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“Ready to Drink Yet? The Chemistry of How Wine Flavor Changes During Aging”

Gavin Sacks
Associate Professor of Enology, Department of Food Science, Cornell University

Beth Burzynski
PhD Candidate in Food Science, Cornell University

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“Ready to Drink Yet?”
The Chemistry of How Wine Flavor Changes During Aging

Gavin L. Sacks
Associate Professor of Enology
Department of Food Science
Cornell University

Moderator: Beth Burzynski, PhD Candidate in Food Science, Cornell University
Let’s get started . . . consider the range of wine labels available to US consumers

This is a wine label

Wine labels can include info on
• Producer/brand
• Production region (e.g. Bordeaux)
• Grape variety (e.g. Chardonnay)
• Vintage year
• Alcohol content, “contain sulfites”, government warnings

In the US, wine labels must be pre-approved prior to bottling or importation by the Tax and Trade Bureau (TTB)

Audience Challenge Question
ANSWER THE QUESTION ON BLUE SCREEN IN ONE MOMENT

How many different wine labels are approved for sale each year in the United States?

• 2,000 to 4,000
• 6,000 to 10,000
• 15,000 to 30,000
• 40,000 to 60,000
• 75,000 to 125,000
Premium wine is a “craft” product, not a “commodity”. Variation in sensory (and thus chemistry) is expected.

**Taste** (Gustation)
- salty, bitter, sweet, acidic, and umami compounds

**Smell** (Olfaction)
- Volatile compounds, of which 50-75 create most wine-like aromas

**Touch** (Chemesthesia)
- Compounds responsible for pungency, cooling, astringent, viscous and related tactile sensations

**Appearance**
- Pigments (mostly red and yellow) or light-scattering haze particles

**Popular appeal of wine - Explanations**
- Its variation
- Its deliciousness, particularly with food
- Its sense of place
- It contains ethanol
- Its mystique, including its longevity

The reputation for “age-ability” persists even though the majority of wines are meant to be, and are, consumed within 0.5-2 years of production.

*(the occasional bottle of 170-year old shipwrecked Champagne excepted)*
“Age-worthy” does not have a well agreed upon definition among enologists

**Working definition:** “assuming two wines (A and B) are equally liked at some time point, Wine A ‘ages better’ if it is better liked than Wine B at a later time point.”

Maybe, youthful characteristics are preserved in Wine A
- Desirable sensory compounds could be more stable
- Fewer undesirable compounds could form

Or, new “aged” characteristics appear in Wine A
- Undesirable sensory compounds could be lost more rapidly during storage
- More desirable compounds could form

This Talk: We will consider case studies of wines that vary in how they age

But, first let’s talk about some basic wine chemistry
Chemically, what’s in a typical dry red wine?

Everything else, a non-comprehensive list
- Glycerol (6-12 g/L)
- Organic acids (5-8 g/L) → wine pH is 3-4
- Hexose sugars (0.5-3 g/L)
- Minerals, e.g. K⁺ (0.5-2 g/L)
- Important odorants (ng/L to mg/L)
- Phenolics (0.5-2 g/L)
  - Anthocyanins [color]
  - Tannins [astringency]
- Sulfur dioxide (20-40 mg/L)
- Glutathione (15-100 mg/L)

Water (86%)  Ethanol (11%)  Everything else (3%)

Adapted from Understanding Wine Chemistry; Waterhouse, Sacks, and Jeffery. 2016

What reactions can happen at pH 3-4, 10-12% EtOH, reducing conditions, 20 °C, over 6-24 months?

Reactions involving water or ethanol, often acid catalyzed
- Hydrolysis, ethanolysis (below)
- H₂O/EtOH addition or elimination

Reactions between wine nucleophiles and electrophiles
- Example nucleophiles = SO₂/HSO₃⁻, H₂S, thiols, polyphenols
- Example electrophiles = carbonyls (e.g. acetaldehyde); tannin hydrolysis products; quinones

Other textbook reactions, e.g. Grignard? Not likely 😊
Case Study 1: Varietal wines “not aging well” due to loss of desirable compounds

Muscat-type wines vs. Gewürztraminer

Both types of wines have floral aromas when young, but they have different reputations for aging

Consider two monoterpene-rich, “floral” smelling grape varieties, with dissimilar fates during aging

Muscat-type grapes, e.g. Moscato bianco from Asti

“Serving recommendations [for Moscato d’Asti] . . . drink it young and fresh!” - winefolly.com

Key odorant = linalool (“lily-of-the-valley”)

Gewürztraminer

“While you often can drink Gewürztraminers young, some benefit from 2-4 years worth of aging.” - wineintro.com

Key odorant = (-) cis-rose oxide “rose/litchee”
The key “floral” monoterpenes in Muscat-type wines, e.g. linalool, are unstable in wine matrix.

Linalyl glycosides
Non-odorous precursors

Partial release during fermentation
Slow non-enzymatic release during storage

$\frac{t_\frac{1}{2}}{\text{months}}$ @ wine pH = months

Linalool
“lily-of-the-valley”
Threshold = 50 ppb

$\alpha$-Terpeniol
“resin, pine”
Threshold = 400 ppb

Acid catalyzed ring closure
$\frac{t_\frac{1}{2}}{\text{weeks}}$ @ wine pH = weeks

By comparison, the key Gewürztraminer monoterpene is stable or increases during storage.

Monoterpene diol precursors
Odor threshold = very high

Enzymatic reduction
Acid-catalyzed ring closure

$(-)$ cis-rose oxide
“rose/litchi”
Threshold = 0.5 ppb

Stable

Belitz, Grosch, Schieberle 2004
Recap: Wines with strong floral aromas often do not “age well” if you desire the odor to stay floral

- Monoterpenes, particularly linalool, contribute “floral” aromas, but are not stable in aqueous acidic matrices (WINE!)
  - In particular, Muscat-type varieties (e.g. Muscat bianco)
  - Stonefruit notes of some wines (e.g. Viognier) do not persist
- A monoterpane exception: cis-rose oxide
  - Gewürztraminer can continue to have “litchee” aromas, even with age

Case Study 2: Production practices lead to desirable odorants that are readily lost during storage

**Beaujolais Nouveau vs. Standard red wines**

*Beaujolais Nouveau is known for intense “cherry, banana” type aromas and is intended for sale within weeks of fermentation*
**Red winemaking: The 30 second story**

**Standard Red Wine Maceration**
*Grapes are crushed and fermented by yeast in the presence on skins and seeds*

**Carbonic Maceration** (common for Beaujolais Nouveau)
*Whole grape clusters are blanketed in CO$_2$, grapes auto-ferment to ~2% alcohol. Then, grapes are pressed, and juice is fermented to dryness by yeast*


**Several differences in odorant concentrations between carbonic (CM) and standard maceration, including...**

- **Isoamyl acetate**
  - “Cherry/banana”
  - Higher in CM wines

- **Isovaleric acid**
  - “Cheesy, sweaty”
  - Lower in CM wines
    (although, below sensory threshold in most wines)

Data from Antalick, et al. AJEV 2014.
**Ester hydrolysis / Esterification in Wine**

Just like in your Organic Chem I Class

\[
R'\text{O}_{\text{esters}} + OH^+ \rightarrow R'O + H_2O
\]

\[
K_{eq} = \frac{[\text{alcohol}][\text{fatty acid}]}{[\text{ester}][\text{H}_2\text{O}]} \approx 0.25
\]

Working through the math... and assuming [EtOH] = ~2M or 12% v/v, acetic acid = ~5 mM, [H_2O] = ~50M

- Isoamyl acetate and other acetate esters decrease during storage
- Ethyl isovalerate and some other ethyl esters can increase during storage
Organic Chem I, cont., kinetics

Ester hydrolysis/esterification follow pseudo-first order kinetics in wines

At pH 3.6, \( T=25^\circ C \),

- \( t_{1/2} \) for hydrolysis of an ester is 3-5 months
- \( t_{1/2} \) for esterification of a fatty acid is 1-2 yrs

How to slow down ester reactions?
- Raise pH (causes other problems)
- Store cold, especially during transit
- Drink quickly!
  - Beaujolais on Thanksgiving

Recap: wines which rely heavily on acetate esters will likely “not age well”

Examples of wines with high acetate esters
- Beaujolais Nouveau and other carbonic wines
- ’Tropical fruity’ whites, e.g. many Pinot Grigio, Sauvignon blanc, and stainless steel Chardonnay

Standard red winemaking
- more branched chain fatty acids
- more branched chain ethyl ester formation during storage

From Antalick, et al AJEV 2014
An aside on additional hydrolytic reactions, before returning to case studies...

“Is it good aging? Or bad aging”?

Dimethyl sulfide (DMS) and 1,1,6-trimethyldihydronaphthalene (TDN)

DMS, TDN . . . do we want these forming in our wines?

Hypothetical responses of two consumers (#1, #2) to spiked wine

“only the dose makes a thing not a poison”
- Paracelsus

Dimethyl sulfide (DMS)
“canned corn, truffle, asparagus”
Segurel, et al JAFEC 2004

S-methylmethionine (SMM)
Higher in riper grapes

Glycosylated C<sub>13</sub>-norisoprenoids
Higher in Riesling, sun exposed grapes

1,1,6-trimethyldihydronaphthalene (TDN)
“kerosene”
Can we accelerate these aging reactions through some sort of physiochemical treatment?

OK, we have reactions that can occur during storage...

Which of these approaches to accelerated aging have been patented?

More than one correct answer may exist

- Agitation
- High-temperature
- Ionizing radiation
- Ultrasound
- Electric Currents

Image: Wikimedia Commons
“Rapid aging” approaches do not appear to duplicate conventional aging (say, in a 12 °C cellar)

Example 1: UV or gamma ray irradiation generates free radicals
→ volatile sulfur compound formation → “burnt hair” aromas

Example 2: High temperature storage promotes different reactions at different rates
→ Wine reactions differ in activation energies

Unfortunately, higher temperatures often accelerate unwanted reactions to a greater extent!

<table>
<thead>
<tr>
<th>Reaction in beverage model system</th>
<th>Activation energy, $E_a$ (kJ/mol)</th>
<th>Fold-increase in reaction rate compared to 12 °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acid hydrolysis of proanthocyanidins (tannins)</td>
<td>45</td>
<td>3</td>
</tr>
<tr>
<td>Esterification or ester hydrolysis</td>
<td>62</td>
<td>5</td>
</tr>
<tr>
<td>Hydrolysis of SMM to DMS</td>
<td>186</td>
<td>106</td>
</tr>
<tr>
<td>Acid hydrolysis of anthocyanin pigment</td>
<td>118</td>
<td>19</td>
</tr>
<tr>
<td>Formation of ethyl carbamate from urea and ethanol</td>
<td>118</td>
<td>19</td>
</tr>
</tbody>
</table>

- Possibly desirable
- Unlikely to be desirable
Case Studies 3 and 4, Oxidation:
Why do wine producers use SO₂?
And why are reds thought to age better than whites?

Audience Challenge Question
ANSWER THE QUESTION ON BLUE SCREEN IN ONE MOMENT

In the current model of wine oxidation, which wine component directly reacts with O₂?

- [Fe(III)] complexes
- SO₂ (in the form of bisulfite, HSO₃⁻)
- Polyphenolics, particularly condensed tannins
- Glutathione and related sulfhydryls
- Alcohols, particularly ethanol
Distribution of SO₂ species in wine

- At wine pH, most SO₂ exists as bisulfite.
- Smaller portion (<5%) exists as ‘molecular’ SO₂.
- Almost no sulfite, SO₃²⁻.
- Some bisulfite is covalently bound to wine electrophiles like acetaldehyde.

Role of sulfur dioxide (SO₂) in wine

**Anti-microbial:** Neutral, “molecular” form can cross the cell membrane.

- Inside cell, pH>5
- SO₂ → HSO₃⁻

**Anti-oxidant:** Bisulfite (HSO₃⁻) reacts with products of oxidation.

- In wine, pH<4
- SO₂ → HSO₃⁻

- Lower pH favors molecular form.

- Best predictor of aroma quality = low levels of off-odorants from spoilage organisms or oxidation (San Juan, et al; JAF 2012)

- Methional, “baked potato”
  - Via abiotic oxidation
  - Via microbial spoilage
Focus on abiotic oxidation during aging
How is $O_2$ getting into a bottled wine?

Same Semillon wine with one of 14 closures

Oxygen can . . .
- Be present in headspace during bottling
- Migrate through or around the closure

Adapted from P. Godden, et al, AJWGR 2001

Wine Oxidation – Production of $H_2O_2$

$[Fe(II)]$ $O_2$ $\rightleftharpoons$ $[Fe(III)-O_2^-]^{2+}$ $\rightarrow$ $2 [Fe(III)] + H_2O_2$

$[Fe(II)]$ $H_2O_2$ $\rightarrow$ $^\cdot OH$ $\rightarrow$ $^\cdot OH$

“Fenton reaction”

$R-OH$ $\rightarrow$ $\rightarrow$

Aldehydes, Ketones
Catechin

Browning

Typical wine: $\sim 1 mg/L Fe$

Oxidized aromas
(“baked potato, bruised apple, cardboard”)

From Understanding Wine Chemistry, 2016
Wine Oxidation – Formation of Quinones

Oxidation of diphenols to quinones by [Fe(III)] is not thermodynamically favorable in wine. Wine nucleophiles “trap” quinones and drive oxidation forward. By comparison, most distilled spirits are low in nucleophiles (and often transition metals and phenolics, too). Oxygen consumption is very slow.

Quinones can react with many nucleophiles, including desirable “fruity” smelling thiols.

3-SH is critical for the fruitiness of many wines, particularly Sauvignon blanc. Result: loss of quality, unless another nucleophile is present (enter SO₂).

Nikolantonaki and Waterhouse, JAFC 2012
The role of SO₂ as a wine antioxidant - Scavenge wine oxidation products

**Scavenge H₂O₂ and aldehydes**

\[
\text{H}_2\text{O}_2 + \text{HSO}_3^- \rightarrow \text{HSO}_4^- + \text{H}^+ \\
\text{RCHO} + \text{HSO}_3^- \rightarrow \text{RCHSO}_3^- + \text{H}_2\text{O}
\]

**Scavenge quinones**

Once HSO₃⁻ is mostly depleted (< 10 mg/L), oxidation products accumulate.

---

So why would red wines potentially age better than white wines?

- In part, red wines are less dependent on thiols as key aroma compounds.
- Also, polyphenols (anthocyanins, tannins) can react with oxidation products, e.g., malodorous carbonyls, or quinones.

Ex:

**Malvidin-3-glucoside** (anthocyanin) + Acetaldehyde → **Vitisin B**
Recap: Antioxidants like SO$_2$ and/or polyphenols may help a wine age better, but not because they directly react with O$_2$

- Oxidation of wine is catalyzed by the presence of transition metals like Fe(II)

- Oxidation generates H$_2$O$_2$ and polyphenol quinones.
  - These species can result in loss of desired aroma compounds, production of oxidized aroma compounds (mostly carbonyls), and browning
  - SO$_2$ (as bisulfite) can react directly with H$_2$O$_2$, quinones, and carbonyls
  - Polyphenols can react with quinones and carbonyls

- Counterintuitively, the presence of SO$_2$ and polyphenols increases the rate at which O$_2$ is consumed

There’s much left to learn about wine aging...

Some recent exciting work from the literature
**Ongoing Studies:** What happens to condensed tannins (chemically, sensorially) during aging?

- **Condensed tannins, aka proanthocyanidins**
  - Polymers of flavan-3-ols, e.g. catechin
  - Present in grape skins and seeds – at higher concentrations in red wines

- **Responsible for “astringency” (drying, puckering sensation) in wines**
  - Mechanism = non-specific covalent binding of proteins, including salivary proteins

During storage, tannins can undergo both acid-catalyzed and electrophile/nucleophile reactions:

- **How do these reactions affect tannin sensory attributes?**
  - Decrease in astringency, most likely?
  - Other changes, i.e. decrease in in-mouth persistence (“stickiness”)?

---

Ongoing Studies: What else forms during storage and contributes to the “bouquet” of aged wines?

- From before: certain ethyl esters, TDN, DMS
- Recent papers using GC-O/MS have suggested additional candidates

Ongoing Studies: Why do wines differ in relative amounts of key malodorous compounds following oxidation?

Methional (“baked potato”) and phenylacetaldehyde (“honey”) have been implicated as key wine oxidation odorants

- Low sensory thresholds
- Relatively weak SO₂ binders as compared to other aldehydes

Wines can differ by over an order of magnitude in the amount of these aldehydes formed during oxidation and HSO₃⁻ loss

Does high bound methional explain why some wines “age badly”?
Wrapping Up: Take-Home Messages

• “Aging well” is a not single concept
  o New desirable compounds could be formed; or else lost more slowly
  o Or, new undesirable compounds could be avoided; or else lost more quickly
  o And, what’s desirable to one consumer may not be to another

• A limited number of types of reactions can occur in aging. Key classes
  o Solvent-mediated, such as ester hydrolysis/esterification
  o Reactions between nucleophiles and electrophiles, e.g. addition of bisulfite to oxidation products

• There’s much to learn still: Tannin changes, odorants responsible for aged wine aroma, differences in observed oxidation products among wines

Still Thirsty?: If you like this talk, and want to learn more, consider the textbook

*Understanding Wine Chemistry*

Andrew L. Waterhouse, Gavin L. Sacks, and David W. Jeffery

Published in 2016 by John Wiley & Sons
References


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