CHEMICAL ENGINEERING TRANSACTIONS
VOL. 75, 2019
A publication of AIDIC
The Italian Association of Chemical Engineering
Online at www.cetjournal.it
Guest Editors: Sauro Pierucci, Laura Piazza

ISBN 978-88-95608-72-3; **ISSN** 2283-9216
DOI: 10.3303/CET1975047

Use of Potassium Polyaspartate for the Tartaric Stabilization of Sicilian White Wines

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Cold stabilization is a common method used to avoid tartaric acid crystals from forming in bottled wine, but this technique shows some inconveniences on the sensory characteristics and energy cost. In the present research, the tartaric stabilization in Sicilian white wines, with a recently permissible molecule in oenology, was studied: the potassium polyaspartate (PAK). The PAK has a negative charge at wine pH and allows to sequester the K^+ cations; consequently it inhibits the formation and the growth of potassium bitartrate crystals. PAK is a relatively small polymer; perfectly microfiltrated and does not allow the filling phenomena in filtration membranes. The adding of PAK allowed to reduce almost all the tartaric precipitations on very unstable wines, regardless of the chemical-physical characteristics of the treated white wines. The stability of PAK, after thermal stress of the wine added to this polymer, was confirmed evaluating different analytical parameters such as pH, total acidity and buffer power, while variations in the conductivity of wines seemed to indicate a residual tartaric instability. In conclusion, the use of PAK makes possible to improve the sensory characteristics of wines, considering lower losses of tartaric acid and potassium, which are important for acid perception and acid persistence (buffer power), as well as reducing production costs and low environmental impact.

1. Introduction

The formation of potassium bitartrate (KHT) is the main cause of precipitate in unstable bottled wines due to the crystallization process, which occurs in two phases: nucleation and crystalline growth (Moutounet et al., 1997). Nucleation is formed by a crystalline germ when conditions of over-saturation occur. The solubility of KHT decreases as the alcohol content increases and the temperature decreases: for instance, it is 2.9 g/L at 20 °C in a 10 % (alcohol by volume); thus, at the end of the alcoholic fermentation, wine is in a state of over-saturation. Some of the macromolecules of wine (pectins, proteins, mannoproteins), called "colloid protectors", hinders KHT precipitation but the wine remains in a state of instability, as the effect of these protective colloids is not permanent and can vary during time (Moine-Ledoux et al., 2005; Moine-Ledoux et al., 2002; Lubbers et al., 1993). The precipitation of KHT excess in wine bottle is a problem to be avoided, as the consumers do not accept it. Hence, tartaric stabilization of wines before bottling, achievable with subtractive or additive techniques, is needful; the former reduces the concentration of tartaric acid and/or potassium through cold, electrodialysis or ion exchange resins, the latter consists in the addition of crystallization inhibitors such as metatartaric acid, carboxymethyl cellulose, mannoproteins and, lastly, potassium polyaspartate, a recently approved, and in this study evaluated, polymer (Crachereau et al., 2001; Corona et al., 2014; Bosso et al., 2015; Gerbaux, 1996). The subtractive techniques modify the sensory quality of wines (Lambri et.al, 2013) decreasing the "freshness" taste (less tartaric acid) or the persistence of the acid sensation (less potassium). Cold stabilization can cause colloidal precipitation reducing polyphenols and polysaccharides, key compounds for colour, volume and structure of wine. In addition, quality white wines have a great impact on global warming potential during the refining phases, including cold stabilization, which can last several months at controlled temperature (Iannone et al., 2014). Potassium polyaspartate is the potassium salt of polyaspartic

Paper Received: 15 March 2018; Revised: 23 August 2018; Accepted: 12 December 2018

Please cite this article as: Cinquanta L., Zarzana D., Planeta D., Liguori L., Albanese D., Di Matteo M., Corona O., 2019, Use of Potassium Polyaspartate for the Tartaric Stabilization of Sicilian White Wines, Chemical Engineering Transactions, 75, 277-282 DOI:10.3303/CET1975047

acid, produced from L-aspartic acid and potassium hydroxide. The thermal process transforms the aspartic acid into an insoluble polysuccinimide, which is treated with potassium hydroxide, allowing the opening of the ring and the polymerization to obtain potassium polyaspartate of the units with an average molecular weight of 5 kDa (Figure 1).

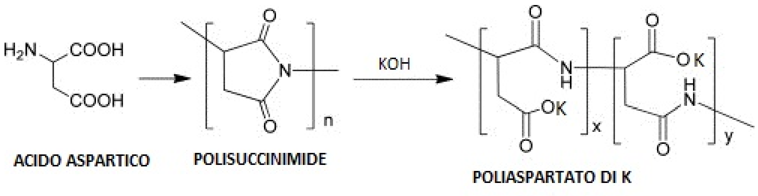

Figure 1: Process to obtain potassium polyaspartate

The last stage is spray drying, which transforms it into a light brown powder that is very soluble in water and is marketed in a 10% solution. PAK has a negative charge on the pH of the wine, which allows it to seize K^+ cations and, consequently, inhibit the formation and growth of potassium bitartrate crystals. Similarly, this negative charge makes it very reactive to proteins that may be present in the wine, causing clouding. Therefore its use must be preceded by a perfect protein stabilization and filtration. Being a relatively small polymer, it is perfectly microfiltrable and does not give rise to phenomena of membrane filling. In brief, polyaspartate has a stabilizing effect due to its negative charge at the pH of wine, which enables to sequester K cations and, thus, to inhibit the formation and the growth of crystals (Joentgen et al., 2005; Thombre et al., 2005). The aim of this work was to evaluate the effectiveness of the potassium polyaspartate (PAK) for tartaric stabilization of different white wines. Furthermore, the stability over time of the PAK effect was assessed by means of a heat treatment at 50 °C for 7 days.

2. Material and Methods

Eight white wines (2017 vintage) of grapes cultivars Catarratto, Zibibbo, Grillo, Pinot Grigio, Chardonnay and *Viognier*, were studied for trials. The winemaking process was carried out on industrial scale in duplicate, at vinery Vini Vaccaro Agricola SRL in Salaparuta (Trapani, Sicily, Italy). All samples were previously subjected to protein stabilization with bentonite, which effectiveness was verified by a hot protein stability test. The stability of wine samples to tartaric precipitation was evaluated by means of the mini contact test, which showed that all wines were unstable. Alcohol content, reducing sugars and volatile acidity were determined by means of Winescan (FOSS, Napa, CA, USA) according to the OIV Compendium of International Methods of Analysis of Wine and Musts (2007). To evaluate the effectiveness of PAK, the wine samples were subjected to the cold stabilization technique, in absence and presence of PAK (Figure 2).

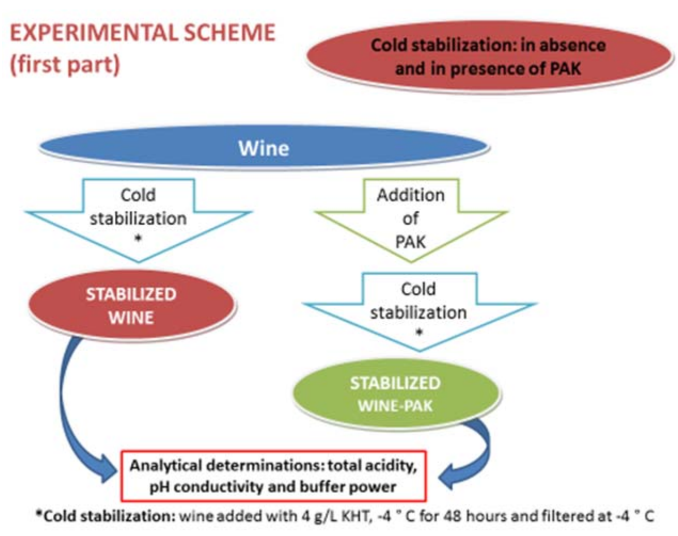

Figure 2: Diagram of the first experimental phase of PAK addition

Cold stabilization was carried out on 500 mL samples at -4 °C for 48 hours with the addition of 4 g/L of micronized potassium bitartrate (KHT) and periodic shaking. The addition of PAK (100 mg/L) was made on 500 mL samples. Total acidity, pH, conductivity and buffering power (EEC-Community Analysis Methods to Use in Wine Sector, 1990; Liguori et al., 2018; 2015) were determined on wines in duplicate, before and after each treatment. To verify the different stability of the wines after cold stabilization treatment and addition of PAK, the samples were then subjected to thermal stress (50 °C for 7 days) and, again, to cold treatment to verify variations in the level of tartaric stability, owing to the presence or not of PAK. As a control, the same wine samples not added with PAK, were subjected to cold stabilization and then subjected to heat treatment and again cold stabilized (Figure 3).

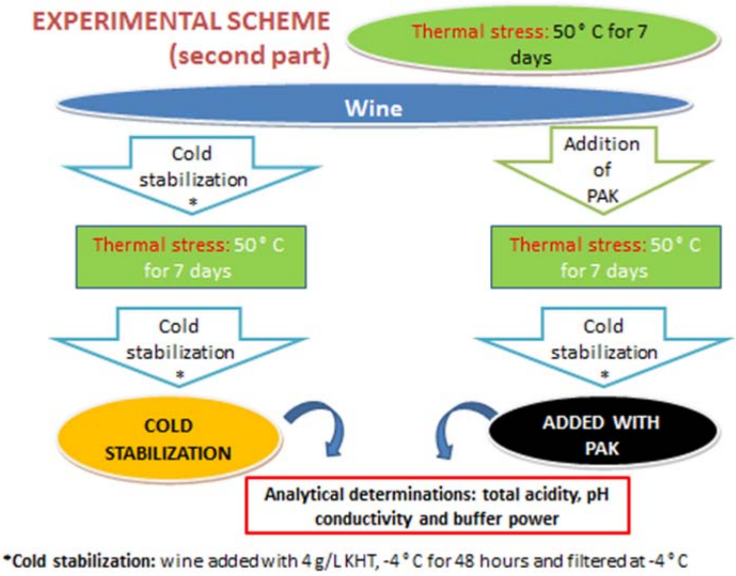

Figure 3: Diagram of the second experimental phase of PAK addition after thermal stress.

3. Results and Discussion

The wines analyzed in this study showed different chemical-physical compositions (Table 1). In particular, they were different in terms of ethanol content (from 10.60 to 14.82 % v/v), total acidity (from 4.40 to 5.60 g/L), pH (from 3.26 to 3.50), conductivity (from 1450 to 1681 μS/cm) and buffering power (from 29.4 to 35.7 meq/L). The same parameters were then reported as differences between the cold stabilized wines, in absence or in presence of PAK. As for the total acidity, cold-stabilized wines have undergone an appreciable reduction (from 0.45 to 0.75 g/L), whereas in wines added with PAK the acidity decrease (Figure 4) was smaller (from 0.10 to 0.50 g/L wine). These results showed the stabilizing effect of the PAK, confirmed by the pH values too (Table1).

Table 1: Ethanol, pH, total acidity, conductivity and buffer power of wines.

Cultivars	Ethanol (% v/v)	Free SO_2 (mg/L)	Total SO_2 (mg/L)	Volatile acidity (g/L as acetic acid)	pH	Total acidity (g/L as tartaric acid)	Conductivity (μS/cm)	Buffer power (meq/L)
Pinot grigio	12.84	42	84	0.38	3.37	5.10	1538	31.3
Catarratto 1	13.13	39	79	0.32	3.37	5.60	1569	33.3
Catarratto 2	12.72	39	83	0.35	3.26	5.50	1466	35.7
Chardonnay	14.44	40	88	0.40	3.44	5.60	1681	35.7
Viognier	14.82	41	76	0.38	3.44	4.90	1520	31.3
Zibibbo	13.16	40	79	0.32	3.50	4.50	1617	29.4
Grillo 1	11.60	42	82	0.28	3.38	4.40	1592	29.4
Grillo 2	13.94	40	88	0.34	3.33	5.10	1450	33.3

To evaluate the stability over time of the PAK, an accelerated aging test was carried out heating both wines at 50 °C for 7 days. Subsequently, wines underwent to another cold tartaric stabilization to evaluate any further variations. Therefore, total acidity, pH, conductivity and buffer power were again determined. It was observed, in the cold stabilized wines, that the thermal stress caused a further reduction in total acidity (from 0.10 to 0.25 g/L) and, consequently, a slight increase in pH, while there were no variations or negligible in wines added with PAK (Figure 5). Wines added with PAK and subjected to heat treatment showed a decrease in conductivity from 44 to 118 mS/cm, higher than those found in cold-stabilized wine samples (Figure 6). The lowering of the conductivity would suggest salts precipitation; however, this finding was not supported by analytical data of both pH and acidity. Rather, it could be hypothesized a higher sensitivity of colloids to heat in wines added with PAK. As for the buffer power, there were negligible variations and only in some wines. Overall, the results obtained confirmed what was reported by Bosso et al. (2015), which showed a similar stabilizing efficacy of PAK compared to metatartaric acid (MTA), commonly used as an oenological adjuvant, but with an effect over time greater than MTA. Lastly, in order to reduce any risk of precipitation in wines with high tartaric instability, an intermediate solution could be hypothesized*, i.e.* a lower intense cold stabilization supported by the addition of PAK.

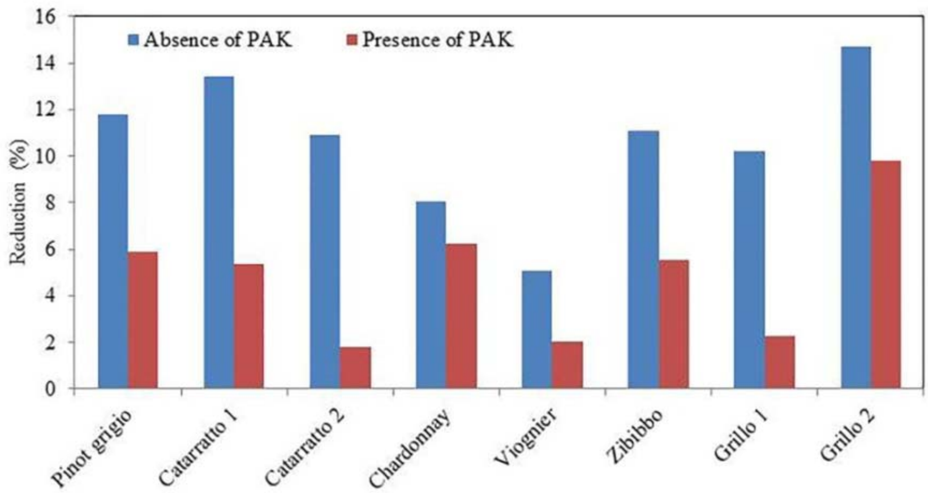

Figure 4: Percentage reduction in total acidity between cold stabilized wines, in absence or in presence of PAK.

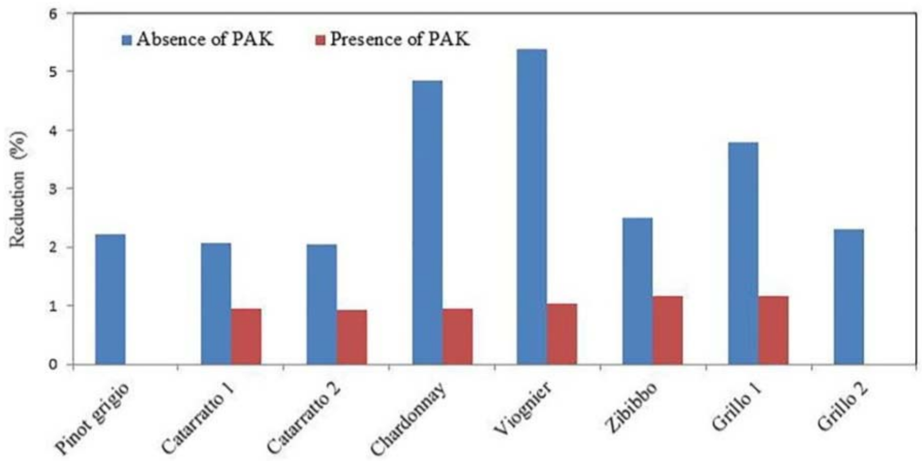

Figure 5: Percentage reduction in total acidity between cold stabilized wines, in absence or in presence of PAK, after accelerated aging test (50°C for 7 days).

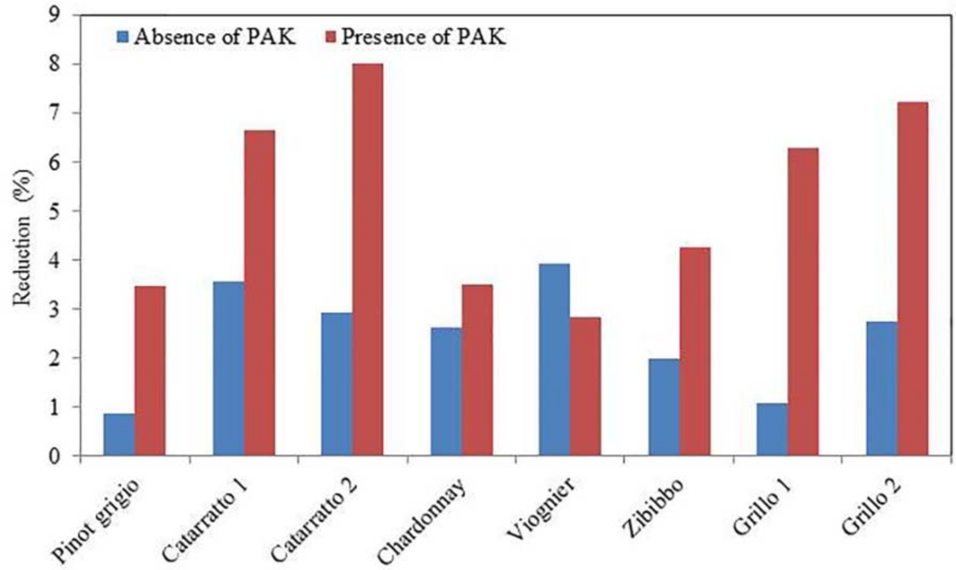

Figure 6: Percentage reduction in Conductivity (µS/cm) between cold stabilized wines, in absence or in presence of PAK, after accelerated aging test (50°C for 7 days).

4. Conclusions

The results of this research showed that tartaric stabilization could be effectively dealt with potassium polyaspartate (PAK). In fact, this additive was able to reduce almost all the tartaric precipitations on white unstable wines of different cultivars. Compared to the classical cold stabilization, considered as subtractive technique, the use of the PAK allows reaching a satisfactory tartaric stability without the loss of tartaric acid and potassium, important for the sensory quality (perception and persistence of the acid sensation). Moreover, the use of potassium polyaspartate is labour, energy and water efficient, and hence cost competitive. The significant reduction of total acidity caused by cold stabilization, in wines with low acidity, such as those used in this study (recurrent situation in hot regions), produced wines with the legal limit lower than that of DOC (controlled designation of origin) considered. Finally, as regards the stability in time of the PAK on white wines, accelerated aging tests confirmed the effectiveness of this additive over time.

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