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Melon fruit distillates: comparison of different distillation methods

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Abstract

Melon fruits (*Cucumis melo*) were crushed under different conditions, to give different substrates with around 11 °Brix, and fermented at controlled temperature (20 °C) by a *Saccharomyces cerevisiae* (commercial strain). Afterwards, the fermented material was double-distilled in two different ways: in a distillation column and in a copper pot, yielding a distillate with around 55% alcohol (v/v). The concentrations of the volatile compounds were different, depending on the fermentation conditions and the way of distillation.

Keywords: Melon fruit; Spirits; Distillation; Fruit fermentations

1. Introduction

The most important distilled spirits are elaborated from diverse raw material, such as grapes (brandy, grappa, orujo), malt (whiskey) or cane sugar (rum). Other distilled beverages come from the distillation of fermented fruits, named in some zones "wine fruits", such as cherries, apples or pears.

In Spain, the distillation of grape wine and its by-products is frequent, but the distillation of "wine fruits" is unusual. Maceration of agricultural products, generally fruits, or the addition of essences and aromas to wine spirits, is done to elaborate different kinds of liquors.

Castilla La Mancha (Spain) is a region which produces a great number of melon fruits (*Cucumis melo*) which must be commercialised in a very short period of time. The fermentation of melons and distillation to produce genuine spirits could be a solution to the problem of the market saturation.

In previous research we studied this type of fermentation on a laboratory scale (Briones, Hernandez Gomez, & Ubeda, 2002) and the results obtained supported the aim of this work: the evaluation of different fermentation processes using different melon substrates and the calculation of the yields. Also, two different ways of distillation, in a copper pot or in a rectification column, to obtain a spirit with adequate flavour and aroma following the European Union rules were studied (European Union Rules No. 1567/89, 1989).

2. Materials and methods

2.1. Fruit juices

"Sancho" cultivar melon fruits were collected from La Mancha region (Spain, 2000). Melon "*Cucumis melo*" is a fleshy fruit of the *Cucurbitaceae* family, forming an outer skin, pulp and pips. Its chemical composition (% w/w) is as follows: water content (89.8), carbohydrates (8.4), proteins (0.9), fibre (0.8) and lipids (0.3×10^{-3}) with certain vitamins and a high content in potassium and calcium according to the USDA.

The fruits were washed and divided into three sets processed in different ways to obtain three substrate types ready for their fermentation:

- The fruits were cut into pieces manually, after that crushed and peeled, and then pressed in a vertical press. The product obtained was called "juice".

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- The fruits were cut into pieces manually, after that crushed and peeled (not pressed), to give one substrate called "paste without skin" (pws)
- The fruits were cut into pieces, and crushed (not pressed and not peeled), yielding a "paste"

The steps of fruit processing are shown in Fig. 1.

2.2. Analysis of juice and pastes

Conventional parameters, such as °Brix and pH, were measured in every substrate (juice, pws and paste) (Recueil des Méthodes Internationales d'Analyse des Vins, 1969).

2.3. Fermentation process

Fermentation of juice, pws and paste was carried out in 100 l vessels each filled with 80 l of different substrate. Temperature was controlled at 20 °C. The substrates were inoculated with a selected and commercial yeast (*S. cerevisiae* UCLM 325) up to a concentration of approximately 10^6 cells ml^{-1}. The process was monitored daily by measuring residual sugars. The end of fermentation was determined on the basis of the sugar consumption.

In order to control the acetic acid and lactic bacteria growth, the pH was adjusted to around 4, it being necessary to add about 9 g l^{-1} of citric acid.

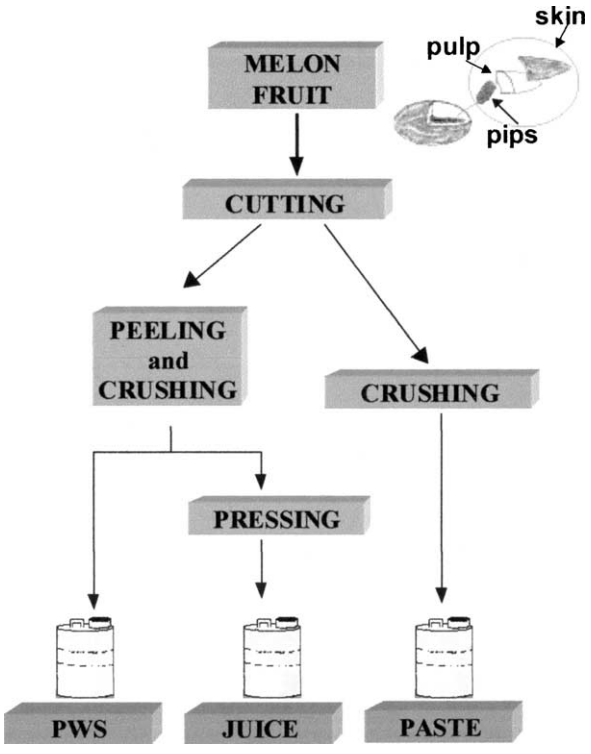

Fig. 1. Fruit processing to obtain different substrates to be studied.

Volatile acidity and alcoholic degree were measured in every fermented batch (Recueil des Méthods Internationales d'Analyse des Vins, 1969).

2.4. Distillation

Upon completion of alcoholic fermentation, the fermenteds were immediately distilled with yeast lees in two ways: in a column or in a copper pot.

- Column: 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 homogeneous heat distribution during the distillation process, boiling stones were added and the flow rate was adjusted to 10 ml min^{-1}.
- Copper pot: a 30 l French type copper pot filled with 15 l of fermented fruit was used and the flow rate was adjusted to 25 ml min^{-1}.

In both cases, the fermented materials were double-distilled. The first distillation was stopped when the alcohol degree was lower than that of the fermented fruit, to give 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 by adding the fractions ranging from 30% (v/v) to 5% (v/v). The distillate was collected in fractions of 1 l for the copper pot and 400 ml for the column. In order to avoid the loss of aromas, all the fractions were collected on ice and kept at 4 °C until their analysis.

2.5. Distillates analysis

A total of 24 fractions were analysed. In all of them the alcohol degree was determined by means of the electronic densimetry method (European Union Rules No. 2870/2000, 2000).

Major volatile components were analysed by gas chromatography (GC). A Perkin Elmer chromatograph and a packed column VINICOL (González & González, 1994) were employed. 20 mg l^{-1} of 3-pentanol (Sigma Chemical Co), as internal standard, were used. The conditions were: injector temperature 200 °C, FID detector temperature 225 °C, carrier gas, nitrogen (flow 15 ml min^{-1}). Temperature programme was as follows: 40 °C for 3 min, thereafter increasing at 6 °C min^{-1} to 100 °C. As the next step, the temperature was maintained for 5 min and then increased at 6 °C min^{-1} to 125 °C, maintained for 14 min, giving a total running time of 36 min. 1 μl of sample was injected. The volatile compounds analysed were: acetaldehyde, methanol,

1-propanol, 1-butanol, 2-butanol, 2-methyl-1-propanol, 2-methyl-1-butanol (2M1B), 3-methyl-1-butanol (3M1B), ethyl acetate, ethyl butyrate and ethyl lactate.

2.6. Statistical analysis

Multivariate principal component analyses (PCA) were performed using the SPSS statistical package (10.0 version).

2.7. Sensory analysis

Flavour quality of distillates was evaluated by an expert panel of judges. The samples were diluted to 30% v/v, and 30 ml of each was tasted in a taster room. The judges were asked to determine their preferences for the different distillates.

3. Results and discussion

The yield of the processed fruits depended on the substrate assayed. Therefore, in the case of the paste, it reached 100%, 70% (30% of skin) for the pws and only 50% for the juice.

In Table 1, pH and °Brix values of different substrates, volatile acidity (g l^{-1} of acetic acid) and alcohol degree (% v/v) of the fermented products are shown. The initial pH of the different substrates varied between 4.4 and 4.9. It was corrected before the fermentation with citric acid to reach values around 4 in order to inhibit lactic acid and acetic bacteria, as in previous research significant numbers of both of them in the ''paste'' fermentation were observed (data not shown). Judging from the initial °Brix, an alcohol degree of 5% (v/v) could be expected. Nonetheless, experimental data showed that the sugar 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 contamination by acetic bacteria.

After the fermentation, each ferment was divided into two sets. One was distilled in a copper pot and the other in a column. The distillate was collected in different fractions, as explained in Section 2.4. The heart fraction alcohol degree values are shown in Table 2. In both distillations (copper pot and column) the alcohol degree of the paste was lower because the initial degree of the ferment was also inferior (Table 1).

The concentrations of different volatile compounds, for both distillation types, are shown in Table 3. Acetaldehyde concentrations ranged from 153 to 243 mg l^{-1} of ethanol. The most important compound to control in the spirits is the methanol. In some quantities, this substance can be dangerous due to its oxidation to ethanal (or formaldehyde) and formic acid which slowly reach high concentrations in the human body (Raposo, 1986). Two types of enzymes can act upon pectins: polygalacturonases, which bring about cleavage of chains at the glycosidic bons; and pectinmethylesterases, which catalyze hydrolysis of the chemical function esterified,

Table 1
pH, °Brix, volatile acidity and alcohol degree of different substrates studied

	Not fermented				Fermented		
	°Brix		Initial pH		Volatile acidity (g l^{-1} of HAc)		Alcohol (% v/v)
	X	S.D.	X	S.D.	X	S.D.	X
Juice	10.0	0.25	4.8	0.36	0.54	0.06	4.2
Pws	11.0	0.35	4.4	0.30	0.47	0.07	4.2
Paste	10.4	0.15	4.9	0.20	1.81	0.11	3.4

X, mean value. S.D., standard deviation.

Table 2
Alcohol degree of second distillation for different fractions collected in the copper pot and in the column

	Alcohol degree (% v/v)			
	1st fraction	2nd fraction	3th fraction	Average value
Copper pot				
Juice	68.6	61.5	44.2	58.1
Pws	69.8	62.8	44.6	59.1
Paste	65.6	52.1	27.9	48.5
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 3
Concentration of volatile compounds (mg l^{-1} of ethanol) in the final copper pot and column distillates

	Copper pot			Column		
	Juice	Pws	Paste	Juice	Pws	Paste
Acetaldehyde	153	237	182	243	163	230
Methanol	1539	618	4564	699	695	4749
1-Propanol	297	479	625	808	591	822
2 Methyl-1-propanol	229	215	221	310	280	261
1-Butanol	1.7	0.0	3.4	0.0	0.0	0.0
2-Methyl-1butanol	418	143	142	220	183	155
3-Methyl-1butanol	658	685	691	881	909	746
Total Has	1603	1530	1681	2219	1963	1984
Ethyl lactate	379	368	902	744	649	274
Ethyl acetate	40.8	154	74.4	74.1	64.6	154
Ethyl butyrate	1.1	0.0	0.0	0.0	0.0	0.0

and thus release methanol (Cortés Diéguez, Gil de la Peña, & Fernández Gómez, 2000; Lee, Robinson, Burren, Acree, & Stoewsand, 1975; Ribéreau-Gayon, Glories, Maujean, & Dubordieu, 2000). Methanol concentrations ranged from 618 for pws to 4749 mg l^{-1} of ethanol for paste. The paste distillate showed the highest methanol values which overruns limits allowed for in the present legislation (European Union Rules No. 1567/89, 1989), probably owing to its high skin contents or the action of certain pectin enzymes. Nonetheless, concentrations of methanol in pws and juice distillate types were below the threshold level and even inferior to the ones present in the orujo, grapa and bagaceira distillates (Silva, Macedo, & Malcata, 2000).

Higher concentrations of the high alcohols (HAs) in distillates can render the flavour of the product unpleasant, due to their strong, pungent smell and taste (Boulton, Singleton, Bisson, & Kunkee, 1995). The HAs quantified in this study (1-propanol, isoamyl alcohols (2M1B and 3M1B), 1-butanol and 2-methyl-1-propanol) were higher in the column than in the copper pot distillates.

Propanol concentrations range from 297 to 822 mg l^{-1} of ethanol. The highest concentrations were for the paste type with either a column or copper pot distillation. The concentrations of 2-methyl-1-propanol were similar; a slightly lower content in the copper pot was observed. 1-butanol was only detected in a very low concentration in the juice and paste copper pot distillates.

Iso-amyl-alcohols (2M1B and 3M1B), are the components which are usually produced in largest amounts in this kind of beverage (Boulton et al., 1995; Nykänen & Suomalainen, 1983). In this study, the distillates in the column in all cases showed higher contents than the ones in the copper pot, except for the juice distillate type.

High levels of 2-butanol in spirits are usually associated with a low quality of raw materials (Cortés Diéguez et al., 2000; Orriols & Bertrand, 1990), but in none of the distillates was it detected.

Esters are associated with a pleasant smell, especially ethyl acetate which, in very low doses (50–80 mg l^{-1}), contributes to the smell complexity and has a positive impact on the product quality (Steger & Lambrechts, 2000). It ranged from 40.8 to 154 mg l^{-1} of ethanol but all the distillates, except the paste in the column and pws in the copper pot, showed a content below 80 mg/l. Ethyl butyrate was only detectable in "juice" type, distilled in copper pot, because it might be included in the head fraction (Reibéreau-Gayon et al., 2000). The presence of ethyl lactate in distillates can be linked to a lactic fermentation and therefore it should be more strictly controlled during the process.

The evolution of the main volatiles in different fractions (1, 2, 3 and tails) collected during the distillation in the copper pot is shown in Fig. 2. Methanol appears in all the fractions due to the formation of azeotropic mixtures (Orrials, 1994). HAs have boiling points lower than 200 °C and are alcohol-soluble and also completely or partially water-soluble so they distil mainly in the heart fraction. For this, the concentration of these compounds decreases as the distillation process progresses. The total ester content mainly appears in the first fraction while "paste" distillate type increases at the last distillation stage.

PCA (principal component analysis) of the major volatiles shown in Table 3 was applied to determine the degree of differentiation between the substrate types and the distillation methods. Ethyl butyrate and 1-butanol were not considered. Components 1, 2 and 3 explained 86.4% of the variance. Table 4 lists the variables which

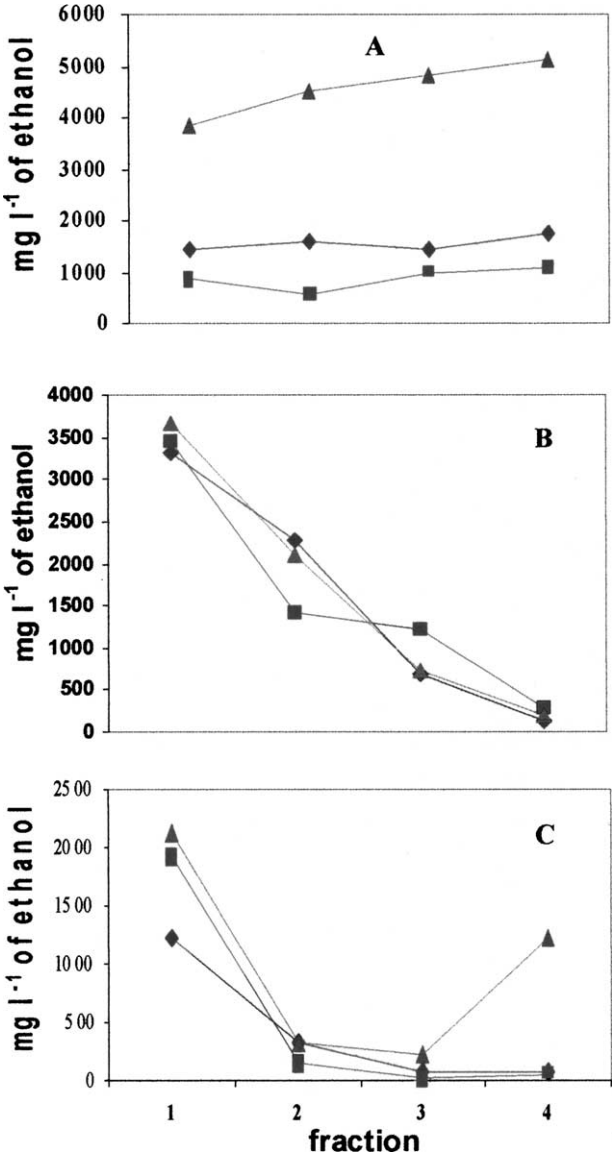

Fig. 2. Evolution of volatile compounds in four different fraction distillates in the copper pot. (A) Methanol, (B) higher alcohols and (C) total esters; juice (◆), pws (■) and paste (△).

Table 4
Principal component analysis applied to major volatile components

Principal component	Variance explained (%)	Cumulative proportion (%)	Best variable correlations and "loadings"
PC1	38.59	38.59	3M1B[a] (0.96) 2M1P[b] (0.95) Propanol (0.62)
PC2	31.04	69.63	Ethyl acetate (0.95) Acetaldehyde (0.85) Ethyl lactate (−0.59)
PC3	16.81	86.44	Methanol (0.78) 2M1B[c] (−0.68) Propanol (0.60)

[a] 3M1B (3-methyl-1-butanol).
[b] 2M1P (2-methyl-1-propanol).
[c] 2M1B (2-methyl-1-butanol).

correlated best with every principal component, along with the contribution of each variable to the corresponding principal component. Fig. 3 plots the samples on the coordinate grid defined by the three main components (PC1, PC2, PC3) and shows that the samples were clustered together, depending on the distillation method followed. In this way, the column samples are separated from the rest by their higher concentrations of volatiles which contributed to PC1 (propanol, 2-methyl 1-propanol and 3-methyl-1-butanol).

The sensory analysis of the distillates offered conclusive results with regard to the distillation type. 100% of tasters preferred the samples distilled in the copper pot. Within the copper pot distillates, the juice sample

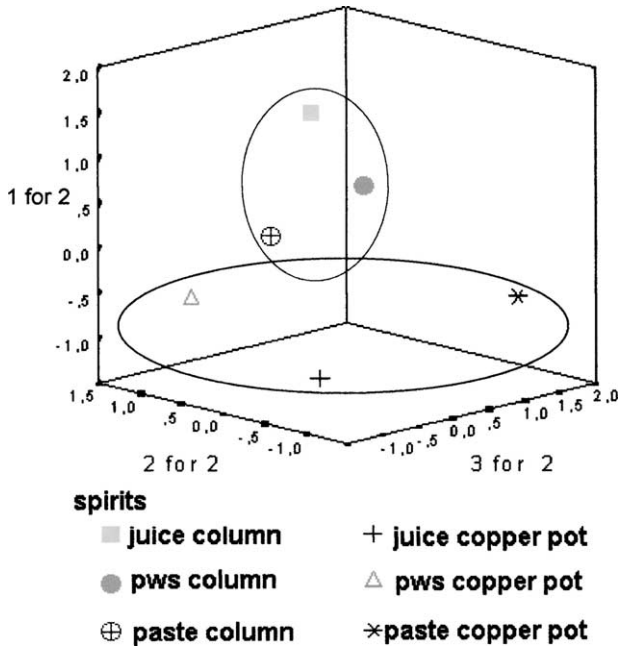

Fig. 3. Principal component analysis of the volatiles in the column and in the copper pot distillates.

was more appreciated due to its aroma intensity, while the rest of the samples were rejected on the basis of their pungent and/or "not sufficiently intensive" aroma.

4. Conclusions

The distillation methods resulted in differences of chemical characteristics of the distillates. In order to get a melon spirit with an appropriate sensorial profile, the use of the copper pot distillation was preferred.

With regard to the type of substrate, even though the "paste" type offers a better yield in the process, it does not seem to be the ideal substrate due to the sluggish fermentation, high methanol content and negative sensory characteristics.

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