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## Spirits and Liqueurs from Melon Fruits (*Cucumis melo* L.)

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### 1. Introduction

Melon fruits, *Cucumis melo* L, are perishable and must be brought to market very quickly, which leads to saturation of the market and a surplus of melons. Increasing melon production has brought a serious marketing problem. Its fermentation and distillation could represent a potential solution to the problem. A review of the literature and other information sources failed to turn up prior experience with transformation of melon by means of alcoholic fermentation, making it necessary to develop a research protocol for investigating the suitability of melon juice, paste without or with skins and seeds as a fermentation substrate and behaviour of the resulting melon wine during distillation, with a view to obtaining a melon spirit or liqueur with appropriate flavour and aroma attributes.

Distillation of fermented fruit wines has been used in some countries for many years to obtain palatable beverages with high alcohol contents. The most important distilled spirits are elaborated from diverse raw material as grapes (Brandy, Grappa, Orujo), malt (Whiskey), cane sugar (Rum), etc. Other distilled beverages come from the distillation of fermented fruits, named in some zones "wine fruits", such as cherries, apples or pears. Several countries produce spirits obtained by steam distillation of the anaerobically fermented grape pomace, left over after grapes have been crushed during wine-making. These spirits contain 30-45 % (v/v) alcohol and are highly appreciated, especially after gourmet meals.

In Spain, the distillation of the grape wine and its by-products is frequent, but is unusual the distillation of "wine fruits". Actually, the maceration of agricultural products, generally fruits, or the addition of artificial essences and aromas to wine spirits is made to elaborate different kinds of liquors, though natural cherry or pear spirits have been developed years ago.

Most commercially available spirits (grappa, orujo, bagaceira, kirsch, plum, brandy) are filtered, bottled, and sold without maceration. However, on occasion fruits, seeds, and leaves are used as substrates to modify the product and improve its sensory qualities diversifying their range and attributes. This process is used in Spain to make a number of drinks, such as "pacharán" (maceration of sloe, *Prunus spinosa*, berries) or "anissette" (maceration of anise, *Pimpinella anisum*, seeds). During maceration, aromatic substances are

leached out of the fruit into the spirit, which may then be redistilled or bottled as the finished product. Maceration time is a key factor both for component extraction and for achieving the right sugar content and colour of the spirit. The amount and the parts of the fruit used in maceration are two other aspects to be considered. For fleshy fruits like pears, apples, raspberries, strawberries, and cherries, pieces of fresh fruit may be used, or the seeds or nuts (blackthorn, hazelnuts, almonds) may be employed. Substrate conditioning is also an important factor, and may include pieces of fruit of different sizes, seeds, placenta, or skins depending on the type of product being manufactured.

Melons are a major crop in the La Mancha region (Spain) and the large crop size results in high levels of surplus production which must be commercialised in a very short period of time. The fermentation of melons and its distillation to produce genuine spirits could be a solution to the problem of the saturation of the market that would prevent wastage. The process of developing a new product has to be undertaken step by step, and for that reason trials to examine fruit processing, clarification, fermentation, column and alembic distillations were performed at laboratory and pilot scale (Briones, et al., 2002; Hernández Gómez, et al., 2003). Chemical and sensory analyses were carried out to assess the quality of the spirits and liqueurs produced comparing the results with those in other commercially available spirits.

## 2. Fruit processing

Healthy melons, *Cucumis melo* L, of the varieties “Piel de sapo”, “Ruidera” and “Sancho” coming from cultivars of La Mancha region in Spain were processed. Melons were washed and divided into three sets and processed in different ways to obtain three substrate types ready for their fermentation:

\* Juice: The melons were hand-peeled, cut up into pieces manually, after that crushed and peeled in a horizontal des-temmer with rollers to produce a paste that was then pressed in a pneumatic vertical press

\* Paste without skin (“pws”): The melons were processed as described above, but not pressed.

\* Paste: The unpeeled melons were cut up into pieces directly and then crushed to form a paste that included the skins.

The yield of an industrial process must be calculated carefully since it is of great economic importance as well as conventional fermentation parameters such as °Brix, reducing sugars, volatile acidity, pH and alcohol degree. The yield (w/w) of the processed fruits depended on the substrate assayed. Therefore in the case of the paste it can reach 100%, 70% (30% of skin) for the pws and only 50% for the juice. This was mainly caused by the percentage of melon removed as skin in the case of the “pws” and by difficulties in filtering the melon paste to produce the “juice”. Juice extraction yields of up to 75 % have been attained for other fermented products, such as grape because of the small weight of the skins. These yields were improved and an increase of 10 % were recorded for the juice and pws substrates, perhaps thanks to optimization of procedures. By-product yields were 21 % (w/w) of skins for the pws substrate and 40 % (w/w) skins and pressed pulp for the juice substrate (Hernández-Gómez et al, 2005a).

The initial pH of the different substrates varied between 4.4 and 5.2. and this supposes a problem for the easy growing of lactic and acetic acid bacteria; for that, pH was adjusted or not before the fermentation by adding citric acid to reach values around 4 in order to inhibit these bacteria.

### 3. Characterisation of fermented from melon fruits

Fermentation of the juice, pws and paste was carried out at 20 °C. The substrates were inoculated with a commercial yeast (*Saccharomyces cerevisiae* UCLM 325) up to a concentration of approximately 10<sup>6</sup> cells/mL. The process was monitored daily by measuring residual sugars, and the end of fermentation was determined on the basis of the sugar consumption (OIV, 1969). The initial assimilable nitrogen was measured using the NitroGenius® kit.

Judging from the initial °Brix, between 10,0-10,2 an alcohol degree of 5% (v/v) could be expected. Nonetheless, experimental data showed that the ethanol yield was acceptable only in juice and pws (4.2 % v/v), being very low (3.4 %) in the case of the paste, possibly due to the complexity of the structure of the fermentation media.

The fermented paste showed the highest values of acetic acid possibly as a consequence of a contamination by acetic bacteria. Adjusting the pH, successfully bacterial growth in all three melon wines produced was diminished. Under unadjusted pH conditions, the bacterial populations increased in the pws and paste substrates but decreased in the juice. This may be attributable to a higher level of contamination from the melon skins or to sluggish fermentation in a complex media like paste substratum.

In this kind of alcoholic beverages, concentrations of the volatiles has to be refereed to the ethanol content. Otherwise, the volatile composition is closely related to the type of substratum, the conditions of the fermentation and the yeast strain used. Respecting the major volatiles, acetaldehyde ranged widely from 243 to 1196 mg/L of ethanol, being highest in the pH-unadjusted substrates, possibly due to the action of spoilage microorganism (Silva et al., 2000). Methanol is not a direct product of fermentation (Ribéreau-Gayon et al., 2000). Two types of fruit enzymes are able to act upon pectins to release methanol: polygalacturonases, by cleavage of the glycosidic bonds on the chains; and pectin-Methylesterases, by catalyzing hydrolysis of the esterified chemical function (Hernández Gómez et al., 2003). The presence of high amounts of methanol in the wine fruit produced from the paste at both pH levels may be the result of the action of these enzymes in the skin.

In general the higher alcohols (HAs) quantified [1-Propanol, isoAmyl alcohols , 1-Butanol, and 2-Methyl-1-Propanol] were higher with pH adjusted, especially in the case of the wine made from the pws. 1-Butanol and 2-Butanol were not detected, a highly positive finding, because these two substances adversely affect the final aroma of the distillate. Total esters were higher in the pH-unadjusted wine made from paste than in the rest of the wines. Ethyl lactate was the main contributor to this high value and probably depends on the initial count of lactic acid bacteria, present in this kind of substratum (Briones et al. 2002).

When ANOVA statistical analysis was applied it was noticed that except for Ethyl acetate, there were differences in the volatiles for all the melon wine types, especially between the wines made from the paste and the rest.

#### 4. Distillation procedure and analysis of spirits

Upon completion of alcoholic fermentation, the fermented were immediately distilled with yeast lees in two ways in previous assays: in rectifying glass column and in French type copper pot. In the first case, a glass column of 50 cm of length and filled up to 50% with Raschig rings and a round bottomed 10 L flask were used. The flask was filled with 5 L of every type of fermented fruit. To ensure a homogenous heat distribution during the distillation process, boiling stones were added and the flow rate was adjusted at 10 mL min<sup>-1</sup>. In the second case, a 30 L French type copper pot filled with 15 L of fermented fruit was used and the flow rate was adjusted at 25 mL min<sup>-1</sup>. In both cases the fermented were double distilled. The first distillation was stopped when the alcohol degree was lower than the fermented fruit, obtaining a distillate around 17-20% (v/v). In the second distillation, the first phase was the collection of 0.8% of distillate (heads) which was discarded. This distillation was stopped at around 30% (v/v), so the final distillate (heart fraction) reached an alcohol concentration around 55% (v/v). The tails were formed adding the fractions ranging from 30 % (v/v) to 5 % (v/v). The distillate was collected in fractions of different volums depending of the equipment used. In these kind of processes and in order to avoid the loss of aromas all the fractions were collected on ice and kept at 4°C until their analysis. The percentage alcohol content in all of them was determined by electronic densimetry in all the fractions (European Union, 2000). The heart fraction alcohol degree values are shown in Table 1. In both distillations (copper pot and column) the alcohol degree of the paste was lower because the initial degree of the fermented was also inferior.

		Alcohol degree (% v/v)			
		1 <sup>st</sup> fraction	2 <sup>nd</sup> fraction	3 <sup>th</sup> fraction	Average value
Copper pot	Juice	68.6	61.5	44.2	58.1
	Pws	65.6	52.1	27.9	48.5
	Paste	69.8	62.8	44.6	59.1
Column	Juice	79.5	60.5	12.3	50.8
	Pws	80.0	61.3	11.2	50.8
	Paste	75.0	54.0	10.6	46.5

Table 1. Alcohol degree of second distillation for different fractions collected in the copper pot and in the glass column

Respecting the chemical analysis of major volatiles, the glass column samples possessed higher concentrations of Propanol, 2-Methyl 1-Propanol and 3-Methyl 1-Butanol. Moreover, the sensory analysis of the first batch of distillates offered conclusive results so as to the distillation type. 100% tasters preferred the samples distilled in the copper pot due to its aroma intensity while the other ones coming from glass column were rejected on basis of their pungent and /or “not sufficiently intensive” aroma.

With regard the type of substrate, even though the “paste” type offered a better yield in the process, it does not seem to be the ideal substrate due to the sluggish of fermentation, a high methanol content and the negative sensory characteristics. Juice distillate was more appreciated due to its aroma intensity (Hernández-Gómez et al., 2008).

To compare the results with those from previous years using a glass column, and thus narrow down the production processes, a pilot-plant copper pot, *alquitara*, was used. At the

same time, the volatile compounds in the distillates obtained were compared with those in other commercially available spirits.

The fermented juice was immediately distilled in a traditional 130-L “alquitara”, (reflux still) (Silva, Macedo & Malcata, 2000) filled to 70-80 % of capacity, equipped with a series of temperature sensors. Distillation flow rate was set at 170 mL/min, and the condenser was kept at below 21 °C throughout. The distillate was collected in volumes of 1 L each, except for the head fraction. The first distillation was stopped when the alcohol content in the volume collected had reached 8-10 % (v/v), which yielded a distillate with an alcohol content of 18.5-25 % (v/v), depending on the source substrate from which it had been made. The head-fraction (200 mL), usually discarded, was not rejected.

The second distillation was carried out in a traditional 30-L alembic copper still filled with 15 L of the first distillate. Distillation flow rate was set at 35-40 mL/min. The heads, 0.8 % of the distillate, were discarded, and distillation was stopped at 40 % (v/v), thus yielding a final distillate (hearts) of 58-69 % (v/v), again depending on the source substrate from which it had been made. The tails comprised the fractions from 40 % (v/v) to 5 % (v/v). For all the distillates the alcohol content of the last volume collected was between 9.2 and 11.8 % (v/v), and the final alcohol content was between 18.5 and 25 % (v/v). In the second distillation, the alcohol content decreased from 74.5 % (v/v) to 40.0 % (v/v) in the last volume collected. The total distillation time for all the fractions was around 4 h. The highest value was for the juice distillate (pH-adjusted) and the lowest for the paste (pH-unadjusted) distillate.

After the second distillation the major volatiles present in the heads, hearts, and tails fractions of the different spirits are depicted in Figure 1.

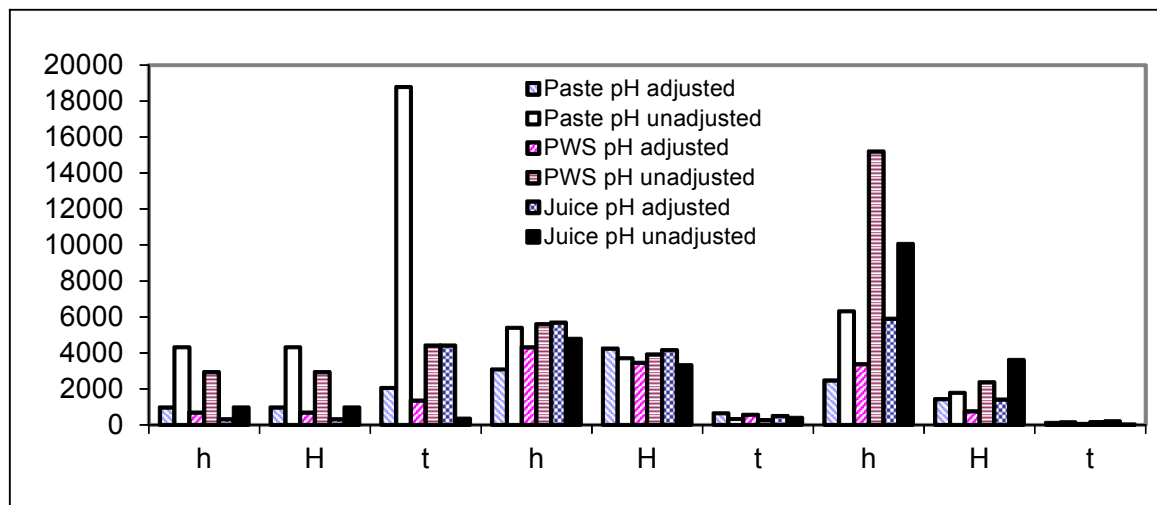


Fig. 1. Evolution in major volatile compounds during the second distillation. Methanol. Higher alcohols: 2M1P, 2M1B, 3M1B, and 1-Propanol. Esters: Ethyl lactate, Ethyl acetate, and Ethyl butyrate (mg/L of EtOH). h, heads; H, hearts; t, tails. pws = paste without skins

Methanol was collected in nearly the same proportion in all the fractions, most likely due to the formation of azeotropic mixtures (Orriols, 1994). Nevertheless, methanol concentrations were higher in the distillates from the pH-unadjusted wines except for “juice” tails. High levels of methanol in the paste distillate were observed (pH-unadjusted). The higher alcohols (HA)

content was higher in the heads and hearts than in the tails. All the distillates displayed the same behaviour, with no considerable differences among them. In addition, the HA content was not related to the fermentation pH and substrate types. The ester content (Ethyl acetate, Ethyl lactate, and Ethyl butyrate) was higher in the heads, decreasing in the hearts and the tails. Fermentation pH had a pronounced influence on the ester content.

Respecting the concentrations of the volatiles in the final spirits, high amounts of methanol and 2-Butanol were noticed. They can make spirits hazardous to consumers health. Moreover, methanol imparts a cooked cabbage odour, with a threshold of 1200 mg/L (Ribéreau-Gayon, et al., 2000). The methanol content was higher in the distillates made from the pH-unadjusted in all substrates. The paste distillates exhibited the highest levels, probably owing to the action of certain pectinases on the substantial amount of melon skins present (Cortés Diéguez, et al, 2000). However, in no case did the levels exceed the limits for fruit spirits set by the legislation currently in force (European Union, 1989).

Higher alcohols are responsible for imparting complex sensory attributes to spirits (Silva et al., 2000). The Amyl alcohols and 2-Methyl 1-Propanol contribute positive to the sensory characteristics (Bertrand, 1975; Orriols, 1992, 1994). They are detectable organoleptically at concentrations below 15 mg/L of ethanol (Tourliere, 1977). 1-Propanol has a pleasant, sweetish odour, but excessive concentrations will introduce solvent notes that mask all the positive notes in distillates (Fundira, Blom, Pretorius, & van Rensburg, 2002). The highest 1-Propanol contents were recorded in the distillates made from the pH-adjusted fermentation substrates, in contrast, 2M1P was lower in the pH-adjusted distillates at levels similar to those reported in previous years. Amyl alcohol contents were very similar in all cases. 1-Butanol has a heavy, penetrating odour, and 2-Butanol is associated with low-quality raw materials (Orriols & Bertrand, 1990; Cortes Dieguez et al., 2000). 1-Butanol concentrations were higher in the pH-adjusted distillates. Conversely, 2-Butanol was not detected in any of the distillates produced. 2-Phenylethanol imparts a very clinging, rose-like aroma (Nykänen & Suomalainen, 1983), and was not influenced by pH, except in the juice distillates. 1-Hexanol, cis-3-Hexen-1-ol, and 3-Methyl-3-buten-1-ol impart strong herbaceous aromas. Hexanol and cis-3-Hexen-1-ol perception thresholds in spirits are 20 mg/L and 3.5 mg/L, respectively (Jouret & Cantagrel, 2000), and the concentrations did not exceed those values. Smoked or burnt wood aroma is conferred by 4-Methyl-guaiacol (Dubois & Dekimpe, 1982), and it was present in all distillates except the pH-adjusted juice. Benzyl alcohol is related to the quantity of benzaldehyde, the latter being important because it imparts a bitter almond aroma to wines at levels above 2-3 mg/L (Blaise, 1986). In the melon spirits, concentrations were nowhere near that perception threshold reported for wines.

Otherwise, Acetaldehyde is 90 % of the total aldehyde content in distillates (Versini, Monetti, dalla Serra & Inama, 1990, Orriols, 1991; Silva, Malcata, & Hogg, 1995). More than 1200 mg/L of ethanol is evidence of oxidation of the ethanol during alcoholic fermentation or a enzymatic pyruvic acid decarboxylation (Baro & Quiros-Carrasco, 1977; Cantagrel, Lablanquie, Snakker, & Vidal, 1993). Its importance derives from its pungent odour and its chemical reactivity (Silva et al., 2000). pH had no effect in juice and pws substrates, in contrast, it was doubled in pH-adjusted paste distillate. Furfural may be formed as a result of oxidation of ascorbic acid (Bayonove, Baumes, Crouzet, & Günata, 2000). A slightly higher furfural content in the distillates from the pH-unadjusted substrates was observed.

Esters are associated with pleasant odours. This is particularly true of Ethyl acetate, which contributes to aroma complexity and has a positive impact at very low levels (50-80 mg/L) (Steger & Lambrechts, 2000). Ethyl acetate was higher in the distillates pH-unadjusted. Ethyl lactate contributes intense, long-lasting aromas (Tourliere, 1977). The content in distillates is linked to lactic fermentation, in general it was lower in pH-adjusted distillates (Briones et al., 2002). Ethyl butyrate adversely influence the organoleptic quality of distillates (Soufleros, 1978,1987). Paste distillates at both pH had the highest concentrations. Concentrations of the minor esters, other than Ethyl lactate and Ethyl acetate, were higher in the pH-adjusted distillates, due principally to Ethyl caprylate, Ethyl palmitate, Ethyl caproate, Ethyl laurate and Ethyl decanoate.

Respecting carboxylic acids, short-chain (C4-C12) fatty acids produce unpleasant odours, and high concentrations are an indicator of poor quality (Orriols, 1992,1994). Decanoic and octanoic acids were the most abundant fatty acids and were present at higher concentrations in the pH-adjusted distillates.

#### 4.1 Comparison with commercial spirits

The major volatile compounds of melon spirit were compared with other commercial spirits such grappa, orujo, and other fruit distillates from cherry, raspberry and pear (Hernández Gómez et al, 2005a) . In general, 1-Propanol was higher in melon distillates, being similar to those in grappa and cherry spirit. In like fashion, Ethyl acetate was much higher in the juice distillate with a concentration nearly three times more than in the other spirits. Acetaldehyde and Ethyl lactate were present in similar amounts in most of the spirits considered, with the exception of much higher concentration of Ethyl lactate found out in the cherry spirit, most probably as a result of the lengthy maceration time. 2-Methyl 1-Propanol and Amyl alcohols were somewhat higher in the melon distillates, pear spirit, and grappa. The melon distillates had appreciably lower methanol contents than the rest of the spirits considered, particularly as compared to the pear spirit (11216 mg / L of ethanol), most likely because of the high pectin content of pears. 1-Butanol was detectable in the pear spirit and, to a lesser extent, in the melon distillates. 2-Butanol and Ethyl butyrate were not present in detectable amounts in any of the spirits analysed.

Sensory analysis was performed in a standard tasting room [Spanish standard 87004:1979 (Aenor, 1977). Distillates were diluted with distilled water to alcohol strength of 28 % (v/v) and served at a temperature of 15 °C. Evaluations were carried out at daily sessions between 10:00 a.m. and 12:00 noon to prevent taster fatigue.

There were significant differences between juice distillates and pws and conversely, there were no significant differences between the paste distillates. The tasters preferred pws or juice distillates, but there was no preference between them. The expert tasters from a distilling company likewise expressed no preferences, possibly because the spirits being tested were new to them and had an unfamiliar flavour.

### 5. Maceration for improving melon spirit

The effect of maceration in increasing and improving aroma intensity in a melon (*Cucumis melo* L.) spirit was studied.



Double-distilled melon spirit (alcohol content 56 % v/v) was macerated using different melon parts (pieces of melon flesh, skinned and then sliced (MF) (72.5 % w/v), and seeds+placenta (MSP) (23.5 % w/v). Contact times were 8 days, 18 days, and one year. The solid percentage was selected based on literature data (Xandri, 1958) and personal communications. After steeping, the distillate was chilled and filtered and the solids pressed and 0.3 g/L bentonite was added for clarification.

Sugar levels in the double-distilled spirit macerated with sliced melon flesh (MF) were four to five times higher than in the spirit macerated with the seeds+placenta (MSP), possibly due to the higher solids content (72.5 % in the MF batches compared with 23.5 % in the MSP batches). Total sugar levels stayed practically constant throughout maceration, though after maceration for one year saccharose levels fell while glucose and fructose levels rose by 80 and 60 %, respectively, perhaps because of hydrolysis of the disaccharide.

Dry matter (DM) was much lower (0.10 g/L) in the unmacerated control batch (C) than in the macerated spirit. The spirit macerated with sliced melon flesh for one year (MF1Y) had the highest values (26.95 g/L), compared with the spirit macerated with the melon seeds+placenta also for one year (MSP1Y), (21.53 g/L), because of the larger amount of macerated solids.

The alcohol content (% v/v) decreased in the macerated double-distilled spirit, the decrease being more pronounced in the macerates made with the sliced melon flesh, possibly attributable to absorption by the melon tissue and the release of larger amounts of water from the pieces of melon flesh.

Respect to the major volatiles, Methanol levels decreased with maceration but in all cases were within the limits set by the legislation currently in force (European Union, 1989) The higher alcohols (HAs), namely, 1-Propanol, 2-Methyl-1-Propanol, 2-Methyl-1-Butanol, and 3-Methyl-1-Butanol, and the esters Ethyl lactate and Ethyl acetate fell appreciably during maceration. These decreases were much more pronounced in the spirit macerated with the sliced melon flesh (MF8 and MF18) than in the spirit macerated with the seeds+placenta (MSP8 and MSP18). Ethyl butyrate was not detected. 1-Butanol, which has a very pungent and heavy aroma (Nykänen & Suomalainen, 1983), was present in lower concentrations in the macerated spirits, and their decrease could have a positive influence on the aroma of the final spirit.

Levels of Benzyl alcohol, 1-Hexanol, t-3-nonenol, c-3-Hexen-1-ol, 2-Phenylethanol, and geraniol were higher in the batches macerated with the sliced melon flesh and the seeds+placenta. 2-Phenylethanol imparts an aroma of roses (Nykänen & Suomalainen, 1983), and the concentration of this compound was three to four times higher than in the control spirit. At high levels 1-Hexanol and c-Hexen-1-ol may contribute herbaceous aromas, while at low levels they may exert a positive influence (Bertrand, 1975, Orriols, 1992, 1994).

On the whole, the ester content in the macerated spirits were lower than in the control spirit, with substantial decreases in Ethyl caproate, Ethyl caprylate, Ethyl decanoate, and Ethyl laurate. At the same time, Ethyl palmitate decreased in the spirit macerated with the sliced melon flesh but remained constant in the spirit macerated with the seeds+placenta because of the quantities that leached out of the melon seed (Al-Khalifa, 1996) . Ethyl linoleate and Ethyl linoleate were also related to the seeds and increased considerably in the spirit macerated with the seeds+placenta.

The concentration of total acids was lower in the macerated spirit, especially because of pronounced decreases in hexanoic acid, octanoic acid, and decanoic acid. Lauric acid levels increased considerably following maceration with both the sliced melon flesh and the seeds+placenta, possibly as a result of hydrolysis of Ethyl laurate.

Furfural is produced by acid hydrolysis or during the heating of polysaccharides containing hexose or pentose fragments, the highest concentrations being found in alcoholic beverages (1-33 mg/kg). This compound is currently allowed, since it is naturally present in fruits and other foodstuffs (European Union, 2002). Furfural and hydroxyMethyl furfural increased substantially in the spirit macerated with the sliced melon flesh as compared to the control spirit, but values were still within allowable limits.

The concentration of phenolic compounds was higher in the macerated spirit. This was particularly true for 4-Methyl-guaiacol, which increased appreciably in the spirit macerated with the sliced melon flesh. In wine this compound has a negative impact on aroma at levels higher than 4 mg/L but a positive impact at concentrations between 1.2 and 2.4 mg/L (Etiévant, et al., 1989).

Benzaldehyde, a carbonyl compound, decreased in the macerated spirit. Concentrations of this substance higher than 2-3 g/L are related to a bitter almond flavour in wines (Blaise & Bruns, 1986).

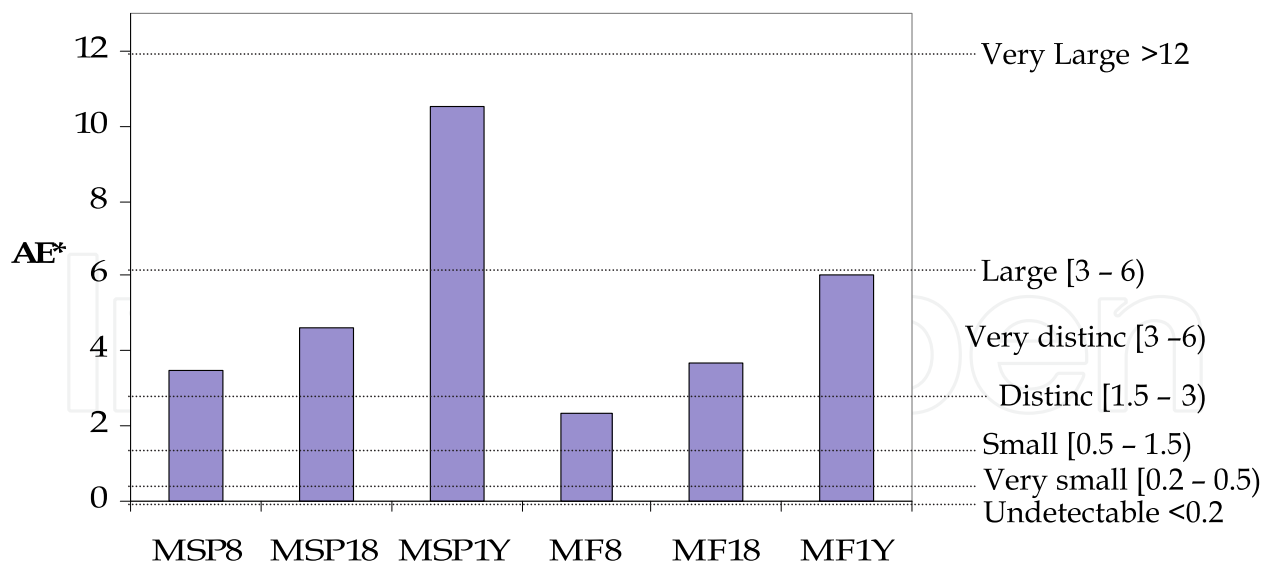
Increased levels of acetoin (3-hydroxy-2-butanone) act as an indicator of oxidation of 2-3 butanediol during ageing (Jouret & Cantagrel, 2000). The increase was higher in the spirit macerated with the sliced melon flesh.

In the colour attribute determinations, the tristimulus values, to derive the rectangular ( $L^*$ ,  $a^*$ ,  $b^*$ ), cylindrical ( $L^*$ ,  $C^*$ ,  $h^*$ ) and the chromaticity ( $x$ ,  $y$ ,  $z$ ) coordinates were used (CIE, 1986). The coordinates  $a^*$ ,  $b^*$ ,  $h^*$ , and  $C^*$  yielded the two-dimensional (CIELAB) colour space, where  $h^*$  is the angle formed with the  $a^*$  axis and  $C^*$  is the distance to the origin. Sample MSP1Y had the highest colour intensity ( $C^*$ ) values, and in terms of chromaticity values, and MSP18 was similar to sample MF1Y. Variation in the angle ( $h^*$ ) was minimal, with angles in the range of 90.14 to 94.46. All the samples fell in the region pale, with macerated sample MSP1Y exhibiting the highest colour intensity and thus falling closer to the region of lightness. Thus, maceration affected both lightness ( $L^*$ ) and colour intensity ( $C^*$ ), with the unmacerated spirit and the spirit macerated with the sliced melon flesh being paler than the spirit macerated with the seeds+placenta.

Figure 2 represents the differences in colour ( $\Delta E^*$ ) between the macerated spirit and the control spirit and also lists the gradations in visual perception according to Schmidhofer, et al., (1994). The longest maceration time (1 year) exhibited higher  $\Delta E^*$  values than the shorter maceration times, and the three macerations carried out with the seeds+placenta exhibited higher  $\Delta E^*$  values than the macerations carried out with the sliced melon flesh.

The results of sensorial analysis thus indicate that maceration did have an influence on the final product and that the panelists perceived distinct differences between the samples. The distillate macerated for 11 days (MF11) and the unmacerated control batch were significant at the 99.9-% level.

The preference tests failed to yield any preference for either the macerate spirit or the control sample. This result is ascribable; on the one hand, to the diverse make-up of the taste



MSP8: seed+placenta 8 days macerate; MSP18: seed+placenta 18 days macerate; MSP1Y: seed+placenta 1 year macerate; MF8: flesh 8 days macerate; MF18: flesh 18 days macerate; MF1Y: flesh 1 year macerate

Fig. 2. Colour differences ( $\Delta E^*$ ) between the macerated double-distilled spirit samples and the unmacerated control spirit (C) and perception levels as per Schmidhofer (1994).

panel and, on the other, to the fact that some panelists preferred a "flatter" spirit with traditional distillation aromas, while others preferred a spirit with an intensely "fruity" melon aroma.

### 5.1 Optimization of maceration process

A trial run at industrial scale is the final step in new product development. Maceration time and substrate are important factors, and the latter may include pieces of fruit of different sizes, seeds, placenta, or skins depending on the type of product being manufactured. For that preliminary trial macerations using melon (Hernandez-Gomez et al., 2005b) yielded positive results in terms of extraction of colour and typical melon aromas, suggesting that macerated spirit could be used to produce an authentic liqueur reminiscent of the fruit employed in production.

Different proportions of fruit were tested based on the results of previous maceration trials, taking unmacerated distillate and adding:

- Melon seeds + placenta in a proportion of 0.18 kg/L [MSP11], 0.23 kg/L [MSP12], and 0.28 kg/L [MSP13]
- Melon pieces + seeds + placenta in a proportion of 0.40 kg/L melon pieces + 0.10 kg/L seeds + placenta (MPSP11) and 0.30 kg/L melon pieces + 0.20 kg/L seeds + placenta (MPSP12)

Maceration time was 16 d, and the sugar content was measured at 0, 4, 8, 12, and 16 days of maceration.

Respect to total sugars (fructose, glucose, and saccharose) at the end of the maceration period, the total sugar content in the macerated with melon pieces and seeds+placenta was double that of the batches macerated with seeds+placenta only basically as a result of

diffusion out of the melon pieces and the larger proportion of macerated substrate. Analysis of each of the sugars separately revealed that due to hydrolysis of the disaccharide by the fruit enzymes, the saccharose content gradually decreased from day 8 on, while the glucose and fructose contents rose.

According to main volatile components concentrations of esters and higher alcohols, of great organoleptic importance (Baro & Quiros-Carrasco, 1977), were higher in the batches macerated with seeds and placenta and with melon pieces + seeds and placenta. Neither n-Butanol nor Ethyl butyrate were detected in any of the batches, a highly positive finding in that these compounds are deemed to produce off flavours when they are present in distillates.

For the colour measurements, the value of  $L^*$  ranges from 0 for black to 100 for white and was extremely high for the spirits considered here, between 91.9 and 95.5, the highest value being recorded for the unmacerated distillate, which also had the lowest Chromaticity ( $C^*$ ).  $C^*$  values increased with substrate content and maceration time, while lightness ( $L^*$ ) decreased with maceration time and was unrelated to the amount of macerated substrate.

All the batches can be grouped close together in the greenish yellow quadrant, though in the region closer to yellow. The macerated with melon pieces were greenish in colour and those with seeds+placenta were yellow-orange in colour because of the higher proportion of carotenoids.

Finally, the results of the preference test carried out on all the macerated batches as part of the sensory analysis indicated a slight though non-significant preference by panelists for the kirsch over the raspberry and melon spirits.

## 6. Melon liqueur

At the same time, production of an authentic melon liqueur was addressed. Three melon liqueurs were prepared (Hernández-Gómez et al., 2009). The alcohol content was adjusted to 280 ml/L (28% v/v), and the saccharose content was 100 g/L (CAE, 1997).

The resulting liqueurs were thus:

- "Melon juice liqueur": distillate + 200ml/L melon juice
- "Melon spirit liqueur": distillate + saccharose and distilled water to the corresponding alcohol content
- "Macerated liqueur": macerated spirit + saccharose

Sensory analysis of the three liqueurs was based on preference tests in the same conditions as the sensory analyses described above. Nevertheless, none of the liqueurs was statistically preferred at the 95 % significance level, though the macerated melon liqueur received the highest scores. The product was novel and unrelated to the panelists' prior experience, and as a result while some of the panelists preferred the unmacerated distillate on account of its "clean" aroma, others preferred the macerated melon liqueur on account of its fruity aroma and melon flavour.

## 7. Conclusion

Melon in the form of juice or paste without skin, constitutes an appropriate fermentation substrate, the sugars being consumed during fermentation to produce alcohol yields in

accordance with expectations. The paste substrate offers a better yield in the process, but it does not seem to be an ideal substrate.

Differences were observed in the contents of the major volatiles in the different melon wines. The fermentation pH brought about perceptible differences based on both the chemical and the sensory analysis. Adjusting the pH brought about substantial decreases in the acetaldehyde and methanol contents, a facet that will have to be borne in mind in the case of methanol in view of the maximum limits set by regulations. Nevertheless, the tasters expressed no preference between paste without skin and juice according to fermentation pH.

From an industrial standpoint, the paste without skin substrate can be regarded as preferable, because it produces less waste with a lower environmental impact and it is not necessary press the paste to obtain it.

Since no preferences were observed for any of the samples of first distillate macerated in between the two distillations, this procedure would not seem to be warranted to improve distillate quality, while conversely it would increase production costs by requiring higher melon consumption during maceration, greater investment in equipment (maceration tanks, presses, and screens), longer production times, and more processing steps.

In contrast, maceration of the final double-distilled spirit did enhance the colour and aroma attributes of the final product, hence further research will be needed to focus on achieving the right sugar levels, aroma intensity, and colour.

On the basis of the results obtained in the present study, maceration is considered unsuitable in that it is too costly for industrial purposes, would yield too much production waste requiring alternative uses, and failed to attain the golden colour of the spirits with melon pieces +seeds+placenta and with melon seeds+placenta.

From a sensory standpoint the taste panelists did not evince any preference between the two spirits, and consequently we would recommend melon seeds+placenta spirit for production on account of its deeper colour, its suitability for filtration, and its typical melon aroma, as well as its lower production costs.

Lastly, comparing the melon liqueur to other commercially available fruit spirits indicated that the acceptability of this product was similar to that of the other liqueurs tasted.

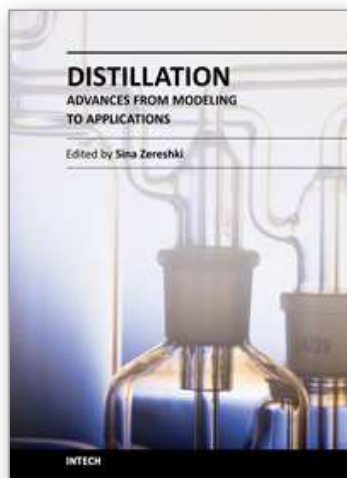
Consequently, it may be concluded that using fruits for spirit production may offer a viable industrial alternative that will help keep fruit output from going to waste.

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## **Distillation - Advances from Modeling to Applications**

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Distillation modeling and several applications mostly in food processing field are discussed under three sections in the present book. The provided modeling chapters aimed both the thermodynamic mathematical fundamentals and the simulation of distillation process. The practical experiences and case studies involve mainly the food and beverage industry and odor and aroma extraction. This book could certainly give the interested researchers in distillation field a useful insight.

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