Additives

Is KPA the magic bullet for tartrate instability in wines?

Neil Scrimgeour, Thomas Almond and **Eric Wilkes** from the Australian Wine Research Institute (AWRI) share the findings from their research into the potential of the additive potassium polyaspartate (KPA) as an effective inhibitor of tartrate crystallisation in winemaking.

Introduction

Tartrate instability is one of the most common issues seen in winemaking and can manifest itself as the precipitation of potassium hydrogen tartrate (KHT) crystals in bottled wine. The deposit sometimes resembles shards of glass, which can cause alarm for consumers.

Potassium and tartaric acid are both natural constituents of grapes and grape juices typically contain a high concentration of dissolved KHT (Boulton *et al.* 1996, Rankine, 1989, Zoecklin *et al.* 1995). A significant proportion of this is, however, insoluble in wine, due to the limited solubility of KHT in alcoholic solutions, with the solubility progressively decreasing as the alcohol concentration increases. KHT can remain in a supersaturated state in wine and, unless stabilised in some manner, can precipitate out after bottling, especially if bottled wines are refrigerated prior to consumption.

There are three different types of tartrate stabilisation processes commonly employed to prevent post-bottling KHT precipitation:

1. Processes that use refrigeration followed by filtration to remove KHT deposits. These include cold stabilisation (cold chilling), seeding with KHT crystals and various continuous contact processes.
2. Processes that remove one or more of the compounds involved in KHT precipitation, such as ion exchange or electrodialysis.
3. Processes that use additives to inhibit or impede crystallisation, such as metatartaric acid, yeast mannoproteins and carboxymethylcellulose (CMC) products.

Cold stabilisation, with seeding by KHT, is the most widespread option used by wine producers. Although it is effective, the refrigeration required can add significantly to the operational cost and environmental impact. This type of process can also result in the loss of wine colour. Processes such as ion exchange and electrodialysis require a very high capital investment and a significant degree of skill to operate effectively. Utility, water and maintenance costs can also be high. Recent advancements in tartrate stabilisation have focused on the development of compounds that can inhibit tartrate crystallisation. However, to date, there have been limited options suitable for achieving long-term stability in both white and red wines, without the loss of colour and/or positive sensory attributes.

Potassium polyaspartate (KPA)

Potassium polyaspartate (KPA) is a synthetic polyamide (Figure 1), produced industrially by thermal polymerisation of aspartic acid in both the acid form and as the potassium salt. Polymer chain length and structure depend on the production process used. It is purported to be effective in inhibiting tartrate crystallisation in white, rosé and red wines and in maintaining long-term tartrate stability in wines. KPA has been approved for use in winemaking since 2016 (OIV International Code Of Oenological Practices) and in 2019 was approved for use in Australian wines by Food Standards Australia New Zealand.

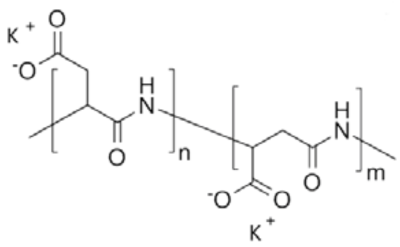

Figure 1. Structure of potassium polyaspartate (KPA)

The KHT crystallisation process depends upon the concentration of KHT as well as the presence of other crystallisation nuclei. Certain wine components such as proteins, polyphenols and polysaccharides can act as natural crystallisation inhibitors, all of which aid in the increased tartrate stability of red wines compared to white wines. White wines tend to become tartrate unstable more rapidly, due to the lack of natural crystallisation inhibitors. As KPA is a negatively charged polymer at wine pH, it can interact with the positively charged potassium ions (K+) of KHT, inhibiting the growth and formation of crystals.

Testing KHT stability

The potential for potassium bitartrate to precipitate from a wine can be predicted, with varying degrees of reliability, by a number of tests. These include:

4. Exposing a wine to low temperatures over a set period of time and visually inspecting for signs of crystal formation.
5. Inducing crystal formation by freezing and thawing the wine.
6. The determination of concentration product (CP) values.
7. Measurement of the change in conductivity of a sample held at low temperature after seeding with KHT crystals.

The AWRI recommends the use of the refrigerated cold stability (cold hold) test as an industry standard. This involves holding the wine at -4°C for three days and requires little outlay on capital equipment.

Assessing the efficacy of KPA

A recent trial was conducted to evaluate the performance of KPA for achieving tartrate stability in wines, with 13 white, rosé and red wines from various regions in Australia and the USA treated with a commercial KPA product (Zenith Uno, Enartis) and tested for tartrate stability across a six-month period, alongside untreated (unstable) control wines.

The 13 wines were filtered (0.45 µm polyether sulfone filters, 800 cm^2 flow) to achieve turbidities of < 2 NTU and were treated with the KPA product at a dose rate of 1 mL/L. Treated samples were analysed in duplicate alongside control (untreated) samples immediately after treatment (t0), and then again after three months (t3) and six months (t6) of storage at controlled cellar temperature (15°C). The following tests were conducted at each time point:

- titratable acidity
- potassium concentration
- turbidity
- colour profile*
- conductivity (mini-contact test)
- cold hold (3-day)
- cold hold (20-day)

* White wines were analysed using absorbance measurements at 280, 320, 420, 520 and 620 nm. Red wines were analysed using the Modified Somers method.

Three of the wines that were deemed to be protein (heat) stable were also subjected to elevated temperature (40°C) for a 14-day period, to mimic the potential effect of heat exposure experienced by wines during shipment to international markets.

The cold hold tests were performed on samples placed in a sedimentation flask and held at -4°C for three days. Following visual analysis, samples were returned to cold storage and subsequently re-analysed after a total time of 20 days.

Mini-contact testing was performed by an external laboratory according to the following procedure:

- 250 mL of sample chilled to -2.2°C; initial conductivity recorded (micro Siemens, μS)
- sample subsequently dosed with 2.5 g potassium bitartrate (KHT)
- sample held at -2.2°C for 20 minutes and conductivity subsequently measured
- final conductivity compared to the initial conductivity and the difference calculated ($\Delta\mu S$).

The following criteria were used to determine tartrate stability of wine samples:

- $\Delta\mu S < 25$, very stable
- $25 \leq \Delta\mu S < 40$, stable
- $40 \leq \Delta\mu S < 60$, at risk
- $\Delta\mu S > 60$, unstable

All wines in the trial were pre-bottling samples and had not undergone any form of stabilisation. Wine samples were analysed for basic chemical attributes (% alcohol, pH, titratable acidity, glucose + fructose, cold stability, heat stability, potassium, calcium, tartaric acid, turbidity, colour profile and free and total SO_2) prior to treatment.

KPA performance results

In the cold hold tests, no tartrate precipitated out of any of the KPA-treated wine samples, even after 20 days at -4°C, up to and including three months post-treatment. In contrast, the control samples showed significant tartrate precipitation when examined after three days at -4°C. Figure 2 shows the appearance of precipitated KHT crystals in the control (untreated) samples after three months' cellar storage, while the KPA-treated samples showed no sign of precipitation three months post-treatment.

Figure 2. Comparison of untreated (top images) and KPA-treated (bottom images) samples after three months' storage. a) White wine control (untreated) sample after three months' storage (t3). b) KPA-treated white wine sample three months post-treatment (t3). c) Rosé wine control (untreated) sample after three months' storage (t3). d) KPA-treated rosé wine sample three months post-treatment (t3). e) Red wine control (untreated) sample after three months' storage (t3). f) KPA-treated red wine sample three months' post-treatment (t3).

Conductivity testing results after three months' storage showed that all of the treated samples were either 'very stable' or 'stable', except for White_Wine_07 and White_Wine_08, which were borderline 'at risk', according to their ΔμS values. All control samples were either 'unstable' or 'at risk', with the exception of Red_Wine_01, which was designated as 'stable' and Red_Wine_03, which had one replicate sample recorded as 'stable' These two red wines did not show any tartrate precipitation in the cold hold test, but did show slightly higher ΔμS values than their KPA-treated counterparts, suggesting that they may have the potential to become tartrate-unstable over time.

There were no significant differences in the basic chemical attributes for the 13 wines tested, pre- and post-KPA treatment. Importantly, there were no changes to the colour profile of the red wines tested (i.e. no colour loss) and potassium concentration levels in the control and treated wines were comparable across the sample set.

Further analysis of tartrate stability has already been carried out on a proportion of these wines six-months post-KPA treatment, with the remainder due for testing in April 2020. To date, all wines treated with KPA continue to exhibit tartrate stability, with no discernible tartrate crystals formed during the cold hold tests and relatively low conductivity changes indicating ongoing tartrate stability.

Summary

Potassium polyaspartate (KPA) appears to be an effective inhibitor of tartrate crystallisation and has been shown to be effective at achieving tartrate stability in a range of white, rosé and red wines over a six-month period. It is recommended that future studies include a formal sensory evaluation of untreated and KPA-treated wines, to evaluate any effects on aroma and flavour caused by the treatment.

Acknowledgement

The AWRI acknowledges the support of Enartis for the provision of the KPA product (Zenith Uno) used in the performance trials. The AWRI's communications are supported by Australia's grapegrowers and winemakers through their investment body Wine Australia, with matching funds from the Australian Government. The AWRI is a member of the Wine Innovation Cluster in Adelaide.

Disclaimer

This article should not be interpreted as an endorsement of any product.

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Table 1. Results from conductivity testing of control and KPA-treated wines in the trial after three months

Sample Name	Variety	Region	Control		KPA-treated	
			Conductivity (ΔμS/cm)	Rating	Conductivity (ΔμS/cm)	Rating
White_Wine_01	Riesling	Riverland, SA	189	Unstable	40	Stable/at risk
White_Wine_02	Chardonnay	Sonoma, California, USA	232	Unstable	41	At risk
White_Wine_03	Chardonnay	Riverina, NSW	187	Unstable	39	Stable
White_Wine_04	Chardonnay	Murray Darling, Vic	239	Unstable	45	At risk
White_Wine_05	Sauvignon Blanc	Riverina, NSW	141	Unstable	25	Stable/very stable
White_Wine_06	Pinot Grigio	Barossa, SA	109	Unstable	23	Very stable
White_Wine_07	Semillon	Riverina, NSW	255	Unstable	126	At risk
White_Wine_08	Chardonnay	Riverina, NSW	241	Unstable	53	At risk
Rosé_Wine_01	Pinot Noir, Grenache, Mataro	Barossa, SA	124	Unstable	21	Very stable
Rosé_Wine_02	Cabernet Sauvignon	Riverland, SA	155	Unstable	31	Stable
Red_Wine_01	Shiraz	Riverland, SA	45	At risk	31	Stable
Red_Wine_02	Cabernet Sauvignon	Riverland, SA	56	At risk	30	Stable
Red_Wine_03	Merlot	Riverina, NSW	36	Stable	22	Very stable