STUDIES REGARDING THE UTILIZATION OF WOOD CHIPS ON THE QUALITY IMPROVEMENT OF SOME NATURAL SPIRITS

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ABSTRACT - *In a series of papers we have studied some methods of accelerated maturation of raw young fruit distillates in contact with oak wood. Total acidity exhibited a differentiated evolution. For the majority of samples a moderate long-term increase was often noticed, although there were samples for which the total acidity remained the same after 170 days. The alcohol concentration diminished in a differentiated manner. The esters content had the most interesting values, as they reflected the non-linear transformations and chemical reactions that occurred. The dry extract increased in all cases, but too much in the case of defective apple distillate. The defective distillate was successfully improved, while another distillate, with real initial qualities, which presented a superior estimate at the half of the interval (at 93 days), evolved differentiated at the end.*

Key words: young apple spirits, wood chips, qualitive improvement, accelerated maturation

INTRODUCTION

The purpose of distillate aging is obtaining some raw materials of superior quality for the preparation, on the account of complex recipes, of some fine distilled drinks. The traditional aging is made in oak barrels of 300-500 liters, where certain substances are extracted from the oak stave.

By the rapid aging of distillates one can solve the problems related to the reduction of price, by maintaining the quality of the aged distillate at a level not too far from the one obtained by the classical method.

The use of oak chips of different types (Ţârdea et al. 2000, Pomohaci et al., 2001) is an orientation in the process of defining and regulating in the case of

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distillates (Beceanu et al., 2004), with a real potential of qualitative melioration and a wide palette of options, taking into account the diversity of the assortment (Beceanu, 2000; Pomohaci et al., 2001; Țârdea et al., 2000)

MATERIALS AND METHODS

The studied samples are represented by apple distillates from the Fruit-Growing Farm 1 within the Fruit-Growing Research and Development Station of Iaşi. It is a young distillate, obtained in 2004, after fermentation as marc, in a traditional discontinuous installation made of copper. The apple distillate from the Bucium Trade Company of Iaşi, obtained in 2004, in a Charente installation, from fermented apple juice, is the raw material for Rameros (a distilled drink like Calvados).

The wood chips used are made of oak wood of the following type: Small Forte, Big Forte and Medium +, produced by the company UNICOM HOLDING, according to a technology kept confidential, but which may be inferred according to their names.

The chips were put into contact with the raw apple distillate in polyethylene containers (PET). The dose was of 10g/l. The containers were closed airtight, letting a void space of 30%, and were introduced in a drier at 50°C. The variants were stirred weekly for oxygenation. The contact duration of the distillate with the chips was first of 93 days going up to 170 days, and the constant temperature was maintained for the entire period of the distillates contact with the chips.

The following variants were obtained: apple distillate (Fruit-Growing Research and Development Station of Iaşi), Small forte chips, apple distillate (Fruit-Growing Research and Development Station of Iaşi), Big forte chips, apple distillate (Fruit-Growing Research and Development Station of Iaşi), Medium + chips, apple distillate Bucium Small forte chips, apple distillate Bucium Big forte chips, apple distillate Bucium Medium + chips. In parallel, one variant of each sample was kept at the same temperature for only a week.

The determination of the alcoholic concentration and the relative density were made by the electronic densimeter Anton Paav DMA 5000. The determination of the total dry extract was made according to the STAS 184/3-70. The determination of the total acidity was made according to the STAS 184/5-70, through the potentiometrical method, by titration of the distillate sample with a solution of NaOH 0.1 n, after the preliminary elimination of CO_2. Conventionally, acidity is expressed in g/l acetic acid, g/l sulphuric acid or in meq/l. The determination of esters was made according to the STAS 184/6-70 by titration with hydroxide sodium, after the elimination of CO_2 from sample. The esters are saponified with hydroxide sodium in excess (*** 1998; *** 1999).

RESULTS AND DISCUSSION

The total acidity (*Table 1*) manifested a differentiated evolution. An obvious increase from 19 to 36 meq/l appeared at the sparged apple distillate from Farm 1. For the rest of the samples, one could notice a moderate increase on a long-term, though there were samples with total acidity having the same values after 170 days. If we take into account the intermediate values determined after 93 days, we notice small values in certain cases, signaling chemical reactions in full process.

Table 1

Total and volatile acidity for the experimental variants

No.	Sample	Total acidity		Volatile acidity g acetic acid / 100 cm^3
		meq/l	g acetic acid / 100 ml alcohol anh.	
1	Raw apple distillate from Fruit-Growing Farm 1	19	0.21	0.114
2	Apple distillate F1 sparged for7 days	36	1.39	0.126
3	Pilot Farm 1 (after 93 days)	18	0.20	0.042
4	Farm 1 Small Forte (after 93 days)	17	0.20	0.036
5	Farm 1 Big Forte (after 93 days)	17	0.20	0.036
6	Farm 1 Medium + (after 93 days)	16	0.18	0.042
7	Pilot Farm 1 (after 170 days)	21	0.22	0.08
8	Farm 1 Small Forte (after 170 days)	19	0.21	0.05
9	Farm 1 Big Forte (after 170 days)	19	0.21	0.06
10	Farm 1 Medium + (after 170 days)	18	0.20	0.05
11	Raw apple distillate from Bucium	4	0.03	0.021
12	Apple distillate from Bucium sparged for 7 days	3	0.04	0.012
13	Bucium pilot (after 93 days)	4	0.03	0.012
14	Bucium Small Forte (after 93 days)	4	0.03	0.009
15	Bucium Big Forte (after 93 days)	4	0.03	0.009
16	Bucium Medium + (after 93 days)	3	0.02	0.012
17	Bucium pilot (after 170 days)	5	0.04	0.02
18	Bucium Small Forte (after 170 days)	5	0.04	0.02
19	Bucium Big Forte (after 170 days)	5	0.04	0.01
20	Bucium Medium + (after 170 days)	5	0.04	0.009

The volatile acidity (*Table 1*) has obviously decreased, except for a single sample (the sparged apple distillate from Farm 1) that had superior values. The diminution was high in all samples after 170 days of thermal treatment, which was a natural and expected phenomenon.

The alcoholic concentration (*Table 2*) has diminished, the phenomenon being generalized and differentiated. The most obvious decreases were noticed in

the sparged distillates, first in the distillate from the Fruit-Growing Farm 1. This case of the sparged distillates made us abandon their study after a week, being a non-economic method of treatment. For the other studied groups, we noticed the following decreases:

- The distillate from the Fruit-Growing Farm 1 diminished its alcoholic concentration after 93 days, at percentages between 92.8 and 96.7 from the initial value, and after 170 days, we noticed smaller values, between 88.35 and 98.91%. The values were not contradictory, but they indicated the mobility of components and the deployment of some biochemical modifications besides the natural process of evaporation that could not be hindered.

- In the distillate from Bucium, after the first 93 days, the decreases were between 86.69 and 90.43% from the initial alcoholic concentration and after 170 days, we have noticed that the greatest decrease was between 70.11 and 88.96%.

Table 2

Alcoholic concentration and density in the experimental variants

No.	Sample	Alcoholic concentration (rounded at 1 decimal)		Density (rounded at 4 decimals)
		vol alc. %	percentage	g/cm^3
1	Raw apple distillate from Fruit-Growing Farm 1	54.0	100	0.9220
2	Apple distillate F1 sparged for 7 days	15.5	28.7	0.9790
3	Pilot Farm 1 (after 93 days)	52.2	96.7	0.9181
4	Farm 1 Small Forte (after 93 days)	50.1	92.8	0.9201
5	Farm 1 Big Forte (after 93 days)	50.9	94.26	0.9201
6	Farm 1 Medium + (after 93 days)	50.5	93.52	0.9202
7	Pilot Farm 1 (after 170 days)	53.41	98.91	0.9131
8	Farm 1 Small Forte (after 170 days)	51.45	95.28	0.9147
9	Farm 1 Big Forte (after 170 days)	51.30	95.00	0.9140
10	Farm 1 Medium + (after 170 days)	47.71	88.35	0.9144
11	Bucium raw apple distillate	72.1	100	0.8803
12	Bucium apple distillate sparged for 7 days	49.3	68.38	0.9316
13	Bucium pilot (after 93 days)	65.2	90.43	0.8789
14	Bucium Small Forte (after 93 days)	62.7	86.96	0.8771
15	Bucium Big Forte (after 93 days)	63.0	87.38	0.8781
16	Bucium Medium + (after 93 days)	62.5	86.69	0.8773
17	Bucium pilot (after 170 days)	53.97	74.85	0.8692
18	Bucium Small Forte (after 170 days)	61.94	85.91	0.8698
19	Bucium Big Forte (after 170 days)	50.55	70.11	0.8680
20	Bucium Medium + (after 170 days)	64.14	88.96	0.8680

The density (*Table 2*), being correlated to the alcoholic concentration, had the same behaviour. The ester content (*Table 3*) presented the most interesting

values in terms of evolution since it reflected the transformations and the non-linear reactions that occurred, often with higher intermediate values, with precipitations and sedimentations, with final contents much different as against the initial one (Popa, 1985; Puech, 1985; Stănciulescu, 1970).

Table 3

Ester contents and the dry extract at the experimental variants

No.	Sample	Ester contents (rounded at 1 decimal)		Dry extract	
		g ethyl acetate / 100 ml anhydrous alcohol		g/l	%
1	Raw apple distillate from Fruit-Growing Farm 1	0.0574	100	0.0018	100
2	Apple distillate F1 sparged for 7 days	0.0687	119.68	nd	
3	Pilot Farm 1 (after 93 days)	0.1233	214.80	0.0473	2628
4	Farm 1 Small Forte (after 93 days)	0.2520	439.02	0.0949	5272
5	Farm 1 Big Forte (after 93 days)	0.0617	107.49	0.1826	10144
6	Farm 1 Medium + (after 93 days)	0.2185	380.66	0.1139	6328
7	Pilot Farm 1 (after 170 days)	0.1688	294.07	0.0420	2333
8	Farm 1 Small Forte (after 170 days)	0.1749	304.70	0.2680	14889
9	Farm 1 Big Forte (after 170 days)	0.1692	294.77	0.2760	15333
10	Farm 1 Medium + (after 170 days)	0.1575	274.39	0.3400	18889
11	Bucium raw apple distillate	0.0261	100	0.0182	100
12	Bucium apple distillate sparged for 7 days	0.0474	181.60	nd	
13	Bucium pilot (after 93 days)	0.1092	418.39	0.0306	168
14	Bucium Small Forte (after 93 days)	0.1762	675.09	0.1246	685
15	Bucium Big Forte (after 93 days)	0.0441	168.96	0.1265	695
16	Bucium Medium + (after 93 days)	0.0176	67.43	0.1011	555
17	Bucium pilot (after 170 days)	0.0130	49.80	0.0420	231
18	Bucium Small Forte (after 170 days)	0.0345	132.18	0.2320	1275
19	Bucium Big Forte (after 170 days)	0.0242	92.72	0.1860	1022
20	Bucium Medium + (after 170 days)	0.0322	123.37	0.1600	879

The two different distillates behaved specifically and in a very different way in case of these treatments. The apple distillate from Fruit-Growing Farm 1 has increased after the first 93 days its content of esters with values between 107-381%. But, after 170 days we have noticed a more uniform increase between 274 and 305%. We can assert that the distillate was the most obviously transformed. The Bucium apple distillate registered in the first 93 days an evolution that also included diminution in the ester contents, oscillating between 67 and 675%. After 170 days, the values were more homogenous but with content increases and decreases, between 50 and 132% compared to the initial value.

The dry extract (*Table 3*) has increased its values in all cases, but at much more important values in the case of the apple distillate from the Fruit-Growing Farm 1. Increases of the initial values from 2628% up to 10144% were noticed. After 170 days, greater increases were found especially at the variants with chips having increases between 14189 and 18889%, while the control has increased with 2333%.

A more dynamic situation is noticed in the Bucium apple distillate where the control has increased its dry extract at 168% and 231%, respectively. The variant with chips has increased in the first stage (93 days) by 555- 696% and in the second stage by 879-1275%.

The sensorial appreciation also highlighted an obvious evolution of quality.

Comparing the control experiments from the Farm 1 after 93 days and after 170 days, respectively, one could finally notice the disappearance of some tastes and flaws that still persisted in the initial phase (of 93 days).

Comparing the control assays from Bucium after 93 days and after 170 days, respectively, we noticed a positive evolution, the appearance of smoothness, harmony and pleasant taste.

Comparing the samples with Small forte chips from the Farm 1 after 93 days and after 170 days, respectively, we noticed the interesting colour and the maintaining of the taste of smoked prunes with a smooth flavoured nuance.

By comparing the samples with Small forte chips from Bucium after 93 days and 170 days, respectively, we could distinguish some positive characteristics, but the taste nuances were perceived sometimes as disharmonious, though an obvious smoothness was observed.

Comparing the samples with Big forte chips from the Farm 1 after 93 days and 170 days, respectively, we noticed only positive effects of the treatment, a less obvious smoke smell and a clear harmony, with the persistence of the fruit taste.

By comparing the samples with Big forte chips from Bucium after 93 days and 170 days, respectively, we could notice that the positive assessment after 93 days was not entirely maintained until the end. Although the smoothness and the fruit flavour remained, one could not notice the fineness of taste.

The comparison between the samples with Medium + chips from the Farm 1 after 93 days and 170 days, respectively, resulted in the observation of a positive evolution, which at the final phase, has determined a pleasant bright yellow colour, with a fine flavour of smoked fruits and a smooth harmonious taste.

The samples with Medium + chips from Bucium after 93 days and 170 days, respectively, were characterized by the yellowish colour, with a bright hue and a smooth taste, relatively harmonious, a slight bitter tint after tasting, which was pleasant

CONCLUSIONS

The use of oak chips in the experimental variants resulted in a melioration of the biochemical constants of the treated apple distillates and a general positive evolution of their sensorial qualities, estimated by us as being similar to a period of aging of more than 5 years.

The total acidity manifested a differentiated evolution. An obvious increase is noticed at the sparged apple distillate from Farm 1. For the rest of the samples we noticed a moderate increase on a long term. There are samples where the total acidity has the same values after 170 days. Finally, **the volatile acidity,** has greatly decreased in all samples.

The alcoholic concentration diminished, the most obvious decreases being found in the sparged distillates. At the end of the thermal treatment we obtained values of the alcoholic concentration of 88.35 and 98.91% from the initial value, in the case of the distillate from Fruit-Growing Farm 1, respectively 70.11 and 88.96% from the initial value in the case of the distillate from Bucium.

In the case of the apple distillate from Fruit-Growing Farm 1, **the ester contents** increased during the entire treatment, in the first part of the period by 107-381%, then registering a more uniform increase between 274 and 305%, compared to the initial value. The Bucium apple distillate registered in the first 93 days an evolution of the ester contents between 67 and 675% compared to the initial value. After 170 days, the values became more homogenous, between 50 and 132% compared to the initial value.

The dry extract increased its value in the case of the apple distillate from the Fruit-Growing Farm 1 from about 26 up to 189 times, while the control increased only by 23 times. A situation less dynamic is registered in the Bucium apple distillate, where the control increased its dry extract by 2,3 times, and the variants with chips only by 9-13 times.

A distillate with flaws meliorated with very good results, whereas another one, with real initial qualities, that had presented a superior evaluation at 93 days, evolved in a different way and sometimes with a disharmonious tint at the end.

REFERENCES

Beceanu D., 2000 – *Romanian assortment of distilled alcoholic beverages from fruits*. Scientific works, vol. 43. University of Agricultural Sciences and Veterinary Medicine, series Horticulture. Ion Ionescu de la Brad Publishing house, Iaşi

Beceanu D., Coşofreț S., Apetăchioaiei V., Roman Camelia, Radu Roxana, 2004 - *Études concernant les technologies d'améloration acélérée des distillats frais de pomme*. Scientific works, University of Agricultural Sciences and Veterinary Medicine. Series Horticulture. vol. 48, p. 877-886, Iaşi

Pomohaci N. et al., 2002 – *Brandy and natural spirits.* Ceres, Bucharest

Popa A., 1985 – *Producing and ageing of wine distillates.* Scrisul Românesc, Craiova.

Pomohaci N. et al., 2001 – *Operations and treatments applied to wine. Oenology vol. 2.* Ceres, Bucharest

Potec I., Anghel Gh., Potec Elena, Beceanu D., Savu Maria, 1988 – *Model technological scheme for obtaining distillates from fruits.* Scientific works, Agronomical Institute, series Horticulture, vol. 31, Iaşi,

Puech J.L., Jouret C., Goffinet B., 1985 - *Evolution des composés phénoliques du bois de chêne au cours du viellissement de l'armagnac.* Sciences des aliments, no. 5.

Stănciulescu Gh., Rusnac D., Borteş Gh., 1975 – *Technology of alcoholic distillates from fruits and wine.* Ceres, Bucharest

Stănciulescu Gh., 1970 – *Making of natural alcoholic beverages.* Tehnica Publishing house, Bucharest

Tanner B., 1982 – *La distilation moderne des fruits.* Heller

Teodorescu Şt., Iliescu Lucia, Ionescu Al., 1958 – *Evolution of chemical composition of wine distillates during ageing.* Annals of Institute of Agricultural Research from Romania vol. XXVI.

Ţârdea C. et al., 2000 – *Wine making treatise.* University of Agricultural Sciences and Veterinary Medicine, Ion Ionescu de la Brad Publishing house, Iaşi

. 1992*** *Elaboration et connaissance des spiritueux. Recherche de la qualite,* ***Tradition et Inovation****.* BINC, TEC&DOC, Paris

***** **1996** ***Spiritulosen Jahrbuch. Versuchs und Lehranstalt fur Spirt. und Ferment****.,* Berlin

***** **1998** *Book of Romanian standards. Ethylic alcohol and alcoholic beverages. Quality conditions. Methods of analysis.* Romanian Institute for Standarization COPCIA, Bucharest

***** 1999** *Professional standard. Alcoholic beverages..* MAA – DGPA. COCPCICA Publishing House