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## Separation & Purification Reviews

Publication details, including instructions for authors and subscription information:

<http://www.informaworld.com/smpp/title~content=t713597294>

## APPLICATION OF ION EXCHANGE TECHNIQUES TO INDUSTRIAL PROCESS OF MUST ACIDIFICATION

V. M. Palacios<sup>a</sup>; I. Caro<sup>a</sup>; L. Pérez<sup>a</sup>

<sup>a</sup> Department of Chemical Engineering, Food Technology and Environmental Technologies, Faculty of Sciences, Cádiz, Spain

Online publication date: 28 February 2001

**To cite this Article** Palacios, V. M. , Caro, I. and Pérez, L.(2001) 'APPLICATION OF ION EXCHANGE TECHNIQUES TO INDUSTRIAL PROCESS OF MUST ACIDIFICATION', *Separation & Purification Reviews*, 30: 1, 143 — 156

**To link to this Article: DOI:** 10.1081/SPM-100102987

**URL:** <http://dx.doi.org/10.1081/SPM-100102987>

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## APPLICATION OF ION EXCHANGE TECHNIQUES TO INDUSTRIAL PROCESS OF MUST ACIDIFICATION

V. M. Palacios, I. Caro, and L. Pérez

Department of Chemical Engineering, Food Technology  
and Environmental Technologies, Faculty of Sciences,  
Campus Río San Pedro, PB 40,  
Puerto Real, Cádiz 11510 Spain

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## INTRODUCTION

The grape variety cultivated in the mayor area of sherry (95%) is *Palomino fino*. This variety exhibits, among other special genetic characteristics, a low level of acidity in the must of mature grains (3.7-3.9 pH), due mainly to its low content in malic acid <sup>1,2</sup>. This situation is worsened by the typical warm climate of the area, which enlarge the malic consumption in the cells of grains by respiratory metabolism <sup>3</sup>. For this reason a correction in the pH level of the fresh musts becomes indispensable in the winemaking industry. The addition of organic acid to the grape juice is a normal prefermentative operation, in order to obtain balanced and healthy musts, and to situate the culture media in appropriate acidity for the fermentative process.

Nowadays the industrial correction of pH in sherry must is generally carried out by addition of tartaric acid in quantities that depend on the vintage <sup>4</sup>, normally 2 to 4 kg/m<sup>3</sup>. This correction doesn't affect the yeast metabolism during the fermentation stage (with tolerance until pH 2.8) <sup>5,6,7</sup>, provided additions are well controlled. A direct consequence of this operation is the continuous appearance of potassium tartrate and bitartrate precipitates during different stages of the industrial process, resulting in an important increase in the total costs.

An interesting alternative to this additive procedure is the application of modern separation techniques like percolation through ion exchange resins. In the second half of this century, several works have been published on the use of ion-exchange in the winemaking industry for avoiding tartrate precipitates <sup>8,9</sup>, and for correction of the wine acidity <sup>10,11</sup>. To a smaller degree, ion-exchange allows for elimination of metal ions <sup>12,13</sup>. In these papers it was well demonstrated that the most effective cationic resins for these applications are strongly acidic ones, such as those containing sulfonic functional groups (-SO<sub>3</sub>H). The International Office of Vineyard and Wine (OIV) has recently authorized the use of these resins in the winemaking industry <sup>14</sup>. Presently these ion-exchange techniques are being practiced in Australia <sup>15</sup>.

Under a new industrial perspective of using ion exchange techniques in fresh must acidifications, we have carried out a set of experiments at laboratory and pilot plant levels. Using the general theoretical model for continuous multi-stage processes, this study also contemplates the engineering design and the economic balance of an industrial process based on ion exchange columns, in acidification of sherry musts.

## EQUIPMENT AND METHODS

### Must and Ion Exchange Resin

The natural media to be conditioned during the laboratory tests was obtained from *Palomino fino* variety grapes, using a piston type press (7 kg/cm<sup>2</sup>), and later



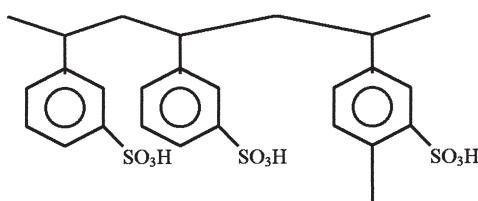
**Table I.** Composition of the Must Used in the Laboratory Experiments

Parameter	Value
density	12.3 °Be
total acidity	4.9 gTH <sub>2</sub> /L
pH	3.82
optical density 470 nm	0.284
optical density 280 nm	0.560
protein index	1.34
K <sup>+</sup>	1500 ppm
Na <sup>+</sup>	22 ppm
Ca <sup>2+</sup>	145 ppm
Mg <sup>2+</sup>	60 ppm
Fe <sup>3+</sup>	3 ppm
Cu <sup>2+</sup>	0.4 ppm
tartrate	3500 ppm
sulphate	401 ppm
phosphate	182 ppm
chloride	36 ppm

TH<sub>2</sub> = tartaric acid; °Be = Baumé degree.

the must was decanted. The composition and principal characteristics of this sherry must are detailed in Table I.

In the laboratory experiments a strong-acid cationic resin was used. It consisted of a matrix of polystyrene reticulate with divinylbenzene (8%), with sulfonic functional groups (Figure 1). The main physical-chemical characteristics of this resin are summarized in Table II<sup>16,17</sup>. The pore size is in the range of the microporous structure (10-20 Å) and the total pore volume can be calculated from the data of real and apparent density. The resolution OENO 4/95, by the OIV Commission of Enology, includes the employment of this resin for tartaric stabilization of wines<sup>14</sup>.

**Figure 1.** Structure of the sulfonic resin used in the laboratory experiments.

### Operation Conditions and Experiments

In a complete acidification cycle it is necessary to distinguish four different phases. The first one is the service phase. During this operation the ion exchange takes place among cations dissolved in the must and protons fixed to resin. The second is the wash phase. Once the resin is completely loaded, it is necessary to wash the microchannels in the bed with water countercurrently to eliminate remains of must and retained particles. The third operation is the resin regeneration phase. This is normally carried out with a HCl solution (10%) in co-current flow. During this operation, the dissolved protons in solution are exchanged with the fixed cations in the resin. The last operation is to clear phase. It is necessary to clear the rest of HCl solution retained into the resin bed, using distilled water in co-current flow. This is needed to start a new acidification cycle, and to avoid acid additions to the product. Once this operation is finished, the resin is ready for a new service phase.

The laboratory experiments have been carried out using a tubular pyrex glass reactor, with an internal diameter of 1.5 cm and filled up to 29 cm height with resin (205 mL). Table III summarizes the operation conditions imposed during the acidification cycle. The conditions are within range that the resin manufacturers recommend for general purposes. A programmable peristaltic pump is used to control the flow.

Three experimental acidification cycles were ran at conditions shown in Table III. In each cycle approximately 32 L must per L resin was eluted. This quantity was fixed in previous experiments based on the total equivalent ion charge of the must. This is the volume of must that is necessary to elute in order

**Table II.** Physical-Chemical Characteristics of the Ion Exchange Resin Used in the Laboratory Experiments

Parameter	Value
normal particles form	spherical
color	dark amber
matrix	styrene-divinylbenzene
active group	sulfonic
structure and pore size	microporous (10-20 Å)
normal supply mode	sodium mode (Na)
real density (Na mode)	1.28 g/cm <sup>3</sup>
apparent density (Na mode)	0.85 g/cm <sup>3</sup>
normal particle size	0.3 – 1.2 mm
total exchange capacity	2.0 Eq/L
maximum temperature	120 °C
pH interval	1 – 14



**Table III.** Operation Conditions of the Different Phases in the Laboratory Experiments

Phase	Fluid	Flow <sup>*</sup>	Time (min)
Service	must	6	300
Wash	water	10	6
Regenerate	HCl (10%)	5	10
Clear	distilled water	10	3

\* Flow as dilution rate ( $L_{\text{fluid}}/h L_{\text{resin}}$ )

to obtain an effluent pH equal than feeding pH. This assumes the exhaustion of the exchange capacity of the resin in the column. During all the experiments we took continuous samples of the effluent (100 mL) for later analysis.

Finally, we carried out an experiment at industrial scale to optimize the acidification process and to check process viability. In this experiment, we used a stainless steel tubular reactor, with internal diameter 43 cm and height of 175 cm resin (1 m<sup>3</sup>). The must used and the operation conditions were the same ones employed at the laboratory scale.

### Analysis

The following analyses have been carried out on each effluent sample in all the experiments. The pH was measured with an electronic pH-meter. The concentration of several cations: K<sup>+</sup>, Na<sup>+</sup>, Ca<sup>2+</sup>, Mg<sup>2+</sup>, Cu<sup>2+</sup> and Fe<sup>2+</sup>, were measured by atomic absorption spectrophotometry<sup>18,19</sup>. The color index (CI) was measured by determination of the optical density at 470 nm<sup>20</sup>. Finally, the protein index (PI) was obtained by the modified Lowry method<sup>21</sup>.

## RESULTS AND DISCUSSION

### Effects on the pH

The variation of pH during the acidification experiments are shown in Figure 2. It can be observed there are two well-differentiated phases. There is a first phase with a fixed pH, that is extended from the beginning of the elution until approximately 20 bed volumes. This can be called the “load phase”. Later a second phase appears, that is extended from this point until the end of the experiment (30 bed volumes). This can be called “unload phase”.

During the load phase, important exchange reactions take place among the cations dissolved in the must and the protons fixed into the resin. The outlet pH in



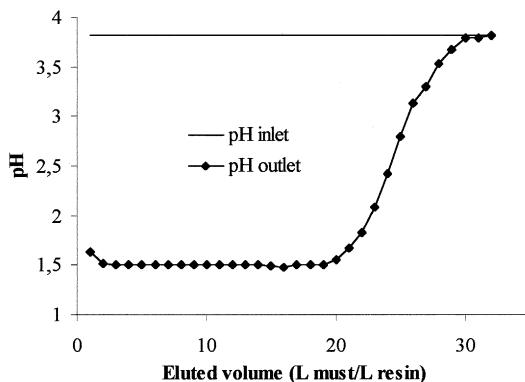


Figure 2. Variation of pH in the outlet must during the acidification experiments.

the must is around a constant value of 1.51. Next, in the unload phase, new ion exchange reactions occur among the dissolved cations with higher affinity and the fixed cations with less affinity. No more protons are transferred from the resin to the liquid. As a consequence, the outlet pH of the must increases again toward the inlet value. In this phase the resin returns part of the low-affinity fixed cations.

#### Effects on the Concentration of Alkaline Ions

As it seen in Figure 3, the variation of  $K^+$  and  $Na^+$  concentrations in effluent presents a very similar behavior to the one for pH.

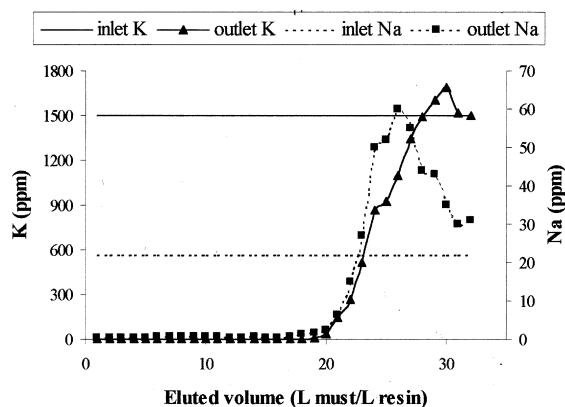


Figure 3. Variation of the concentration of major alkaline cations ( $K^+$  and  $Na^+$ ) in the eluted must.



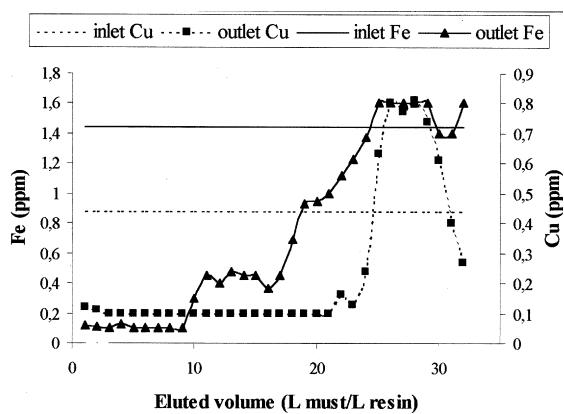
During the load phase the  $\text{Na}^+$  and  $\text{K}^+$  concentrations stabilize at very low levels with respect to the value of the inlet must (below 1 ppm for both cations). However, during the unload phase the concentrations increase significantly due to the low affinity of these alkaline cations. The concentrations can go up above the feed values and, for a short period, the resin unloads a higher concentration than it loads. The resin loses  $\text{Na}^+$  cations more quickly than  $\text{K}^+$  cations, but both present an exchange curve very well defined. These curves exhibit the characteristic pattern of the axial dispersion and mass transfer limitation in tubular reactors.

For  $\text{Ca}^{2+}$  and  $\text{Mg}^{2+}$ , it is necessary to point out that both ions a low, constant concentrations in the outlet must during the whole experiment. Such behavior is not different to that of the expected: trend an initial, stable value followed by an increase. However, there is no time in 30 volumes to observe the unload phase for these divalent cations, due to the lower initial concentration and their higher affinity for the resin. In consequence, they are practically undetectable in the eluted must during the whole process and their concentration remain under 1 ppm even during the unload phase.

#### Effects on the Concentration of Metal Ions

The variation of the metals concentration ( $\text{Fe}^{2+}$  and  $\text{Cu}^{2+}$ ) in the eluted must during the acidification experiments doesn't differ much from the behavior of major alkaline cations, as shown in Figure 4.

The concentration of the metals remain significantly low during load phase (less than 0.2 ppm) and later an increases to the inlet values or more. The  $\text{Cu}^{2+}$



**Figure 4.** Variation of the concentration of major metal cations ( $\text{Fe}^{2+}$  and  $\text{Cu}^{2+}$ ) in the eluted must.

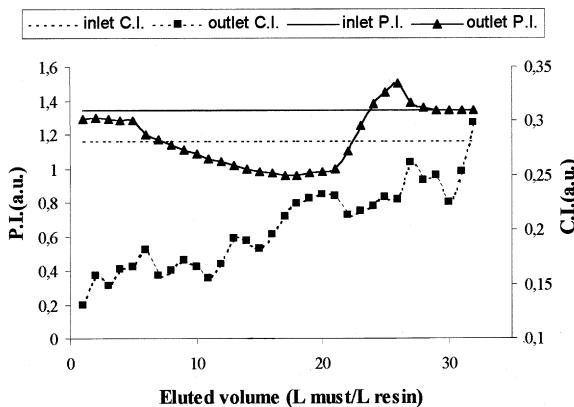


cation begins to come out at 20 bed volumes of eluted must, which is the start point of the unload phase. On the other hand,  $\text{Fe}^{2+}$  seems to come out slightly before, which is probably due to its lower affinity for the resin. The partial elimination of these metals in the conditioned musts is an interesting approach order to enhance the redox stability of the future wines.

### Effects on Other Parameters

It is also of interest to evaluate the effect of the ion exchange operations on the compounds responsible for the color of wines (polyphenols). During the acidification the optical density at the characteristic wavelength for must color (470 nm) was monitored. In Figure 5 it can be observed that the color index (CI) diminishes initially to 25% of the inlet value. However, this decrease is slowly recovered during the entire load phase. At the end of the unload phase, the optical density of the eluted must is practically the same than the one of the feed must. This behavior shows that the resin exercises a certain retention of polyphenolic compounds, but many cations are able to displace it. As a consequence, the overall CI reduction of the must is under 10%, that don't affects the product quality. The Sherry industry considers that a must with a CI under 0.1 can be abnormal. Moreover, the poliphenolic compound reduction in wine can be positive for the future redox stability of this type of wine.

Figure 5 it also shows the variation of the protein index (PI) of the eluted must during the experiments. Proteins seem to be retained only in a very small quantity and principally only at the end of the load phase. Later, at the beginning of the unload phase, part of the retained proteins are released. This effect is prob-



**Figure 5.** Variation of the protein index (PI) and color index (CI) in the outlet must.



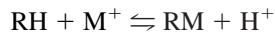
ably due to the change of the pH in the column at this point, which modifies the net charge of the protein molecules and its affinity for the resin. As a consequence, the overall PI reduction of the must is under 3%, that don't affects the fermentative potential of the must, since it can be considered that a must with a PI under 0.5 can have fermentation problems.

### Standard Model for Ion Exchange Operations

The theoretical model for the ion exchange process can be treated similarly to the general model for multistage process, which is classically applied to gas-liquid or solid-liquid operations. The steady state ion exchange process can be modeled with the resin loaded into the column circulating continuously in upward flow, while the liquid continuously circulates in downward flow. Multiple equilibria can occur between resin and solution in the multiple theoretical stages in the continuous contact equipment. To complete the mathematical model of the process, we must obtain, the equilibria equations and the operating lines. Secondly, we calculate the height equivalent to a theoretical stage (or plate) for each cation equilibrium state involved in the multicomponent system. We assume that the presence of a cation does not affect the ionic equilibria of other cations.

Because the acidification cycle described above is not a true continuous operation, we must apply the continuous model only to the first phase (service phase) in the laboratory experiments. Since during the service phase ion concentrations remain approximately constant at the column inlet and outlet, we can consider a pseudo-stationary state of the process.

The equilibrium equation of a stage in a ion exchange multistage process relates the molar concentration of ions in solution with the molar concentration in the resin at the outlets of each equilibrium stage. The form of this equation will depend on the type of interchange produced between the resin and solution. In our particular case monovalent ions or divalent ions are involved. The general reaction for the interchange of monovalent  $M^+$  ions is:



Where RH represents the initial form of a ligand in the resin (protonated) and RM is the final state of that ligand. The equilibrium equation for this process is:

$$\bar{X}_M = \frac{K_H^M X_M}{1 + X_M(K_H^M - 1)}$$

Here,  $\bar{X}_M$  is the molar fraction of the ion into the resin and  $X_M$  is the molar fraction of ion in solution. Further  $K_H^M$  is the specific selectivity coefficient of  $M^+$  with regard to  $H^+$  for the resin used. In Table IV the values of different selectivity coefficients are shown.



**Table IV.** Chemical Data and Design Data for the Different Cations Under Study

i	K <sub>H</sub> <sup>i</sup>	HETP	R%	C%	C <sub>i</sub>
Na <sup>+</sup>	1.5	0.147	97.15	1.00	18
K <sup>+</sup>	2.5	0.054	99.94	36.44	990
Mg <sup>2+</sup>	2.5	0.073	99.97	4.06	45
Fe <sup>2+</sup>	2.6	0.192	78.32	0.10	2
Cu <sup>2+</sup>	2.9	0.290	76.91	0.02	0.2
Ca <sup>2+</sup>	3.9	0.152	99.07	6.33	90

K<sub>H</sub><sup>i</sup>: Selectivity coefficient for the i cation.

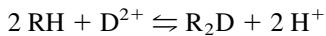
HETP: height equivalent to a theoretical plate (m).

R%: reduction grade of the cation concentration in the must (%).

C%: final charge of the cation in the resin (%).

C<sub>i</sub>: cation concentration in the final industrial must (ppm).

Similarly, the general equations for the interchange of divalent D<sup>2+</sup> ions are:



$$\frac{\bar{X}_D}{(1 + \bar{X}_D)^2} = K_H^D \frac{\bar{T} X_D}{T(1 - X_D)^2}$$

Here  $\bar{T}$  represents the total molar concentration of ions on the resin (D<sup>2+</sup> plus H<sup>+</sup>) and T the total molar concentration of ions in solution.

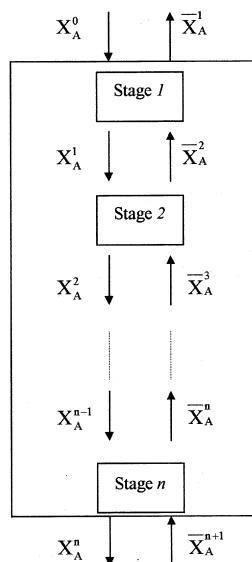
The operating line in an ion exchange multistage process relates the molar concentration of ions in solution with the molar concentration in the resin, at a point between any two consecutive equilibrium stages. If we assume that the ion exchange column behaves as a system with N stages, the operation line is obtained from the values of molar concentration at the bottom of the column (liquid outlet and resin inlet) and the top of the column (liquid inlet and resin outlet). Figure 6 shows the sequence of values in the ion exchange column. The equation of the operation line is the following:

$$\bar{X}_A^{i+1} = \frac{\bar{X}_A^1 - \bar{X}_A^{n+1}}{X_A^0} - X_A^a (X_A^i - X_A^a) + \bar{X}_A^{n+1}$$

where n is the number of the last equilibrium stage and i is the number of any stage.

Using the equilibrium equation and the operating line, one can obtain graphically or numerically an estimation of the number N of theoretical equilibrium stages necessary to carry out the required. The height equivalent to a theoretical stage or theoretical plate for each cation (HETP) can be calculated from dividing





**Figure 6.** Molar fraction of cations in the liquid and into the resin, at different equilibrium stages in an ion exchange column.

the linear height of the column by the number of theoretical stages. In Table IV the different equivalent heights for each cation are calculated.

In each case, the number of theoretical stages in the column will fundamentally depend on the affinity of the resin for that cation (selectivity constant) and on the initial and final concentrations of that cation (molar fractions). Under the usual industrial conditions, significant differences exist for the concentrations of the different ions, and therefore the obtained number of theoretical stages are different, even in the case of very similar selectivity constants. In Table IV it is also shown the reduction grade of each cation concentration in the must and its final charge in the resin.

### Optimization of the Industrial Process

The experiments carried out at industrial scale corroborate the obtained result at laboratory stage. The behavior of the different parameters doesn't differ from the one shown to scale laboratory. According to our results, the process of must acidification at the industrial scale must be carried out in an effective way during the general load phase. In this phase the outlet pH remains constant around 1,5 and principal metal ions of the must are retained with considerable effectiveness. Therefore, seems in principle a logical approach to establish a cycle volume



of the process corresponding to the load phase (20 bed volumes). However, if the treatment is prolonged going into in the unload phase (30 bed volumes), some cations initially retained by the resin will be recovered without modifying substantially the pH of the must. A further expansion of the cycle volume only leads to effectiveness losses.

Further, at the industrial level it is not necessary of course the completely treat all of the must processed in vintage, due to the very high effectiveness of this technique. It is advisable to partially treat the vintage and blend with the rest of the product. Operating in this way yields a double effect, first the acidification process is more capitalized, and second, the treated must can be compensated in alkaline cations and other parameters (color and protein index). For the sherry industry, it could be necessary to treat 30-35% of the vintage.

Table V shows an estimation of the cost savings that would result from the substitution of the classic acidification with tartaric acid for the ionic exchange operation. According to this economic balance, ionic pH regulation decreases the operation cost to less than 10% of the classical method.

The advantages of the ion exchange process, is that the treatment doesn't produce any significant alteration in the organoleptic properties of the future wines, providing greater stability to the products. Besides, in the classical treatments, the acidification is usually caused by addition of chemicals, while in the proposed treatment it is caused by substitution.

The added excess of tartaric acid also leads to the formation of crystalline precipitates of potassium bitartrate and calcium tartrate in the deposits and pipes. This causes difficulties in the temperature control of fermenters and in the cleaning operations, which now become more expensive.

Another important advantage of the proposed industrial operation is that it produces wastewaters that are very easy to control, contrary to the treatment with

**Table V.** Estimated Calculations of Acidification Costs

Supplies*	Expense (Kg/m <sup>3</sup> )	Unit cost (US\$/Kg)	Total cost (US\$/m <sup>3</sup> )
Acidification With Tartaric Acid			
Tartaric acid	3	6.00	18.00
Acidification With Exchange Resin			
Mineral regenerants	2	0.30	0.60
Cleaning water	100	0.005	0.50
Column amortization	0.2	3.00	0.60
Total			1.70

\* Supplies and total cost per one m<sup>3</sup> of conditioned must.



tartaric acid. Furthermore, the different outlet currents in the wash, regenerate, and clear phases of the operation can be neutralized and used as liquid fertilizer for agricultural lands (including the vineyard), due to the high concentration in mineral salts. This type of industrial methodology, based on the total recycling of the processed elements, is perfectly integrated inside the recommendations of the ISO14000 normative.

### CONCLUSIONS

The application of the ion exchange technology for the acidification of grape juice, using strong acidic cationic resins, is shown as an effective procedure for the pH regulation of musts, and in particular of the warm areas. This technique allows substituting the traditional practice of tartaric acid addition without producing appreciable alterations in the enological qualities of the products.

At the industrial level, the partial treatment of the vintage with blending of the untreated part with the rest of the raw material constitutes an important economic improvement with regard to the traditional technique.

Finally, the ion exchange technologies for the must acidification processes in warm areas have been shown as an alternative technology consistent with the current industrial idea of sustained development.

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