BIO Web of Conferences **3**, 02010 (2014)
DOI: 10.1051/bioconf/20140302010

Use of the evapo-destillation of reverse flow to obtein new products derived from grape

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Abstract. The aim of this communication is to show the advantages of the use of wine alcohol as a preservative of must and its recovery through a system of evapo-destillation of reverse flow. Currently, the industry uses sulfur dioxide in the conservation of musts destined for concentration. The problem about the use of sulfur dioxide in the conservation of musts is that not ensure an anti-fermentative stability whole, but mainly in that it is not recovered, thereby producing a consequent pollution. Moreover concentrated musts usually contain a small presence of ethanol from micro-fermentations.

At laboratory scale it was verified an excellent state of preservation of alcoholizated must, and all the alcohol used was recovered by distillation.

The proposed system achieved a high reduction of production costs which is demonstrated by simulation process software, where it can show the energy savings compared to the conventional process for the same volume and concentration of product obtained.

Another important advantage of this system is that higher alcohols and flavors would not be lost because is not necessary a desulfitation process, thus obtaining better quality products.

1. Introduction

This work is based on the patent: "Process and equipment for the production of concentrated fruit juice by evapo-destillation" of the Chem. Eng.. Armando Kamal Neme [1].

1.1. Market situation of musts in Argentina, the agreement Mendoza-San Juan

The Argentine provinces of Mendoza and San Juan, which produce over 90% of the grape production Argentina, annually fixed by agreement with the government a minimum percentage of grapes that must be spent on the production of musts, respct to the total grape entered to winery during the season. In Table 1 shown the percentages from the last years.

Moreover due to the crisis in wine consumption in recent years have fallen must exports. Fig. 1.

Also in Table 2 and in Fig. 2, it can be seen that practically between 50 and 70% of must concentrated have destination the exportation.

This situation cause annually a large amount of excess production of musts so as of wines.

Table 1. Minimum percentage of grapes destined for the production of musts in Mendoza-San Juan agreements.

Year	Percentage of grape from agreements Mendoza-San Juan
2009	20%
2010	20%
2011	30%
2012	30%
2013	32%
2014	18%

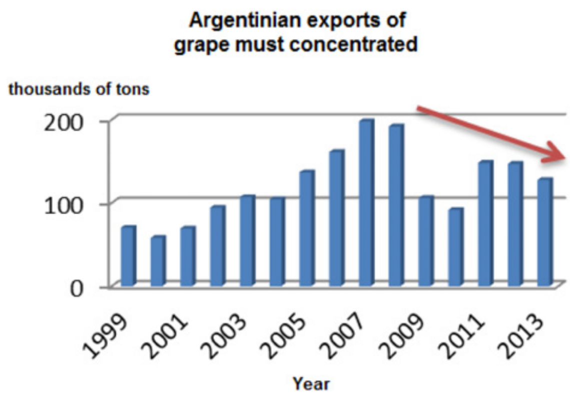

Figure 1. Argentine exports of grape must concentrated (GMC).

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Table 2. Production and Export. Of GMC in Argentina.

Year	Production of GMC in million liters	Exportation of GCM in millions liters	%exported compared to produced
2008	232	143	62
2009	163	77	47
2010	136	68	50
2011	149	109	73
2012	151	109	72
2013	192	94	49

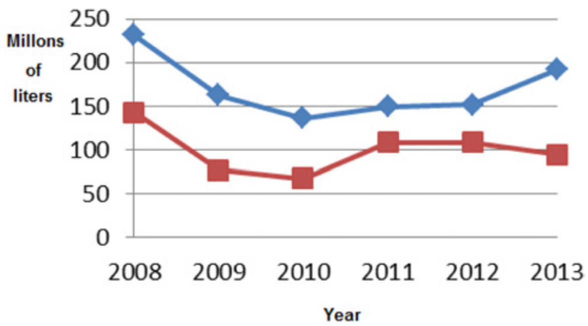


Figure 2. MCU production and exports in Argentina.

1.2. Problems in the typical process of concentration of must in Argentina

Harvested grapes must be processed in a very short time and also this concentrated has little stability.

That was the reason why the production of GMC must be based on the use of SO_2 for its conservation. The dosage of sulfur dioxide for this purpose is of 2g/L.

But sulfation of grape must and its conservation for an extended period not completely secures antifermentative stability and requires use of epoxy resins for pools currently questioned by possible generation of Phthalates.

Since desulfitation of must, which removes most of the SO_2, but not all, is performed by stripping with water vapor in distillation columns, generally plates perforated, in counterflow between a descending flow of must and a water vapor upward, the alcohol and the aromatic substance or flavor precursors also volatile, are lost along with detached SO_2 and water vapor used. The SO_2 produces a consequent environmental pollution.

Furthermore, the musts concentrated usually contain a small presence of ethanol from microfermentations also lost by evaporation.

Also is important to note that in the usual process of concentration three thermal processes are performed: desulfitation, preconcentration and concentration, the last two are normally conducted in the same multiple-effect evaporator.

2. Proposed system: Conservative alcohol and evapo-destillation

To solve the problems outlined in the previous sections, the proposal is the use of wine alcohol of about 90% v/v as a preservative of musts that are destined for concentration, in

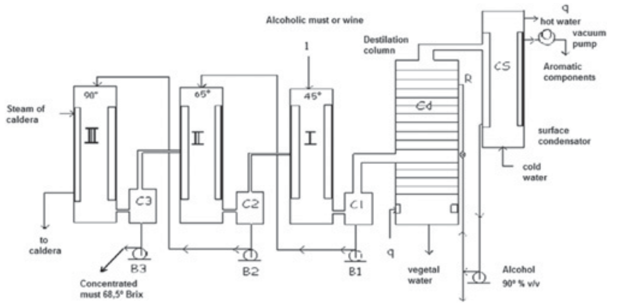


Figure 3. Evapo-destillation: Diagram of equipment and flows. References: q: heat; I-II and III: falling film evaporators; C1-C2-C3: cyclone separator of vapors; B1-B2-B3: circulation pumps of must; A: reflux; Cd: distillation column.

the place of sulfation. This alcohol will be recovered and reused.

The alcoholization should be conducted at a concentration between 15 and 18% v/v.

Also is proposed for the concentration of alcoholized must implementing the "evapo-destillation" it was highly developed in Europe in the mid-twentieth century in the maize alcohol industry.

It consists in the use of an evaporator connected to a body of evaporators: the alcoholic must enter at the first stage of evaporation, where a gaseous fraction rich in alcohol goes to the distillation column to recover the wine alcohol (Fig. 3).

In the second and third effect the must is concentrated to reach 68.5 ° Brix. The type of evaporators used would be falling film. Furthermore, these same units would be used for the concentration of wine in obtaining the wine alcohol necessary for alcoholizations.

The direction of flow would be reversed, that is, the liquid to evaporate and concentrate circulates in the opposite direction to the steam flow. This type of flow is used to process juices that containing aromatic components to be recovered and then reinstated the concentrated juice.

En este caso, el componente aromático, volátil, es el alcohol contenido en el vino o en el mosto alcoholizado.

In this case, the aromatic volatile component is the alcohol contained in the wine or alcoholic must.

3. Tests on laboratory scale

In these tests, sulfur dioxide is added to a fraction of must and wine alcohol to another fraction.

The addition of SO_2 was 2150 mg/l and the alcoholic fraction was carried to 18% v/v.

Determination of reducing sugars was performed, after dilution, by chemical method by titration with liquor Felling [8], with a tolerance of ±7% [9]. Alcohol was analyzed by aerometric method [10] with a tolerance of ±0.3 [11]. The analytical technique used to determine the total sulfur dioxide was oxidation in acid medium [4,6] with a tolerance of ±35 mg/l [7].

As expected, the added alcohol insolubilizes largely of tartaric salts, forming a precipitate easy to separate from the liquid fraction. This property would enable to separate tartaric salts avoiding the cooling and destartarization

Table 3. Variables entered and results.

	Classic process of concentration		Evapo-destillation
Base of calculation	5000 kg/h of sulphited must		5930 kg/h = 5000 kg/h of must + 930 kg/h wine alcohol
	Precon-centration.	Concen-tration.	
Temp. 1st effect (°C)	90,7	92,9	64,3
Pressure 1st effect (bar)	0,7	0,7	0,3
Temp. 2nd effect (°C)	77	79,6	75,5
Pressure 2nd effect (bar)	0,4	0,4	0,4
Temp. 3rd effect (°C)	71,1	74,4	95,8
Pressure 3rd effect (bar)	0,3	0,3	0,7
Concentrated must	1470 kg/h		1475 kg/h
Final sugar concnetration (weight fraction)	67,7		67,8

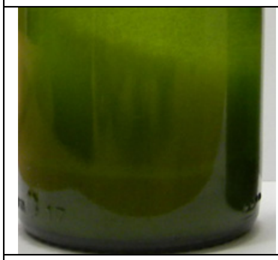
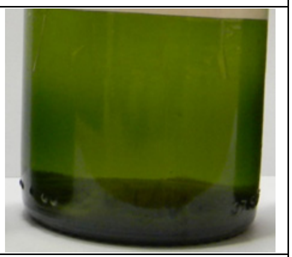
Sulphited must	Alcoholic must
	
It was noted that the particles remained in suspension	It was observed a precipitate of tartaric salts which can be separated easily from the liquid fraction formed.

Figure 4. Observations in laboratory scale.

step carried out in the conventional concentration process (Fig. 4). Mosto Sulphiting Mosto Alcoholic

After 5 months it is proceeded to analyze the samples and the distilled alcohol recovery was 100%.

As it was seen in Fig. 5, the result of alcoholization was very satisfactory, it evidenced that must sugar did not ferment and thus the alcoholization fulfilled the preservative function expected.

4. Simulation

The evapo-destillation of must alcoholized and the classic process of concentration practiced in Argentina for sulphited grape musts was simulated by the soft CHEMCAD 6, it was used typical values of this industry.

It was started with a base of calculation of 5000 kg/h of must and for heating it was used steam at 100 °C and 1.013 bar.

The parameters that are taken to simulate and results can be seen in Figs. 4 and 5.

As shown in Table 5 the consumption of steam in the process of "evapo-destillation" was 20% less than typical method of concentration, with similar totals heat exchanged in the heating equipment, heat exchangers and evaporators, see Table 6. It should be clarified that the current of concentrated must not was used to preheat the raw material in either of the two processes but the vegetable water of the base of distillation tower in evapo-destillation process was used for this purpose. See Fig. 6.

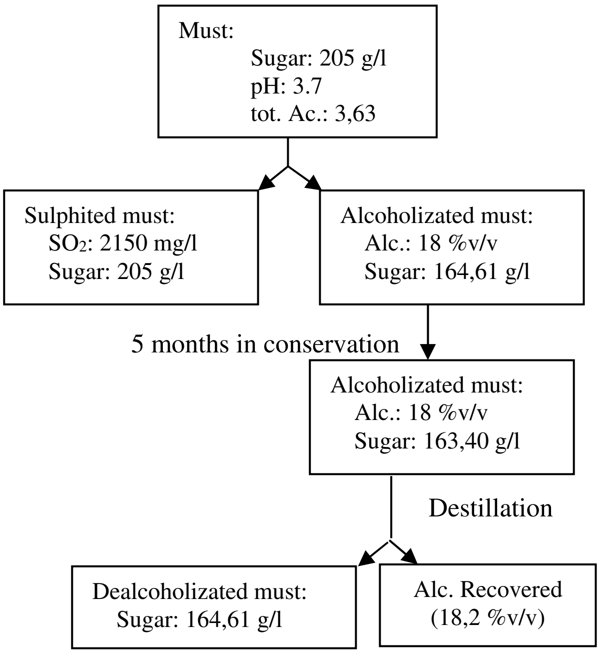


Figure 5. Schematic of laboratory-scale test.

5. Industrial scale

Samples were taken before and after of a concentration by the typical system of must concentration in evaporator of five effect and the analytical data are shown in Table 7.

Table 4. Variables Distillation Column.

Number of plates	12
Reflux in condenser	4,5
Reflux in boiler	0,3
Alimentation plates	7,9 y 11
Concentration of vegetal water	99,9% H_2O
Alcohol concentration at the top of the tower	92,6%v/v of wine alcohol

Table 5. Flow of caldera steam used in heat exchangers and evaporators.

Typical system of concentration in Argentina	kg/h	Evapo-destillation	kg/h
Preheater for preconcentration	500	Preheater	100
1st effect of preconcentration	1120	3rd evaporator	1510
Preheater for concentration	60	TOTAL	1610
1st effect of concentration	320		
TOTAL	2000		

Table 6. Energy exchanged.

Typical system of concentration in Argentina	thousand kcal/h	Evapo-destillation	thousand kcal/h
Preheater for preconcentration	280	Preheater	5
Desulfitator	9	1st effect of concentration	710
1st effect of preconcentration	616	2nd effect of concentration	773
2nd effect of preconcentration	516	3rd effect of concentration	852
3rd effect of preconcentration	417	TOTAL	2340
Preheater for concentration	28		
1st effect of concentration	181		
2nd effect of concentration	99		
3rd effect of concentration	99		
TOTAL	2245		

Table 7. Analytical data of must sulfited and concentrated.

	°Brix	mg/l SO_2
Must sulfited	20,6	2086
Preconcentrated	40,5	41
Concentrated	68,5	90

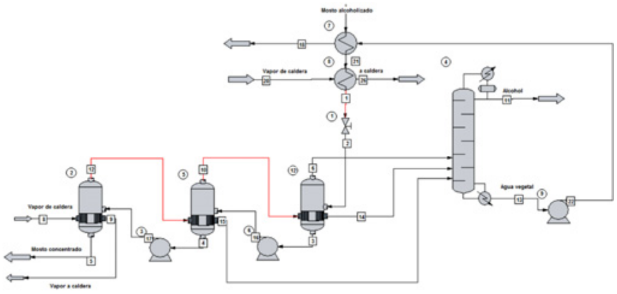


Figure 6. Evapo-destilation: flowsheet of simulation in CHEMCAD 6.

Table 8. Stages of "Concentration of must typical in Argentina" and "Alcoholization/Evapo-destillation".

Stages	Typical concentration process in Argentina	Alcoholization/Evapo-destillation
raw material	Sulfited Must	Fresh mosto
Filtration	Filtration	Centrifugation (or other method of rapid clarification)
Adjust acidity / Elimination of salts	Cation exchange (increased acidity)	Alcoholization. Formation and separation of colloidal sediments and potassium tartrate. Adjustment of acidity
Desulfitation	Desulfitation (sieve plate columns of perforated plates, direct steam stripping)	————
Preconcent.	Preconcent., 45° Brix	————
Cooling	Cooling	————
Filtration	Filtration	————
Evaporation	Reconcentra-tion, 68,5° Brix	Concentración direct to 68,5° Brix
Filtration	Filtration	Filtración
Packaging	Packaging	Packaging

Sugar determination was made by refractometer, while the analytical technique used to determine the total sulfur dioxide was oxidation in acid medium [4,6] with a tolerance of ±35 mg/l [7].

The Table 7 shows that the grape juice concentrated has a remaining amount of SO_2 that was not eliminated. It is important to note that still has not been done an industrial scale trial of alcoholization and evapo-destillation, but it is planned soon to perform in a plant in the east of Mendoza.

6. Conclusions

6.1. Advantages found if the proposed system is adopted

- The must concentrated would be free of chemical preservatives, since ethanol used would be of the same origin.
- There would have no transfer of sulphite compounds to the environment, whereas that in desulfitation process, sulfur compounds are released into the atmosphere or solution.
- There would be no loss of higher alcohols and aromatics componets of the grapes for desulfitation. Using wine alcohol as a preservative, the volatiles would be condensed and reused together with the alcohol.
- In the case of concentrated wine, vinasse pollution is eliminated, getting a marketable wine extract.
- A single heat treatment increase performance by elimination of losses in the process of desulfitation process, double concentration and double filtration. See Table 6.
- Steam consumption 20% less than the typical concentration system in Argentina.
- Can be stored in unlined pools.
- The product obtained by evapo-destillation is more natural, in this case well called juice, while today the term must is used, in the practice and in the wine legislation.
- Do not introduce mineral water.
- The reverse flow allows that most part of the concentration of salts (from wine) or sugars (from alcoholizated must) occurs in the hottest area of the evaporator, thereby the fouling by tartaric salts are reduced.
- It is obtained an alcohol unrectified but totally genuine and usable for conservation antifermentativa of musts. This wine alcohol (90%v/v) can be recycled.
- No presence of residual amounts of SO_2 in the final product.
- The cost of production of wine alcohol is significantly lower than the wine alcohol 96° GL rectified which currently is obtained from distilleries from wines.
- Losses of alcohol during storage and processing of alcoholizated musts are estimated between 5 to 7% of the alcohol used.
- At laboratory scale, with the alcoholization was observed the formation of a precipitate and a limpid liquid unlike of the sulfitation which remains a turbid must.

6.2. Disadvantages of the proposed system

- Initial inversion to generate the volume of alcohol required.
- Initial inversion for alcohol recovery equipment and storage tanks.

6.3. Other consequences of the proposed system

- It shall legislate on the destination and use of the "wine alcohol preservative", wine extract and vegetal water.
- Concentrating wine for the production of alcohol and generating a vinous extract of high commercial value; the application of this system would eliminate the problem from wine surplus that are produced every year in Argentina, revalued at the same time the price of the must as wine product.

We thank the following professionals for collaborate and provide information to this work: Ms. Carla Aruani, Ms. Raquel Gargantini from National Wine Institute and Mr. Ramón García from Guanacache S.A.

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