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Commercial Winemaking Production Series

Fining with Bentonite

Clay is one way to remove proteins that could make wine hazy

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The grape berry contains a large variety of nitrogen compounds, mainly amino acids, peptides (short amino acid chains) and proteins (long amino acid chains). They serve various biological functions within the grape such as enzymes, cell wall components, etc. The nitrogen content of grapes varies greatly by variety, rootstock, vintage, climate, pruning and crop levels, fertilization practices, etc.

Winery lab bench tests

Amino acids are soluble, and wine yeast can use them to grow and ferment the grape's sugars into alcohol. Amino acids, together with ammonium ions, are referred to as yeast-available nitrogen (YAN). Peptides and proteins are not considered YAN because they cannot be metabolized by yeast. Their solubility decreases with the wine's alcohol content. This may lead to precipitation of agglomerated proteins in the form of a visible amorphous haze. This effect is accelerated or triggered by exposure to elevated temperatures, e.g., when a customer buys a bottle of wine in the tasting room and leaves it in the car over the weekend. Protein hazes cannot be tasted; they are a purely aesthetic, visual problem in wine. However, while it is a natural effect, most consumers prefer a wine free from unappetizing-looking protein instabilities.

The winemaker's options to prevent protein instabilities

- Bentonite clay in different forms can irreversibly adsorb various sizes of proteins and has been the protein-fining agent of choice. It takes about six times the quantity of clay to take out the relevant amounts of protein. Since the protein content of wine ranges from around 10 to 300 mg/L, bentonite additions range from 60 to 1,800 mg/L.
- Heat exposure, such as a high temperature–short time (HTST) treatment, can denature proteins in unfermented juices and limit the need for additional fining of the wine. An HTST treatment, similar to a milk

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pasteurization at 80°C (176°F) for 5 seconds, generally does not affect the quality of the fermented wine. Such a treatment is also advisable for juices from grapes with heavy *Botrytis* infections, because the polyphenol oxidase (PPO) laccase — a browning enzyme — in the fungus does not respond to traditional sulfur dioxide treatments at the crusher the way grape PPOs do.

- Tannins derived from chestnut galls that contribute no color and low astringency have the potential to precipitate grape proteins as well as *Botrytis* laccase.
- Mannoproteins from yeast cell walls may act as protective colloids, bind to grape protein and prevent their flocculation.
- Regeneratable cation exchange resins that reversibly bind proteins from wine and do not create solid waste have been used only experimentally.

Potential problems with bentonite fining

Excess amounts of bentonite added to wine can bind not only proteins but also desirable aroma compounds or colloidal materials. Proper bench testing to determine the minimal effective amount to add is important, as that will not have a detrimental effect on the wine. Each individual wine has separate dosage requirements. Routine additions of bentonite will certainly lead to over- or under-dosing, as the requirements may vary by more than an order of magnitude (60 to 1,800 mg/L = 0.5 to 15 lb/1,000 gal). In addition, oxidative damage to the wine may occur if the mixing in of the bentonite slurry allows for air exposure during transfer operations, from the tank headspace, or via subsequent filtration steps.

The heat tests

The quickest (and laziest) test for protein stability after fining treatment is a heat test that exposes a treated and filtered wine sample to a high temperature for a short period of time, e.g. 49°C (120°F) for 48 hours or 90°C (194°F) for one hour, followed by a period of cooling. Such tests are trying to simulate the precipitation of proteins at a proper, cool storage temperature over the lifespan of the wine. It is a rather uninformed assumption that these two scenarios are identical in their outcome. Moreover, the resulting over-stabilization of commercial wines against excessive heat exposure has significant consequences for the sensory quality of the wine. If the wine experiences extreme temperatures during shipping and storage, its aroma will be damaged but there may be no visible indicators that the wine was treated poorly. Temperature data logging and tracking of shipments can help identify sources of heat exposure within the distribution chain.

A more representative way to assess the effect of fining treatments on the stability of a wine under normal storage and aging conditions is the Boulton ethanol assay that measures the stability of all colloidal materials. It can assess the effect of a bentonite fining treatment on a wine though a titration with successive quantities of ethanol while using a nephelometric turbidity meter to quantify the resulting cloudiness.

Bentonite fining of juice before fermentation may lead to a sluggish fermentation due to its clarification effect on the treated juice and the possibly stripping of certain growth factors, such as fatty acids, phospholipids, and sterols. An extended fermentation can lead to increased amounts of residual fructose in the wine. Since fructose is twice as sweet as glucose, this may affect the perception of the wine's dryness.

Rosé/blush wines

When treating blush/rosé wines, make sure that the bench trials are also evaluated for any loss or change of color associated with the bentonite treatment.

Storage

Proper storage of bentonite — much like filter pads — in a clean, dry environment and in a resealable container is crucial. Bentonite will absorb odors from the air, e.g. the cork taint component TCA, and release them into the treated wine.

Waste issues

Bentonite creates a solid waste problem if separated with the lees. If flushed out of the tank, it can clog drains and sewer lines. In the winery/irrigation pond, it will settle and enhance the seal of the pond bottom. Over the years, however, sludge will gradually accumulate and make the pond more shallow, thereby enhancing light penetration and algae growth.

Bentonite and barrel leaks

A small amount of bentonite can be directly added to any wine and used to seal small leaks in a barrel that was previously dry-stored or has a more leakage-prone, e.g. fortified, wine in it.

Questions about bentonite application

Should I use sodium or calcium bentonite?

It doesn't really matter, as long as the enologist performs an ethanol titration or one of the arbitrary heat stability bench tests on each wine and determines the smallest effective dose to satisfy the test.

Bentonite clay is the most widely used fining agent against heat-instable grape proteins in white wines. In red wines from *Vitis vinifera*, the inherent tannins usually denature these proteins enough to cause precipitation during aging. However, anthocyanin-rich but tannin-deficient red European-American varieties, as well as blush/rosé wines and red viniferas from very cool climates, should be tested for protein-stability.

There are two different forms of bentonites commercially available: sodium-rich ones and calcium-rich ones. Suppliers of sodium bentonites argue that this form has a protein fining capacity twice as high as its calcium cousin. Suppliers of calcium bentonites argue that their form swells less in water, and it creates fewer lees and a smaller loss of wine when racking.

Excess additions of sodium to wine are undesirable, as sodium consumption may contribute to high blood pressure and heart disease. For the same reason, the use of sodium metabisulfite for sulfur dioxide additions or sodium bicarbonate (baking soda) for deacidification purposes is not permitted by the U.S. Tax and Trade Bureau (CFR Title 27 Part 24 § 24.246 *Materials authorized for the treatment of wine and juice*). The quantities added to wine by a heavy bentonite treatment can double the amount of sodium naturally present in grape juice (10 to 20 mg/L), but even then wine is still considered a "very low sodium" beverage.

On the other hand, excess release of calcium into a wine from bentonite via exchange with grape proteins may increase the risk of calcium tartrate instability. For example, an addition of 1,920 mg/L (16 lbs/1,000 gal) calcium bentonite — equivalent to 960 mg/L (8 lbs/1,000 gal) sodium bentonite — to a batch of protein-rich Gewürztraminer would result in an additional potential for 114 mg/L calcium tartrate. Since calcium tartrate does not respond as readily to cold stabilization as potassium bitatrate, this may mean the difference between a stable wine and a wine throwing a glass-like precipitate that may worry consumers.

Should I rehydrate my bentonite in water or in wine?

Water. Bentonite, independent of type, should be rehydrated with clean, chlorine-free hot (140°F, 60°C) water. It must be added under immediate, vigorous mixing to the water (not the other way around) and allowed to swell for at least four hours. The lump-free slurry shouldn't sit longer than overnight, as this may encourage microbial growth. A maximum of 16.7 L of water may be used to dissolve each kilogram of bentonite (2 gallons of water per pound). Note that the total amount of water introduced from all processing sources during the winemaking should not exceed 1 percent of the wine. For bench trials in the winery lab, a mixing ratio of water to bentonite of 16 to 1 (60 g per 1 L) results in an easily pipettable 6 percent w/v slurry (Table 1).

Table 1: Bentonite (6%) slurry additions for bench trials

g/hL	lbs/1,000 gal	mL per liter wine	mL per 750mL wine	mL per gal wine
12	1	2	1.5	8
24	2	4	3.0	15
36	3	6	4.5	23
48	4	8	6.0	30
60	5	10	7.5	38
72	6	12	9.0	45
84	7	14	10.5	53
96	8	16	12.0	60
108	9	18	13.5	68
120	10	20	15.0	76

Rehydrating with wine doesn't allow the bentonite to fully swell, thereby reducing its fining capacity. In addition, it is a waste of wine that cannot be recovered.

However, if one would use 6 percent slurry at additions above 60 g/hl (5 lbs/1,000 gal), the amount of water added to the wine exceeds 1 percent. Thus in practice, bentonite is typically dissolved at ratios of about 8 to 1 (1 kg bentonite per 8 L of water; 1 lb/gal) which allows for bentonite additions of up to 120 g/hl

(10 lb/1,000 gal). Above this addition level, a wine/water mix can be used for rehydration to keep the processing water addition below 1 percent of total wine volume. Alternatively, rehydrating with too little water will limit the amount of swelling and makes for a difficult-to-stir, lumpy slurry.

Does a change in pH due to acidification/deacidification or cold stabilization change the protein stability of my wine?

Yes. Even small changes in pH can significantly alter the protein solubility and thus the wine's bentonite requirements. Protein stability must be reassessed after any treatment that changes the acidity of the wine in question. Especially high pH values likely will lead to increased bentonite demand. Calcium bentonites are not recommended for wines with a pH above 3.4.

Can blending two protein-stable wines compromise the stability of the blend?

Yes. Any shift in alcohol content, pH, protective colloid concentration, etc. can potentially render the entire blend unstable. A new test of the mixture must be conducted, and additional fining might be needed.

Can nongrape proteins from fining agents, enzymes or lysozyme treatments, or sur lie aging influence the protein stability of my wine?

- Gelatin and other proteins that are used as fining agents against over-extracted seed or skin tannins may contribute to protein instabilities and are often used in conjunction with other fining agents — such a silica gel (Kieselsol) — that can precipitate any excess fining proteins that didn't bind to the wine's tannins.
- Additions of processing enzymes such as pectinases or glucosidases are usually not relevant sources for protein instabilities. However, they respond well to bentonite fining, which allows the winemaker, e.g., to stop the activity of β-glucosidases. This limits the premature release of sugar-bound varietal aroma precursors such as monoterpenes and a loss of aging potential for the wine.
- Lysozyme additions to inhibit malolactic fermentation can add substantial amounts of nongrape-derived protein to the wine when added at a recommended 250 to 500 mg/L, which is between 2 and 50 times the average concentration of natural grape protein in wine. Note that lysozyme — while potentially unstable — does not respond well to the heat tests.
- Mannoproteins on the other hand, released from yeast cells during aging on the lees or added as commercially available adjuncts, may act as protective colloids and keep unstable grape protein from precipitating.

How fast/long should I mix?

The reaction between protein and bentonite is quick but not instantaneous. Proper mixing is crucial, and it has been shown that mixing speed, time, and temperature affect the efficacy of the treatment. At least 10 to 15 minutes of vigorous mixing is recommended, and the wine temperature should be above 50°F (10°C). To increase the effectiveness of the bentonite fining, the winemaker may choose to do it at a warmer temperature, and then proceed with a cold stabilization against tartrate precipitation thereafter. In this scenario, the dropout of potassium bitartrate may affect the pH of the wine, and the bench test for protein stability should address this by simulating the chilling beforehand.

It is important that any bench trial conditions, especially the mixing speed and the temperature, are representative of the conditions that can be achieved on a large scale in the cellar. Otherwise, an underestimation of the bentonite requirements will result.

Mixing with an inert gas fed in via the racking valve avoids potential oxidative damage due to mechanical mixing if the tank headspace contains traces of air/oxygen.

How long does it take to settle?

Allow one week (depending on tank height) to have all bentonite lees settle to the bottom by gravity alone. Limiting the contact time between wine and bentonite helps to minimize the amount of lead residues that could be extracted into the wine.

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