis was measured and the compounds identified in freshly distilled cognac were thought to display the following descriptors. Table 1.1 shows the volatile compounds in Cognac and their corresponding odour notes.

Table 1.1 Volatile compounds found in Cognacs which are responsible for specific odour notes (Ferrari et al. 2004).

Compound	Odour notes
Ethyl acetate	Solvent, alcohol
2,3-Butanedione	Butter, pastry
Ethyl butyrate	Fruity
2 and 3-Methylbutyl acetate	Banana, pear
2 and 3-Methylbutan-1-ol	Fruity, cacao, sweat
Ethyl hexonate	Strawberry, anise
2-Phenylethyl acetate	Rose
2-Phenylalcohol	Rose
Nerolidol	Dry wood, hay
n-Hexan-1-ol	Green, flowery
β -Citronellol	Hay, tea, dry, spicy,
B-Damascenone	Cooked fruit
Methyl salicylate	Cooked fruit

2.4.2 Volatile fatty acids

Fatty acids and their ethyl esters are generated in fermentation and are passed through the distillation process into the resultant distillate. Only 1-10 carbon atoms are volatile enough to distil over, therefore the composition of the volatile fatty acids in distillates should not vary greatly from the raw material. It can be stated that the formation of acids occurs in the same way for most cases so the raw material does not exert a major influence upon the composition of acids (Nykanen 1968). The only other source of fatty acids is due to the thermal degradation and autolysis of yeast cells during the distillation process.

Increases in concentrations of fatty acids in distillates are a result of wine that is distilled together with the yeast lees, especially those fatty acids with even carbon atoms, (C2-C10) which are products of biochemical metabolism (Von Adam et al. 1996). Along with the even numbered fatty acids, the main volatile acid found in distilled beverages is acetic acid. This acid constitutes 40-95% of the total volatile acids in whisky, 50-75% in Cognac and brandy, and for rum 75-90%. If acetic acid is disregarded then capric acid is the largest component which varies between 20-45%,

after this in descending order is caprylic, lauric and caproic acid (Nykanen, 1968). Octanoic and decanoic acids are also prominent and make up 30%, followed by hexanoic acid.

Studies conducted by Nykanen (1968) show that out of the three brandies evaluated, caprylic and capric acid are the main components and the longer chained acids such as C12 are found only in low percentages. However in whisky myristic, palmitic and palmitoleic acid are the most abundant long-chained fatty acids.

2.4.3 Alcohols

2.4.3.1 Methanol

Methanol imparts a cooked cabbage odour in spirits and has a threshold value of 1200 mg/L (Ribereau-Gayon 2000). High amounts of methanol can be hazardous for humans to consume and therefore strict control of the amount found in alcoholic beverages should be managed. Methanol is produced by the degradation of pectin's found in the raw materials by enzymes known as pectinases and it is their contribution that determines the level. Methanol will distil over mainly in the head section of the distillate; this is why the head fraction is collected separately from the heart fraction to ensure that most of it is eliminated (Porto 1998).

2.4.3.2 Higher alcohols

Higher alcohols or commonly known as fusel oils are alcohols that contain more than two carbon atoms and therefore have a higher molecular weight and higher boiling point than ethanol. They have an important aromatic effect in wines and especially distillates as they are found in higher concentrations (Steger and Lambrechts 2000).

These compounds are produced as a by product from yeast due to their metabolism of sugars and amino acids and are secreted into the fermenting medium (Ayrapaa 1990; Lurton et al. 1995; Riponi et al. 1996). This production of higher alcohols depends on the raw material and the yeast employed, and during the distillation processes the low molecular-weight alcohols increase and the high-molecular-weight alcohols decrease due to the effects of differing volatility during distillation. Figures 1.7 and 1.8 show the expected outcome of some alcohols during the first and second distillation process (Piggott 1983).

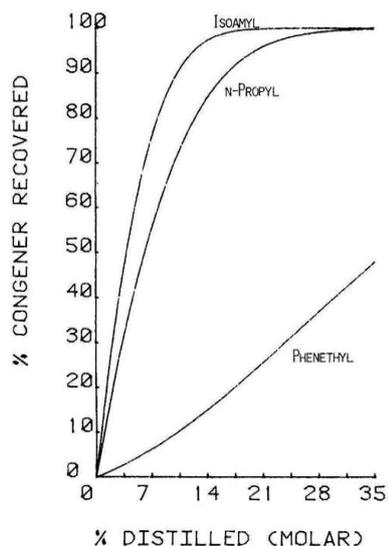


Figure 1.7 Recovery of some alcohols during the first distillation (Piggott 1983).

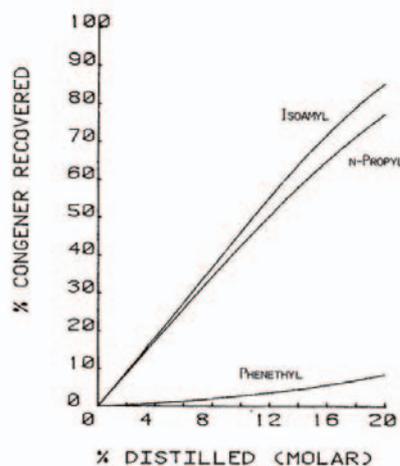


Figure 1.8 Recovery of some alcohols during the second distillation (Piggott 1983).

Major higher alcohols found in wine in order of amounts produced are, isoamyl alcohol, active amyl alcohol, isobutyl alcohol and n-propyl alcohol (Jounela-Eriksson 1981).

Studies conducted by Boscolo et al. (2000) indicate that when looking at the higher alcohol content of wine and spirit, the most important one to consider is isoamyl alcohol as this higher alcohol if in large concentrations can render the product unpleasant. Due to the fact that the distillation technique enhances the amount of higher alcohols found in the distillate, it is important to monitor these levels. Ideally the product should be high in esters, low levels of higher alcohols and have high concentrations of 2-phenethyl acetate (Boulton et al. 2000; Chatonnet et al. 1993).

Average concentrations vary in different distilled beverages. For example in Brazilian Sugar-Cane Spirit, the limit values for total higher alcohols are in the range of 210 mg/L. Studies show that unmaturing spirit beverages will have lower concentrations of higher alcohols and esters because the maturation process will lead to an increase in the production of these compounds (Boscolo et al. 2000). In the whisky industry concentrations vary greatly between different types of whiskies, and in fact the ratio of active amyl alcohol and isoamyl alcohol has been used as a criterion for differentiating between different alcoholic beverages. This ratio was found to be an average of 0.20 for rums, 0.22 for brandies and 0.34 for whiskies (Piggott 1983).

Brandies can be grouped into different categories according to their levels of higher alcohols. Guymon 1972 states that brandies that contain 420-525 mg/L are considered light, those with 525-630 mg/L as medium and those greater than 630 mg/L to be a brandy that is heavy bodied.

Studies conducted by Scheirer et al. (1978) show that fusel oils contribute towards the quality of the alcoholic beverage, and if found in dilute amounts can add complexity and interest to the beverage.

2.4.4 Carbonyl compounds

2.4.4.1 Aldehydes

Aldehydes are said to be the most volatile compounds found in alcoholic beverages and are formed during the fermentation process (Nykanen 1986). These compounds are the main compounds in the biochemical reaction when the yeast uses amino acids and sugars to produce fusel alcohols.

Of the carbonyl compounds, acetaldehyde is the major component and constitutes approximately 90% of the total aldehyde content in alcoholic beverages. The amounts of acetaldehyde vary greatly and relatively large concentrations are found in whisky, cognac, brandy and rum. Guymon (1972) show that commercial brandy distillates are low generally in their aldehyde concentration with a mean score being in the range of 11 mg/L at 50% (5.5 mg/L) alcohol, but low quality brandy have shown amounts as great as 264 mg/L at 50% (132 mg/L) of acetaldehyde. Table 1.2 indicates the aldehyde content in some alcoholic beverages (Guymon 1972).

Table 1.2 Aldehyde content (mg/L 50% alcohol) in distilled alcoholic beverages (Guymon 1972).

Type of distilled alcoholic beverages	Aldehyde content (mg/L 50% alcohol)
American whiskey	a.v ^a 43
Bourbon whiskey	20-60
Canadian whiskey	10-36
Irish whiskey	20-70
Scotch whiskey	20-110
Wine distillate	19-55
Brandy	63-308
Cognac	a.v 105

^a a.v-Average aldehyde content (mg/L 50% alcohol).

Another aldehyde to consider is acrolein. Studies done by Kahn et al. (1968) where the low boiling compounds found in head fractions were analysed using gas chromatography found acrolein to be present. Acrolein is responsible for a “peppery” smell associated with some whiskies and is produced by bacteria from the compound known as glycerol.

According to Soumalainen and Ronkainen (1968) 2, 3-butanedione (diacetyl) is a ubiquitous flavour component in distilled beverages. This compound is particularly important in distilled beverages as its sensory threshold value in beer is said to be in the range of 0.15 ppm. In small quantities it can resemble a “butterscotch” flavour. Scotch whisky and cognac contain an average of 0.16 mg/L of 2, 3-butanedione, and a Martinique rum was found with a concentration of 4.4 mg/L. It has been shown that rectification can decrease the aldehyde concentration in distilled beverages to some degree. Studies conducted by Dieguez et al. (2005), also show that in the production of Galician orujo spirits, if the grape pomace is stored in the presence of oxygen, there is a definite increase in acetaldehyde. Figures 1.9 and 1.10 show the recovery of some aldehydes during the first and second distillations (Piggott 1983).

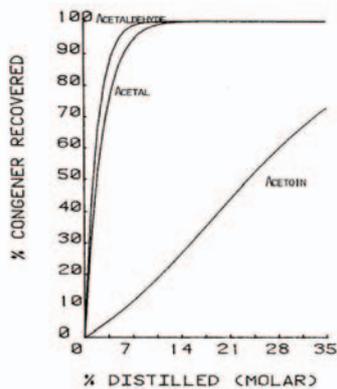


Figure 1.9 Recovery of some aldehydes during the first distillation (Piggott 1983).

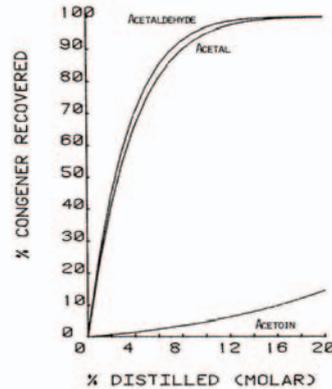


Figure 1.10 Recovery of some aldehydes during the second distillation (Piggott 1983).

2.4.5 TERPENOIDS IN DISTILLATES

Terpenoids in distillates are formed in the grapes and during the fermentation period, and pass into the distillate through the process of distillation (Egorov and Rodopulo 1994). During the aging process linalool is esterified and forms linalyl acetate therefore decreasing the amount of linalool present in the distillate. Terpenoids may have an important contribution by adding “floral” and “fruity” notes to whiskies and the norisoprenoids can impart a “camphor” or “honey-like” note. Studies conducted by Ledauphin et al. (2004) in which a comparison between freshly distilled cognac and calvados was made, showed that there were varying amounts of terpenic and norisoprenoidic derivatives in the distillates. β -Damascenone was found in the distillates and it is said that distillation increases the amounts of this compound. Compounds such as α -terpineol, linalool and its oxidation derivatives are commonly found in distillates but presence of β -citronellol and farnesol is limited.

It was found that there are differences between the terpenic derivatives found in Cognac compared to those found in Calvados. The terpenic derivatives that are specific to cognac are rose oxide, myrceol, γ -terpineol and β -terpinel, and those found in calvados are 4-terpineol, geraniol.

Studies by Ferrari et al (2004) show that besides the volatile compounds such as fatty acids, esters and fusel alcohols, terpenoid compounds which are found in distillates can also greatly influence the organoleptic profile of the product. Compounds like nerolidol is responsible for the “dry wood, hay” odour found in the product. The compound β -citronellol is responsible for the “tea, spicy” aroma. Therefore it is important to qualify and quantify the terpenoid compounds found in the distillates as they may contribute to the profile of the product, and without doing so one can not fully understand the impact on the sensory outcome of these compounds on the final product.

2.5 QUALITY INDICATORS IN BRANDY

Freshly distilled cognac can already contain certain compounds that are assigned to specific odour notes which arise from the distillation process and grapes, but their aromatic quality depends on the association of these compounds together in the mixture, not necessarily an individual compound (Ferrari et al. 2004).

Studies conducted by Cantagrel (1988) show that there are certain limits of the amount that a compound can be within a distillate before it will be considered a defect. There are threshold

values that will affect the sensory perception of the distillate. The taste threshold value for the following compounds are as follows; the amount of ethyl acetate must not exceed 600 mg/L, compounds such as 1-butanol and 2-butanol should range from 6-7 mg/L before it could render the product unsatisfactory. Acetaldehyde is limited to 60 mg/L, ethyl butyrate between 4-5 mg/L and 2, 3-butanediol with an average of 8 mg/L.

Positive indicators for brandy quality include compounds such as isoamyl acetate (0.3-10 mg/L) which display fruity notes in the distillate, the fatty acid ethyl esters are responsible for the floral aroma found in distillates and the value ranges from 10-30 mg/L. Herbaceous characteristics are associated with the compound 3-hexanol, and buttery flavours and aromas are produced from diacetyl and a concentration above 4 mg/L is considered negative.

Infected wines can lead to a defect in the organoleptic properties of the distillate. These infected wines normally amongst others have an increase in ethyl lactate, acetic acid and the fungus known as *Botrytis cinerea* (Cantagrel 1988). Infected wines will exhibit the following characteristics such as a loss of fruitiness, appearance of lactone, sotolon and typical oxidation flavours such as prune and maderized.

Volatile compounds found in the distillate are analysed using gas chromatographic analysis which can ascertain defects such as sourness due to the ethyl acetate and pungency which correlated to the sum of acetal and ethanal (50-200 mg/L). Stagnant flavours and odours can be associated with ethyl butyrate and 1-butanol. Compounds such as acrolein can present a plastic characteristic in the distillate.

Brandies with higher quality also had a lower concentration of esters, fusel oils and aldehydes, whilst still containing higher amounts in total acids. However, the type of acid is important and Quady and Guymon (1973) thus speculated that it was the organic acids that influenced brandy quality.

2.6 SENSORY EVALUATION OF SPIRIT PRODUCTS

2.6. 1 Introduction

Sensory evaluation has been defined as a “scientific method used to evoke measure, analyze, and interpret those responses to products as perceived through the senses of sight, smell, touch, taste and hearing” (Anonymous, 1975). Sensory evaluation consists of a list of techniques that accurately measures the human responses to foods and minimizes the biasing effects that brand identity and other information may have on consumer perception. It is also considered a science of measurement and like other analytical test procedures the concerns are that the results will be accurate, precise, and sensitive whilst avoiding false positive results (Meiselman 1971).

Sensory evaluation can show how in the sensory dimension the competitor’s product differs from yours and these techniques can be used for shelf-life testing, research and product development and how close a new product is to the prototype product (Lawless and Heymann 1995). Consumer perception and ultimately knowledge of the consumer preference can be used to drive the production process for the company’s advantage.

Therefore, sensory evaluation is a critical step in any company’s strategy to determine the quality and authenticity of a product (Jack 2003).

In the sensory evaluation of spirit products one should consider that these products are made up of many different compounds, both volatile and non-volatile. Over 1300 volatile compounds have been identified and if the non-volatile compounds were taken into consideration this number would properly double (Nykanen 1986). The way in which these compounds are derived vary and depend

on many factors which include flavours occurring from the original source, the fermentation procedure, the distillation technique as well as the specific maturation that the product undergoes (Nishimura et al. 1971).

These compounds play an important role in the profile of the spirit product and it is not necessarily the type of compound but the quantity of it and how the compounds interact with each other that will ultimately influence the sensory perception (Steger and Lambrechts 2000). Sensory evaluation should be used together with the chemical analysis of the product so that a correlation can be made between the sensory data and the compounds that play a role in the profile of the product (Jack 2003).

Studies conducted by Ferrari et al. (2004) whereby the odour of freshly distilled Cognac samples were evaluated by nose smelling showed that it was possible to correlate certain descriptors with specific chemical compounds and to therefore determine which compound was responsible for which aroma. This was used to predict what odour notes present in samples are associated with what compound.

The two main categories in sensory evaluation are namely an objective and subjective method. The objective method involves training of a sensory panel. However, the subjective method uses subjects that have not received any formal training. Both of these techniques have different motivations behind the use of them (Lawless and Heymann 1995).

When using Objective testing, the trained analytical panel's evaluations are used to detect and describe the qualitative and quantitative parameters of the product. The panels are used in product development, prediction and research. This technique can be divided into two main tests applications, namely discrimination testing and descriptive testing (Munoz et al. 1998).

Table 1.4 The three main types of test methods that is available for sensory evaluation (Lawless and Heymann 1995)

Class	Question of Interest	Type of test	Panelist Characteristics
Discrimination	Are the products different in any way?	Analytic	Screened for sensory acuity, orientated to test method, sometimes trained
Descriptive	How do products differ in specific sensory characteristics?	Analytic	Screened for sensory acuity and motivation trained or highly trained
Affective	How well are products liked or which products are preferred	Hedonic	Screened for product use, untrained

2.6.2 Discrimination testing

Discrimination testing is a sensory technique that uses qualitative testing. This technique is used to determine if there are differences between samples and once this has been applied, it can be used to decide if further analysis such as descriptive testing should be used to differentiate between the samples. However, the differences between the samples must be small otherwise this form of testing is not valid (Stone and Sidel 1993).

It is important to use this technique due to the fact that it is possible for two samples to be chemically different from one another but this may not be perceived by human perception. There

are three main tests that are applied to the products for sensory evaluation. These include the paired comparison test, duo-trio test and the most common method known as the triangle test (Lawless and Heymann. 1995; Stone et al. 1993).

2.6.2.1 Triangle test

This method is the most well known method when discrimination testing is being discussed. Subjects are presented with three samples, all of which are coded randomly. The purpose of the test is to distinguish which of the three samples is completely different to the other two.

This is a difficult test as subjects have to recall the characteristics of the other two samples before analyzing the final one and then drawing a conclusion (Meilgaard et al. 1991).

2.6.2.2 Duo-Trio Test

In this test, three samples are given to the subject. One of the samples is a reference, and the objective is to decide which of the other two samples corresponds to the reference. This form of testing is normally used when there is an intense aroma or odour associated with the samples (Lawless et al. 1995; Stone et al. 1993).

2.6.2.3 Paired comparison Test

Paired comparison testing is easy to apply and implement. There are two samples provided and the subject must identify which of the samples has more of the designated characteristic. It is important that this characteristic is identified before the test is conducted.

This test is known as a forced choice test as subjects are forced to make a decision and cannot simply state that they choose “neither” of the samples that are presented. (Meilgaard et al. 1991)

2.6.3 Descriptive testing

Descriptive sensory analyses are the most sophisticated tools that are available to the sensory scientist (Lawless and Heymann 1995). These methods were designed to help the sensory scientist to obtain sensory descriptions of products by identifying the underlying process variables and to determine the sensory attributes that indeed play a role in acceptance. This form of analyses is used in many different situations where a detailed specification of a single product is desired.

The major approaches and philosophies of descriptive analysis techniques which are used to evaluate alcoholic beverages involve using the techniques known as the Flavour profile method and Quantitative descriptive analysis (Jounela-Eriksson 1981).

2.6.3.1 Flavour profile method

This technique was developed by Arthur D. Little in the late 1940s to describe complex flavour systems (Meilgaard et al. 1991). This method involves obtaining a consensus from trained panel members by developing a vocabulary that best describes the product. It also involves the analysis of flavour and aroma characteristics for a product regarding their intensities, aftertaste and order of appearance by a panel consisting of trained judges.

Another example is in the whisky industry where a tool is used to assist in the profiling of whisky samples by using the whisky terminology lexicon. A lexicon is a group of descriptors that have been formed by using sensory descriptive analysis technique and is used to describe the flavour of a product or commodity (Drake and Civille 2002). This lexicon is also used to produce a profile of the whisky product (Table 1.5) (Shortreed et al. 1979).

Table 1.5 A Whisky lexicon (Shortreed et al. 1979).

1 st tier term	2 nd tier term	3 rd tier term
Nasal effects	Pungent Prickle Nose-warming Nose-drying	Peppery
Phenolic	Medicinal Peaty Klippery	TCP, Iodine Smokey, mossy Guaiacol, burnt wood
Feints	Leathery Tobacco Sweaty Stale fish	New cow hide, meaty Fresh and stale tobacco Beeswax, piggery Scorched plastic
Cereal	Cooked mash Cooked vegetables Toasted Malt extract Husky	Maize cooker, cooked potato skins Boiled corn Burnt toast, coffee, cocoa - Chaff-like
Aldehyde	Hay-like Leafy Floral	Dry hay, herbal Greens leaves Geraniums, green tomatoes
Estery	Fragrant Fruity Solvent	Perfumed, rose-like Banana, pear drop, i-amyl acetate Paint thinners, ethyl acetate
Sweet associated	Glycerin-like Honey-like Vanilla-like	- - Custard powder, treacle
Woody	New wood Developed extract Defective wood	Sap-like, pine-like Ethyl alcohol, walnut-like Musty, sour associated
Oily associated	Nutty Buttery Fatty Rancid	Benzaldehyde Diacetyl, creamy Soapy, mutton fat
Sour associated	Sickly Cheesy Vinegary	- Lactic Acetic acid
Sulphury	Stagnant Coal-grassy Rubbery Cabbage water	- - New rubber Dimethyl sulphide

Table 1.5 A Whisky lexicon (Shortreed et al. 1979) (continued).

Stale	Metallic Blotting paper Musty Earthy	Inky, tinny Wet filter sheets Mouldy, damp Damp soil
Primary tastes	Sweet Sour Salty Bitter	(Reference-Sugar n water Citric acid in water Salt in water Caffeine)
Mouthfeel effects	Mouth coating Astringent Mouth warming	Oily-feel Mouth drying Mouth prickle, alcohol burn

2.6.3.2 Quantitative descriptive analysis

Quantitative descriptive analysis (QDA) was developed to correct some problems that developed with the Flavour profile analysis (Stone and Sidel 1993). One of the problems that developed was the lack of statistical information. For that reason QDA was developed as this method relies heavily on statistical analysis to determine the procedures and terms as well as the panellists to be used in the analysis (Meilgaard et al. 1991).

In contrast to Flavour profile the data is not generated through consensus discussions, the panel leaders do not play a role and unstructured line scales are used to rate the intensities of the attributes that are generated (Figure 1.12).

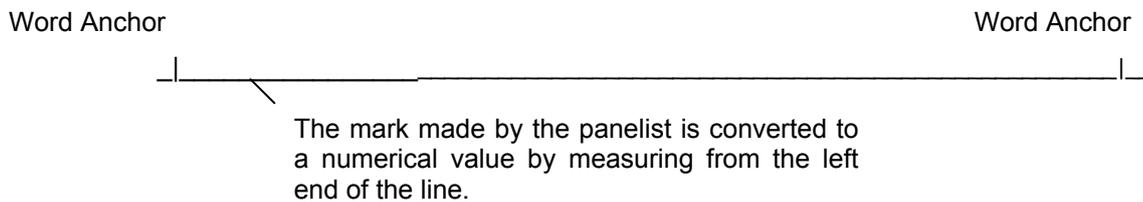
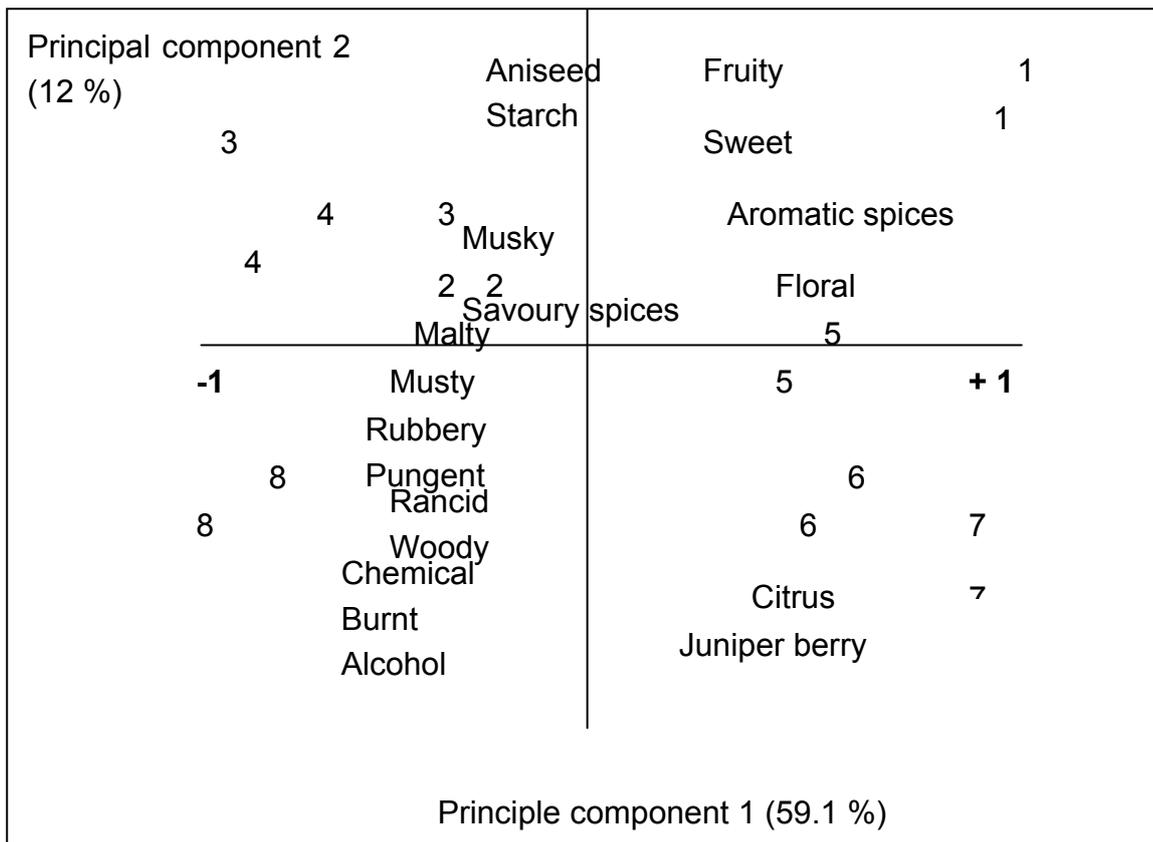


Figure 1.12 An example of the QDA graphic line scale (Lawless and Heymann 1995).



The resulting data can then be analyzed statistically through analysis of variance and multivariate techniques. PCA (Principle component analysis) is a multivariate statistical technique used to determine the relevant power of each attribute. One can see what the meaning of each term is and how it is used (Mc Donnell 2001).

Figure 1.13 indicates a PCA that was generated after the third QDA session was conducted. Eight samples of different distilled beverages were analyzed and one can clearly see that by using PCA some descriptors rank the same between samples, but others are significantly different. Sample 1 is highly correlated with descriptors such as fruity, sweet and aromatic spices. Samples 2, 3 and 4 are associated with attributes such as musky, malty and aniseed starch. Samples 5, 6 and 7 are linked to attributes such as citrus and juniper berry. And finally sample 8 is associated with attributes such as rubbery, rancid and chemical (MC Donnell 2001).

Another study conducted by Zamora and Guirao (2002) whereby Chardonnay wines were evaluated using Quantitative descriptive analysis, showed that this technique was indeed beneficial as descriptors generated were able to be statistically analysed by using PCA. Grouping of samples was produced using PCA and therefore one was able to differentiate between samples and the attributes that were correlated with.

Therefore when applying QDA it is important to decide which type of descriptive terms should be generated that best describe the product. For example, when dealing with whisky, descriptive terms that are related to maturation are the most important (Shortreed et al. 1979).

2.6.4 Senses

Sensory evaluation has two sources of variation (Duerr 1984). Firstly the product is analyzed for chemical composition such as pH or quantity of volatile/non-volatile compounds. The second source that is used to evaluate samples is the human instrument known as the sensory judge.

Together with his/her experience, actual disposition, and certain senses these can be used to evaluate the product. These senses for the purpose of evaluation of spirit products include sight, taste and smell. It is important to understand the concepts of these senses to be able to use them effectively in sensory evaluation (Meilgaard et al. 1991).

2.6.4.1 Visual perception

Visual perception seems to be highly important as it is the initial assessment of quality regarding the product. Some scientific studies show that colour definitely influences the perception of the other attributes, such as aroma, flavour (Lawless and Heymann 1995).

Author Jack (2003) found that the number of correct identifications of flavours decreased when the sample possessed an atypical colour.

Therefore, the visual perception of the product can play a major role in the evaluation and can lead to some biased results (Jack 2003). One can clearly see that correct sample presentation is of crucial importance when assessing products.

2.6.4.2 Taste

The human tongue consists of many taste buds that contain receptors. These receptors pick up certain stimuli and transmit them to the brain where they are interpreted as either sweet, salty, bitter, sour or “umami” (Jackson 2000).

There are many factors that influence the taste reception on the tongue. Firstly temperature can play an important role. If the temperature is too low it can reduce the sensitivity of the individual to sweetness and bitterness (Thorngate 1997). Secondly, some people can possess a genetic trait that does not allow them to perceive a certain sensation, for example sweetness or bitterness. Acuity loss is another factor that influences taste perception; this is a result of old age or health reasons (Lawless and Heymann 1995).

All of these parameters can have a large effect on the final perception of the product and the way in which it is perceived. This is why it is of fundamental importance that the person tasting and evaluating the sample does not possess any deficiencies (Jackson 2000)

2.6.4.3 Mouth-feel

Astringency is not a taste, but instead a touch sensation. It has been defined as “the complex of sensations due to shrinking, drawing or puckering of the epithelium as a result of exposure to tannins or polyphenols”(Bakker 1998).

Both bitterness and astringency are induced by related compounds, but have very different effects. Astringency can have a cumulative effect with an increase of sampling and is the slowest in-mouth sensation to develop (Jackson 2000).

Another criterion that is used to evaluate spirits is Alcohol burn. Due to the increase in ethanol concentration in spirits, it brings about a burning sensation that is noticeable at the back of the throat (Jackson 2000).

Spirits are evaluated according to astringency and alcohol burn as it is important in defining the character of the product (King et al. 2003).

2.6.4.4 Odour

Odour is perceived through the nasal cavity, and this is used to detect the threshold value of a certain compound. The threshold value is that value at which 50% of population can perceive the compound (Lawless and Heymann 1995). The odour of the spirit is highly subjective and variations can occur between individuals. Repeated sampling and training of the subject can lead to less variation and more consistency (Jack 2003).

Many industries rely on analyzing samples solely on aroma. The whisky industry uses a technique called nosing, where the aroma of the sample is evaluated to measure the quality of the product (Jack 2003). Another example where nosing is used is where freshly distilled Cognac is analyzed for quality to establish aromatic profiles (Ferrari et al. 2004). Further studies conducted by Peña y Lillo et al. (2005) also show that sensory evaluation using nosing whereby a profile of the product is produced is far more beneficial as it lead to a decrease in panel fatigue due to less sampling.

Studies by Ferrari et al. (2004) clearly show that this technique was a better form of evaluation for cognac samples as the aroma of cognac was more important in classifying the product than the flavour.

The objective of the company's strategy will ultimately determine the technique employed to evaluate the product.

2.7 CONCLUSION

The production of brandy is a complex process which is influenced by many different factors. It is of utmost importance for a company to produce a product that is consistent and of good quality thereby making it crucial to understand and control those factors that influence this process. These factors include, the geographical origin, cultivar, vinification process, fermentation, maturation as well as the distillation process. The distillation process and the type of apparatus used for the purpose of distillation is a key factor in the production of brandy as this influences the way in which the compounds will distil.

Brandy is made up of many different compounds, both volatile and non-volatile. The quantity and the type of these compounds and the way that they interact with each other will greatly influence the sensory profile of the product. Sensory evaluation is an important tool used to evaluate brandy as this is used to maintain quality within the product and to develop consumer driven production. The main sensory evaluation technique used in the industry today for the evaluation of brandy is Quantitative Descriptive Analysis (QDA). This technique is useful as it produces a profile of the brandy. The sensory profile of the brandy product can therefore be used together with the chemical composition to form a correlation between the sensory profile and the chemical compounds. The correlation between the sensory profile and chemical composition of the brandy can be used to alter production process, including the distillation technique to ultimately produce a product with the desired characteristics.

Therefore control of the key factors that influence the production of the chemical compounds found in the brandy product is vital, however the need for sensory evaluation is also of importance and if used in conjunction with each other, it can produce products that are consistent and of good quality.

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Chapter 3

RESEARCH RESULTS

The influence of pot still design on the chemical and sensory characteristics of unmaturred brandy.

3. THE INFLUENCE OF POT STILL DESIGN ON THE SENSORY CHARACTERISTICS OF UN-MATURED BRANDY

ABSTRACT

The effect of five variations of pot still designs which differed with regards to their heads and swans neck apparatus on the chemical composition and the sensory profile of the resultant distillate was investigated. GC-FID was used to identify the volatile compounds found in the distillates and together with Quantitative Descriptive Analysis (QDA) a distillate profile was produced which was used to differentiate between distillate samples and their pot still designs. The chemical composition of the low wines prior to distillations differed significantly from each other with low wine one containing a larger amount of total esters and carbonyl compounds whilst low wine two contained a larger amount of total higher alcohols and acids. The chemical composition of distillates in both low wine one and two were found to follow a similar chemical composition of the low wine prior to distillation. Variation one of the pot still designs was based on the *Alambic Charentais* method and it was found that this variation was significantly different from the other variations as this variation influenced the chemical composition and the sensory profile of the distillates the most. Distillates produced from this variation contained smaller amounts of esters as well containing a lower intensity of the fruit and sweet associated caramel aromas and flavours. Ethyl acetate and the ethyl esters of the long chained fatty acids were found to correlate with the sensory attributes known as fruit associated aroma, soapy aroma, and spicy aroma and therefore indicated that these compounds influenced the intensity of these attributes. The distillate samples of low wine one and two were also subjected to a sensory classification system whereby they were classified on a sensory classification scale of one until five. For the distillates of low wine one the variations influence the sensory style classifications however this was not observed in the case of low wine two. Overall it was found that low wine one leans towards the sensory style classification one, where as LW2 lies more towards the sensory style classification five. Therefore the shape and size of the still heads and swans neck apparatus were shown to influence the chemical composition of the distillates; however the chemical composition and the sensory characteristics and sensory style classification of the distillates are more dependent on the chemical composition of the low wine prior to distillation. This was confirmed, as the addition of certain esters, carbonyl compounds, higher alcohols and acids in specific ratios could alter the sensory style classification of the distillates.

3.1 INTRODUCTION

Distillation is one of the most important separation processes in the chemical industry and entails a heating of a solution, a mixture of liquid and condensing the resulting vapour into a different vessel (Leaute 1950). The distillation technique that is employed to produce spirit products vary widely and is dependent of the style of final product. There are two main techniques employed for the purpose of distillation (Carnacini 1989). These techniques are known either as pot (batch) or column still distillation. However, the main distillation technique that is used for the production of brandy is pot still distillation.

Pot still distillation is a term used for a distillation that entails distilling a mixture to obtain different component fractions (Bernot et al. 1990). There are two stages of distillation when pot still distillation is used (Leaute 1950). The first stage entails taking wine and distilling it until the alcohol

strength is 28-30% a/v. This is now known as low wine. Low wine can be stored for a long period of time, as it is protected against microbial spoilage. The second stage is distillation of the low wine and collecting it in three fractions. These are known as the heads, hearts and the tails. Each of these fractions contains different amounts and types of compounds. However, it is the heart fraction that is of importance as this is the fraction that is matured. The alcohol strength of the heart fraction ranges from 65-75% a/v (Gold 1972).

In pot still distillation an apparatus known as a pot still is used to distil wine into brandy. Pot stills vary in capacity and shape but are essentially made of the same material i.e. copper. The shape of the pot still is an important factor and can play a large role in the production of the flavour and aroma compounds that make up the final distillate product (Leaute 1950).

Most pot stills consist of the same components, namely the boiler, pot still head, swans neck and condenser (Leaute 1950). Each of these components serves a different purpose but is essential in the make up of the pot still. However, some of these components play a role in the reflux within the system during distillation and therefore influence the final outcome of the product.

Reflux is the term that is used for the process by which the liquid that is placed into the pot still boils; the vapours will rise and start to move upwards through the still head towards the condenser (Hampel and Hawley 1982). If the still head has a surface area that is either too large or long, then the vapours will cool and condense and run back down into the original liquid inside the pot still and be reboiled (Kister 1992). This reflux in the system ultimately influences the amount and type of compounds that will distil over into the distillate or un-matured pot still brandy (Hampel and Hawley 1982). Studies conducted by Carnacini (1989) show that pot still distillation enhances the aromatic quality of the original wine. The reason for this is that pot stills have more reflux in their system.

For the production of Cognac the type of pot still that is used is known as *Alambic Charentais* (Leaute 1950). This pot still traditionally has a pot still head with a typical "onion" shape. This pot still head has a large surface area and therefore a greater degree of reflux. Studies by Leaute (1950) show that the shape and surface area of the pot still head and swans neck play a large role in the compounds found in the final distillate. He mentions that pot still heads that are narrower and have a smaller surface area, will have a smaller degree of reflux, therefore making it easier for compounds to move up and over into the condenser and consequently the distillate.

It is widely known that spirit products consist of many compounds (Nykanen 1986). Both the volatile and non-volatile compounds play a large role in the organoleptic perception and quality of the product. Other factors that have been shown to influence the sensory quality of the final product are, the cultivar used for the production of the wine, fermentation procedure, yeast strain used, maturation together with the distillation technique (Steger and Lambrechts 2000). It is not an individual factor, but the combination of these that will influence the type and amount of the compound found in the distillate. The compounds found to contribute mainly towards the organoleptic quality of spirit products are esters, higher alcohols, fatty acids and aldehydes (Nykanen 1986).

The purpose of this study was to investigate the role and influence that different pot still designs will have on the chemical and sensory profile of resultant distillate. There have been limited studies which indicate that different surface areas as well as different shapes will influence the outcome of the product due to a change in reflux. Therefore it was decided to investigate the difference between five different variations of pot still heads and swans necks on the sensory characteristics of distillates. The effect of the addition of certain chemical compounds on the sensory style classification of specific distillates was also investigated.

3.2 MATERIALS AND METHODS

3.2.1 Pot still heads and swans necks variations

The figures below show the five types of pot still variations used. Type A is variation two and consists of a swan's neck that is fitted directly to the boiler. Type B is a representation of original pot still head and swans neck that is used for the production of Cognac (*Alambic Charentais*). This pot still head was used as the control and known as variation one. Type C is variation four and consists of a vertical pot still head and a straight swan's neck. Type D is variation five and consists of the original pot still head together with the straight swan's neck. Type E is variation three and consists of the vertical pot still head and the rounded swan's neck from the original pot still (Figure 3.1) (van der Merwe 2008).

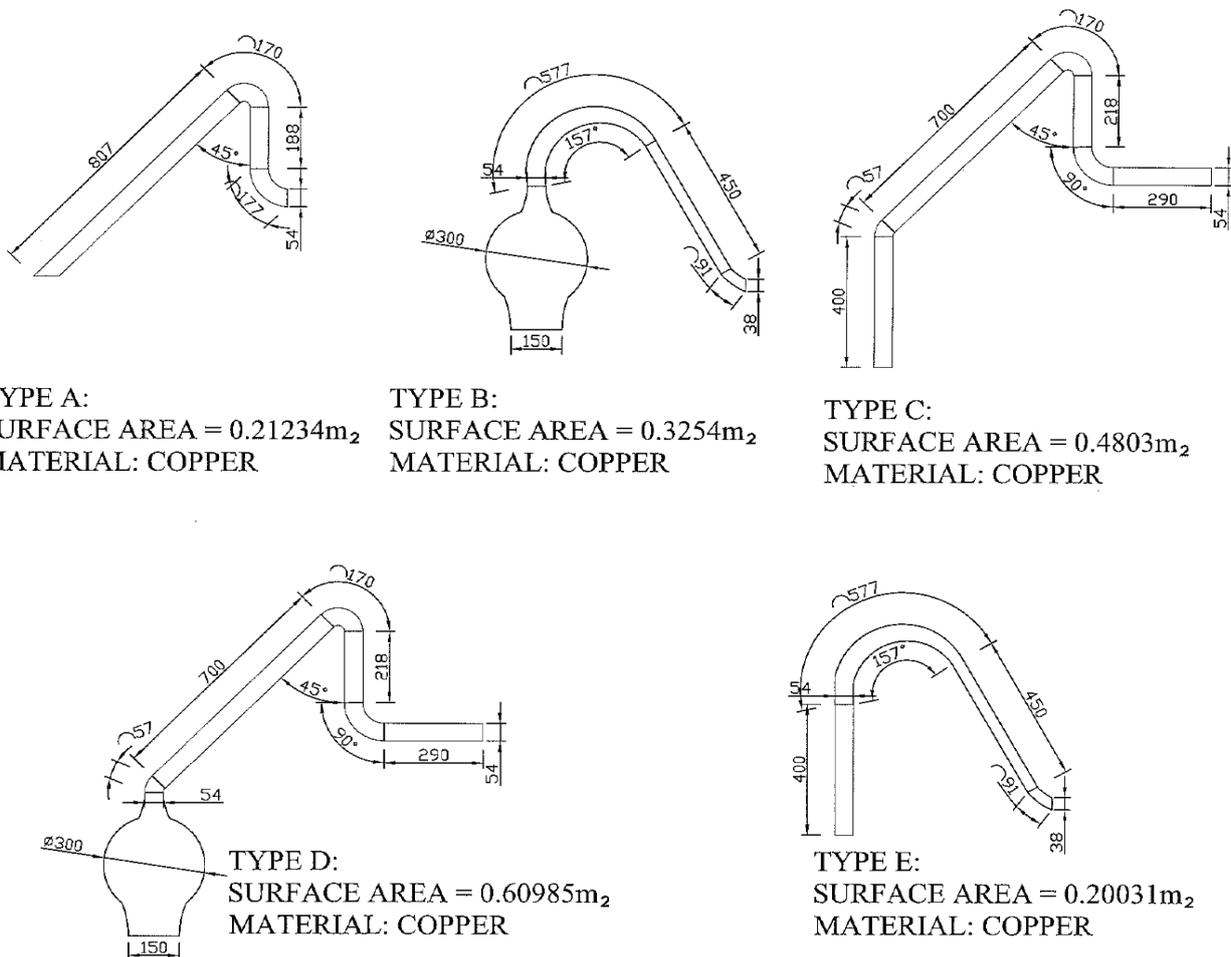


Figure 3.1 The dimensions of the five different pot still variations used for the distillations. A-Variation 2; B-Variation 1; C-Variation 4; D-Variation 5; E-Variation 3. (van der Merwe 2008).

3.2.2 Distillations

Low wine with an alcohol concentration of between 38 and 40% a/v was obtained from a commercial distillery. Two different batches of low wines were used, both from the 2007 vintage. The first batch of low wine (LW1) was distilled in the beginning of the season i.e. March and the second batch of low wine (LW2) was distilled at the end of the season i.e. November. The reason for this was to test the effect of the storage on the chemical composition of the low wines. Each batch of low wine consisted of a total amount of 1500 L.

A 140 L Prulho pot still was used for the purpose of the distillations. Distillations were repeated three times to obtain triplicates for a variation. During the distillations, all variables were kept constant. According to the industry guidelines, the water temperature of the condenser was kept between 18 and 23°C. The head fraction was collected during the first fifteen minutes of the distillation. The heart fraction was collected after the head fraction until the temperature of the boiler reached 92°C. The tail fraction was collected from temperatures 92°C until 101°C.

For each distillation, 100 L of low wine mixture was used. This mixture consisted of ± 70 L low wine and ± 30 L of heads and tails mixture. An original distillation was conducted to obtain the heads and tails which were to be used in the experiment. Each fraction, namely the heads, hearts and tails was collected separately. With the collection of each fraction, the alcohol concentration, temperature of pot still and the temperature of distillate were recorded. The temperatures were measured using a thermometer, and in the case of the alcohol, a hydrometer was used. The flow rate for low wine one and two were measured and noted. Table 3.1 represents the code for each of the distillate samples indicating which low wine as well as which variation of pot still design was employed for the purpose of the distillation.

Table 3.1. Sample codes of the 10 distillate samples selected for sensory profiling using Quantitative Descriptive Analysis (QDA).

Sample number	LW ^a	V ^b
LW1V1	1	1
LW1V2	1	2
LW1V3	1	3
LW1V4	1	4
LW1V5	1	5
LW2V1	2	1
LW2V2	2	2
LW2V3	2	3
LW2V4	2	4
LW2V5	2	5

^a LW-Low wine used for the purpose of distillation; ^b V-Variation of the pot still designs.

3.2.3 Chemical analysis

Triplicate samples of the distillate samples were analyzed on a GC-FID to identify and quantify the volatile compounds found in the sample. Although all three fractions of the distillate were analyzed as well as the initial low wine prior to the distillations, only the heart fraction will be discussed as this fraction is used for maturation of brandy.

A volume of 5 mL of each head and heart fraction ca. 70% (a/v) distillate sample was spiked with 0.25 mL of the internal standard solution (4-methyl-2-pentanol at a concentration of 2 g/L in

absolute ethanol) and transferred into 2 mL crimp-top autosampler vials. The vials were sealed and a volume of 2 μ L was injected into the GC inlet.

A volume of 10 mL of each tail fraction ca. 30% (a/v) distillate sample was pipetted into a screw-capped glass test tube and spiked with 0.4 mL internal standard (4-methyl-2-pentanol at a concentration of 1.2 g/L in absolute ethanol). A volume of 6.5 mL diethyl ether was added to each test tube. The test tubes were capped and placed in a rotary mixer, where after the samples were extracted for 30 minutes at a speed of 60 rpm. The upper diethyl ether layers were transferred into 2 mL crimp-top autosampler vials, the vials were sealed and a volume of 3 μ L of each extract was injected into the GC inlet.

The prepared samples/extracts were analyzed by GC-FID using Agilent HP6890 series gas chromatograph, equipped with a 60 m x 0.32 mm ID x 0.5 μ m film thickness, HP-Innowax fused silica capillary column, connected to a flame ionization detector (FID). The split/splitless injector was set at 200°C. Split injectors were used for both the 70% (a/v) samples and the 30% (a/v) extracts, viz. split ratios of 15:1 and 10:1, respectively. The initial oven temperature was set to 30°C (for 17 min), then increased to 240°C (for 5 min) at a heating rate of 8°C/min. The carrier gas was hydrogen 5.0 (Air Products) with a nominal initial pressure of 77.3 kPa with an average linear velocity of 45 cm/s for the 70% (a/v) samples and a nominal initial pressure of 83.5 kPa with an average linear velocity of 48 cm/s for the 30% (a/v) samples. A constant flow mode was employed. The detector temperature was set at 250°C, with an air (medical air, Air Products) flow of 400 mL/min, a hydrogen flow of 30 mL/min and nitrogen 5.0 (Air Products) make up gas flow of 30 mL/min.

Normal chemical analysis of the alcohol concentration was also carried out on the low wines prior distillation as well as on the final distillates in low wine one and two. This was conducted by using a hydrometer which was calibrated with a 96% a/v ethanol solution.

Table 3.2 shows a list of the chemical compounds that were identified in the distillate samples. The abbreviations of these compounds are given as these will be used to indicate the respective compound throughout the text.

Table 3.2 Chemical compounds and their abbreviations identified in the distillates of LW1 and LW2.

Higher alcohols	Abbreviations
n-Propanol	n-PrOH
i-Butanol	i-BuOH
n-Butanol	n-BuOH
Isoamyl Alcohol	i-AmOH
Methanol	MeOH
Hexanol	Hexanol
2-Phenyl ethanol	2-PhEtOH
Esters	
Ethyl acetate	EtAc
Ethyl butyrate	EtBut
i-Amyl acetate	i-AmAc
Ethyl caproate	EtC6
Ethyl caprylate	EtC8
Ethyl caprate	EtC10
Ethyl lactate	EtLact
Hexyl acetate	HexAc
Di-ethyl succinate	Di-EtSucc
2-Phenylethyl acetate	2-PhEtAc
Carbonyls	
Acetaldehyde	Ac

Table 3.2 Chemical compounds and their abbreviations identified in the distillates of LW1 and LW2 (continued).

Acids	
Acetic acid	Acetic acid
i-Butyric acid	i-But Acid
n-Butyric acid	n-But Acid
Hexanoic acid	Hex Acid
Octanoic acid	Oct Acid
Decanoic acid	Dec Acid

3.2.4 Sensory analyses

Both discrimination and descriptive testing was used for the sensory evaluation. Triangle tests were employed for the discrimination testing and for the descriptive testing, the technique known as Quantitative Descriptive Analysis (QDA) was carried out. All sensory tests were conducted in Distell's sensory laboratory.

Samples were prepared according to research guidelines for the sensory evaluation of food products (Lawless and Heymann 1995, Jack 2003). Care was taken to ensure uniformity of each sample (volume served and serving temperature) of each replication of the different samples. All samples were randomized to exclude any bias due to the position effect. Studies by Jack (2003) mention that samples should be diluted to 23% a/v as this is the optimum alcohol percentage for sensory evaluation of whiskey products, however a tasting was conducted whereby the distillate samples were broken down to 23% a/v, 20% a/v and 15% a/v. The results obtained from these tasting showed that the samples that contained 20% a/v were at an alcohol concentration that was best for the sensory evaluation of the un-matured pot still brandy. Jack (2003) also mentions that samples should be prepared at least three hours prior to evaluation, as diluting with distilled water causes the samples to increase in temperature as well as increasing the volatility of the compounds resulting in a negative effect on the sensory evaluation of the samples. Therefore the distillate samples were diluted to 20% a/v with distilled water not more than three hours prior to serving and were served at room temperature (21°C). A volume of 40 mL of each sample was served in Vitria standardized 250 mL a clear wine glass, covered with a Petri dish and coded with a three digit random number. Samples were randomized and presented monadically to panellists via the serving pass-through. Between samples, panellists were instructed to cleanse their pallets with distilled water served at room temperature. Unsalted crackers were also available to panellists as palate cleansers. After rating three samples in this manner, a five minute break was introduced in order to avoid sensory adaptation. Marked reference samples of the descriptors found in the lexicon of which were previously evaluated by the panelists were prepared and served at the beginning of the session as warm up samples. (Table 3.3)

All sensory evaluations were conducted in a sensory laboratory. This laboratory was equipped with 12 separate tasting booths designed according to American Society for Testing and Materials (American Society for Testing and Materials 2001) standard requirements. Panellists evaluated products one at a time and data was recoded using the computerized data collection software Compusense five Release 4.6 (Compusense Inc, Guelph, Canada). It is important to mention that the five distillate variations of each low wine were evaluated separately due to the fact that each batch of low wine was distilled at different times. Different distillation times resulted in the distillates of LW1 being stored for a longer period of time than LW2, therefore a combined sensory evaluation could not be conducted on LW1 and LW2.

Triangle tests were employed prior to the descriptive testing to test for significant differences between replications within variation of freshly distilled brandy. No significant differences ($p \leq 0.05$) were noted between replications and therefore the replications were pooled to yield one representative sample per variation (Addendum A).

Quantitative Descriptive Analysis (QDA) was used in order to determine sensory differences between the five distillate samples of low wine one and low wine two and to determine the direction of the differences. The treatments of the distillates of low wine one were evaluated separately to low wine two. Due to the time constraints of the panelists, the distillates of low wine one were only evaluated three months after distillation; however the distillates of low wine two were evaluated immediately after distillation. Twelve panellists were selected to participate in the profiling of the ten distillate pot still brandy samples, based on their taste and smell acuity, interest, ability to discriminate between the four basic tastes and availability for the entire project.

In this study the judges were trained according to the procedures outlined in Lawless and Heymann (1995). During the 12-day training sessions (one hour per day), panellists received representative samples of the different distillate samples and were trained to increase their sensitivity and ability to discriminate between specific samples and the sensory attributes of each product. A clear definition of each attribute was developed to describe the specific product attribute to be evaluated. The panel used a 100 mm unstructured line scale, with nil (0) denoting "none" (e.g. no Vanilla aroma) and hundred (100) "intense" (e.g. intense Vanilla like aroma) to evaluate the sensory characteristics of the different distillate samples (Addendum B).

In the industry brandy is evaluated using a sensory style classification test after the three years of maturation. The sensory style classification system consisted of classification one, two, three, four or five. It is important to note that the sensory classification system used here to evaluate the distillate samples is an internal classification system used at Distell and therefore due to the constraints regarding the confidentiality of these classification systems, it is not possible to reveal the specifications of this classification system. This test was conducted by Distell's internal expert classification panellists who were presented with two reference samples. These reference samples represented two classes from Distell's internal classification system for distillate brandy samples. The ten distillate samples were subjected to tasting and then classified as one, two, three, four or five. The sensory style classification one was on the opposite side of the scale being furthest away from sensory style classification five. Panellists were presented with three digit coded samples presented randomly to them. All samples were broken down to 20% a/v using distilled water and served in Vitria standardized 250 mL clear wine taster glasses, covered with a Petri dish. The panelists were subjected to these blind tasting on 4 separate occasions. The number of panel members consisted of 3 people which have had numerous experiences evaluating un-matured pot still brandy. However it should also be noted that there is no preference regarding the sensory classification scale from 1 until 5 of these distillates. These classifications are merely regarded on a scale from 1 until 5 and not on their desirability of the sensory profile of the product.

Table 3.3 lists the attributes plus the definition for each attribute as they were described during the training sessions. Each panellist was provided with a copy of the lexicon during the sensory evaluation of the distillate brandy samples.

Table 3.3 Definition of attributes for the descriptive sensory analyses of distillates.

Aroma and flavour	Definition	Reference	Dosage
Fruit associated	A general fruity aroma/flavour associated with dried fruits, which can not be recognized as any specific dried fruit.	SAD dried peaches and apricots.	100 mL of the stock solution (6 peaches and 8 apricots in 150 mL spirit base ^a , saturated for 48 hours and decanted) and an additional 100 mL of spirit base.
Soapy	The aroma/flavour associated with unscented soap.	Ethyl Caprate	75 mL of a 0.005% solution and an additional 100 mL of spirit base.
Coconut aroma/flavour	The aroma/flavour of coconut that has a slightly soapy undertone.	Ethyl Caprylate	150 mL of a 0.005% solution and an additional 50 mL of spirit base.
Spice	The aroma reminiscent of general ground spices which cannot be recognized as any specific spice.	Robertson's mixed spice.	0.1 g/100 mL mixed spice and an additional 100 mL of spirit base.
Herbaceous	The sweet dry herbaceous aroma/flavour associated with thatch or straw.	Dry thatch pieces covered with a Petri dish.	n/a
Sweet associated	An overall term reminiscent of sweet associated aromas and flavours such as caramel and vanilla.	Moir's Caramel Essence.	1 mL/100 mL and an additional 100 mL of spirit base.
Fatty/Oily	The fatty/oily aroma note perceived in the top layer.	n/a	
TASTE			
Sweet	A taste and aftertaste on the tongue stimulated by sugars.	n/a	
Bitter	A sharp taste and aftertaste experienced at the back of the throat, e.g. caffeine, aloe and tonic water.	n/a	
MOUTHFEEL			
Alcohol burn	The lingering, sharp burning sensation caused by high levels of alcohol that is experienced in the back of the entire mouth cavity and lips.	n/a	
Mouth coating/oily	A sensation of an oily layer formed in the oral cavity.	n/a	

^a Spirit base- A spirit base consisting of 250 mL of cane spirits (96% a/v), 130 g of liquid sugar, 1.5 g of tartaric acid and 620 mL of distilled water. The total volume of this solution equals 1 L of spirit base. n/a (Not applicable). Sensory attributes mentioned here are the descriptors used for the QDA sessions for sensory evaluation of the un-matured pot still brandy.

3.2.5 The addition of certain compounds to the distillates to investigate the effect on the sensory style classification.

The previous investigations of the chemical composition of low wine one and low wine two. indicated that there were no correlations between the sensory style classifications and the chemical compounds. Therefore it was decided to investigate the effect of the addition of certain chemical groups in higher concentrations on the sensory style classifications. The variation LW1V1 was considered to fall under the sensory style classification one and LW2V4 under the sensory style classification five. Therefore both of these variations were used as references samples for

sensory style classification one and five respectively when evaluating the addition samples. There were two experiments which were employed to evaluate the effect of the additions of certain compounds on the sensory style classification system. Distell's internal panel evaluated these samples. Samples were tasted blind, and experiment one was replicated three times and experiment 2 was replicated 4 times to produce reliable and consistent results. The chemical composition of the distillates was obtained using GC-FID and this was used to calculate the quantities of the required chemical compounds to add to the respective distillate samples.

Experiment one was to determine if the addition of (a) esters, (b) aldehydes and (c) esters and aldehydes would change the sensory style classification five of LW2V4 into a sensory style classification one. LW1V1 was classified as sensory style classification one. Therefore this variation was used as indication of what the amounts and type of esters and carbonyl compounds should be present in LW2V4 after the additions. The amount and type of esters and aldehydes that were to be added to LW2V4 was calculated by subtracting the amounts of esters and aldehydes in LW1V1 from LW2V4 and then adding this to LW2V4 to obtain the same desired amount in LW1V1. The amount and the type of esters and aldehydes that were added to LW2V4 are indicated in table 3.15.

In experiment two, certain higher alcohols and acids were added to LW1V1 to investigate the effect on the sensory style classification. Table 3.16 shows the chemical composition of the original LW1V1 and LW2V4 as reference samples and prior to the additions of the compounds. The amount of higher alcohols needed to increase the concentration of LW1V1 to LW2V4 was calculated by using the ratio of higher alcohols to esters in LW2V4 i.e. 0.49 and then adding the amounts of higher alcohols needed to LW1V1 to obtain the same amount present in LW2V4. The amount and type of acids that was to be added to LW1V1 was calculated by subtracting the amounts of acids in LW2V4 from LW1V1 and then adding this to LW1V1 to obtain the same amount in LW2V4.

3.2.6 Statistical analysis

Statistical analyses were performed using the software known as Statistica version 9. The univariate data was analysed and a one-way ANOVA (Analysis of Variance) was performed which tests the hypothesis that the means for the chemical compounds and sensory attributes are equal using the variations as the independent variable. Standard residuals and outliers were calculated using the Shapiro Wilk test (Shapiro and Wilk 1965).

The ANOVA was performed using Microsoft XLStat. to test for significant differences between the variations of low wine one, the variations of low wine two and between the variations of both low wine one and two together. A Fischer's least significant difference (LSD) at a 5% significance level was used. Further multivariate data analysis was carried out Principal Component Analysis (PCA). This data analysis method produces a graphical representation of the interrelationships of the variations and the chemical compounds/sensory attributes/sensory classifications or a combination thereof.

Sensory data obtained from the sensory panel was exported from the Compusense data collection program into a Microsoft Excel spreadsheet. For each attribute, the following was calculated from the raw data obtained through sensory evaluation of the 10 distillate samples: the mean, minimum, maximum and standard error of the mean. Data was subjected to a two-way analysis of variance (ANOVA) for a complete block design to assess attributes significantly different between the distillate samples. A paired t-test was conducted to test for significant differences between

treatment means. Differences with a significance level of 5% ($p \leq 0.05$) were considered as significant (Ott 1998; SAS 2002). Product means were also subjected to multivariate analysis using principal component analysis (PCA) (Piggott and Sharman, 1986). Significance in the PCA was conducted using a two way ANOVA and the replicate scores were averaged before plotting mean product spaces.

The interpretation of descriptive sensory evaluation is often simplified with the assistance of multivariate statistical procedures such as a principal component analysis (PCA). PCA is a bilinear modelling method which gives an interpretable overview of the main information in multidimensional data table. The information carried out by the original variables is projected onto a smaller number of underlying latent variables called principal components (Esbensen 2002). Through PCA, the correlation structure of a group of multivariate observations is analyzed and the axis along which maximum variability of the data occurs is identified and referred to as the first principal component or F1 (horizontal axis). The second principal component or F2 (vertical axis) is the axis along which the greatest amount of the remaining variability lies subject to the constraint that the axes must be perpendicular (at right angles) to each other (Meilgaard et al. 1991). By plotting the principal components, one can view interrelationships between different variables, and detect and interpret sample patterns, groupings, similarities or differences (Esbensen 2002).

A correlation coefficient (R of approximately 0.7 and higher) is regarded as indicating a fairly strong association between the sensory attributes/chemical compounds and the variations of the different pot still designs. Only correlations with values at a 5% level of significance will be discussed. The positive associations indicate that an increase in a specific attribute/chemical compound will result in an increase in the corresponding associated sensory attribute/chemical compound and vice versa. A negative association indicates that as a specific attribute/chemical compound increases the corresponding associated attribute/chemical compound decreases.

3.3 RESEARCH RESULTS AND DISCUSSION

3.3.1 Low wines prior to distillation

3.3.1.1 Chemical composition

The chemical compositions of the two low wines (LW) which were used in the distillation experiment are presented in Table 3.4.

Table 3.4 shows that the total esters found in LW1 are significantly different ($p \leq 0.05$) to LW2. LW1 contains the highest level of total esters and LW2 the lowest level. Of the total esters, the only esters that are not significantly different ($p \leq 0.05$) between LW1 and LW2 are ethyl butyrate and hexyl acetate. In the case of the total higher alcohols, LW2 is significantly different ($p \leq 0.05$) to LW1 with LW2 containing the highest level of total higher alcohols when compared to LW1. The only higher alcohol that is not significantly different ($p \leq 0.05$) when comparing LW1 and LW2 is n-butanol. LW2 also contains the highest level of total acids and is significantly different ($p \leq 0.05$) to LW1, where LW1 contains the lowest level of total acids. The acids i-butyric acid and n-butyric acid are however not significantly different ($p \leq 0.05$) between LW1 and LW2. In the case of total carbonyl compounds, LW1 contains the highest level of total carbonyl compounds and is significantly different ($p \leq 0.05$) from LW2 where LW2 contains the lowest level of total carbonyl compounds. Therefore it is evident that the chemical composition of the two low wines prior to distillation differ significantly ($p \leq 0.05$) from each other.

Table 3.4 Means of the chemical compounds found in LW1 and LW2 prior to distillation.

Compounds (mg/L)	P value (≤ 0.05)	LW ^a 1	LW2
Higher alcohols			
n-Propanol	0.03	133.43	150.81
i-Butanol	n/d ^b	n/d	n/d
n-Butanol	0.98	4.20	4.20
Isoamyl Alcohol	0.000	769.96	814.38
Methanol	0.003	90.94	84.34
Hexanol	0.005	3.96	10.34
2-Phenyl ethanol	0.003	26.17	31.63
Total	<0.0001	1028.66	1095.70
Esters			
Ethyl acetate	<0.0001	175.16	146.06
Ethyl butyrate	0.48	2.37	2.72
i-Amyl acetate	0.002	24.35	16.03
Ethyl caproate	0.06	3.16	4.46
Ethyl caprylate	0.001	3.05	7.15
Ethyl caprate	0.004	1.13	6.55
Ethyl lactate	0.001	53.29	64.65
Hexyl acetate	0.39	0.92	1.12
Di-ethyl succinate	0.001	4.77	9.14

Table 3.4 Means of the chemical compounds found in LW1 and LW2 prior to distillation (continued).

Compounds (mg/L)	P value (≤ 0.05)	LW ^a 1	LW2
2-Phenylethyl acetate	0.12	1.16	0.90
Total	0.001	268.20	258.79
Aldehydes/Carbonyl			
Acetaldehyde	0.002	33.29	24.47
Acetoin	0.002	13.60	5.45
Total	<0.0001	46.88	29.92
Acids			
Acetic acid	<0.0001	160.68	209.62
i-Butyric acid	0.32	5.72	5.52
Compounds (mg/L)	P value (≤ 0.05)	LW^a 1	LW2
n-Butyric acid	0.42	4.49	4.21
Hexanoic acid	0.000	31.85	26.22
Octanoic acid	0.000	92.01	65.70
Decanoic acid	0.06	56.61	55.98
Total	0.000	351.37	367.25

^a LW-Low wine used for the purpose of distillation; n/d^b-not detected. Shaded rows indicate those compounds found to be not significantly different between the two low wines ($p \leq 0.05$).

3.3.2 Distillation conditions

3.3.2.1 Alcohol and temperature measurements throughout the distillations.

During distillations, temperatures of the different fractions (heads, hearts and tails) at collection time should be kept constant as this has a large influence on the separation of the compounds which will ultimately be found in the distillate. Table 3.5 shows the alcohol % as well the temperature (°C) of the heads, hearts and tail fractions throughout distillations. The alcohol concentration of the heart fraction should be in the range of 65 until 75 % a/v.

Table 3.5 The alcohol % and temperature (°C) of the distillate at each replication measured throughout the distillations of LW1 and LW2.

Low wine 1									
Variation	Alc % Heads	Temp of distillate	Boiler temperature	Alc % Heart	Temp of distillate	Boiler temperature	Alc % Tails	Temp of distillate	Boiler temperature
Variation1									
Rep ^a 1	74.5	11	84.7	74.5	11	85.5	59	17.5	92
Rep 2	74.5	13.5	84.8	74.5	13.5	85.7	58	16	92
Rep 3	74	13	84.6	74	13	85.1	59	18	92
Variation 2									
Rep1	71	11	84.6	71	11	85.4	60	17	92
Rep2	71.5	10	84.6	71.5	10	85.2	58	16	92
Rep3	74.5	11	84.9	74.5	11	85.6	60	17	92
Variation 3									
Rep1	74	11	84.6	74	11	85.4	60	18	92
Rep2	72.5	11	84.6	72.5	11	85.1	59	17	92
Rep3	74	12	84.6	74	12	85.3	59	18	92
Variation	Alc % Heads	Temp of distillate	Boiler temperature	Alc % Heart	Temp of distillate	Boiler temperature	Alc % Tails	Temp of distillate	Boiler temperature
Variation 4									
Rep1	73.5	11	84.6	73.5	11	85.3	59	17	92
Rep2	72.5	11	84.5	72.5	11	85.3	59	16.5	92
Rep3	74.5	11	84.2	74.5	11	85.1	60	17	92
Variation 5									
Rep1	72	10	84.7	72	10	85.4	61	16.5	92
Rep2	72.5	10	84.7	72.5	10	85.4	59	17.5	92
Rep3	73	11	84.3	75	13	85.1	60	16	92
Low wine 2									
Variation	Alc % Heads	Temp of distillate	Boiler temperature	Alc % Heart	Temp of distillate	Boiler temperature	Alc % Tails	Temp of distillate	Boiler temperature
Variation1									
Rep 1	75.5	11	84.8	75	11	85.7	60	19	92
Rep 2	74.5	11	84.7	74.5	11	85.5	59	17.5	92
Rep 3	74.5	13.5	84.8	74.5	13.5	85.7	58	16	92

Table 3.5 The alcohol % and temperature (°C) of the distillate at each replication measured throughout the distillations of LW1 and LW2.

Variation 2									
Rep1	74	13	84.6	74	13	85.1	59	18	92
Rep2	71	11	84.6	71	11	85.4	60	17	92
Rep3	71.5	10	84.6	71.5	10	85.2	58	16	92
Variation 3									
Rep1	74.5	11	84.9	74.5	11	85.6	60	17	92
Rep2	74	11	84.6	74	11	85.4	60	18	92
Rep3	72.5	11	84.6	72.5	11	85.1	59	17	92
Variation 4									
Rep1	74	12	84.6	74	12	85.3	59	18	92
Rep2	73.5	11	84.6	73.5	11	85.3	59	17	92
Rep3	72.5	11	84.5	72.5	11	85.3	59	16.5	92
Variation 5									
Rep1	74.5	11	84.2	74.5	11	85.1	60	17	92
Rep2	72	10	84.7	72	10	85.4	61	16.5	92
Rep3	72.5	10	84.7	72.5	10	85.4	59	17.5	92
Standard deviation	1.31	0.99	0.16	1.26	0.99	0.19	0.80	0.76	0

^a Rep-Replication.

A one-way ANOVA was conducted on the alcohol % and temperature (°C) throughout the distillations for all variations within LW1 and LW2 and results indicated that there are no significant differences ($p \leq 0.05$) between the alcohol % and temperature (°C) of LW1 and LW2 as the p-value for LW1 and LW2 are ($p=0.208$) and ($p=0.103$) respectively.. The alcohol concentrations of the 10 un-matured distillate samples were between 75 and 76 %a/v.

3.3.2.2 Flow rate of distillates

Flow rate is influenced by the surface area of the pot still head and swans neck of the pot still. The flow rate is correlated to the amount of reflux within the system. Studies by Leaute (1950) show that the larger the surface areas of the still head, the greater the degree of reflux.

All of the different pot still variations varied with regards to their total surface area. Type D had the largest surface area (0.60985 m^2) and variation type E the smallest surface area (0.20031 m^2). A one-way ANOVA was conducted as this showed that there were no significant differences ($p \leq 0.05$) between the flow rates of the different variations in LW1 and LW2 as their p-values are ($p=0.154$) and ($p=0.161$) respectively. Table 3.6 shows the flow rates for each distillation in both low wine one and two. Studies by Leaute (1950) show that pot still heads with a larger surface area i.e. Type D, should have a larger degree of reflux in the system and therefore a slower flow rate. However, it can be seen that none of the flow rates are found to differ from each other in low wine one and two.

The flow rate of the distillates during the distillation for each replication within variations of both low wine one and two were measured (Table 3.6). This was conducted by measuring the time taken to fill a 4.5 L container after the head fraction was collected.

Table 3.6 The flow rates measured for each distillation for the 5 different variations of pot still designs of both low wine one and two.

Variation	Flow rate min and seconds/4.5L	Variation	Flow rate min and seconds/4.5L
Low wine 1		Low wine 2	
Variation1		Variation1	
Rep ^a 1	13min, 35sec	Rep 1	13min, 12sec
Rep 2	13min, 45sec	Rep 2	12min, 59sec
Rep 3	12min, 49sec	Rep 3	13min, 32sec
Variation 2		Variation 2	
Rep1	13min, 32sec	Rep1	13min, 44sec
Rep2	13min, 12sec	Rep2	13min, 20sec
Variation	Flow rate min and seconds/4.5L	Variation	Flow rate min and seconds/4.5L
Low wine 1		Low wine 2	
Rep3	14min, 5sec	Rep3	13min, 33sec
Variation 3		Variation 3	
Rep1	13min, 45sec	Rep1	13min, 25sec
Rep2	13min, 37sec	Rep2	13min, 08sec

Table 3.6 The flow rates measured for each distillation for the 5 different variations of pot still designs of both low wine one and two (continued).

Variation	Flow rate min and seconds/4.5L	Variation	Flow rate min and seconds/4.5L
Low wine 1		Low wine 2	
Variation 4		Variation 4	
Rep1	13min, 23sec	Rep1	13min, 43sec
Rep2	13min, 31sec	Rep2	13min, 29sec
Rep3	13min, 13sec	Rep3	13min, 38sec
Variation 5		Variation 5	
Rep1	12min, 55sec	Rep1	13min, 12sec
Rep2	13min, 9sec	Rep2	13min, 08sec
Rep3	12min, 55sec	Rep 3	13min, 05sec

Rep^a-Replication.

3.3.3 Low wine one

3.3.3.1 The effect of the five pot still heads and swans neck variations on the chemical compounds of the distillates of low wine one.

Table 3.7 indicates that some compounds are found to be significantly different ($p \leq 0.05$) between variations whilst others do not differ at all.

Table 3.7 indicates that LW1V1 is significantly different ($p \leq 0.05$) from the other variations as it contains the lowest amount of total esters, and more specifically the lowest amount of ethyl acetate, i-amyl acetate, ethyl caproate, ethyl caprylate, ethyl caprate and hexyl acetate. LW1V1 is also found to be significantly different ($p \leq 0.05$) from the other variations with regards to the higher alcohols, as this variation contains the lowest amount of total higher alcohols and more specifically n-propanol, i-butanol, n-butanol and i-amyl alcohol. When considering the total acids, LW1V1 contains the lowest amount of octanoic acid and largest amount of decanoic acid and is found to be significantly different ($p \leq 0.05$) to the other variations with regards to these two compounds.

Table 3.7 shows that overall the variation that contrasts with LW1V1 the most is LW1V3. LW1V3 is highest in total esters and more specifically ethyl acetate, i-amyl acetate, ethyl lactate and hexyl acetate. This variation is also found to be significantly different from all the other variations with regards to these compounds. LW1V3 is also significantly different ($p \leq 0.05$) to the other variations and contrasts LW1V1 the most with this variation containing the highest amount of hexanoic and octanoic acid. LW1V3 contrasts LW1V1 the most with regards to its higher alcohol concentration, with this variation being significantly different ($p \leq 0.05$) from the other variations and therefore containing the highest amount of total higher alcohols and more specifically n-propanol, i-butanol, n-butanol and i-amyl alcohol. LW1V1 is also lowest in methanol and is significantly different ($p \leq 0.05$) to LW1V3 as this variation contains the highest amount of methanol. Therefore it can be concluded that the LW1V1 contrasts LW1V3 the most and is lowest in esters, higher alcohols and acids and LW1V3 contains the highest esters, higher alcohols and acids.

Table 3.7 Means for the chemical compounds as influenced by the different pot still designs in the distillates of LW1.

Compound (mg/L)	LSD ^a	P value (p<0.05)	LW1V1	LW1V2	LW1V3	LW1V4	LW1V5
Ethyl acetate	3.61	<0.0001	437.42e	503.35d	532.42a	515.12c	527.58b
Ethyl butyrate	0.15	<0.0001	2.98c	3.59b	3.85a	3.92a	3.91a
i-Amyl acetate	0.20	<0.0001	35.60e	41.24d	44.63a	44.28b	43.56c
Ethyl caproate	0.07	<0.0001	7.14d	8.03c	9.06b	9.17a	9.08b
Ethyl caprylate	0.05	<0.0001	14.40e	15.62d	18.65b	18.76a	18.12c
Ethyl caprate	1.13	<0.0001	18.59c	20.55b	24.11a	23.22a	21.27b
Hexyl acetate	0.02	<0.0001	1.82e	2.02d	2.22a	2.18b	2.15c
Compound (mg/L)	LSD ^a	P value (p<0.05)	LW1V1	LW1V2	LW1V3	LW1V4	LW1V5
Ethyl lactate	0.32	<0.0001	24.78c	27.52b	28.08a	23.80d	23.60d
2-Phenylethyl acetate	0.00	n/d ^b	n/d	n/d	n/d	n/d	n/d
Di-ethyl succinate	0.24	0.02	1.91ab	1.90ab	2.10a	1.72b	1.69b
Total Esters	4.16	<0.0001	544.66e	623.85d	665.15a	642.21c	650.98b
Acetic acid	1.86	0.001	17.54ab	17.56ab	19.20a	14.01c	16.09b
i-Butyric Acid	0.00	n/a	Not detected				
n-Butyric Acid	0.97	0.05	0.62ab	0.94ab	1.48a	0.37b	0.00b
Hexanoic Acid	0.40	<0.0001	17.10d	19.59b	20.18a	17.51c	15.88e
Octanoic Acid	0.55	<0.0001	13.77e	15.97b	17.09a	14.42d	15.37c
Decanoic Acid	0.38	<0.0001	15.55a	11.92b	11.67b	10.47d	11.27c
Total Acids	3.75	<0.0001	62.92b	66.00ab	69.64a	56.78c	58.62c
Acetaldehyde	2.81	0.012	28.14c	30.26bc	31.00b	33.83a	32.02ab
Acetoin	0.13	0.0003	3.42ab	3.32b	3.46a	3.17c	3.07c

Table 3.7 Means for the chemical compounds as influenced by the different pot still designs in the distillates of LW1 (continued).

Total Carbonyls	2.85	0.019	31.56c	33.58bc	34.47ab	37.01a	35.09ab
n-Propanol	0.62	<0.0001	221.85e	230.51c	237.27a	229.65d	231.36b
i-Butanol	0.25	<0.0001	183.45e	194.58b	198.97a	191.76d	193.52c
n-Butanol	0.04	<0.0001	4.78d	4.95b	5.07a	4.87c	4.89c
i-Amyl alcohol	2.80	<0.0001	948.95e	998.93b	1017.74a	974.45d	978.09c
Hexanol	0.08	<0.0001	8.75c	9.12b	9.27a	8.76c	8.75c
2-Phenyl ethanol	0.29	0.0012	2.93a	2.76ab	2.64b	2.31c	2.20c
Total Higher Alcohols	2.34	<0.0001	1370.73e	1440.77b	1470.98a	1411.82d	1418.82c
Methanol	1.98	<0.0001	298.26d	310.08c	321.44a	310.27c	312.43b

^a LSD- Least significant difference at a 5 % level of significance. Compounds with different letters in the same row are found to be significantly different ($p \leq 0.05$). Shaded areas indicate those compounds that are either the highest or lowest in the different variations .n/d^b (not detected).

The interrelationships of the chemical compounds of the five distillate samples of LW1 are shown in figure 3.6. The first principal component (F1) accounted for 59.10% of the total data variation and the second principal component (F2) accounted for 35.68% of the total data variation.

Biplot (axes F1 and F2: 94.78 %)

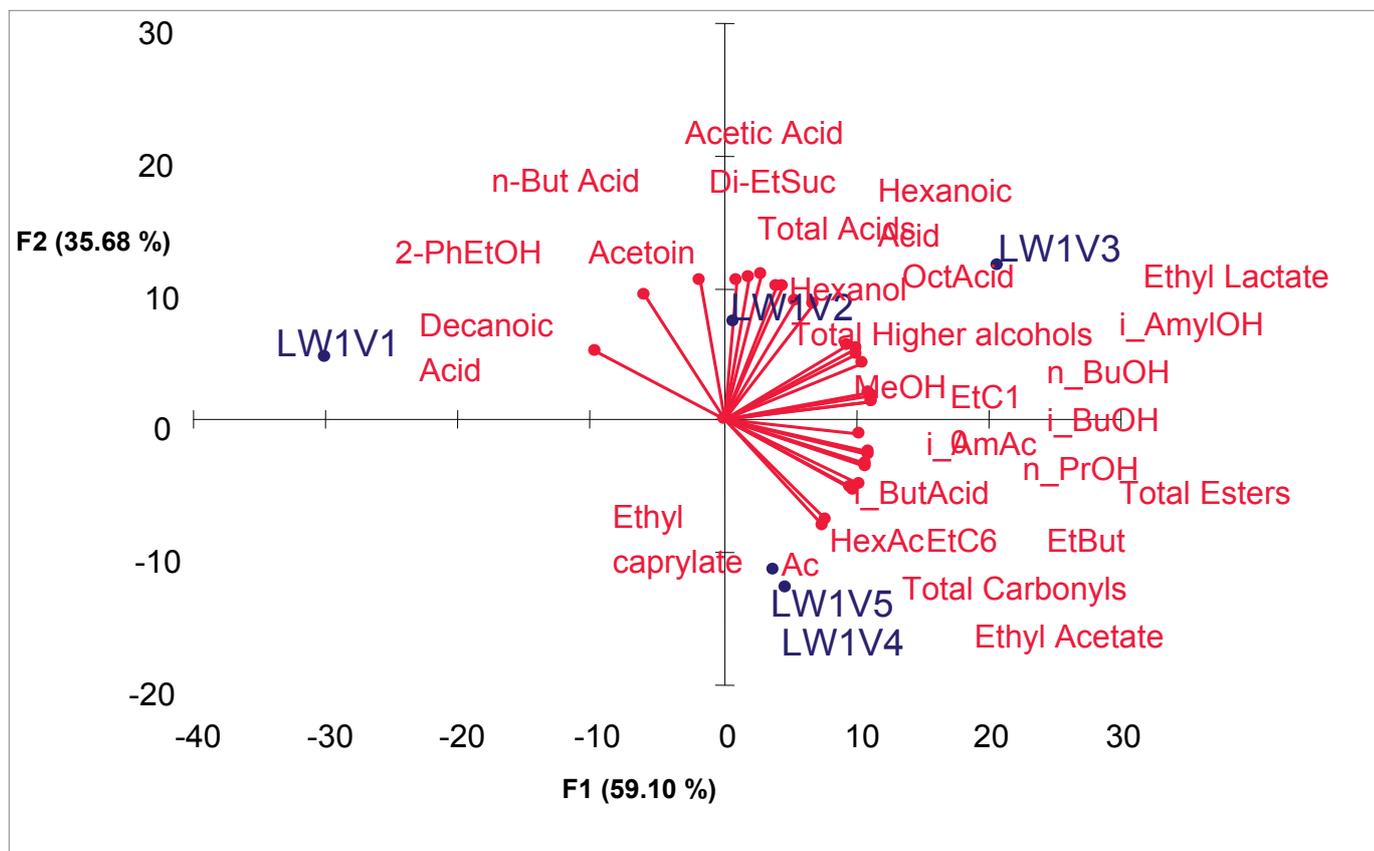


Figure 3.6 PCA Biplot showing the relation between chemical compounds and the five un-matured distillates of LW1 on F1 and F2.

F1 indicated that LW1V3 (factor score=5.09) contrasted the most with LW1V1 (factor score=-7.38).

LW1V3 is positively associated to the compounds methanol ($r=0.981$), n-propanol ($r=0.980$), total esters ($r=0.974$), i-butanol ($r=0.969$), ethyl acetate ($r=0.960$), i-amyl acetate ($r=0.949$), hexyl acetate ($r=0.946$), total higher alcohols ($r=0.915$), ethyl caprylate ($r=0.912$), ethyl butyrate ($r=0.899$), i-amyl alcohol ($r=0.880$), n-butanol ($r=0.874$), ethyl caproate ($r=0.871$), ethyl caprylate ($r=0.845$), octanoic acid ($r=0.828$) and negatively associated to the compound decanoic acid ($r=-0.849$). Variation LW1V1 is therefore found to be positively associated to decanoic acid and negatively associated to the compounds that LW1V3 is positively associated to.

F2 indicated that LW1V2 (factor score=2.33) and LW1V3 (factor score=3.68) are found to contrast the most with LW1V4 (factor score=-4.00) and LW1V5 (factor score=3.54). Therefore LW1V2 and LW1V3 are positively correlated to the compounds total acids ($r=0.965$), di-ethyl succinate ($r=0.950$), acetic acid ($r=0.925$), acetoin ($r=0.919$), ethyl lactate ($r=0.895$), n-butyric acid ($r=0.892$), 2-phenyl ethanol ($r=0.829$), hexanoic acid ($r=0.785$) and negatively correlated to acetaldehyde ($r=-0.706$). This is opposite for LW1V4 and LW1V5.

Even though LW1V1 contrasts the most with LW1V3, figure 3.6 shows that LW1V1, LW1V2 and LW1V3 are associated to each other but only with regards to their total acids, di-ethyl succinate, acetic acid, ethyl lactate, acetoin, ethyl butyrate, hexanoic acid and 2-phenyl ethanol. F1 shows

that all the variations on the right hand side of the plot are associated with higher concentrations of most of the chemical compounds while LW1V1 on the left hand sided of the plot contain lower concentrations of these compounds but a higher concentration of decanoic acid. F2 differentiates variations at the top, notably LW1V3, from the variations at the bottom (LW1V4 and LW1V5) based on differences in acetic acid, total carbonyl etc. Therefore it seems that LW1V1 in general is the variation that is found to differ the most from the other variations.

3.3.3.2 Sensory characteristics of the distillates produced by the different pot stills of low wine one

The distillates of low wine one were profiled using a trained panel and certain sensory attributes were generated. Table 3.8 shows the means of the sensory attributes found in the variations of LW1.

Table 3.8 indicated that the sensory attributes fruit associated aroma and sweet associated caramel aroma are significantly different between the variations. For the attribute fruit associated aroma, LW1V1 significantly differs ($p \leq 0.05$) from LW1V2, LW1V4 and LW1V5 with LW1V1 containing the lowest intensity of this attribute. Sweet associated caramel aroma is significantly different ($p \leq 0.05$) in LW1V1 when compared to the other variations with this variation containing the lowest intensity of this attribute.

Even though the attributes fruit associated flavour ($p=0.066$) and spicy flavour ($p=0.063$) are not significantly different ($p \leq 0.05$) amongst the variations, these attributes do follow a trend and are close to the significantly different value ($p \leq 0.05$), it can be concluded that LW1V1 does not differ from LW1V3 and LW1V4 but does differ from LW1V2 and LW1V5 for the sensory attribute fruit associated flavour, where LW1V1 and LW1V3 contain the lowest intensity of this attribute. Similarly the attribute spicy flavour is also different between variations, with LW1V1 and LW1V2 are not significantly different ($p \leq 0.05$) from each other, but are from LW1V3, LW1V4 and LW1V5 with LW1V1 and LW1V2 containing the lowest intensity of the spicy attribute. Therefore it seems that LW1V1 contains the lowest intensity of fruit associated aroma, sweet associated caramel aroma, fruit associated flavour and spicy flavour and is consequently less intense in these attributes. These results correspond with the findings of Leaute (1950) whereby he states that pot still heads with the typical *Alambic Charentais* (Variation one) still head produces brandy that is less intense and less aromatic.

The reason of LW1V1 containing the least amount of the fruit associated aroma, sweet associated caramel aroma, fruit associated flavour and spicy flavour could be due to this variation containing the lowest amount of esters. Studies by Steger and Lambrechts (2000) and Ferrari et al. (2004) mention that esters are responsible for the fruity and floral characteristics in distilled beverages.

Table 3.8 Means for the sensory attributes of distillates as influenced by different pot still designs for LW1.

Attribute	LSD ^a	P-value ($p \leq 0.05$)	LW1V1	LW1V2	LW1V3	LW1V4	LW1V5
Soapy Aroma	7.6	0.6	45a	48a	42a	45a	45a
Fruit Associated Aroma	3.9	<0.001	30b	37a	34ab	35a	35a
Coconut Aroma	4.6	0.4	22a	24a	22a	22a	25a
Spicy Aroma	3.6	0.3	26ab	27ab	25b	27ab	29a
Thatch Aroma	3.0	0.6	9a	10a	10a	8a	11a

Table 3.8 Means for the sensory attributes of distillates as influenced by different pot still designs for LW1 (Continued).

Sweet Associated Caramel Aroma	7.4	<0.001	41b	50a	50a	50a	52a
Fatty/Oily Aroma	3.2	0.8	10a	11a	10a	11a	12a
Sweet Taste	4.3	0.1	33a	35a	35a	30a	35a
Bitter Taste	4.8	0.4	29a	28a	32a	27a	30a
Alcohol burn	6.5	0.3	45a	49a	51a	46a	49a
Mouth coating oily	3.7	0.4	16a	15a	13a	13a	14a
Fruit Associated Flavour	4.0	0.1	31b	36a	31b	34ab	36a
Soapy Flavour	3.9	0.5	26a	28a	28a	27a	29a
Coconut Flavour	3.3	0.2	13b	14ab	16a	13ab	15ab
Spicy Flavour	3.3	0.1	26b	28ab	30a	30a	31a
Thatch Flavour	3.2	0.5	10a	13a	10a	11a	12a
Sweet Associated Caramel Flavour	5.0	0.5	34a	38a	37a	35a	35a

Means in the same row with different letters are significantly different ($p \leq 0.05$). ^a LSD-Least significant difference. Shaded areas indicate those sensory attributes that are found to be significantly different amongst variations.

PCA was performed to obtain a graphical representation of the interrelationships of the sensory attributes of the 5 distillate samples (Figure 3.7). The first principle component (F1) explained a total of 45.73% of the variation of the data. The second principal component (F2) explained a total of 29.47% of the variation of the data.

Biplot (axes F1 and F2: 75.20 %)

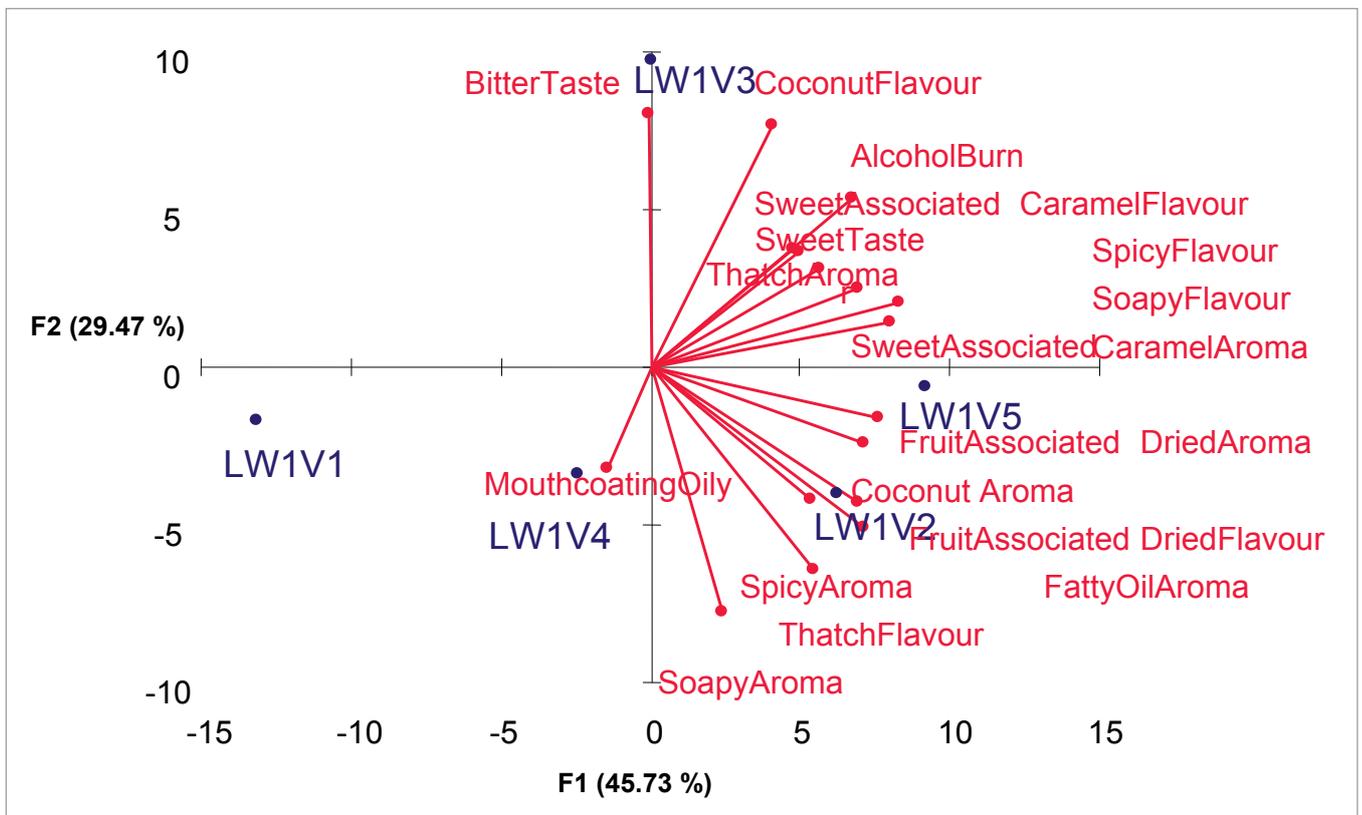


Figure 3.7 PCA Biplot showing the relation between sensory attributes and the five un-matured distillates of LW1 on F1 and F2.

F1 indicated that LW1V1 (factor score=-4.72) contrasts the most with LW1V2 (factor score=2.23) and LW1V5 (factor score=3.28) (Figure 3.7). Figure 3.7 indicates that LW1V1 is found to be negatively associated to soapy flavour ($r=0.953$), sweet associated caramel aroma ($r=0.915$), fruit associated aroma ($r=0.867$), coconut aroma ($r=0.815$), fruit associated flavour ($r=0.811$), fatty oily aroma ($r=0.792$), spicy flavour ($r=0.789$) and alcohol burn ($r=0.770$) but positively associated to mouth coating oily ($r=-0.723$) and thatch aroma ($r=-0.789$). This variation is found to have the lowest intensity of fruit associated aroma and sweet associated caramel aroma (Table 3.6).

F2 indicated that LW1V2 (factor score=-1.79) and LW1V4 (factor score=-1.53) contrasted the most with LW1V3 (factor score=4.34). LW1V3 is found to be positively associated to bitter taste ($r=0.916$) and coconut flavour ($r=0.880$). The reason for this variation having the highest intensity of these attributes could be due to the fact that this variation contains the highest amount of total higher alcohols as studies by Boscolo et al. (2000) show that if total higher alcohols are present to large amounts it could render the product unpleasant. Of the variations it was seems that LW1V1 is associated with lower amounts of sensory attributes when compared to the other variations (F1) and F2 differentiates samples at the top of the bi-plot, notably LW1V3 from the samples LW1V2 and LW1V5. All of the associations mentioned above were found to be significantly different ($p \leq 0.05$).

3.3.3.3 The effect of the five pot still heads and swans neck variations on the chemical composition and the sensory attributes of the distillates of low wine one.

According to the Pearson's correlation matrix of the five distillates of LW1, certain chemical compounds are found to be correlated to particular sensory attributes. Table 3.9 shows these correlations. Only those sensory attributes that are found to be correlated with a correlation coefficient $r \geq 0.9$ to compounds will be discussed. All of the correlations mentioned were found to be significantly different ($p \leq 0.05$).

Table 3.9 The correlations between the specific chemical compounds and certain sensory attributes as indicated by the Pearson's correlation matrix in low wine 1.

Sensory attributes	Esters	Acids	Carbonyl compounds	Higher alcohols	Methanol
Alcohol burn	n/a ^a	Octanoic acid (r=0.959)	n/a	n-Propanol (r=0.998); i-Butanol (r=0.977); n-Butanol (r=0.986); i-Amyl alcohol (r=0.996); Hexanol (r=0.865); Total higher alcohols (r=1)	Methanol
Soapy flavour	Ethyl acetate (r=0.916)	n/a	n/a	n/a	n/a
Spicy flavour	Ethyl acetate (r=0.957); Ethyl butyrate (r=0.969); i-Amyl acetate (r=0.940); Hexyl acetate (0.932); Ethyl caproate (0.956); Ethyl caprylate (0.9); Total esters (r=0.944)	n/a	n/a	n/a	n/a
Sweet Associated Caramel aroma	Ethyl acetate (r=0.957); Ethyl butyrate (r=0.969); i-Amyl acetate (r=0.940); Total esters (r=0.944)	n/a	n/a	n/a	n/a
Sweet Associated Caramel flavour	n/a	n/a	n/a	i-Amyl alcohol (r=0.946); Total higher alcohols (r=0.928)	n/a

^a n/a-Compound not applicable

Ethyl acetate imparts a floral, fruity odour if found in low concentrations (Steger and Lambrechts 2000). Ethyl caproate is fragrant and has an odour similar to that of banana oil and strawberry (Ferrari et al. 2004); ethyl caprylate is more pungent and less fragrant and resembles crude grape fusel oil; ethyl caprate is less intense and milder with fatty tones and finally ethyl laurate is the least aromatic and had a waxy candle like odour (Guymon 1969). Overall the ethyl esters of the long chain fatty acids display a floral, fruity aroma and some times with a sweet characteristic. The compound responsible for the spicy aroma in Cognacs is β -Citronellol and ethyl butyrate is also associated with a fruity aroma (Ferrari et al. 2004).

Alcohol burn seems to be correlated to higher alcohols, thus indicating that large amounts of these compounds could contribute to the alcohol burn in a distillate sample. The sensory attribute sweet

associated caramel aroma, spicy flavour and soapy flavour are found to be positively correlated to the chemical compound ethyl acetate. This compound is found to be correlated to the sensory attributes, floral and fruity aromas. These findings are indeed valid for the attribute sweet associated caramel aroma (Steger and Lambrechts 2000). However this is not the case with regards to spicy and soapy flavour. Ethyl butyrate, i-amyl acetate and total esters are also correlated to sweet associated caramel flavour. This finding is supported by Ferrari et al. (2004) which mention that ethyl butyrate are associated with a fruity associated aromas. This corresponds with the literature as esters are associated with this sensory attribute (Steger and Lambrechts 2000).

3.3.4 Low wine two.

3.3.4.1 The effect of the five pot still heads and swans neck variations on the chemical compounds of the distillates of low wine two.

The means for chemical compounds of the distillate samples are presented in table 3.10.

Table 3.10 Means for the chemical compounds of distillates as influenced by different pot still designs for LW2.

Compound (mg/L)	LSD ^a	P value (p<0.05)	LW2V1	LW2V2	LW2V3	LW2V4	LW2V5
Ethyl acetate	2.91	<0.0001	289.55d	299.87c	306.17b	297.24c	316.40a
Ethyl butyrate	0.08	<0.0001	1.80c	2.07b	2.05b	2.08b	2.28a
i-Amy acetate	0.04	<0.0001	22.29d	22.28d	23.02c	23.49b	24.89a
Ethyl caproate	0.21	<0.0001	6.05d	6.25cd	6.36bc	6.56b	6.97a
Ethyl caprylate	0.34	<0.0001	13.70bc	13.54c	13.77bc	14.00b	15.10a
Ethyl caprate	3.74	0.604	22.08a	19.99a	21.01a	20.88a	22.55a
Hexyl acetate	0.27	0.896	1.85a	1.84a	1.82a	1.82a	1.93a
Ethyl lactate	2.50	0.12	35.17b	36.32ab	38.04a	35.34b	35.20b
2-Phenylethyl acetate	0.00	n/d ^b	n/d	n/d	n/d	n/d	n/d
Di-Ethyl succinate	0.59	0.09	3.08b	3.58ab	3.88a	3.68a	3.75a
Total Esters	3.70	<0.0001	395.59d	405.79c	416.15b	405.12c	429.10a
Acetic acid	2.99	0.04	22.88a	21.26ab	20.76ab	19.01b	18.33b

Table 3.10 Means for the chemical compounds of distillates as influenced by different pot still (continued).

Compound (mg/L)	LSD ^a	P value (p<0.05)	LW2V1	LW2V2	LW2V3	LW2V4	LW2V5
i-Butyric Acid	0.26	0.0005	0.00c	0.00c	0.75a	0.35b	0.32b
n-Butyric Acid	0.31	<0.0001	0.00c	0.00c	0.69b	0.94b	2.09a
Hexanoic Acid	2.66	0.098	26.02a	24.46ab	25.10ab	23.33b	22.48b
Octanoic Acid	1.86	<0.0001	12.74c	18.11b	19.52ab	20.35a	19.42ab
Decanoic Acid	0.83	0.0087	14.12b	15.68a	15.64a	15.61a	15.46a
Total Acids	4.87	0.11	75.77b	79.52ab	82.47a	79.60ab	78.10ab
Acetaldehyde	2.25	0.351	23.34a	21.64a	21.84a	21.56a	22.84a
Acetoin	0.26	0.681	1.91a	1.83a	1.86a	1.77a	1.76a
Total Carbonyls	2.16	0.288	25.26a	23.47a	23.71a	23.33a	24.60a
n-Propanol	2.26	<0.0001	268.58a	260.50b	254.48d	242.86e	258.09c
i-Butanol	2.13	<0.0001	211.71a	204.02b	199.63c	190.57d	202.89b
n-Butanol	0.08	<0.001	4.93a	4.80b	4.63c	4.40d	4.73b
i-Amyl alcohol	2.61	<0.0001	1046.71a	1011.13b	985.17d	937.69e	996.15c
Hexanol	0.48	<0.001	12.45a	12.14ab	12.07ab	11.15c	11.82b
2-Phenyl ethanol	0.23	0.003	3.26a	3.17a	3.17a	2.84b	2.79b
Total Higher Alcohols	5.23	<0.0001	1547.65a	1495.78b	1459.17d	1389.53e	1476.49c
Methanol	1.14	<0.0001	315.53a	313.46b	311.06c	297.44d	313.78b

^a LSD- Least significant difference at a 5 % level of significance. Compounds with different letters in the same row are found to be significantly different ($p \leq 0.05$). Shaded areas in table indicate those attributes that are found to be significantly different ($p \leq 0.05$) between the variations of pot still design. n/d^b (not detected).

Table 3.10 indicates that LW2V1 is lowest in ethyl acetate, ethyl butyrate, i-amyl acetate, ethyl caproate and overall lowest in total esters where LW2V5 contains the highest amount of total esters and more specifically in ethyl acetate, ethyl butyrate, i-amyl acetate, ethyl caproate. LW2V1 is found to be significantly different ($p \leq 0.05$) to the other variations regarding these compounds. LW2V3, LW2V4 and LW2V5 do not differ significantly ($p \leq 0.05$) from each other with regards to their di-ethyl concentration, but they do differ from LW2V1 with this variation containing the lowest amount of di-ethyl succinate.

For acetic and hexanoic acid, LW2V4 and LW2V5 do not differ significantly ($p \leq 0.05$) from each other but they do differ from LW2V1, with LW2V1 containing the least amount of these compounds.

Octanoic acid is highest in LW2V4. With regards to total acids, LW2V1 is only significantly different ($p \leq 0.05$) from LW2V3. LW2V1 is also highest overall with higher alcohols and more specifically n-propanol, i-butanol, n-butanol, i-amyl alcohol and hexanol. LW2V4 contains the largest amount of total higher alcohols. LW2V1 differs significantly ($p \leq 0.05$) from the other variations with regards to these compounds. Methanol is also highest in LW2V1 and is significantly different ($p \leq 0.05$) from the other variations. LW2V1 is the variation that contains the smallest amount of esters, which is similar to the variation one of LW1 (LW1V1). LW2V1 however does not correspond with having the lowest amount of total higher alcohols as in the case of LW1V1. Therefore the lower amount of esters found in variation one of LW1 and LW2 could be a result of the variation and not the chemical composition of the low wine prior to distillation as this is found in both LW1 and LW2.

PCA bi-plot that was performed on LW2 to obtain a graphical representation of the interrelationships of the chemical compounds of the five distillate samples. F1 explains 54.77% of the total data variations and F2 accounts for 27.43% of the total data variation.

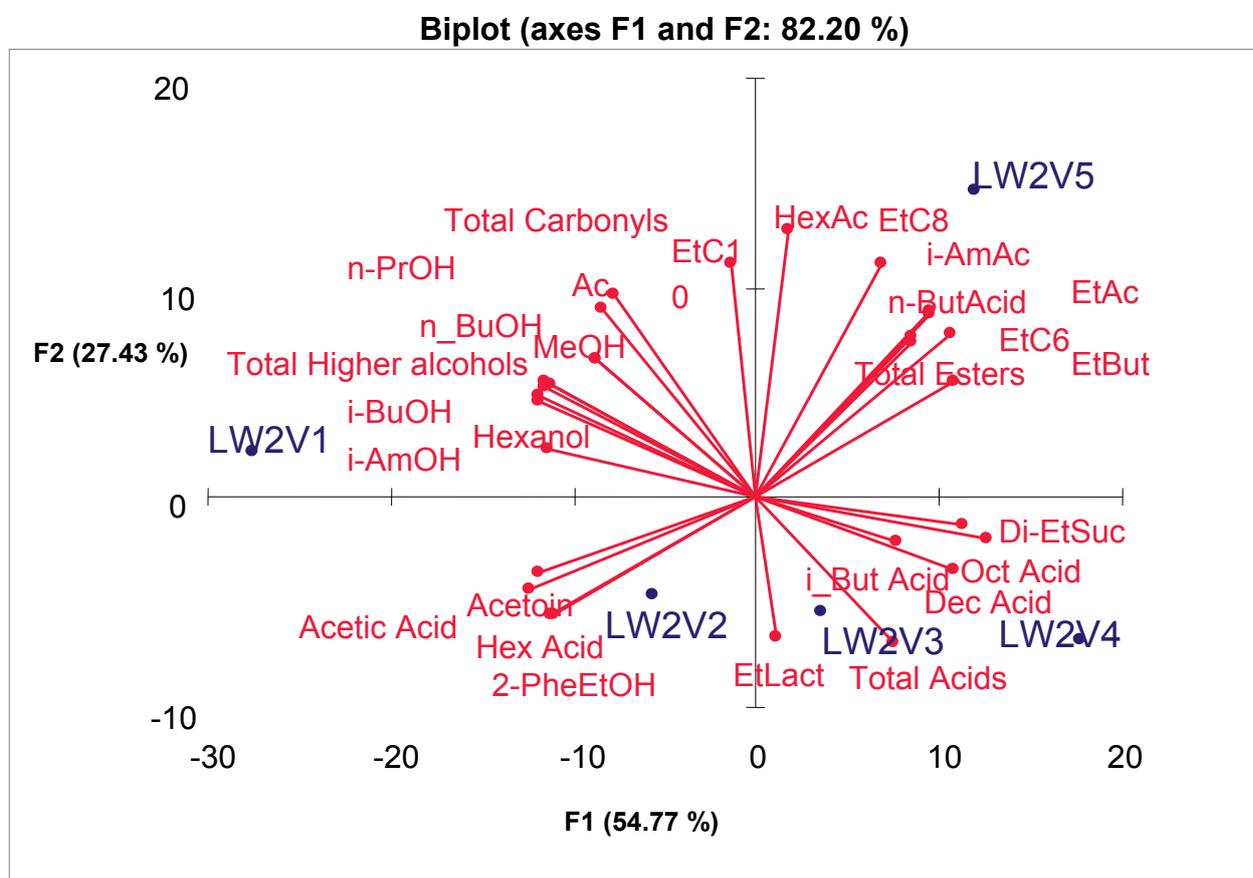


Figure 3.9 PCA Biplot showing the relation between the chemical compounds and the five un-matured distillates of LW2 on F1 and F2.

F1 indicated that LW2V1 (factor score=-6.90) contrasted the most with LW2V4 (factor score=4.44) and LW2V5 (factor score=2.99). Variation LW2V1 (factor score=-6.90) is positively associated to the compounds 2-phenyl ethanol ($r=-0.836$), hexanoic acid ($r=-0.848$), n-butanol ($r=-0.856$), hexanol ($r=-0.868$), n-propanol ($r=-0.876$), i-butanol ($r=-0.880$), total higher alcohols ($r=-0.898$), acetoin ($r=-0.904$) and i-amyl alcohol ($r=-0.904$) and negatively associated to the compounds octanoic acid ($r=0.958$), di-ethyl succinate ($r=0.857$), ethyl butyrate ($r=0.824$), decanoic acid

($r=0.824$), ethyl caproate ($r=0.803$), n-butyric acid ($r=0.718$), i-amyl acetate. This is opposite for LW2V4 and LW2V5.

F2 indicated that LW2V5 (factor score=5.18) contrasted the most with LW2V2 (factor score=-1.64), LW2V3 (factor score=-1.95) and LW2V4 (factor score=-2.38). In the case of LW2V5, this variation contains the largest amount of total esters and is positively associated to the compounds hexyl acetate, ethyl caprylate, ethyl caprate and acetoin. Therefore the variation LW2V5 contrasts LW2V1 the most as this variation has the highest concentration of total esters when compared to LW2V1. It can be concluded that variation one produces distillates that are low in esters which is shown in both LW1 and LW2. Therefore variation one has an effect on the chemical composition of the distillates.

3.3.4.2 Sensory characteristics of the distillates produced by the different pot stills of low wine two

Table 3.11 shows the means for the sensory attributes of the distillates of LW2 as affected by the pot still designs.

Table 3.11 Means for the sensory attributes of distillates as affected by pot still designs in LW2.

Attribute	LSD ^a	P-value	LW2V1	LW2V2	LW2V3	LW2V4	LW2V5
Soapy Aroma	8.0	0.77	43a	45a	44.a	48a	44a
Fruit Associated Aroma	4.6	0.49	29a	32a	31a	32a	29a
Coconut Aroma	6.0	0.97	21a	21a	21a	19a	20a
Spicy Aroma	3.5	0.68	26a	25a	24a	25a	25a
Thatch Aroma	3.0	0.74	11a	9a	10a	11a	11a
Sweet Associated Caramel Aroma	6.9	0.3	44b	49ab	47ab	50ab	51a
Fatty/Oily Aroma	3.9	<0.001	15a	8b	13a	16a	13a
Sweet Taste	4.1	0.06	29b	34a	30ab	31ab	28b
Bitter Taste	5.1	0.02	33ab	30abc	28ab	27bc	34a
Alcohol burn	6.6	0.64	45a	49a	44a	46a	47a
Mouth coating oily	3.7	0.63	13a	14a	12a	13a	15a
Fruit Associated Flavour	3.6	0.45	29a	32a	30a	29a	29a
Soapy Flavour	3.7	0.25	28ab	28ab	29a	27ab	25b
Coconut Flavour	2.6	0.07	14ab	13ab	15a	14ab	12b
Spicy Flavour	2.0	0.64	25a	27a	26a	25a	25a
Thatch Flavour	3.1	0.88	13a	13a	12a	12a	13a
Sweet Associated Caramel Flavour	4.5	0.04	30b	36a	32ab	28b	32ab

Means in the same row with different superscripts are significantly different ($p \leq 0.05$). ^a LSD-Least significant difference. Shaded areas in the table indicate those attributes that are found to be significantly different ($p \leq 0.05$) between variations of pot still design.

Table 3.11 indicates that the sensory attributes fatty oily aroma, bitter taste and sweet associated caramel flavour are significantly different ($p \leq 0.05$) amongst variations. For fatty oily aroma, LW2V2 contains the lowest intensity of this attribute and is significantly different ($p \leq 0.05$) from the other variations. Bitter taste is only significant different ($p \leq 0.05$) amongst the variation LW2V4 and LW2V5, where LW2V5 contains the highest intensity of this attribute. Sweet associated caramel flavour is significantly different ($p \leq 0.05$) between LW2V2 and both LW2V1 and LW2V3. LW2V1 and LW2V3 do not differ from each other, but they do differ from LW2V2 whereby LW2V1 and LW2V3 contain the lowest intensity of sweet associated caramel flavour. Even though the attributes sweet taste ($p=0.064$) and coconut flavour ($p=0.064$) are not significantly different with regards to their p-values, there is a general trend that indicates that variations LW2V1 and LW2V5 do not differ from each other with regards to sweet taste, but they do differ significantly ($p \leq 0.05$)

from LW2V2 where LW2V1 and LW2V5 contain the lowest intensity of sweet taste. In the case of coconut flavour, LW2V3 differs significantly ($p \leq 0.05$) from LW2V5 with LW2V5 containing the lowest intensity of coconut flavour. However LW2V3 and LW2V5 do not differ from the other variations with regards to the attribute coconut flavour.

PCA was performed to obtain a graphical representation of the interrelationships of the sensory attributes of the five distillate samples of LW2. F1 accounted for 39.05% of the total variation of the data and F2 accounted for 34.05% of the total data variation.

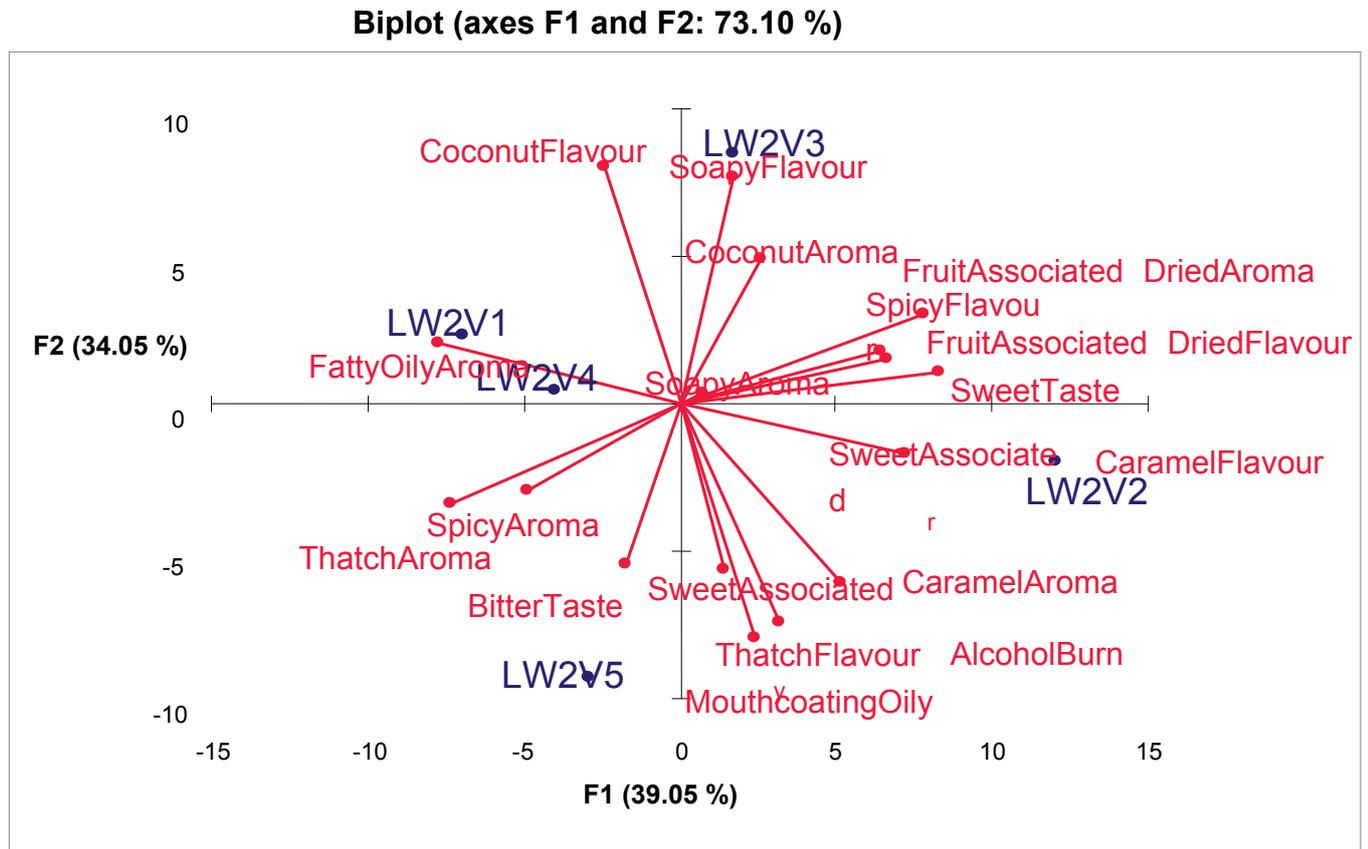


Figure 3.10 PCA Biplot showing the relation between the sensory attributes and the five un-matured distillates of LW2 on F1 and F2.

F1 indicates that LW2V1 (factor score=-2.68), LW2V4 (factor score=-1.53), LW2V5 (factor score=-1.12) contrasted the most with LW2V2 (factor score=4.68). LW2V2 is found to be positively associated to the attributes fruit associated flavour ($r=0.989$), spicy flavour ($r=0.930$), sweet associated caramel flavour ($r=0.868$), sweet taste ($r=0.786$) and fruit associated aroma ($r=0.764$) (Figure 3.10). F1 shows that LW2V2 contains a higher intensity of fruit and sweet associated aromas and flavours when compared to LW2V1. However only the attributes sweet associated caramel flavour, bitter taste, sweet taste, fatty oily aroma and coconut flavour are significantly different ($p \leq 0.05$) between the variations of LW2. Therefore it can be seen that LW2V2 in general contrasts the other variations the most.

3.3.4.3 The effect of the five pot still heads and swans neck variations on the chemical composition and sensory attributes of the distillates of low wine two.

According to the Pearson's correlation matrix of the five distillates of LW2, certain chemical compounds are found to be correlated to particular sensory attributes. Table 3.12 shows those sensory attributes that are found to be correlated to specific compounds in the distillates of LW2.

The attribute coconut aroma is positively correlated to 2-phenyl ethanol. Literature states that this compound is responsible for a rose aroma in distilled beverages (Ferrari et al. 2004), however the information gathered suggests that this compound could also be responsible for the coconut aroma. Studies by Guymon (1969) indicate that the soapy aroma found in alcoholic beverages is a result of the long chain fatty acid esters, more specifically ethyl caprylate; however the information obtained here suggests that the compound hexanoic acid could contribute towards the sensory attribute soapy flavour. Sweet associated caramel aroma in LW1 is correlated to esters such as ethyl acetate, ethyl butyrate, isoamyl acetate together with the total esters. Studies by Steger and Lambrechts (2000) confirm these results. In LW2, sweet associated caramel aroma is correlated to ethyl butyrate. Studies by Ferrari (2004) indicate that this compound is associated with the sensory attribute fruity or sweet aromas, therefore indicating that this compound could play a role of the sensory attribute sweet associated caramel flavour.

Table 3.12 The correlations between the specific chemical compounds and certain sensory attributes as indicated by the Pearson's correlation matrix in low wine 2.

Sensory attributes	Esters	Acids	Carbonyl compounds	Higher alcohols	Methanol
Coconut aroma	n/a ^a	n/a	n/a	2-Phenyl ethanol (r=0.911)	n/a
Soapy flavour	n/a	Hexanoic acid (r=0.912)	n/a	n/a	n/a
Sweet associated caramel aroma	Ethyl butyrate (r=0.932)	n/a	n/a	n/a	n/a

^a n/a-Compound not applicable.

3.3.5 The combination of low wine one and two.

3.3.5.1 The effect of the five pot still heads and swans neck variations on the chemical compounds of the distillates of low wine one and two.

Table 3.13 shows the means of the chemical compounds as affected by the pot still designs in the distillates of LW1 and LW2.

Table 3.13 Means of the chemical compounds as affected by the pot still designs in the distillates of LW1 and LW2.

Compound (mg/L)	LSD ^a	P values (≤ 0.05)	LW1V1	LW1V2	LW1V3	LW1V4	LW1V5	LW2V1	LW2V2	LW2V3	LW2V4	LW2V5
Ethyl acetate	3.03	≤ 0.0001	437.42e	503.35d	532.42a	515.12c	527.58b	289.55i	299.97h	306.17g	297.24h	316.40f
Ethyl butyrate	0.12	≤ 0.0001	2.98c	3.59b	3.85a	3.92a	3.91a	1.80f	2.07e	2.05e	2.08e	2.28d
i-Amyl acetate	0.15	≤ 0.0001	35.60e	41.24d	44.63a	44.28b	43.56c	22.29i	22.28i	23.02h	23.49g	24.89f
Ethyl caproate	0.10	≤ 0.0001	7.14d	8.03c	9.06b	9.17a	9.09ab	6.05h	6.25g	6.36g	6.56f	6.97e
Ethyl caprylate	0.13	≤ 0.0001	14.40e	13.70g	18.65a	18.76a	18.12b	15.62c	13.54h	13.77g	14.00f	15.10d
Ethyl caprate	1.62	≤ 0.0001	18.59f	20.55ed	21.01cde	23.22ab	21.27cde	22.08bcd	19.99ef	21.01cde	20.88cde	22.55abc
Hexyl acetate	0.20	0.048	1.82b	2.02ab	2.22a	2.18a	2.15a	1.85b	1.84b	1.82b	1.82b	1.93b
Ethyl lactate	1.79	0.157	24.78d	27.52c	28.08c	23.80d	23.60d	37.15b	36.32ab	38.04a	35.34b	35.20b
2-Phenyl acetate	0	n/a	0.00a									
Di-Ethyl succinate	0.47	0.087	1.91c	1.90c	2.10c	1.72c	1.69c	3.08b	3.58a	3.88a	3.68a	3.75a
Total Esters	3.58	≤ 0.0001	544.63e	623.85d	665.15a	642.21c	650.98b	359.95i	405.79h	416.15g	405.12h	429.10f
Acetic acid	2.69	0.20	17.54ed	17.56ed	19.20bcd	14.01f	16.09ef	22.88a	21.26ab	20.76ab	19.01bcd	18.33cde
i-Butyric Acid	0.2	0.0002	0.00c	0.75a	0.35b	0.32b						
n-Butyric Acid	0.74	≤ 0.0001	0.62cd	0.94bc	1.48ab	0.37cd	0.00d	0.00d	0.00d	0.69cd	0.94bc	2.09a

Table 3.13 Means of the chemical compounds as affected by the pot still designs in the distillates of LW1 and LW2 (continued).

Compound (mg/L)	LSD ^a	P values (≤0.05)	LW1V1	LW1V2	LW1V3	LW1V4	LW1V5	LW2V1	LW2V2	LW2V3	LW2V4	LW2V5
Hexanoic Acid	1.28	0.0009	17.10fg	19.59e	20.18e	17.51f	15.88g	26.02a	24.46bc	25.10ab	23.33cd	22.48d
Octanoic Acid	1.38	≤0.0001	13.77gh	15.97de	17.09cd	14.42fg	15.37ef	12.74h	18.11bc	19.52a	20.35a	19.42ab
Decanoic Acid	0.55	≤0.0001	15.55a	11.92c	11.67cd	10.47e	11.27d	14.12b	15.68a	15.64a	15.61a	15.46a
Total Acids	4.38	0.0087	62.92ed	66.00cd	69.64c	56.78f	58.62ef	75.77b	79.52ab	82.47a	79.60ab	78.10ab
Acetaldehyde	2.11	0.001	28.14c	30.26b	31.00b	33.83a	32.02ab	23.34d	21.64d	21.85d	21.56d	22.84d
Acetoin	0.15	0.093	3.42a	3.32ab	3.46a	3.17bc	3.07c	1.91d	1.83de	1.86de	1.77de	1.76e
Total Carbonyls	2.17	0.002	31.56c	33.58bc	34.47bc	37.01a	35.09ab	25.36d	23.47d	23.71d	23.33d	24.60d
n-Propanol	1.52	≤0.0001	231.36g	230.15gh	237.27f	229.65h	231.36g	268.58a	260.50b	254.48d	242.86e	258.09c
i-Butanol	1.46	≤0.0001	183.45f	194.58d	198.87c	191.76e	193.52d	221.71a	204.02b	199.63c	190.57e	202.89b
n-Butanol	0.05	≤0.0001	4.78ef	4.95b	5.07a	4.87d	4.89cd	4.93bc	4.80e	4.63c	4.40h	4.73f
i-Amyl alcohol	2.39	≤0.0001	948.95a	998.83d	1017.74b	974.45h	978.09g	1046.71a	1011.13c	985.17f	937.69j	996.15e
Hexanol	0.34	0.0006	8.75e	9.12d	9.27d	8.76e	8.75e	12.45a	12.14ab	12.07b	11.15c	11.82b
2-Phenyl ethanol	0.26	0.553	2.93bc	2.76cd	2.64d	2.31e	2.20e	3.26a	3.17ab	3.17ab	2.84cd	2.79cd
Total Higher Alcohols	3.46	≤0.0001	1370.73j	1440.77f	1470.98d	1411.82h	1418.82g	1547.65a	1495.78b	1459.17e	1389.53i	1476.49c
Methanol	1.52	≤0.0001	298.26f	310.08e	321.44a	310.27e	312.43cd	315.53b	313.46c	311.06de	297.44f	313.78c

^aLSD- Least significant difference at a 5 % level of significance. Compounds with different letters in the same row are found to be significantly different (p≤0.05)

Table 3.13 shows a general trend amongst the compounds found in both LW1 and LW2. An overview of these trends will be discussed. The total esters in LW1 are significantly different ($p \leq 0.05$) to the esters in LW2; more specifically the total esters in LW1V1 and LW2V1 seem to display lower amounts of esters when compared to the esters found in the other variations. Ethyl acetate and ethyl caproate are also overall found to be lower in LW1V1 and LW2V1.

Total acids in LW1 and LW2 do not differ significantly ($p \leq 0.05$) from each other, there is however a general trend that shows that overall LW1 contains less total acids than LW2. Decanoic acid is found to be not significantly different ($p \leq 0.05$) between LW1V1, LW2V2, LW2V3, LW2V4 and LW2V5 but is significantly different ($p \leq 0.05$) to the other variations. In general the total carbonyl compounds overall are higher in LW1 than in LW2, with none of the variations in LW2 differing significantly ($p \leq 0.05$) from each other with regards to their acetaldehyde content.

For total higher alcohols, in general there is a higher amount of n-propanol and i-butanol in LW2 than in LW1. i-Amyl alcohol is lowest in variation 1 of both LW1 and LW2 and this compound is significantly different from the other variations. Hexanol in general is lower in LW1 than in LW2. For the total higher alcohols, all of the variations differ significantly ($p \leq 0.05$) from each other, however no general trend can be noted. Therefore it can be concluded that the chemical composition of LW1 differs from LW2 (after distillation) where by LW1 is lower in esters, less acids and higher carbonyl compounds. It seem that variation 1 in both LW1 and LW2 produce distillates that are lower in esters and more specifically ethyl acetate therefore indicating that this variation can alter the amount of esters found in the distillate irrespective of the chemical composition of the low wine prior to distillation.

PCA was performed on LW1 and LW2 to obtain a graphical representation of the interrelationships of the chemical compounds of the 10 distillate samples after distillation (Figure 3.12). F1 accounted for 62.07% of the total data variation and F2 accounted for 21.40% of the total data variation.

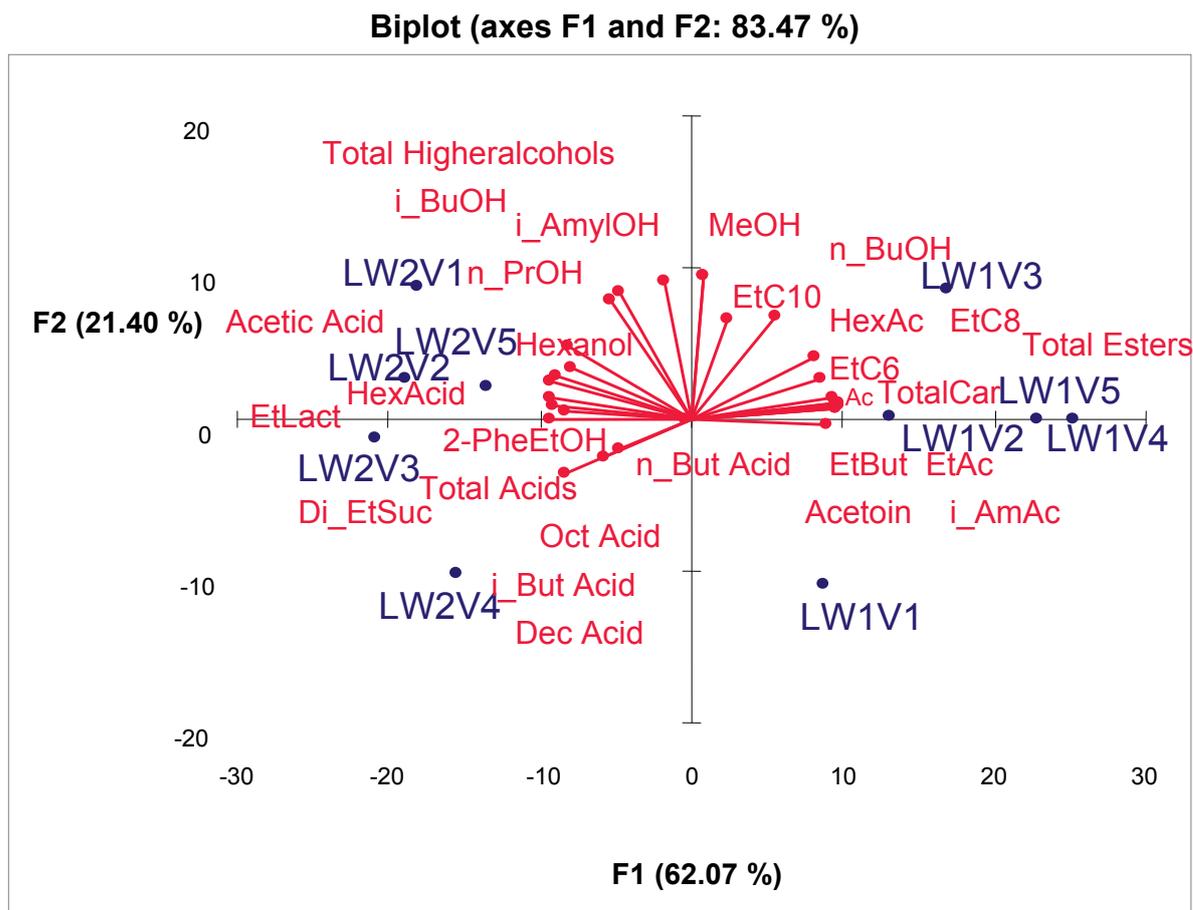


Figure 3.12 PCA Biplot showing the relation between the chemical compounds and the 10 un-matured distillates of LW1 and LW2 on F1 and F2.

F1 indicated that LW1V1 (factor score=2.07), LW1V2 (factor score=3.09), LW1V3 (factor score=3.99), LW1V4 (factor score=5.94) and LW1V5 (factor score=5.38) contrasted the most with LW2V1 (factor score=-4.27), LW2V2 (factor score=-4.46), LW2V3 (factor score=-4.91), LW2V4 (factor score=-3.64) and LW2V5 (factor score=-3.18).

The distillates of LW1 strongly contrast the distillates of LW2. Variations LW1V1, LW1V2, LW1V3, LW1V4 and LW1V5 are positively associated to the compounds total carbonyls ($r=0.900$), acetaldehyde ($r=0.989$), i-amyl acetate ($r=0.988$), ethyl butyrate ($r=0.983$), ethyl acetate ($r=0.981$), total esters ($r=0.978$), ethyl caproate ($r=0.949$), acetoin ($r=0.924$), ethyl caprylate ($r=0.882$) and hexyl acetate ($r=0.841$). Whereas variations LW2V1, LW2V2, LW2V3, LW2V4 and LW2V5 are positively associated to the compounds acetic acid ($r=-0.819$), n-propanol ($r=-0.844$), 2-phenyl ethanol ($r=-0.855$), decanoic acid ($r=-0.864$), hexanoic acid ($r=-0.923$), total acids ($r=-0.943$), diethyl succinate ($r=-0.952$), hexanol ($r=-0.959$), and ethyl lactate ($r=-0.966$). Therefore LW1 is associated with higher ester concentration and therefore is contains positive quality brandy indicators where as LW2 is associated to higher concentrations of higher alcohols which are found in brandies that are of poorer quality. LW2 is also positively associated to the compound ethyl lactate. This could suggest that the wine used for the production of the low wine, was stored for a longer period of time and therefore the production of ethyl lactate could have resulted due to the increase in levels of lactic acid bacteria as a result of malo-lactic fermentation. Therefore the

chemical composition of the low wine prior to distillation is a crucial factor to consider as this ultimately influences the chemical make up of the distillates after distillation.

3.3.5.2 The correlations between the chemical compounds and sensory style classifications of the distillates of low wine one and two, as influenced by the different pot still heads and swans neck variations.

In the industry it is common practice to classify brandy after the three year maturation period, however there is limited information regarding the classification of un-matured distillates thus making it a difficult topic to investigate (Guymon 1972). These sensory style classifications that were obtained are a result of a tasting from Distell's internal tasting panel. Table 3.14 shows the different variations of pot still design together with the low wine used and its respective sensory classification system.

Table 3.14 The sensory style classifications of the 10 un-matured distillate samples of LW1 and LW2 correlating to their chemical compounds.

Variation	Sensory classification
LW ^a 1V ^b 1	1
LW1V2	5
LW1V3	1
LW1V4	5
LW1V5	1
LW2V1	5
LW2V2	5
LW2V3	4
LW2V4	4
LW2V5	5

^a LW-Low wine; ^b V-Variation

Table 3.14 shows that LW1 has a sensory style classification that is found to be on the extreme ends of the sensory style classification scale namely 1 and 5. In the case of LW2 is shown that the distillates in LW2 do not differ greatly with regards to their sensory style classification scale i.e. 4 and 5. LW1 shows that the variations have a larger effect on the sensory style classification system and produce distillates that are on opposite ends of the scale. In the case of LW2, the variations did have an effect on the sensory style classification of the distillates; however the change in style was not as large as in the case of LW1. Therefore because the sensory style classifications differ so greatly between LW1 and LW2 it can be concluded that even though the variations do play a role in the sensory style classifications of the distillates, it is however the chemical composition of the low wine prior to distillation that has the largest effect on the chemical composition and sensory profile of the resultant distillate..

3.3.6 The effect of the addition of certain compounds on the sensory style classification of specific distillates.

The previous investigations of the chemical composition of low wine one and low wine two. indicated that there were no correlations between the sensory style classifications and the chemical compounds Therefore it was decided to investigate the effect of the addition of certain chemical groups in higher concentrations on the sensory style classifications Distillates from LW1 classified as mostly sensory style classification one whereas distillates from LW2 were classified as either sensory style classification four or five. At the same time the chemical composition of

LW1 differed with that of LW2 when total esters, total acids, total higher alcohols and total carbonyls were compared.

3.3.6.1 The effect of the addition of esters and carbonyl compounds on the sensory style classification of LW2V4

Distell's internal panellists were presented with reference sample one (LW1V1) and reference sample five (LW2V4) in order to help them evaluate the three addition samples and therefore to classify them according to the sensory style classification system. Sample one (LW2V4 + esters), sample two (LW2V4 + carbonyls compounds) and sample three (LW2V4 + esters & carbonyl compounds) were evaluated by the trained panellists and all three samples resulted in a shift of the sensory style classification system. Sample one shifted from a sensory style classification four to a three. Sample two shifted from a sensory style classification four to a sensory style classification two, therefore it shifted the least. However, sample three resulted in a shift towards the sensory style classification one, thus this addition of both esters and carbonyl compounds resulted in the greatest degree of shifting.

Therefore it can be concluded that the addition of esters and carbonyl compounds results in a shift of the sensory style classification system from a sensory style classification five to a sensory style classification one. However, it is the carbonyl compounds that results in a greater degree of shifting of the sensory style classification system from a sensory style classification five to one. Therefore the addition of esters and carbonyl compounds can shift the sensory classification to a lower degree of classification such as classification one, two, three or four. Table 3.15 indicates the chemical composition of the variations LW1V1 and LW2V4 with the additional compounds that were added to these references.

Table 3.15 The chemical composition of variations LW1V1 and LW2V4 with the additional esters and carbonyl compounds (mg/L).

Compounds	Standard Deviations	Manufacture	(LW 1V1) ^a	(LW2V4) ^b	(LW2V4+ carbonyl compounds) ^c	(LW2V4+ esters) ^d	(LW2V4+ esters & carbonyl compounds) ^e	(Addition of esters & carbonyl compounds) ^f
Alcohols								
Methanol	2	None	298.27	297.45	286.97	280.73	287.63	None
Higher Alcohols								
n-Propanol	10.11	None	221.86	242.93	244.91	240.31	246.99	None
i-Butanol	22.99	None	183.44	190.68	140.10	190.62	196.87	None
n-Butanol	0.13	None	4.76	4.41	4.48	4.55	4.55	None
i-Amyl alcohol	14.74	None	948.95	937.78	969.27	949.72	972.50	None
Hexanol	1.00	None	8.76	11.18	10.97	10.73	10.96	None
2-Phenyl ethanol	0.12	None	2.88	2.89	3.12	3.02	3.12	None
Total	26.04	None	1370.64	1389.87	1372.86	1398.94	1434.98	n/a ^g
Esters								
Ethyl acetate	67.40	Riedel-de Haen	437.42	297.64	330.93	424.99	442.27	137.66
Ethyl butyrate	0.61	Sigma Aldrich	2.99	2.10	3.12	3.56	3.60	0.89
i-Amyl acetate	7.36	Sigma Aldrich	35.61	23.50	29.44	39.65	41.15	12.12
Ethyl caproate	0.27	Sigma Aldrich	7.15	6.51	6.54	6.52	6.64	0.65
Ethyl caprylate	0.28	None	14.42	14.03	14.78	14.34	14.56	None
Ethyl caprate	3.17	None	18.64	20.60	25.38	24.91	25.49	None

Table 3.15 The chemical composition of variations LW1V1 and LW2V4 with the additional esters and carbonyl compounds (mg/L) (continued).

Hexyl acetate	0.29	None	1.83	1.82	2.36	2.33	2.37	None
Ethyl lactate	4.10	None	24.66	35.34	33.12	32.30	33.08	None
Di-Ethyl succinate	0.95	None	1.79	3.64	4.04	3.86	3.99	None
2-Phenylethyl acetate	0.51	None	0.00	0.00	0.91	0.96	0.93	None
Total	73.52	None	544.50	405.18	450.62	553.41	574.25	n/a
Acids								
Acetic acid	3.75	None	22.07	19.03	22.34	14.56	14.91	None
i-Butyric acid	0.29	None	0.00	0.36	0.76	0.62	0.50	None
n-Butyric acid	0.51	None	0.93	0.94	0.00	0.00	0.00	None
Hexanoic acid	8.75	None	17.11	22.47	4.11	4.10	4.38	None
Octanoic acid	2.86	None	13.78	20.25	19.90	19.43	20.78	None
Decanoic acid	1.24	None	12.45	15.67	13.94	13.24	14.59	None
Total	10.52	None	66.03	78.71	61.05	51.95	55.15	n/a
Carbonyls								
Acetaldehyde	7.58	Fluka	27.64	21.50	36.84	31.71	40.80	6.14
Acetoin	0.63	Fluka	3.32	1.79	2.00	1.89	2.11	1.51
Total	7.53	None	30.95	23.29	38.84	33.60	42.91	n/a

^a (LW1V1)-Reference sample 1; ^b (LW2V4)-Reference sample 4; ^c (LW2V4 +esters)-Reference sample 4 with the addition of certain esters; ^d (LW2V4 + carbonyl compounds)- Reference sample 4 with the addition of certain carbonyl compounds; ^e (LW2V4 + esters & carbonyl compounds)- Reference 4 with the addition of certain esters and carbonyl compounds. ^f (Addition of certain esters & carbonyl compounds)- Amount of the certain esters and carbonyl compounds (mg/L) which were added to reference 4; ^g n/a- Not applicable.

3.3.6.2 The effect of the addition of higher alcohols and acids on the sensory style classification of LW1V1.

Distell's internal panel were presented with two samples. Sample one (LW1V1 +higher alcohols) and sample two (LW1V1 + acids) (Table 3.16). The trained panellists evaluated the samples and came to a consensus regarding the sensory style classification of the spiked samples. Sample one resulted in a shift of the sensory style classification one of LW1V1 to a sensory style classification five. In the case of sample two, the sensory style classification of LW1V1 shifted towards a sensory style classification four. This indicates that the addition of higher alcohols and acids can increase the sensory style classification from one to four or five.

Therefore by changing the ratios between the volatile compounds it is possible to alter the style of the distillates. The chemical composition of the low wine and base wine prior to distillation is an important factor to consider as it will have an effect on the sensory style classification of the distillates after distillation.

Table 3.16 The chemical composition of variations LW1V1, LW2V4 with the addition of higher alcohols and acids (mg/L).

Compounds	Standard deviation	Manufacture	(LW1V1) ^a	(LW2V4) ^b	(LW1V1+ higher alcohols) ^c	(LW1V1+ acids) ^d	(Addition of higher alcohols) ^e	(Addition of acids) ^f
Alcohols								
Methanol	11.87	None	298.27	297.45	279.61	275.44	None	None
Higher Alcohols								
n-Propanol	17.41	None	221.86	242.93	252.56	215.57	26.78	None
i-Butanol	12.75	None	183.44	190.68	209.20	181.05	21.88	None
n-Butanol	0.19	None	4.76	4.41	4.84	4.76	0.58	None
i-Amyl alcohol	76.51	None	948.95	937.78	1095.89	942.71	113.71	None
Hexanol	1.29	None	8.76	11.18	9.67	8.25	1.05	None
2-Phenyl ethanol	0.21	None	2.88	2.89	2.78	3.26	0.37	None
Total	102.76	None	1370.64	1389.87	1575.42	1355.12	n/a^g	n/a
Esters								
Ethyl acetate	84.04	None	437.42	297.64	480.74	467.15	None	None
Ethyl butyrate	0.77	None	2.99	2.10	3.83	3.60	None	None
i-Amyl acetate	6.43	None	35.61	23.50	37.30	35.89	None	None
Ethyl caproate	0.36	None	7.15	6.51	7.36	7.06	None	None
Ethyl caprylate	0.47	None	14.42	14.03	15.17	14.62	None	None
Ethyl caprate	2.10	None	18.64	20.60	23.33	22.53	None	None
Hexyl acetate	0.32	None	1.83	1.82	2.38	2.38	None	None

Table 3.16 The chemical composition of variations LW1V1, LW2V4 with the addition of higher alcohols and acids (mg/L) (continued).

Compounds	Standard deviation	Manufacture	(LW1V1) ^a	(LW2V4) ^b	(LW1V1+ higher alcohols) ^c	(LW1V1+ acids) ^d	(Addition of higher alcohols) ^e	(Addition of acids) ^f
Ethyl lactate	6.15	None	24.66	35.34	22.94	22.10	None	None
Di-Ethyl succinate	0.88	None	1.79	3.64	1.94	1.93	None	None
2-Phenylethyl acetate	0.56	None	0.00	0.00	0.99	0.94	None	None
Total	86.53	None	544.50	405.18	595.97	578.21	n/a	n/a
Acids								
Acetic acid	2.71	None	22.07	19.03	15.67	17.48	None	None
i-Butyric acid	0.18	Fluka	0.00	0.36	0.00	0.00	None	0.36
n-Butyric acid	0.54	Fuka	0.93	0.94	0.00	0.00	None	None
Hexanoic acid	8.59	Sigma Aldrich	17.11	22.47	3.83	7.43	None	5.37
Octanoic acid	3.35	Fluka	13.78	20.25	13.71	18.62	None	6.47
Decanoic acid	20.59	Sigma Aldrich	12.45	15.67	41.84	54.98	None	3.20
Total	15.71	None	66.03	78.71	41.84	54.98	n/a	n/a
Carbonyls								
Acetaldehyde	12.00	None	27.64	21.50	47.15	42.00	None	None
Acetoin	0.81	None	3.32	1.79	3.48	3.44	None	None
Total		None	30.95	23.29	50.63	45.43	n/a	n/a

^a (LW1V1)-Reference sample 1; ^b (LW2V4)-Reference sample 4; ^c (LW1V1 +higher alcohols)-Reference sample 1 with the addition of certain higher alcohols; ^d (LW1V1 +acids)- Reference sample 1 with the addition of certain acids; ^e (Addition of higher alcohols)- Amount of the higher alcohols (mg/L) which were added to reference 1; ^f (Addition of acids)-Amount of the acids (mg/L) which were added to reference 1; ^g n/a- Not applicable.

3.5 CONCLUSION

It can be concluded that of the pot still designs used, variation one influenced the chemical composition and sensory profile of the distillates and differed the most when compared to the other variations with regards to the outcome of the final distillates. This variation produced a distillate that contained a lower amount of esters and more specifically ethyl acetate and also exhibited sensory attributes that were least intense in the sweet associated and fruit associated aromas and flavours. These results correlated with the findings of Leaute (1950) whereby he states that pot still heads with a larger surface area will have more reflux in the system, and therefore produces distillates that are less aromatic. Even though the flow rates of the individual pot still designs did not differ, it was shown that variation one did have an effect on the chemical composition and sensory profile of the distillates. However, the low wine that is used for the purpose of the distillation is the most important factor as this will ultimately have the largest influence on the chemical composition and sensory profile of the resultant distillate. It should be noted that this was a preliminary study and it is strongly recommended that further studies should be conducted as there is very limited research on this topic. Further expansion on the identification of the volatile compounds in distillates could result in a better understanding of those compounds that are responsible for certain attributes. The use of other pot still variations as well as more repetitions of the distillation procedure could produce a larger amount of information regarding the effect of the pot still designs on the resultant distillates chemical composition and sensory profile. Further analysis of the low wine prior to distillation could be beneficial as this would produce a "blue print" of the low wines chemical composition and therefore a possible prediction of the outcome of the chemical composition of the distillate could be obtained and therefore ultimately a sensory profile of the un-matured brandy.

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Chapter 4

GERNERAL DISCUSSION AND CONCLUSION

4. GENERAL DISCUSSIONS AND CONCLUSIONS

4.1 GENERAL DISCUSSION

There are many factors that influence the production of spirit products and more specifically brandy. These factors include the geographical area from which the grapes are sourced, the cultivar of the grapes used, the type of yeast strain used for fermentation, the vinification techniques employed, the type of distillation apparatus as well as the period of maturation of the brandy. However this study focussed mainly on the type of distillation apparatus used for the purpose of distillation and the effect that different types of pot still heads and swans neck apparatus have on the chemical composition and sensory profile of the resultant un-matured brandy.

The two low wines used in this experiment differ in their chemical composition prior to distillation. Low wine one contained a higher amount of total esters and total carbonyl compounds than low wine two. Therefore it is possible that the wine used for the distillation of low wine one was fermented at a lower temperature (Fundira et al. 2002). The total amount of esters found in French distillates were found to be 385 mg/L and in Italian distillates 406 mg/L (Von Adam et al. 1996). LW1 contains 261 mg/L of total esters however ester concentration in un-matured Pot still brandy is dependent on the original concentration prior to distillation. Low wine two contains a larger amount of higher alcohols and acids than low wine one which could possibly be due to a higher fermentation temperature of the wine prior to distillation (Fundira et al. 2002). Higher alcohols are produced by yeast and not the raw material thus indicating that low wine two was fermented with yeast strain that has a high production of higher alcohols (Jounela-Eriksson 1981).

Studies conducted by Nykanen (1986) also mention that if there are problems in the fermentation procedure then the concentration of n-propanol increases which is the case in low wine two. The levels of ethyl lactate are below the amount considered to be caused by malo-lactic bacteria and therefore spoiled i.e. 154 mg/L for both low wine one and two, thus indicating that both wines used for the production of the low wines did not undergo malo-lactic fermentation. Low wine one also contained the highest amounts of the long chain fatty acid esters and since these esters are produced when the wine or low wine is distilled with a larger lees content, it is clear that low wine one was possibly distilled with a larger percentage of lees.

The amount of ethyl acetate found in low wine one and two are greater than the required amount found in distillates to be considered to be microbially spoiled. If the ethyl acetate content is higher than 150-200 mg/L it is considered to be spoiled (Postel and Adam 180). However variation one in both low wine one and two decreases the amount of ethyl acetate when compared to the other variations, thus indicating that this variation can be used to decrease the ethyl acetate concentration found in the un-matured pot still brandy. Variation one in general in both low wine one and two decreases the total concentration of esters found in the final distillate. Similarly the total amount of esters in low wine one is also greater in the final distillate of low wine one, thus showing that the concentration of esters prior to distillation is a good indication of the esters that will be found in the final distillate.

It can be seen that the variations do have an effect on the final chemical composition, however variation one was found to alter the chemical composition of the distillates the most. This variation decreases the total esters in the distillates of low wine one and two irrespective of the original ester concentration of the low wines prior to distillation. Therefore the chemical composition of the un-

matured Pot still brandy is dependent on the chemical composition of the low wine prior to distillation rather than the variations themselves.

With regards to their sensory attributes, low wine one in general contains more of the fruity and sweet associated characteristics when compared to low wine two. This is due to the fact that low wine one contains more esters prior to distillation than low wine two, and it is known that esters have a large effect on the sensory characteristics of alcoholic beverages. LW1V1 is found to be less intense in the fruit and sweet associated attributes, this is due to the lower amount of esters found in this variation as Cantagrel (1992) stipulates that the removal of esters results in a negative effect on the overall intensity. Low wine 2 contains more of the fatty oily characteristics than low wine one, which is due to the increased fatty acid concentration.

LW1V1 is found to be negatively correlated to fruit and sweet associated attributes and total ester concentration, LW2V1 also displays the same trend. However LW2V1 is not negatively correlated to total esters, but is to higher alcohols, thus indicating that higher alcohols have an effect on the sensory profile of the variation. Therefore variation one does produce samples that are less intense in the fruit and sweet associated attributes and also lower esters. It is however still the original chemical composition of the low wine prior to distillation that largely determines the chemical composition of the final sample.

Studies by Leaute (1950), show that pot stills with a still head that has a larger surface area, i.e. variation one, will have a larger degree of reflux. This pot still head produces a distillate that is purer and less aromatic as the denser heavier compounds such as the long chain fatty acids will not be able to move up and over into the distillate. Brandy manufactured using variation one did indeed display these characteristics.

The results also show that low wine two is associated with the sensory classification four and five. The reason for this could possibly be because low wine two contains more of the higher alcohols and total acids than low wine one. Some of the variations in low wine one are also found to be positively associated with sensory classification five; however LW1V1 is found to be positively associated with sensory classification one. All variations influenced the composition and sensory profiles of the end product. However, variation one had an effect on the chemical composition that was different compared to the other variations. This therefore indicates that variation one produces a sample that is less intense in the fruit and sweet associated attributes and the reason for this is the lower amount of esters found in this variation and this is therefore correlated to sensory classification one.

Low wine one and two are found to contrast each other with regards to their chemical composition and sensory style classification, therefore indicating that the sensory classification is dependent on the original chemical composition of the low wine prior to distillation.

Sensory classification five is associated with the sensory attributes soapy aroma, fatty oily aroma, thatch aroma and sensory classification one is associated with the sensory attributes fruit, sweet associated aroma/flavours, sweet taste and spicy flavour, therefore indicating that an association between the sensory style classification, sensory attributes is dependent on the chemical composition of the low wines prior to distillation.

The addition experiments also showed that with the addition of esters and carbonyl compounds the sensory classification shifted from sensory classification five to one. Therefore indicating that esters do indeed play a role in the sensory classification of the sample and the addition of the higher alcohols and acids can also change the sensory classification system from one to four and five. Therefore these compounds do have a large effect on the sensory profile and characteristics

of the un-matured pot still brandy sample. However it is important to note, that chemical composition, sensory attributes and sensory classifications of the un-matured pot still brandy samples are more dependent on the original composition of the low wine prior to distillation rather than the variations themselves. Of the variations, variation one differed the most when compared to the other variations with its effect on the un-matured pot still brandy sample with regards to its chemical composition and consequently its sensory classification and sensory attributes.

4.2 CONCLUSION AND RECOMMENDATIONS

4.2.1 CONCLUSION

Distill produces many different styles of brandy. A need arose to investigate the effect of different pot still designs on the chemical composition, sensory profile and sensory style classification of un-matured pot still brandy due to the lack of information available in the industry thus far. If a specific pot still design could be used to predict the outcome of the final distillate it lead to a better understanding of the brandy production process and it could therefore be possible to manipulate the final chemical composition of the distillate and ultimately the sensory profile.

Of the variations used it seems that variation one has the largest effect on the outcome of the distillates. This variation produces a distillate with a smaller amount of esters and more specifically ethyl acetate. This variation is also found to have a less intense fruit associated and sweet associated caramel aromas, therefore indicating that the esters and more specifically ethyl acetate are responsible for these attributes. This variation can be used to produce a distillate with a lower amount of esters, less fruit and sweet associated aroma irrespective of the chemical composition prior to distillation. These results coincide with the literature of Leaute (1950) that states that this pot still design produces a distillate that is less intense and less aromatic when compared to other distillates. Even though variation one does coincide with the literature of Leaute (1950) and also has a larger surface area when compared to the other variations, it does however not differ with regards to its flow rate.

Distillates of low wine one contained a larger amount of ethyl acetate and acetaldehyde than low wine two. The reason for this could be due to the time of storage of the distillates prior to the sensory evaluation. Studies by Satora and Tuszynski (2008) indicate that the levels of ethyl acetate will increase with an increase in storage time as the dominant alcohol and acid in the distillate are ethanol and acetic acid respectively.

It can be seen that the addition of certain compounds could possibly alter the sensory style classification of the distillates, however further investigation i.e. more replications of the sensory evaluation of the spiked samples is needed to validate this. It does however show that a distillate with a lower concentration of esters and less fruit and sweet associated caramel aromas will result in a distillate of a sensory style classification one as in the case of variation one of the pot still designs.

The chemical composition of the distillates of low wine one seem to display a similar pattern as the chemical composition of the low wine that was used prior to distillation. The chemical composition of the distillates of low wine two are found to have a similar make up as the low wine two prior to distillation. It can therefore be deduced that the chemical composition and the sensory profile of the distillates is not dependent on the different variations of pot stills, but however largely on the original chemical composition of the low wines prior to distillation. It is also shown that the time taken for the storage of the base wine and the low wine prior to distillation can have a large effect

on the chemical composition and sensory profile of the resultant distillate, therefore making this an important factor to monitor during the production of brandy.

4.1.2 RECOMMENDATIONS

It would be recommended that the chemical composition of the low wine prior to distillation be analysed as this will give one an idea of the chemical compounds found in the low wine, and what the possible outcome of the chemical composition of the resultant distillate could be. Analysing the base wine as well as the low wine prior to distillation could also show if there are any compounds associated with spoilage which could indicate incorrect storage of the base wine and low wine prior to distillation. This would therefore insure the selection of good quality base wine and low wine for the production of premium quality brandy. Variation one should be used if the desired result is to obtain a distillate that contains a lower concentration of esters and a lower intensity of fruit and sweet associated aromas and flavours. Less application of different pot still designs should be employed in the industry as it is ultimately the chemical composition of the low wine prior to distillation that has the largest effect on the distillate.

Low wine one was profiled three months after distillation; this could have changed the sensory profile of the product as it could have lead to a possible increase in ethyl acetate and acetaldehyde thus influencing the sensory profile of the distillate. Therefore sensory evaluation of the resultant distillates should therefore be conducted as soon as the low wine is distilled as prolonged storage could alter the chemical composition of the distillate and therefore influence the sensory perception of the un-matured brandy. Profiling of the distillates of low wine one and two simultaneously would have possibly resulted in less repetition of results. This could have been achieved by applying a two-factor ANOVA with interaction testing the effects of LW and V (Variation) and LW x V in the first place. However, this was not a possibility due to the fact of the time constraints of the panel members.

The use of PCA (Principal Component Analysis) is appeared to be a limited multivariate technique with regards to the distillate products. Variable loadings are also influenced by the negative correlations among variables which could result in chemical compound/sensory attribute being “pushed” in a certain location because of this. Partial Least Square Regression (PLSR) forms a better correlation between the variables and with this technique it would have been possible to perhaps predict the outcome of the sensory profile of the distillates by forming a correlation between the chemical compounds and the sensory attributes.

The results obtained during the investigation of this dissertation could serve as an important basis of distillation of premium quality brandies. Future studies in this domain could provide valuable information facilitating the production of brandy of consistent quality and thus the more effective promotion of production of these products. This study provides important scientific insight into the effect of pot still design and low wine quality on the quality of brandy.

4.3 REFERENCES

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ADDENDUM A

Table A shows the amount of correct identifications needed of the odd sample by either 8, 9, 10, 11 or 12 panelists for the results of the tasting to be significantly different at either a 5, 1 or 0.1%.

Table A. The amount of correct observations needed for a significant difference between samples at a 5, 1 or 0.1%.

No. of panelists	5%	1%	0.10%
8	6	7	8
9	6	7	8
10	7	8	8
11	7	8	10
12	8	9	10

Data that was collected from the triangle tests were analyzed and evaluated according to the Roessler tables for triangle tests and the significance level calculated according to a 5% significance level. Table B shows the results obtained from the triangle tests and no significant differences were noted between the five variations of LW1 and LW2.

Table B. Results of the triangle tests for the different variations of pot still designs in LW1 and LW2.

Treatment 1 (Low wine 1)	Test 1 1 vs. 2	Test 2 1 vs. 3	Test 3 2 vs. 3	Treatment 2 (Low wine 1)	Test 1 1 vs. 2	Test 2 1 vs. 3	Test 3 2 vs. 3
Incorrect	6	5	10	Incorrect	7	9	7
Correct	3	4	2	Correct	4	2	4
Total	9	9	12	Total	11	11	11
Significant difference (p≤0.05)	8	8	8	Significant difference (p≤0.05)	8	8	8
Treatment 3 (Low wine 1)	Test 1 1 vs. 2	Test 2 1 vs. 3	Test 3 2 vs. 3	Treatment 4 (Low wine 1)	Test 1 1 vs. 2	Test 2 1 vs. 3	Test 3 2 vs. 3
Incorrect	9	5	8	Incorrect	6	5	7
Correct	1	5	3	Correct	5	5	3
Total	10	10	11	Total	11	10	10
Significant difference (p≤0.05)	8	8	8	Significant difference (p≤0.05)	8	8	8
Treatment 5 (Low wine 1)	Test 1 1 vs. 2	Test 2 1 vs. 3	Test 3 2 vs. 3	Treatment 1 (low wine 2)	Test 1 1 vs. 2	Test 2 1 vs. 3	Test 3 2 vs. 3
Incorrect	4	5	4	Incorrect	7	5	7
Correct	4	3	4	Correct	1	3	1
Total	8	8	8	Total	8	8	8
Significant difference (p≤0.05)	8	8	8	Significant difference (p≤0.05)	8	8	8

Treatment 2 (Low wine 2)	Test 1 1 vs. 2	Test 2 1 vs. 3	Test 3 2 vs. 3	Treatment 3 (Low wine 2)	Test 1 1 vs. 2	Test 2 1 vs. 3	Test 3 2 vs. 3
Incorrect	5	8	4	Incorrect	6	5	6
Correct	3	0	4	Correct	3	4	3
Total	8	8	8	Total	9	9	9
Significant difference (p≤0.05)	8	8	8	Significant difference (p≤0.05)	8	8	8
Treatment 4 (low wine 2)	Test 1 1 vs. 2	Test 2 1 vs. 3	Test 3 2 vs. 3	Treatment 5 (Low wine 2)	Test 1 1 vs. 2	Test 2 1 vs. 3	Test 3 2 vs. 3
Incorrect	8	6	5	Incorrect	7	5	7
Correct	1	3	4	Correct	1	3	1
Total	9	9	9	Total	8	8	8
Significant difference (p≤0.05)	8	8	8	Significant difference (p≤0.05)	8	8	8

ADDENDUM B

Addendum B is an example of a descriptive analysis tasting sheet used for evaluating un-matured pot still brandy.

Name _____

Sample _____

Please evaluate the sample presented for AROMA attributes

Fruit associated dried Aroma

None Intense
|-----|-----|-----|-----|

Soapy Aroma

None Intense
|-----|-----|-----|-----|

Coconut Aroma

None Intense
|-----|-----|-----|-----|

Spicy Aroma

None Intense
|-----|-----|-----|-----|

Herbaceous Aroma

None Intense
|-----|-----|-----|-----|

Sweet associated caramel Aroma

None Intense
|-----|-----|-----|-----|

Fatty oily Aoma

None Intense
|-----|-----|-----|-----|

Please evaluate the sample presented for FLAVOUR attributes

Fruit associated dried Flavour

None Intense
|-----|-----|-----|-----|

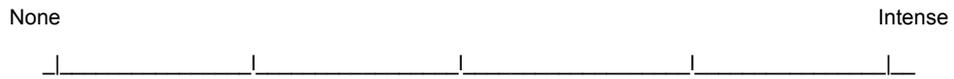
Soapy Flavour

None Intense
|-----|-----|-----|-----|

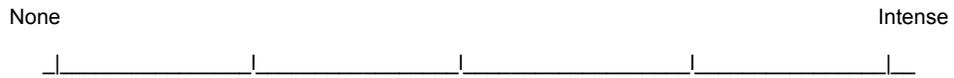
Spicy Flavour

None Intense
|-----|-----|-----|-----|

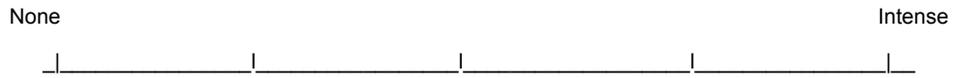
Herbaceous Flavour



Sweet Associated caramel Flavour

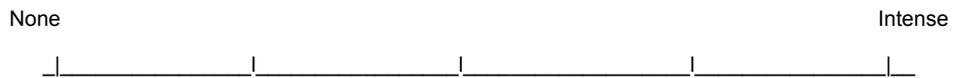


Fatty oily Flavour

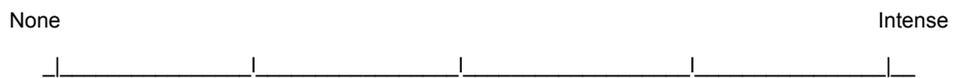


Please evaluate the sample presented for TASTE attributes

Sweet

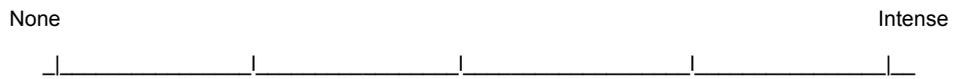


Bitter



Please evaluate the sample presented for MOUTHFEEL attributes

Alcohol burn



Mouth coating

