BOTTLING LAFFORT

PREPARING WINE FOR BOTTLING

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LAFFORT
l'œnologie par nature

PREPARING WINE FOR BOTTLING

- *Introduction*
- *Clarification and Fining*
- *Stabilization*
    - *Microbial Stabilization*
    - *Protein Stabilization*
    - *Color Stabilization*
    - *Tartrate Stabilization*
- *Filtration and Filterability*
- *Bottling Timelines*

LAFFORT

l'œnologie par nature

Introduction

Clarity and Stability

*"Clarity is an **essential quality** required by consumers, **especially for white wines**.*

*Nowadays, the only normally **acceptable deposit** is **red coloring matter** in old wines. Sediment should not appear until the wine is four or five years old, and then only in small quantities, and easy to eliminate by decanting.*

*Wine must **not only be clear** at the time of bottling, but also **maintain its clarity during aging and storage** for an indefinite period, whatever the temperature conditions."*

Pascal Ribéreau-Gayon, Handbook of Oenology.

MOUNT & PAUL
WINE INDUSTRY SOLUTIONS

Key consumers insights

Consumer studies:

- CHINA: 1027 wine consumers of middle-high class,
66% men,
43% 30-39 years old,
80% from Shanghai/Beijing
- USA: 2053 people regularly consuming wine,
56% women,
46% 35-54 years old

In China, **50%** of the consumers had previously ***seen wines with sediments***; **33%** in USA.

In the US and in China, almost **50%** of the consumers surveyed view any ***sediment in bottle in a negative way***.

Although **30%** of the consumers surveyed ***understand*** that sediment in red wine is a consequence of the winemaking process, ***40% would not buy a wine with sediment.***

In both of the consumer groups surveyed,
the presence of tartrate crystals / sediment was perceived in a negative way.

Only **16%** of American and **32%** of Chinese consumers
would buy wine with sediment, including red wine.

INTRODUCTION

Market status on wine stability

Analysis of wines from super market shelves

Study carried out in France:

	In 2010 with 63 bottles (41 red, 12 white & 10 rosé)	In 2016 with 80 bottles (43 red, 32 white & 5 rosé)
Microbiological Stabilization *(Non-compliant: pop > 1 CFU/10 mL)*	36%	47%
Protein stabilization *(Heat test - Δ NTU < 2)*	17%	13%
Tartaric stabilization *(ISTC50 ≤ 3µS or crystallization test)*	66%	59%
Coloring matter stabilization *(Cold test - Δ NTU < 10)*	x	72%

Microbiological stabilization detail, 2010:

AB	LB	*Brett*
52 %	15 %	9 %

Microbiological stabilization detail, 2016:

AB	LB	*Brett*
46 %	11 %	28 %

What do we prepare wines for?

To reach clarity and stability

Before bottling, the aim is to:

- **Obtain total clarity** by appropriate clarification methods.
- **Achieve stability of that clarity** by means of efficient treatments.

  We must understand the consequences of each treatment.

Examples:

- ***Filtration clarifies, but does not stabilize, except from a microbiological standpoint.***
- ***Fining has a double effect: clarifying and stabilizing.***
- ***Certain colloid additions improve and prolong the stability equilibrium but do not clarify the wine.***

Clarification

- Elimination of a current haze
- Short term

↓

~~Existing haze~~

Stabilization

- Colloidal particle precipitation avoiding potential future precipitation
- Long term
- Clarity preservation

↓

~~Existing haze~~
~~Potential haze~~

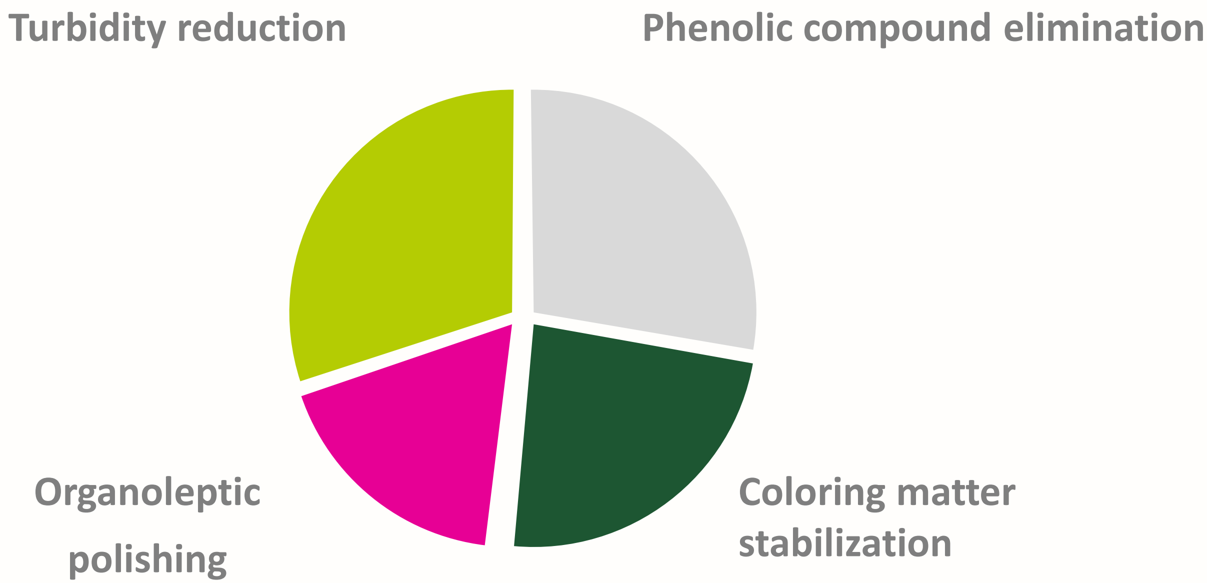
CLARIFICATION & FINING PRINCIPLES

The principle of clarification and fining

“Clarification aims to eliminate wine haze, consisting of visible and/or light absorbing or deflecting particles. These are particulate suspensions of yeasts, bacteria, crystals, vegetal debris visible microscopically or to the eye, but also colloidal solutions.“

Knowing and Making Wine, Jacques Blouin and Emile Peynaud, 2005

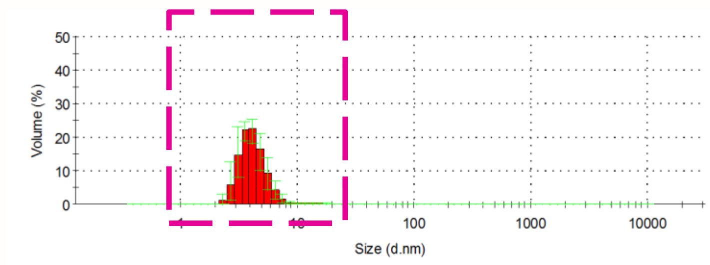
What Does Fining Achieve



Particle size of protein fining agent

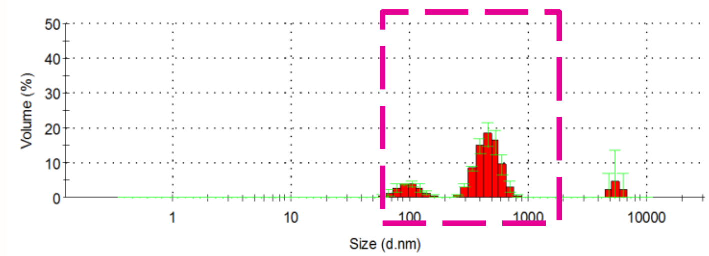
Liquid Gelatin

0.1-1 µm



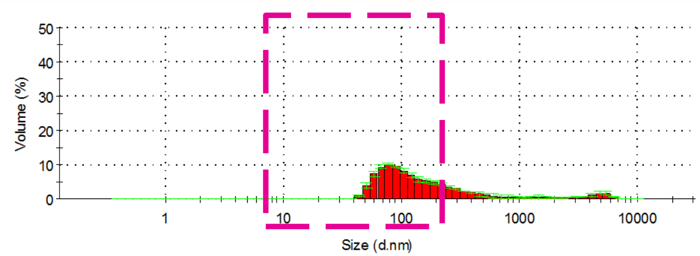
Vegecoll®

< 10 nm



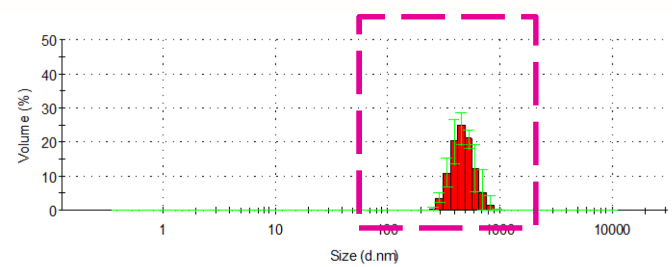
Albumin

< 10 nm



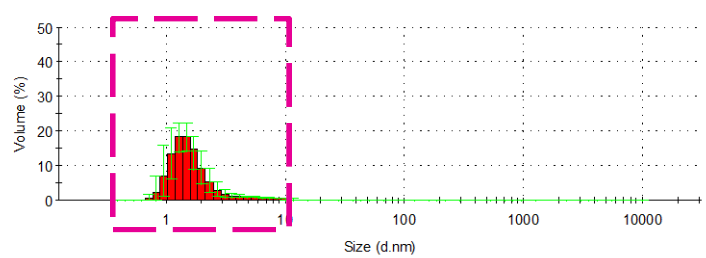
Pea

< 10 nm



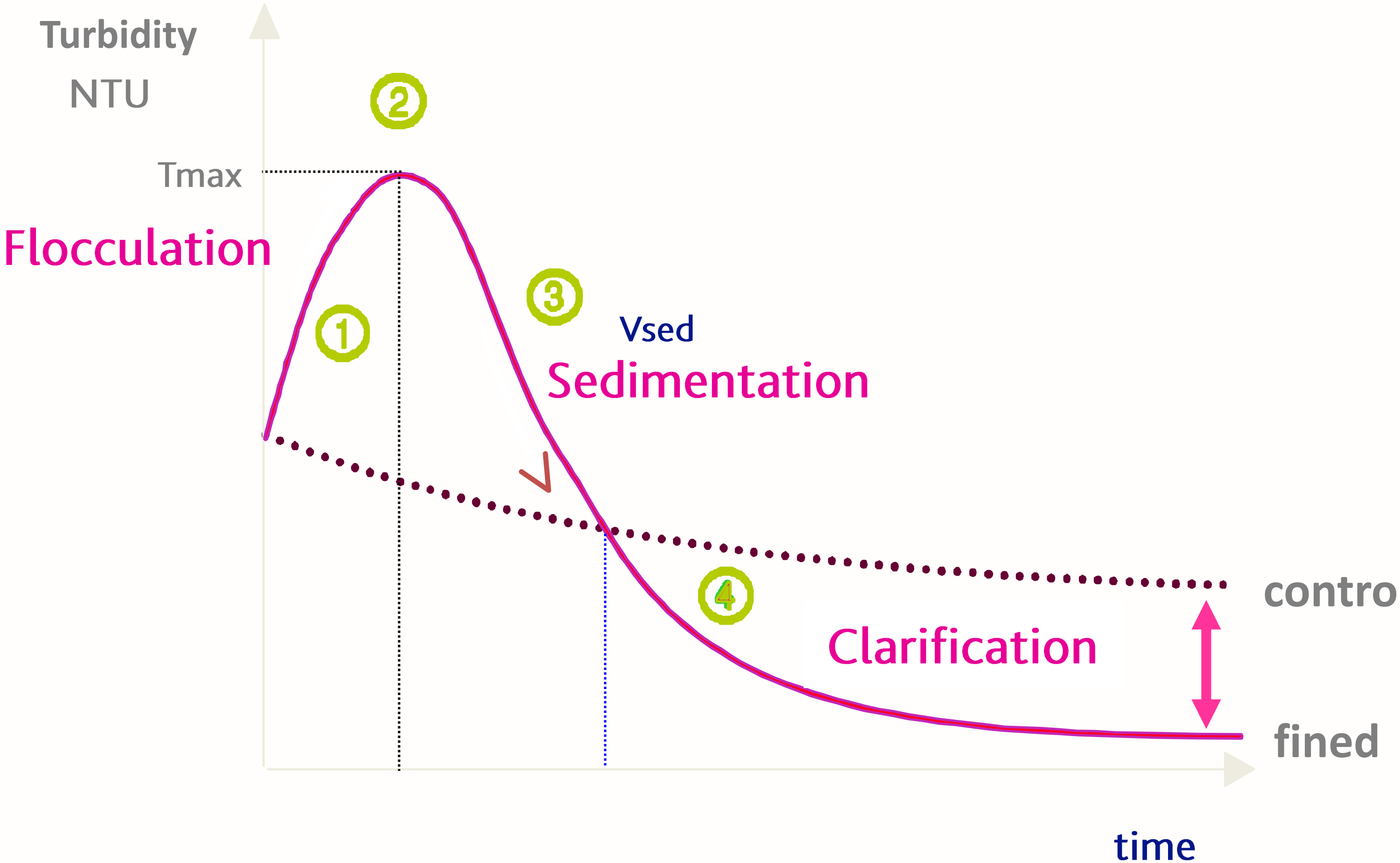
Yeast extract

0.1-1 µm



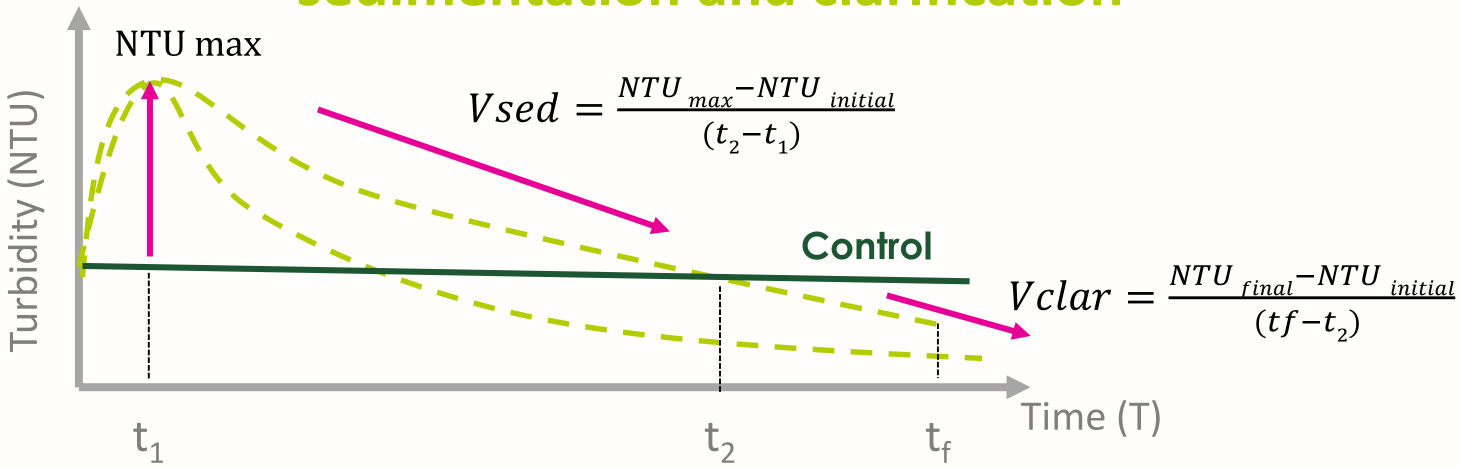
CLARIFICATION

Wine turbidity evolution during fining



It is the compromise between flocculation capacity and sedimentation speed that optimises the clarification effectiveness

Turbidity reduction – sedimentation and clarification

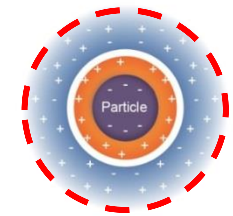


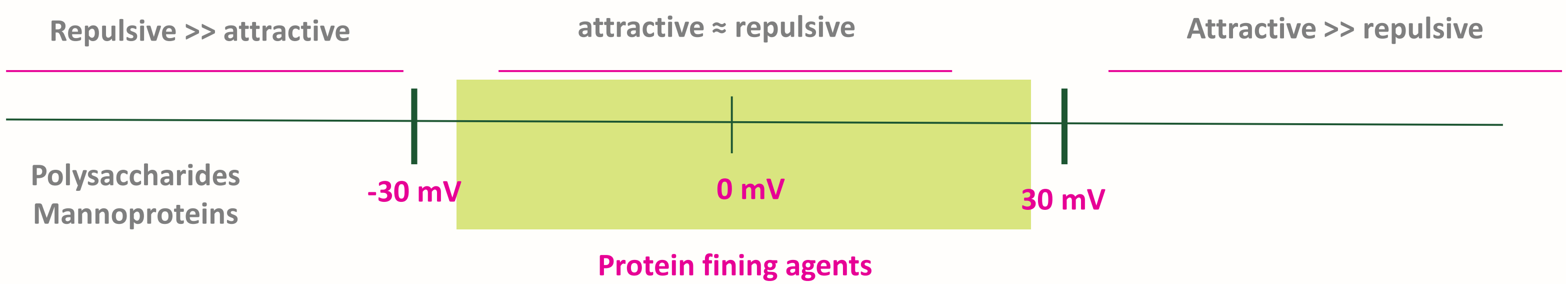
- The **flocculation capacity** depends on the nature and the dose of the fining agent and of the wine
- A high flocculation is not associated with a higher clarification speed

- The ***sedimentation speed*** and the ***clarification speed*** depend on the size and the weight of the flake

It is the compromise between flocculation capacity and sedimentation speed that optimizes the clarification effectiveness

Zêta Potential
Indicates the balance of attractive and repulsive forces of a particle in a medium



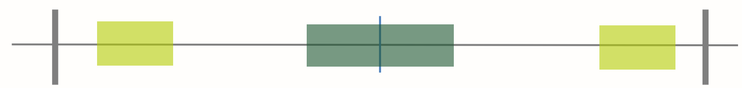


- When none of these forces is predominant, particles flocculate.
- This number helps in the prediction of fining agent reactions, because it indicates the flake formation type.

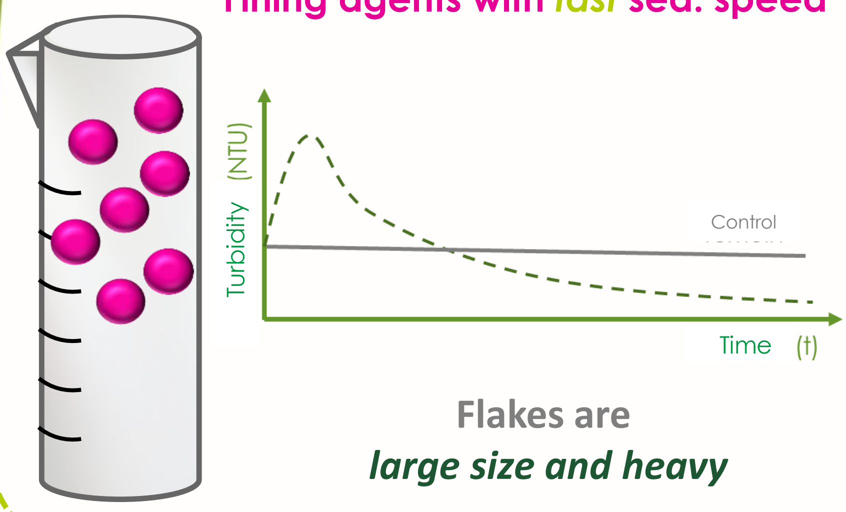
The fining phenomenon

Zêta Potential

Protein fining agents

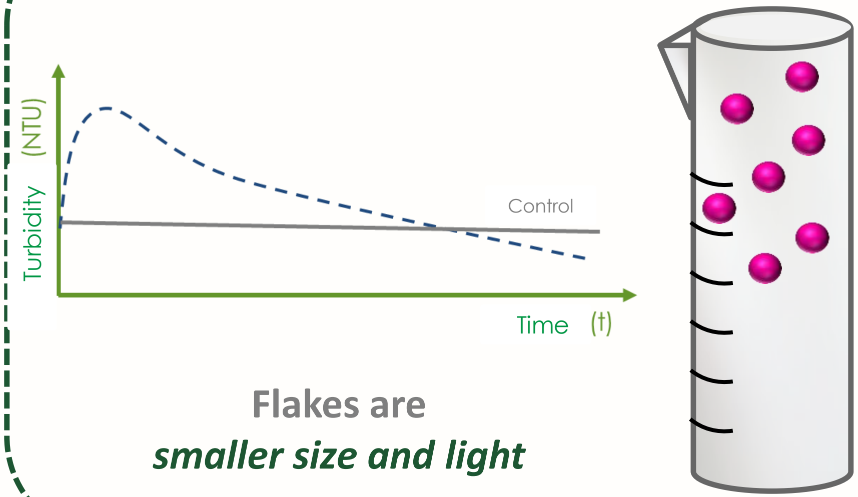


Fining agents with *fast* sed. speed



Flakes are
large size and heavy

Fining agents with *slow* sed. speed



Flakes are
smaller size and light

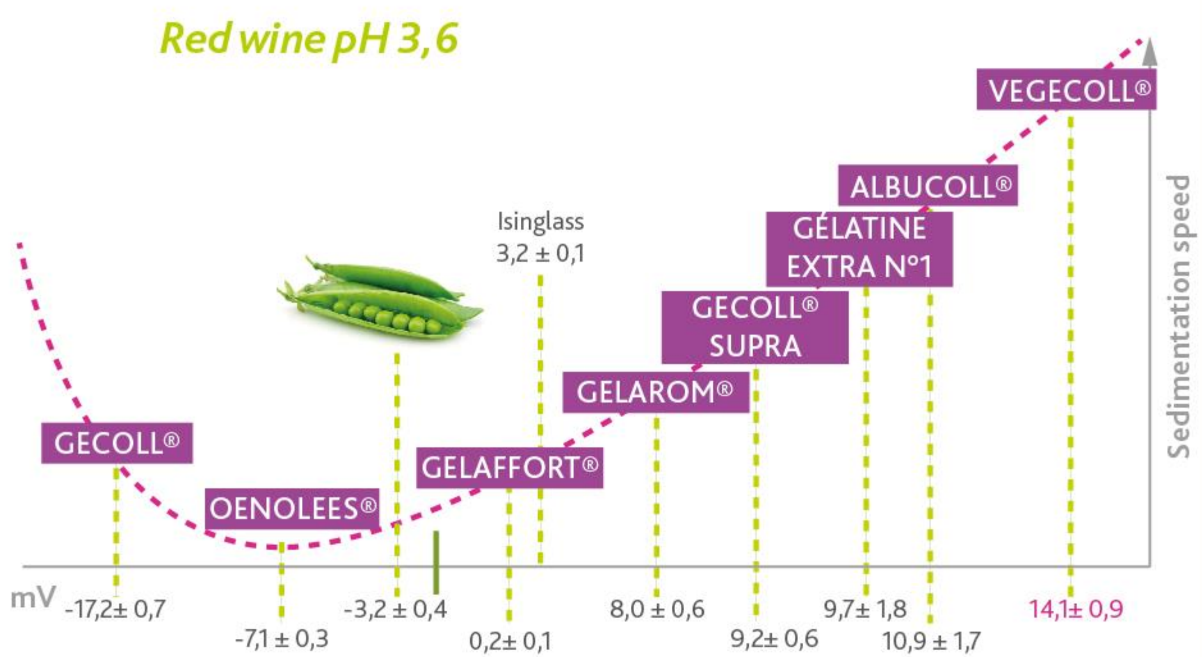
A fining agent that shows a *fast clarification*,
will automatically produce a high volume of lees.

Zeta Potential and Clarification

Classification of fining agents with respect to sedimentation rate

The physical phenomenon can be explained by the Zeta Potential

A fining agent with a high Zeta Potential (positive or negative) clarifies quickly

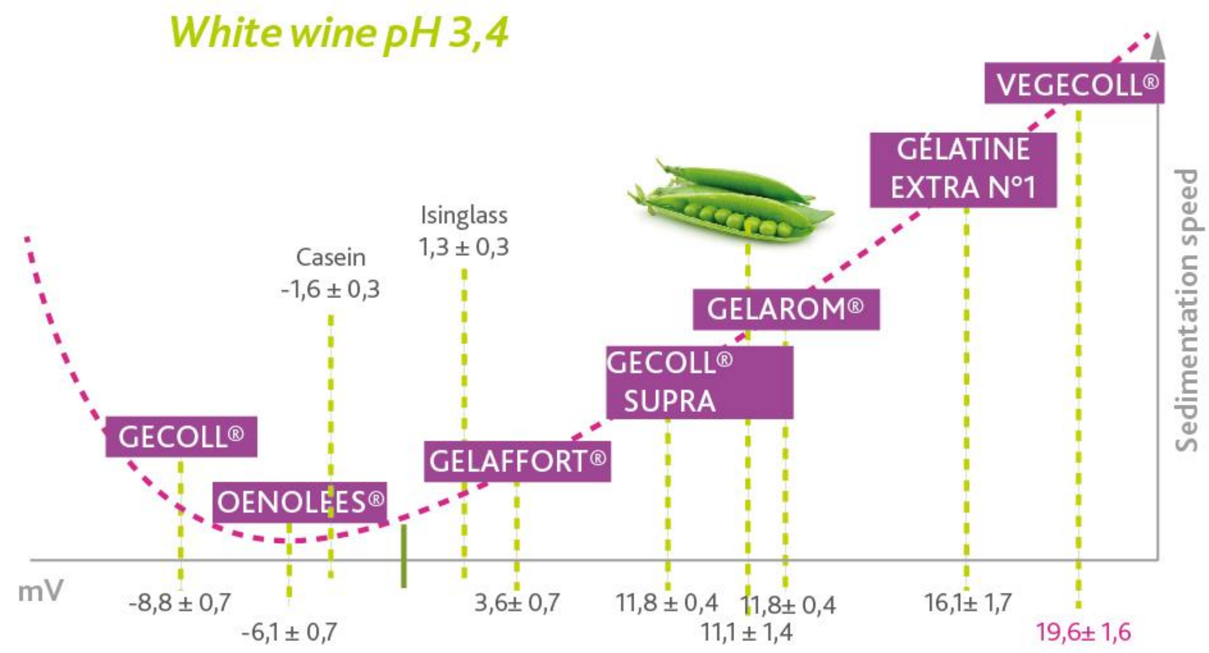


Zeta Potential and Clarification

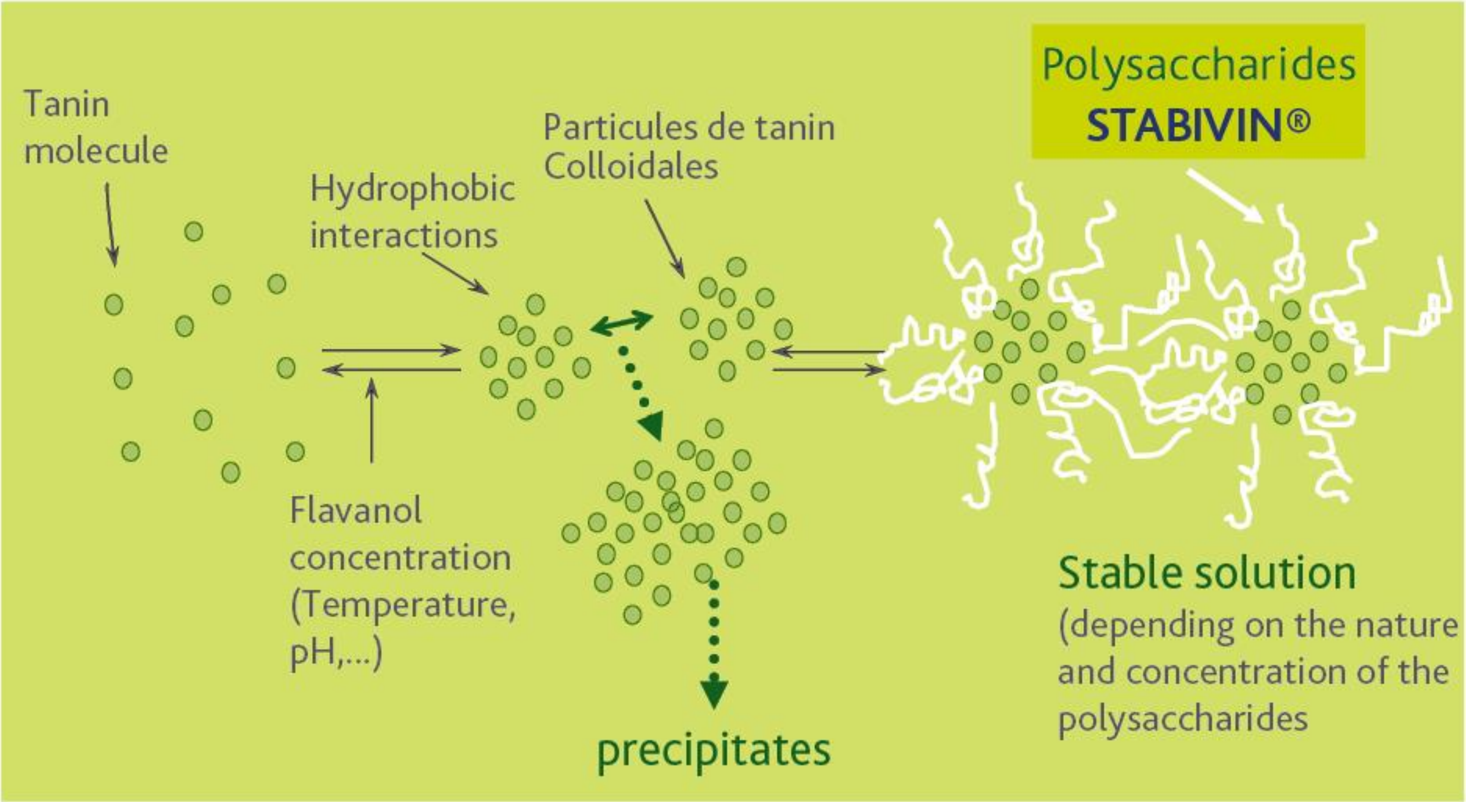
Classification of fining agents with respect to sedimentation rate

The Zeta Potential changes with the wine pH
and the same phenomenon is observed on white wines.

Other physico-chemical parameters have an impact (particle size)



The colloidal stabilisation phenomenon



Source: Cédric Saucier, 1997,
Wine tannins: Study of their colloidal stability –
PhD thesis at Université de Bordeaux II

Fining trial in the lab: DIY

For a successful lab fining trial:

- ✓ Adjust free SO_2 to 30mg/L if necessary
- ✓ Use a 375mL bottle (minimum)
- ✓ Keep a control of each wine batch
- ✓ Keep wines at room temperature
- ✓ Add the fining agent(s)
- ✓ Always try at least 2 different fining agents at 2 different doses
- ✓ Taste blind after 2 to 3 days (and measure turbidity if possible)

Over-fining

What is over-fining?

Part of the fining agent remains in suspension in the wine (gelatin and other fining agents)

To avoid over-fining:

- ✓ Thoroughly homogenize the fining agent in the wine
- ✓ Do not add more fining agent than necessary
- ✓ Keep a treatment temperature lower than 15°C / 60°F
- ✓ Use silica gel prior to the fining agent

What to do in case of over-fining?

Bentonite (white) or tannins (red)

Keys for a successful fining treatment

- **Validation of the absence of glucan in the wine**

- **Choice of the fining agent nature and dosage**

The lab performs a complete analysis of the wine and does the fining trials.

- **Incorporation**

The fining agent must be incorporated homogenously into the entire volume of wine to be treated; using a venturi like Oenodoseur is recommended.

- **Fining timing**

Dependent on: volume to be treated, temperature, initial turbidity and fining agent type.

- **Racking**

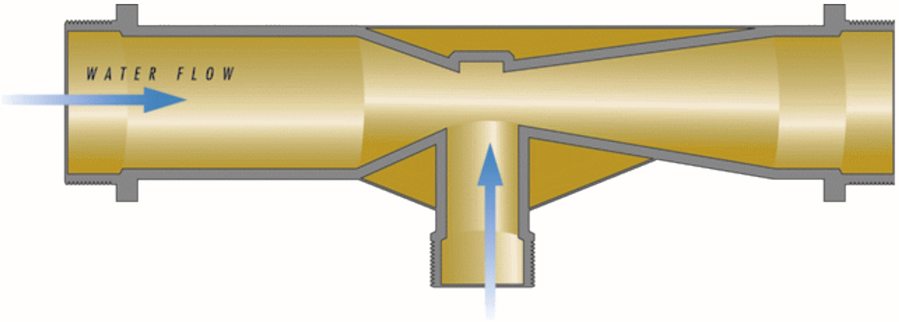
Careful racking to eliminate entire fining lees.

Note: any enzyme treatment beforehand will improve the fining quality

Fining treatment

Incorporation:
- progressive
- homogenous

During a pump-over:
- using a Venturi system (**OENODOSEUR**)



Products	Preparation Addition	Over-fining risk	Contact time before racking and filtration
Gelatins: GECOLL SUPRA, GELAROM, GEL. EXTRA N°1	**Liquid**: Add directly to the wine during a pump-over. **Powder**: Dissolve in warm water (40°C): Add directly to the wine during a pump-over while maintaining the temperature.	Yes	1 to 3 weeks
Egg Albumin: OVOCLARYL	**Powder**: Dissolve in 5 to 6 times its weight in water. Add directly to the wine during a pump-over.	Yes	1 to 3 weeks

> Fining agent use: practical points

Products	Preparation Addition	Over-fining risk	Contact time before racking and filtration
OENOLEES®	Dissolve in 5 to 6 times its weight in water. Add directly to the wine during a pump-over.	No	2 to 4 weeks
ICHTYOCOLLE®	Dissolve in 100 times its weight in water. Let swell for 2 hrs while stirring to ensure a good dispersion. Add directly to the wine during a pump-over.	Yes	2 to 4 weeks
CASÉINE CASÉI +	Dissolve in 10 times its weight in water. Add directly to the wine during a pump-over.	No	1 to 3 weeks
POLYMUST® ROSÉ POLYMUST® PRESS	Dissolve in 10 times its weight in water. Add directly to the wine during a pump-over.	No	5 days to 3 weeks
VEGECOLL®	Dissolve in 10 times its weight in water. Add directly to the wine during a pump-over.	Yes	5 days to 2 weeks

To see VEGECOLL® preparation

Products	Preparation Addition	Over-fining risk	Contact time before racking and filtration
PVPP (VINICLAR®)	Dissolve in 5 to 6 times its weight in water. Add directly to the wine during a pump-over.	No	1 to 3 weeks
MICROCOL® CL MICROCOL® ALPHA	Dissolve in 6 to 10 times its weight in hot water (50°C), keep stiring for 2 hrs, let it swell for 12 to 24 hrs. Add directly to the wine during a pump-over.	No	5 days to 3 weeks
Silica gel (SILIGEL®)	Add directly to the wine or after dilution in water or wine. Shake vigorously. Add prior to the organic fining agent	No	With the fining agent
Tannin	Add prior to the organic fining agent		With the fining agent

To see MICROCOL® ALPHA preparation

STABILIZATION

Questions to ask ourselves:

What are the risks linked to instability?

Regarding my wine:

A. Is my wine stable?
B. What are the treatment options for stabilization?
C. How can I choose the necessary and appropriate treatments?
D. How do I check the efficacy of the treatment?
E. What are the parameters influencing stability?
F. How to best carry out the treatment in the cellar?

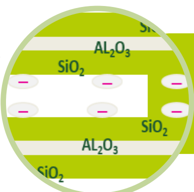
… for each stability!

Stabilization Roadmap

4 to 6 weeks prior to bottling → F → *1 to 2 weeks prior to bottling* → F → *Bottling*

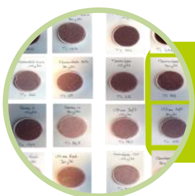
NTU < 5 CI< 20
T° > 15°C (60°F)
Checking Free SO_2, CO_2, etc.,
Complete analysis and tasting

Microbiological stabilization

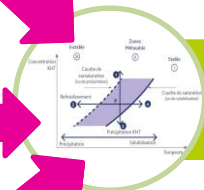


Protein stabilization

Or



Color stabilization



Tartaric stabilization

Enzyme, fining agent, lysozyme, SO_2, chitosan…	Finishing tannins	Bentonite	CMC Mannoproteins	Gum Arabic (D-2) SO_2 & ascorbic acid (D-1) Sorbic acid (D-1)

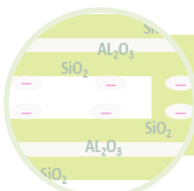
Acidification/ deacidification

Stabilisation roadmap: change in mindset

4 to 6 weeks prior to bottling → F → *1 to 2 weeks prior to bottling* → F → *Bottling*

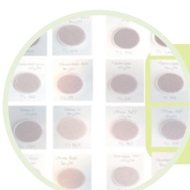
NTU < 5 CI< 20
T° > 15°C (60°F)
Checking Free SO_2, CO_2, etc., Complete analysis and tasting

Microbiological stabilization



Protein stabilisation

Or



Colouring matter stabilisation

Tartaric stabilisation

Filterability index monitoring: turbidity < 5 and CI < 20

Enzyme, fining agent, lysozyme, SO2, chitosan…	Finishing tannins	Bentonite	Metatartaric acid CMC Mannoproteins	Gum Arabic (D-2) SO2 & ascorbic acid (D-1) Sorbic acid (D-1)

Acidification/ deacidification

MICROBIOLOGICAL STABILIZATION

Risks linked to absence of microbiological stability

HAZE IN BOTTLE	**Yeasts**: re-fermentation ***Brettanomyces***: phenolic character **Acetic bacteria**: volatile acidity **Lactic bacteria**: “maladie de la graisse”, biogenic amines *+ formation of a haze on the surface*
TYPES OF WINE CONCERNED	**All types of wine**
HAZE FORMATION FAVORED BY	- Quality of wine preparation to bottling - Molecular SO_2 level (i.e. take into account pH) - Wine storage conditions: exposure to heat - Closure quality - Residual sugar level

Haze / Alterations due to micro-organisms:

Accidents still happen ***TOO*** often!

ANALYSES OF WINES SAMPLED ON SUPERMARKET SHELVES:

Non-compliant wine: pop > 1 CFU/10 mL

In 2010

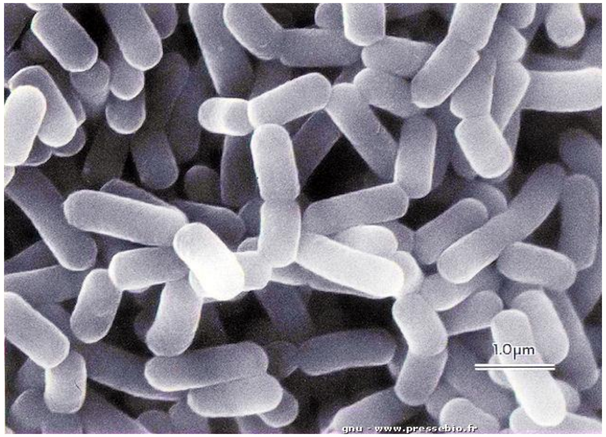
36%

AB	LB	*Brett*
52 %	15 %	9 %

In 2016

47%

AB	LB	*Brett*
46 %	11 %	28 %



A. Is my wine stable from a microbiological standpoint?

Before each bottling, perform a **complete** microbiological assessment on the final blend; by plate cell count on gel medium specific for:

- Yeasts
- Yeasts *Brettanomyces* (red wine)
- Acetic bacteria
- Lactic bacteria

Today there is no regulations regarding the micro-organism population in wine after bottling.

A test post- bottling helps to assess the "quality" of this crucial and definitive step

Thresholds are defined by some buyers:
From < 1 *CFU* / 10 mL to < 1 *CFU* / 750 mL (sweet wines)

B. Options for treatment?

Tools available to reduce microbial load:

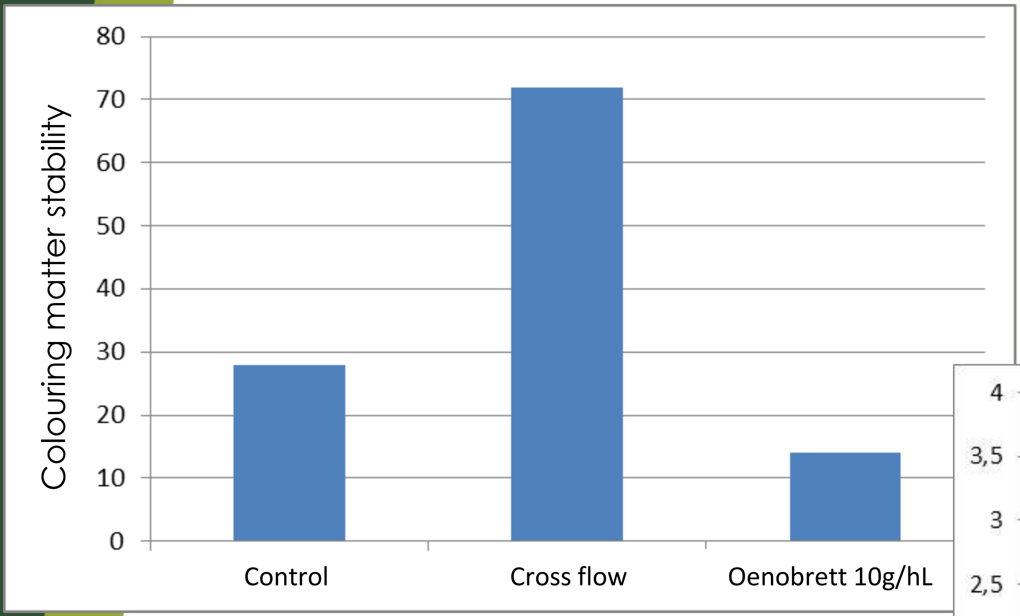
- **SO_2 addition (active SO_2)** — Non selective action
- **Enzyme addition** — Non selective action
- **Fining** — Non selective action
- **Lysozyme** — Selective action on bacteria only
- **Sorbate** — Selective action on yeasts only

Physical treatment : pay attention to potential "collateral" damages when treatments take place early in the aging process

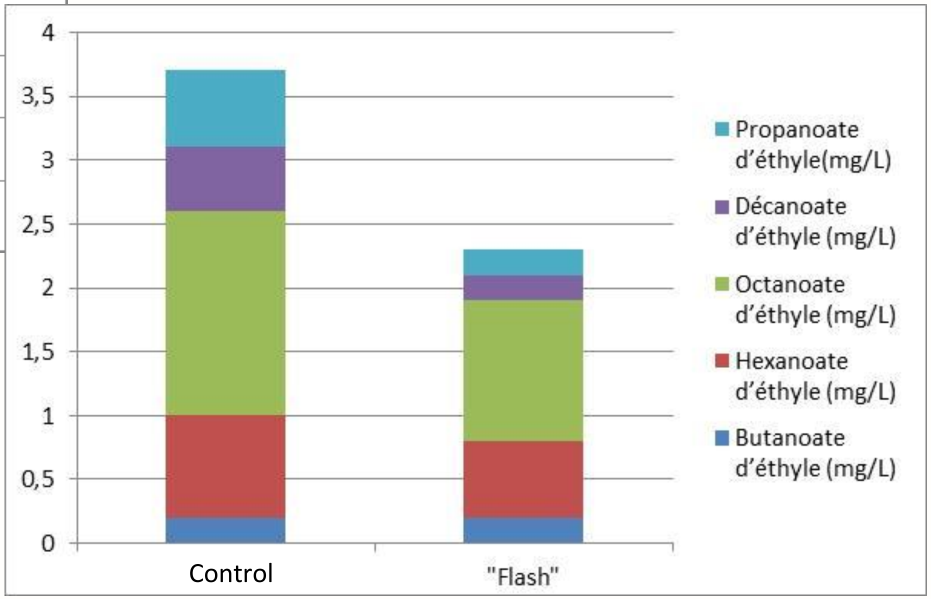
- **Flash pasteurization** — Impact on some aromatic compounds (esters)
- **Filtration (sterile)** — Careful regarding microbiological emptiness
- **Cross flow filtration** — Sterilizing filtration vs sterile filtration + impact on coloring matter stability

- **OENO*Brett*®** — Selective action on Brettanomyces (+ Bacteria)

Impact of crossflow filtration on coloring matter stability (2012 filtered wine in December 2012 measures 15 days after treatment)



Impact of Flash pasteurisation on some esters (2011 wine *flashed* in January 2012. Analysis 10 days after treatment)

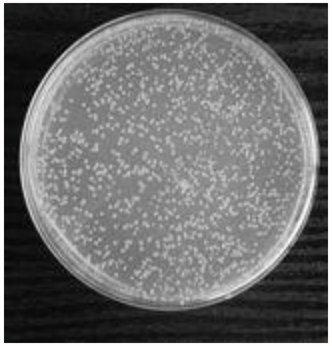


C. How do I check the efficacy of treatment?

Example of reduction of the microbial load through fining

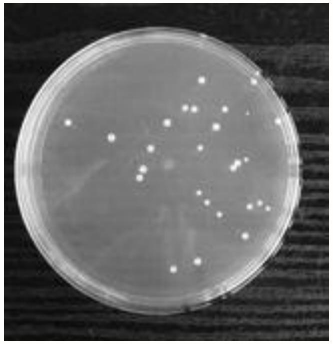
WINE FINED WITH GELATIN

Wine racked, unfined



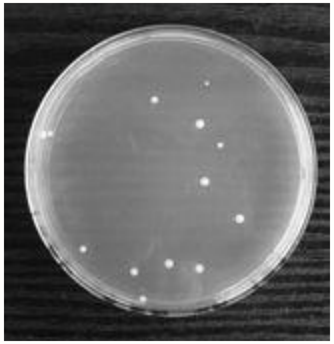
BRETT = 3.1. 10^4 CFU/ mL

20 mL/hL



BRETT = 1.8.10^2 CFU/ mL

40 mL/hL



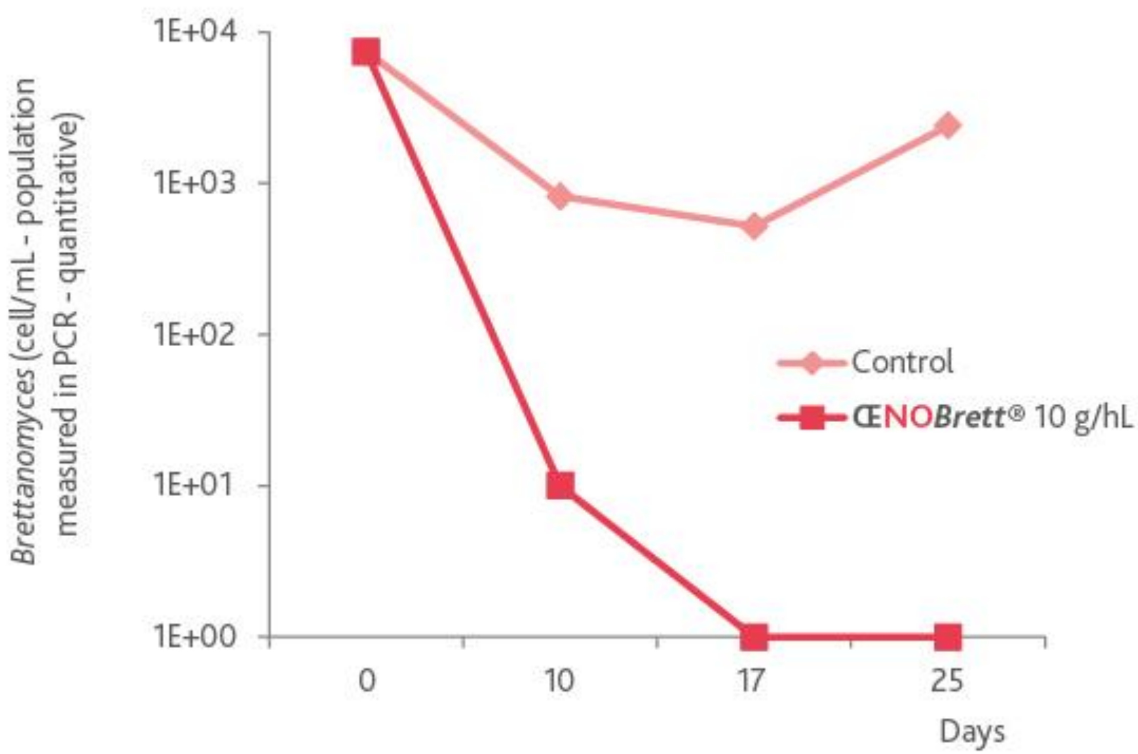
BRETT = 1.10^2 CFU/ mL

Adding a pectinase and ß-glucanase blend

	Unracked control wine	Racked wine	Wine + EXTRALYSE® Then racked
Brettanomyces *(D+10 after operation)*	4.4.10^4 CFU/mL	2.2.10^2 CFU/mL	< 1/10 mL
4-EP + 4-EG *(µg/L)* *(end of aging)*	660	290	120

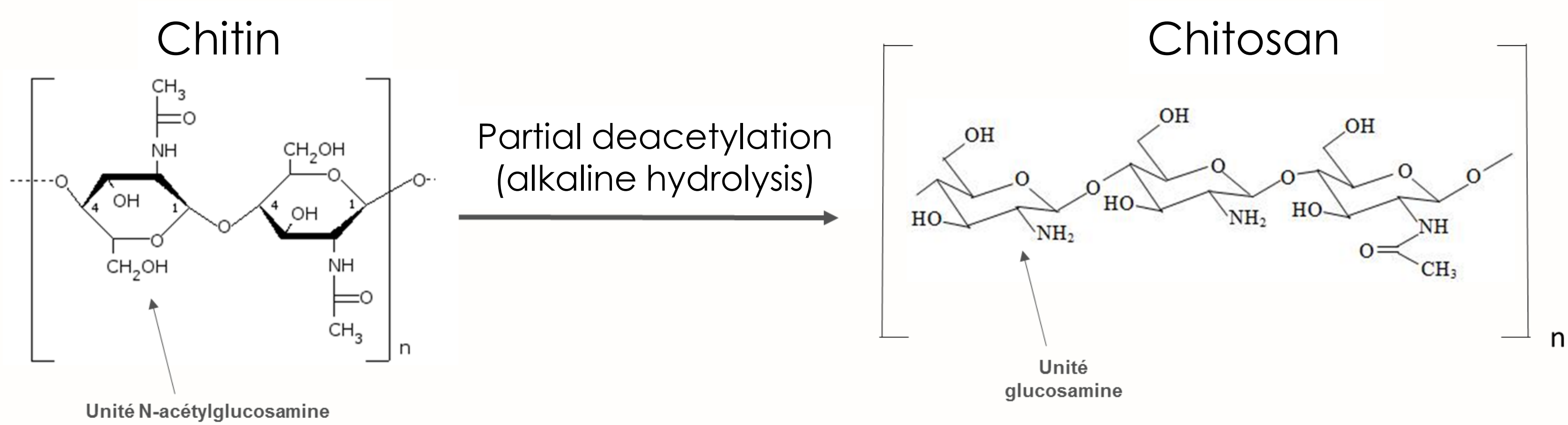
From the work of Vincent Renouf

Curative use of **OENO*Brett*®**



What is Chitosan?

N-acetyl-D-glucosamine & D-glucosamine units.



Chitin and cellulose:

- Most abundant polysaccharides: 10^{11} tonnes/year
- Origin: crustaceans (15-30 % MS), fungus (42 % in *A. niger*)

Characterization of chitosan:

- **Deacetylation degree (DD)/ acetylation (DA)**
- **Molecular weight (MMw or MMn)**

In winemaking: Non-allergenic and natural polysaccharide, non-animal, extract from the fungus Aspergillus niger.

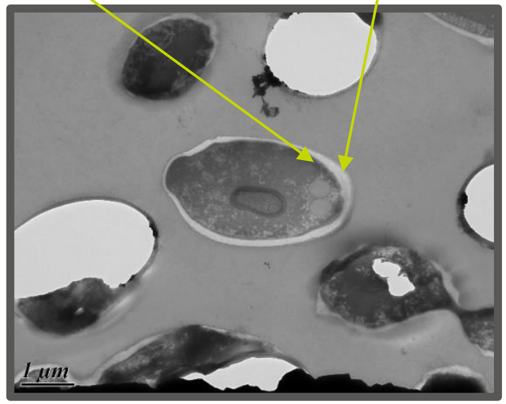
Curative use of Chitosan
Microscopic illustrations

SEM: Scanning Electronic Microscopy

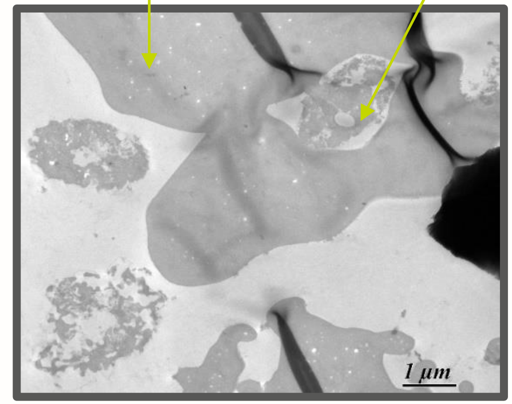
Model medium, (YPG).

Action of the Chitosan on *Brettanomyces*. **8 days after treatment with 100 mg/L.**

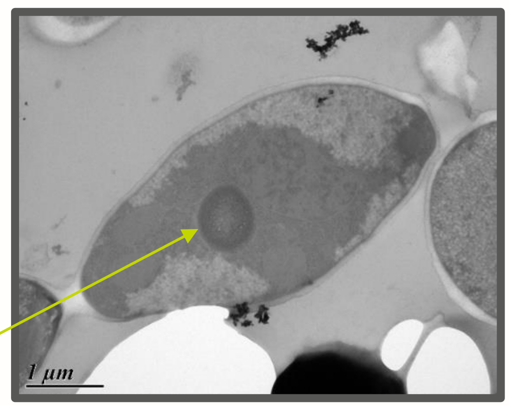
- ✓ **Destruction of the wall and cell membrane.**
- ✓ **Breakdown of the intracellular medium (no more cellular organization).**



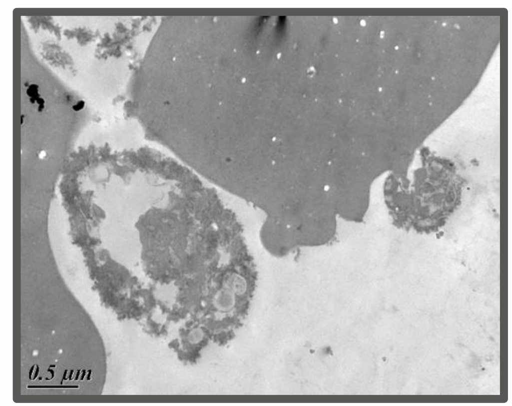
Control T0 – x 20000



Treated terms– x 20000



Control T0 – x 40000



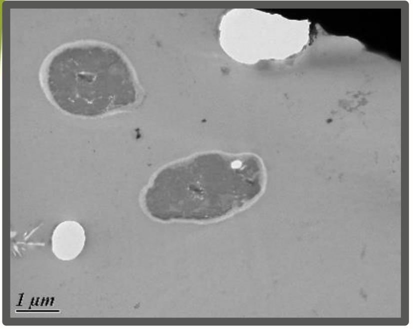
Treated terms– x 40000

Curative use of Chitosan
Microscopic illustrations

In wine

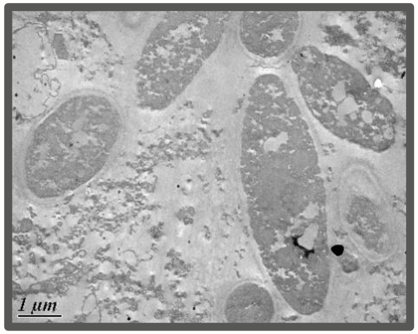
Evolution of the cell structure of *Brettanomyces* within 8 days of treatment with 10 g/hL of Chitosan- SEM (x 20.000)

Day 0 (untreated)



Compared with the model medium, the cells already seem affected by the environment, however, their walls and membranes are normal and the architecture of the cell tends to demonstrate their viability.

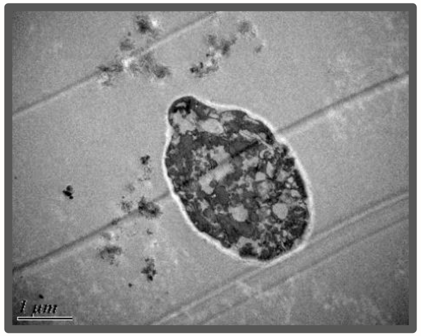
Day + 1 (treated)



The day after treatment cells are already affected by Chitosan

- ✓ Intracellular breakdown.
- ✓ Wall and membrane damage.

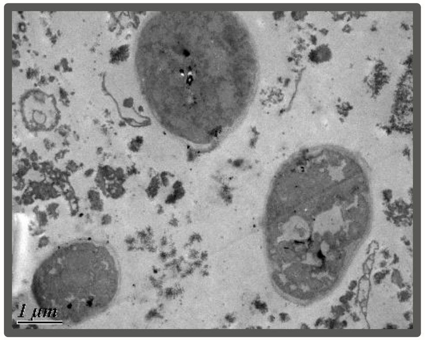
Day + 4 (treated)



Next step in the process of cell death.

The majority of cells do not exhibit a wall and viable membrane structure any more.

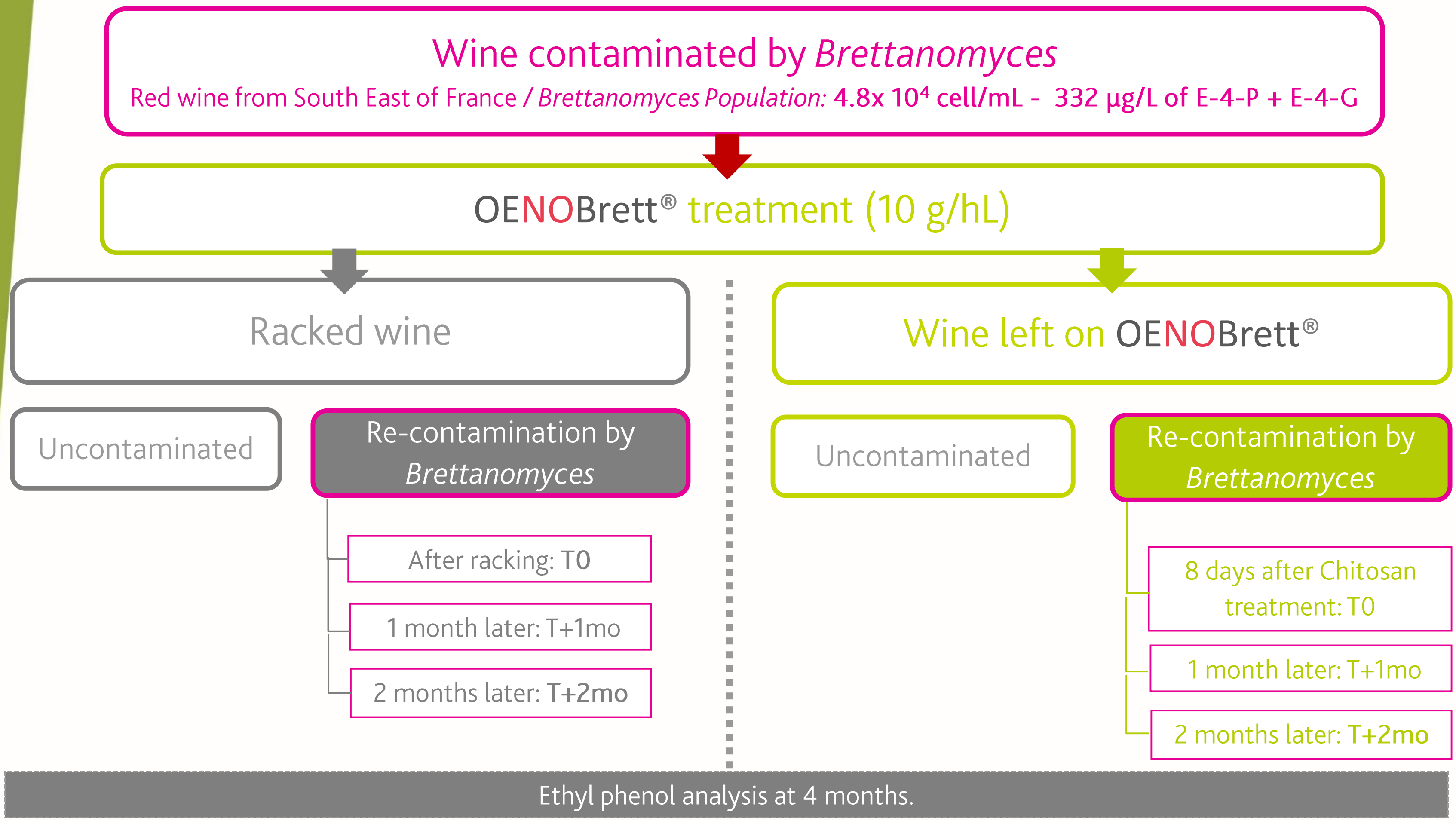
Day + 8 (treated)



Bordeaux Imaging Center

Preventative use of OENOBrett®

Concept of persistence: experimental protocol

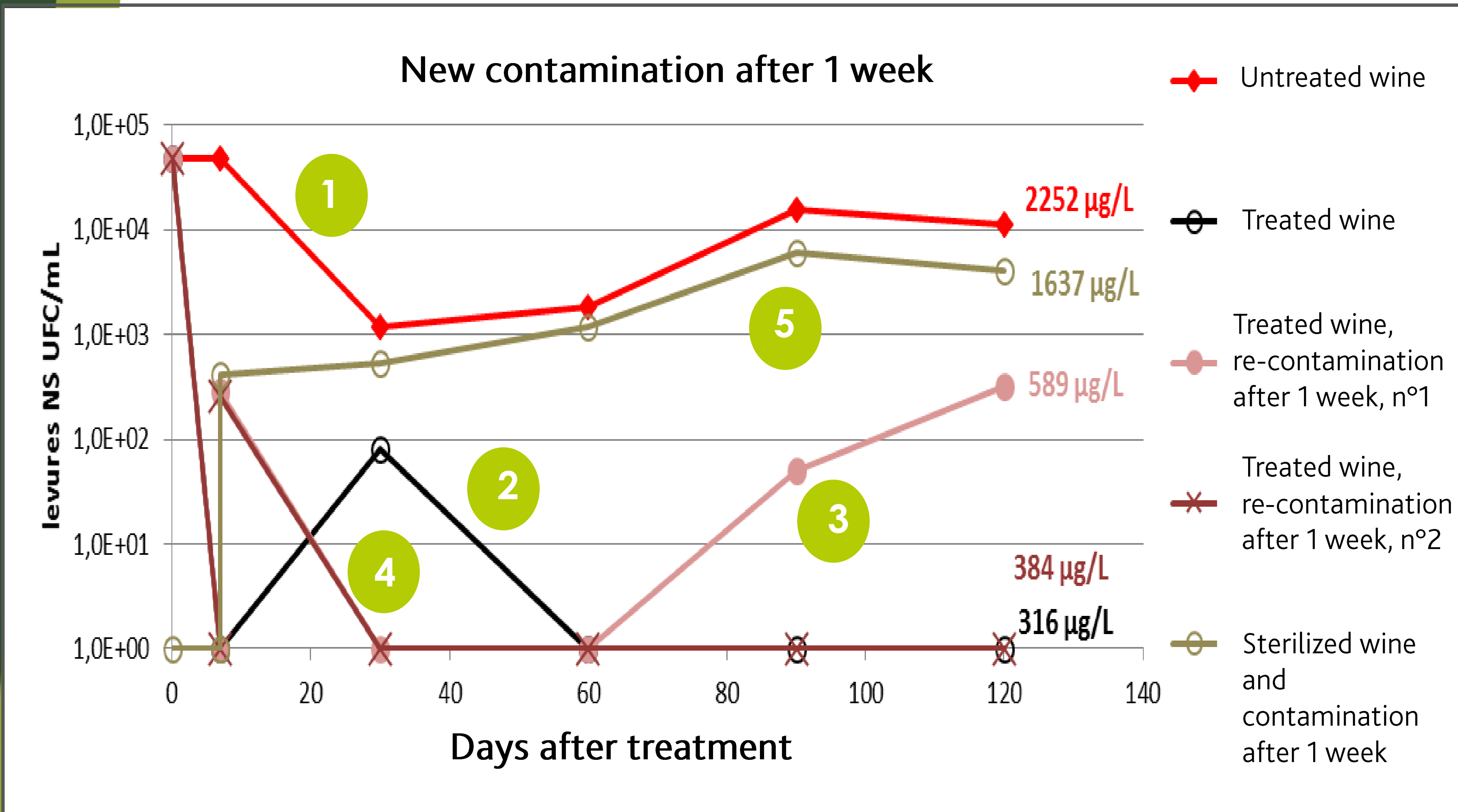


In parallel, two control wines are followed:

- An untreated control, to watch the evolution of *Brettanomyces* populations.
- A sterile filtered control as a physical stabilization alternative.

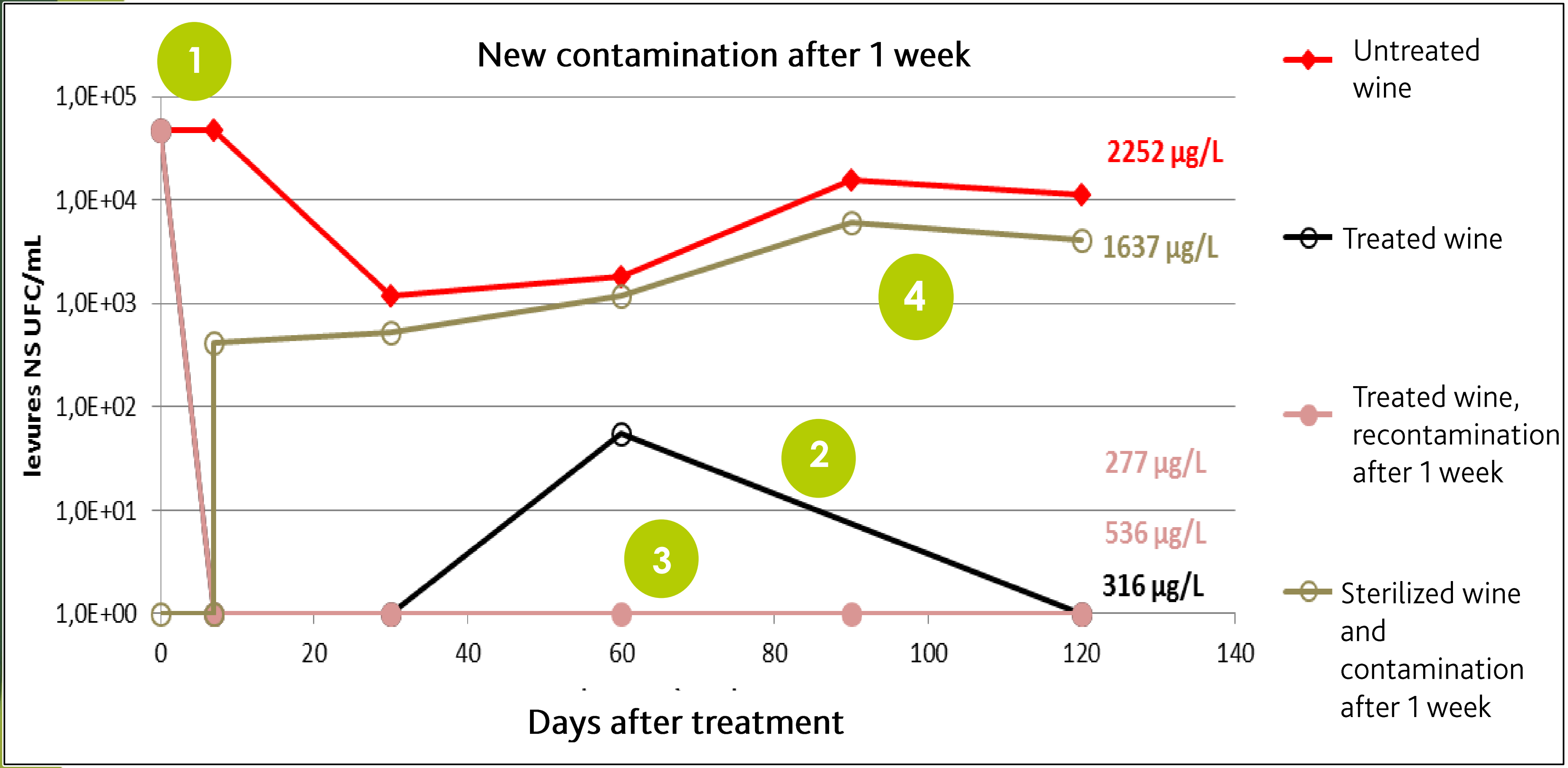
Preventative use of OENOBrett® Concept of persistence

Trial n°1: Treated wine – racked after 8 days.



Preventative use of OENOBrett® Concept of persistence

Trial n°2: Treated wine – unracked.



STABILIZATION

> Microbiological Stabilization

Preventative use of OENOBrett®

Concept of persistence

4EP + 4EG concentration in µg/L	Treated wine, racked after 8 days	Treated wine left on Oenobrett for 3 months	Wine sterilized by filtration (0,45µm)
Uncontaminated	**316 - 350**	**280**	**1750**
Contaminated T0	589 - 384	277 - 536	1637
Contaminated T+1month	1112 - 341	304 - 308	1657
Contaminated T+2month	288 - 294	274 - 300	1631
Untreated wine		**2252**	

The concentrations of 4-ethylphenol and 4-ethyl guaiacol were determined at the end of the fourth month.

B. No change in the concentration of ethyl phenols.

C. Spontaneous development of *Brettanomyces* prompted a significant production of phenols.

D. The wine left on Oenobrett is less sensitive to recontamination than the wine racked after 8 days.

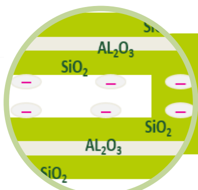
A. High production of ethyl phenol in the untreated wine!

Stabilisation roadmap: change in mindset

4 to 6 weeks prior to bottling → F → *1 to 2 weeks prior to bottling* → F → *Bottling*

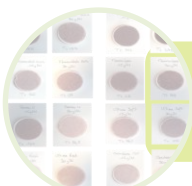
NTU < 5   CI< 20
T° > 15°C (60°F)
Checking Free SO_2, CO_2, etc.,
Complete analysis and tasting

Microbiological stabilisation



Protein stabilization

Or



Colouring matter stabilisation

Tartaric stabilisation

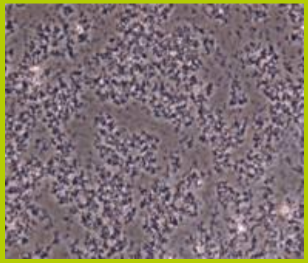
Filterability index monitoring: turbidity < 5 and CI < 20

Enzyme, fining agent, lysozyme, SO2, chitosan…	Finishing tannins	Bentonite	Metatartaric acid CMC Mannoproteins	Gum Arabic (D-2) SO2 & ascorbic acid (D-1) Sorbic acid (D-1)

Acidification/ deacidification

Protein Stabilization

Risks linked to absence of stability

PROTEIN HAZE	Protein denaturation and flocculation under heat conditions in case of an accidental temperature increase
TYPE OF WINE CONCERNED	White and rose wines: off-white flakes formation
HAZE FAVORED BY 	• Inefficient fining • Bottling filtration quality: ***a stable wine before bottling that became unstable by retention of protective colloids in case of a clogging filtration*** • Certain additives like Lysozyme at bottling • Poor quality natural corks: possibility of releasing ***cork tannins***. • ***Wine storage*** conditions: wine exposed to heat.

ANALYSES OF WINES SAMPLED ON SUPERMARKET SHELVES:

In 2010

17%

34 % white / 2 % rosé

In 2016

13%

9 % white / 40 % rosé

A. Is my wine stable from a protein standpoint?

Review of protein stability tests

Over time, different lab tests have been used to evaluate protein haze risk prior to bottling.

They are based on:

- **Flocculation of proteins under different conditions:**
  - Heat
  - In presence of tannins
  - In presence of chemical reagents

- **Protein presence:**
  - Immunological test

A. Is my wine stable from a protein standpoint?

Review of protein stability tests

1. Tests by chemical denaturation:

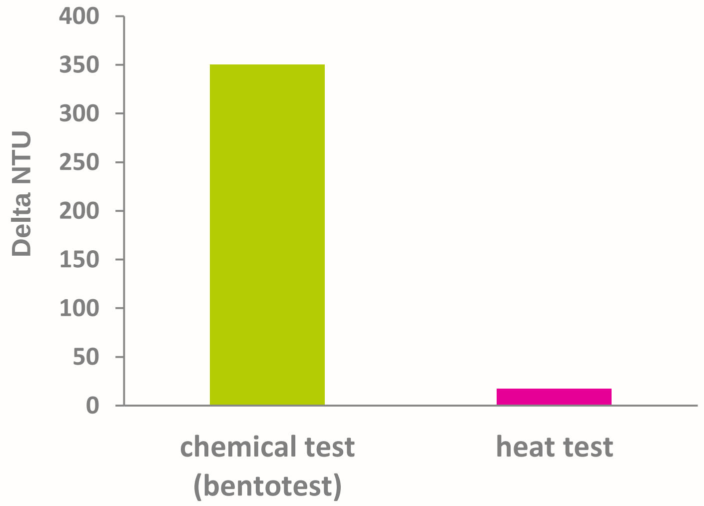
Based on reactions with a chemical agent

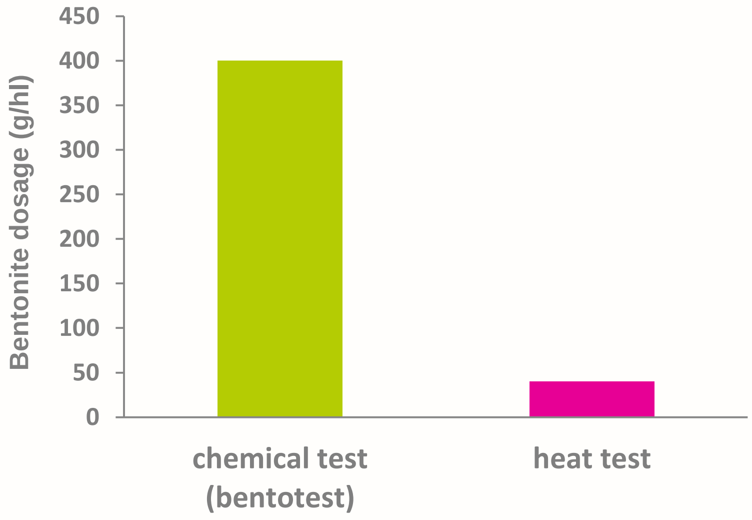
(phosphomolybdic acid, trichloroacetic acid, etc.)

✓ ***Do not model the natural phenomenon of protein haze formation***

Reagents used are not specific of thermo-unstable proteins.

✓ ***These tests systematically lead to an overestimation of the bentonite dose.***





A. Is my wine stable from a protein standpoint?

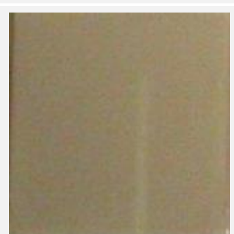
Review of protein stability tests

2. Immunological test:

Based on the reaction with "specific" antibodies

✓ ***Does not model the natural phenomenon of protein haze formation***

Reagents used are not specific of thermo-unstable proteins.

CONTROLS Immunological test		**Sauvignon Bordeaux 2008**	
Positive control	Negative control	Immuno. test	Heat test
			0.9 NTU ***Wine stable***

A. Is my wine stable from a protein standpoint?

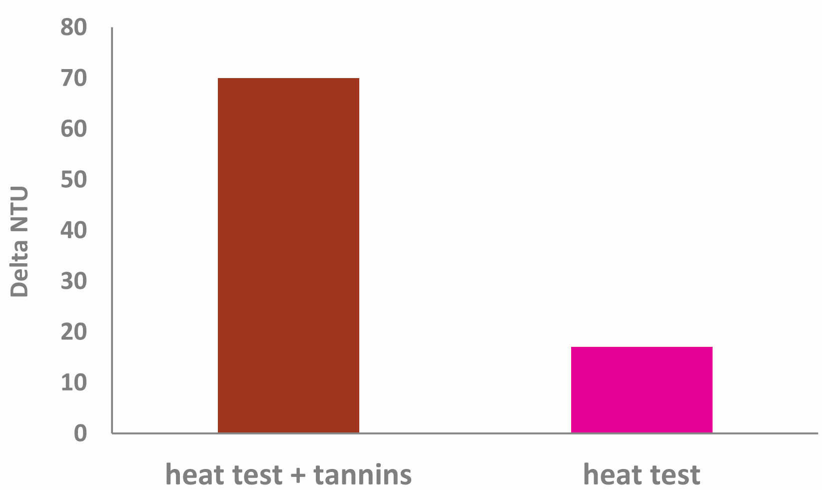
Review of protein stability tests

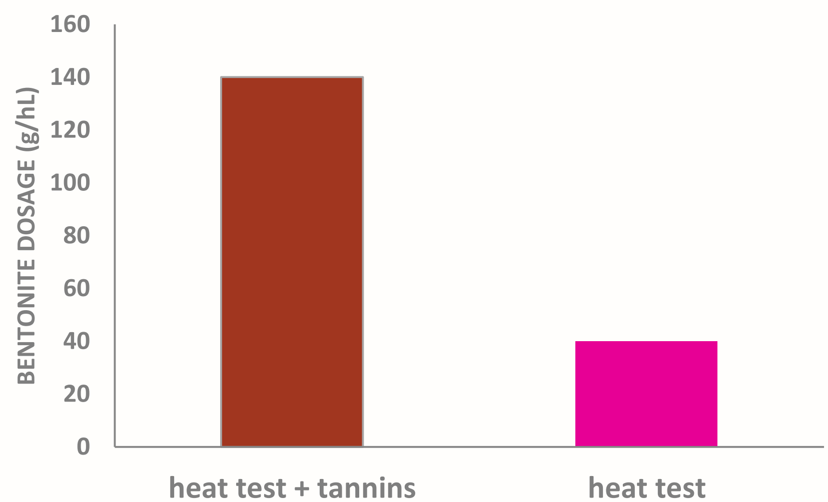
3. Heat test in presence of tannins:

Based on the reaction with tannins and exposure to heat

✓ ***Does not model the natural phenomenon of protein haze formation.***

✓ ***These tests systematically lead to an overestimation of the bentonite dose.***





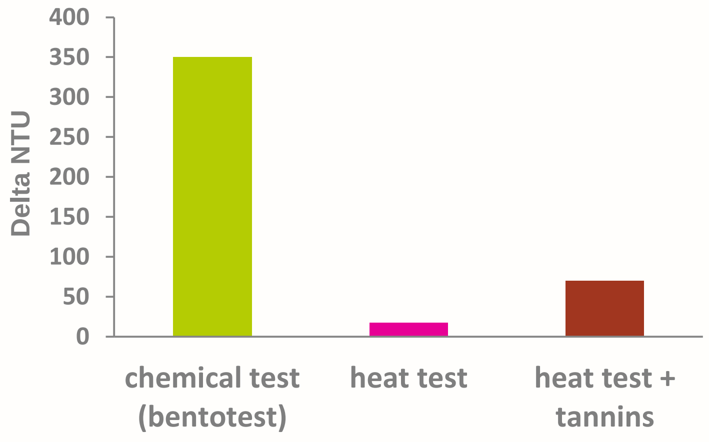
A. Is my wine stable from a protein standpoint?

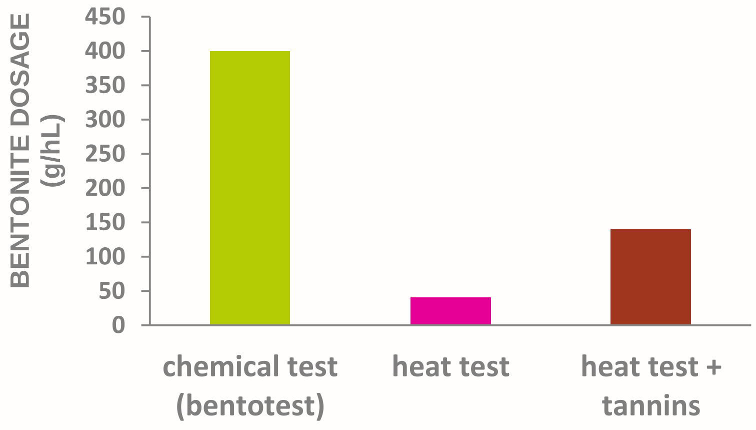
Review of protein stability tests

4. Heat test:

Based on exposure to heat ***in perfectly defined conditions*** *(80°C 30min, room temp. 45 min)*

- ✓ *Only test modelling the natural phenomenon of protein haze formation*
- ✓ *Specific to thermo-unstable proteins*





A. Is my wine stable from a protein standpoint?

Review of protein stability tests

Heat test Protocol

a. Measure the wine turbidity: **if > 2 NTU, filter the wine (cellulose ester membrane, 0.65 µm)** => **turb1**

b. Heat the wine for **30 minutes at 80°C / 176°F.**

c. Let it cool for **45 minutes at room temperature.**

d. Measure the wine turbidity again => **turb2**

***In case of a shorter cooling time** (eg putting the tube under cool running water):*
Risk of under-estimation of the bentonite dose (minor haze).

In case of a longer cooling time than 45 minutes:
Risk of over-estimation of the bentonite dose (formation of a haze not due to the thermo-instable protein fractions).

The wine is UNSTABLE if Δ NTU (turb2 – turb1) > 2

Stability must be measured with an appropriate test

Chemical denaturation or immunological protein stability tests:

- Does not model the “natural” phenomenon of protein haze
- Precipitates all proteins, whether they are heat sensitive (thermo-unstable) or not
- Leads to ***bentonite doses greater than necessary to stabilize the wine (= eliminate thermo-unstable proteins).***

⇒ ***The heat test*** **is the only test modelling the natural phenomenon of protein haze formation**

B. What are my options in terms of treatment?

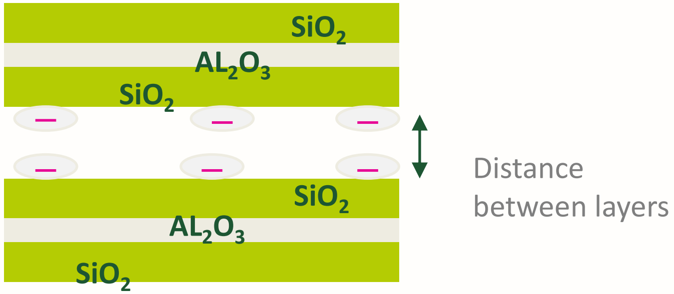
The bentonite treatment is ***the only tool*** preventing protein haze to this day.

There are 3 types of bentonites:

- ✓ Calcium bentonites ($Na^+/Ca^{2+} < 1$)
- ✓ Sodium bentonites ($Na^+/Ca^{2+} > 1$)
- ✓ Calcium bentonites, sodium activated

Inter layer space:

- ✓ Sodium bentonite: 100A
- ✓ Calcium bentonite: 10A



Sodium bentonites swell more and adsorb proteins more effectively.
Calcium bentonites precipitate and clarify more effectively.

All bentonites in the Laffort range are natural for a better stability and shelf life.

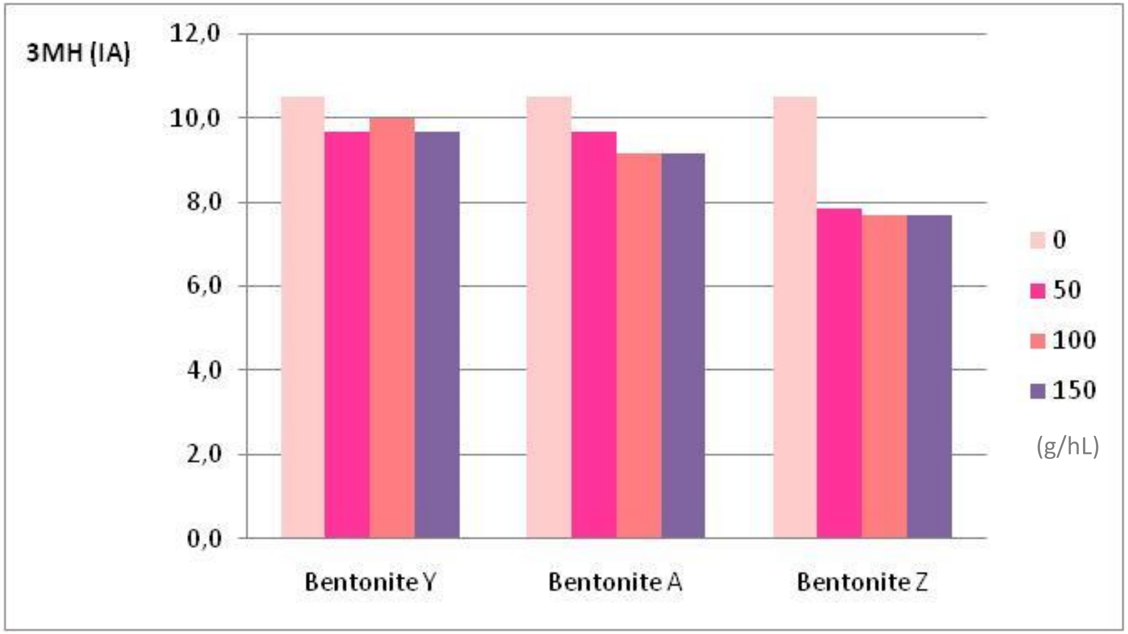
Dominant Cation	Swelling rate	Exchange capacity	Lees sedimentation	Aromatic preservation
Sodium	High	High	Medium Fluffy lees	+++
Calcium	Medium	Low	Rapid Compact lees	+++(+)

Note: VEGECOLL®, or a combination of SILIGEL® and GELAROM®, at a low rate helps improve sedimentation.

B. What are my options in terms of treatment?

Effects of bentonite treatment

An excessive bentonite treatment **does not lead to over-fining**.
But it does, undeniably, have an impact on the wine organoleptic quality.



From Moine-Ledoux, 2006

The necessary bentonite dose for stabilization must therefore be precisely determined.

C. How do I choose the appropriate treatment?

Following the heat test results: ***if Δ NTU > 2***

⇒ Treat the wine with bentonite

Determination of the bentonite dose necessary to stabilisation

a. Double and triple the instability value (Δ NTU).
b. Test 2 to 3 doses of bentonite in order to frame these values.
c. 30 minutes after the bentonite incorporation (small volume), renew the stability test assessment.

Bentonite preparation in the lab:

Use the same bentonite as the one used in the winery!!

- Pick the bentonite dose that reaches Δ NTU (turb2 – turb1) < 2

⇒ Add 10 g/hL (100ppm) to this bentonite dose value for the cellar treatment.

C. How do I choose the appropriate treatment?

Following the heat test results: ***if Δ NTU > 2***

⇒ Treat the wine with bentonite

Bentonite preparation and treatment in the cellar:

Use the same bentonite in the cellar as the one used in the lab!!

- ✓ Prepare a 5 % solution in water.
- ✓ Keep stirring for 2 hours, let it swell for 12 to 24 hours, stir vigorously before use.
- ✓ Incorporate the bentonite into the wine to be treated with a venturi.
- ✓ To accelerate sedimentation, add silica gel and gelatin 24 hours later.
- ✓ Check the treatment efficacy.

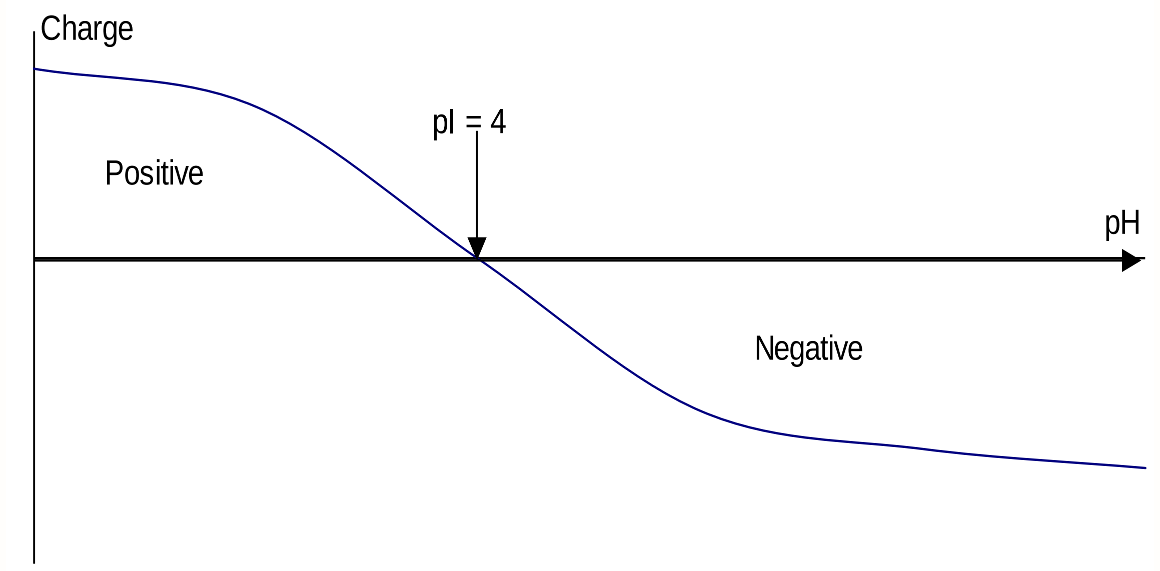
See the bentonite preparation

D. What are the parameters influencing stability?

1. The maturity level influences the protein concentration

- ✓ The concentration in thermo-unstable proteins increases during ripening
- ✓ **The higher the pH, the more bentonite required to stabilize the wine**

Protein charge according to pH



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1. The maturity level influences the protein concentration

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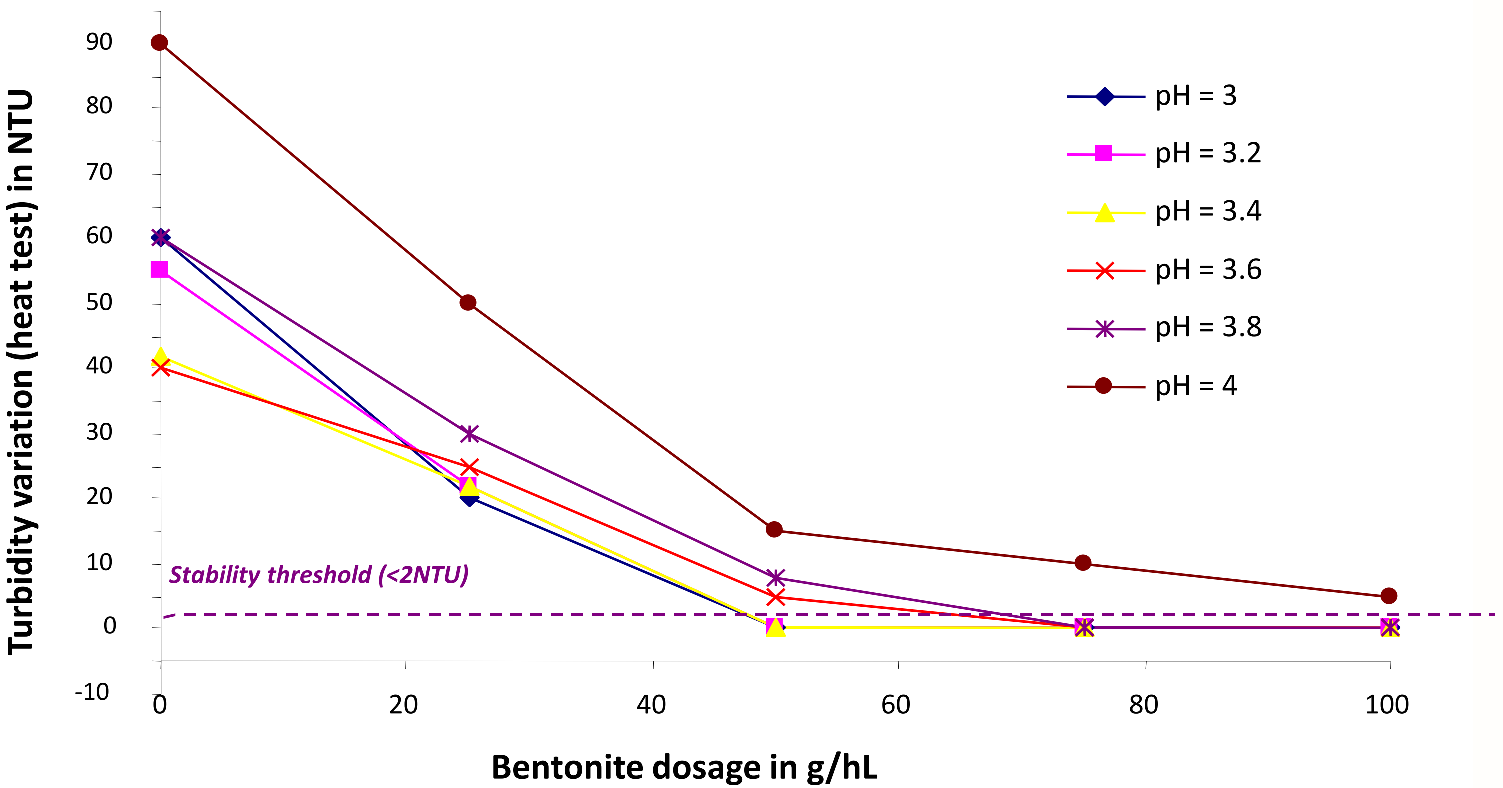
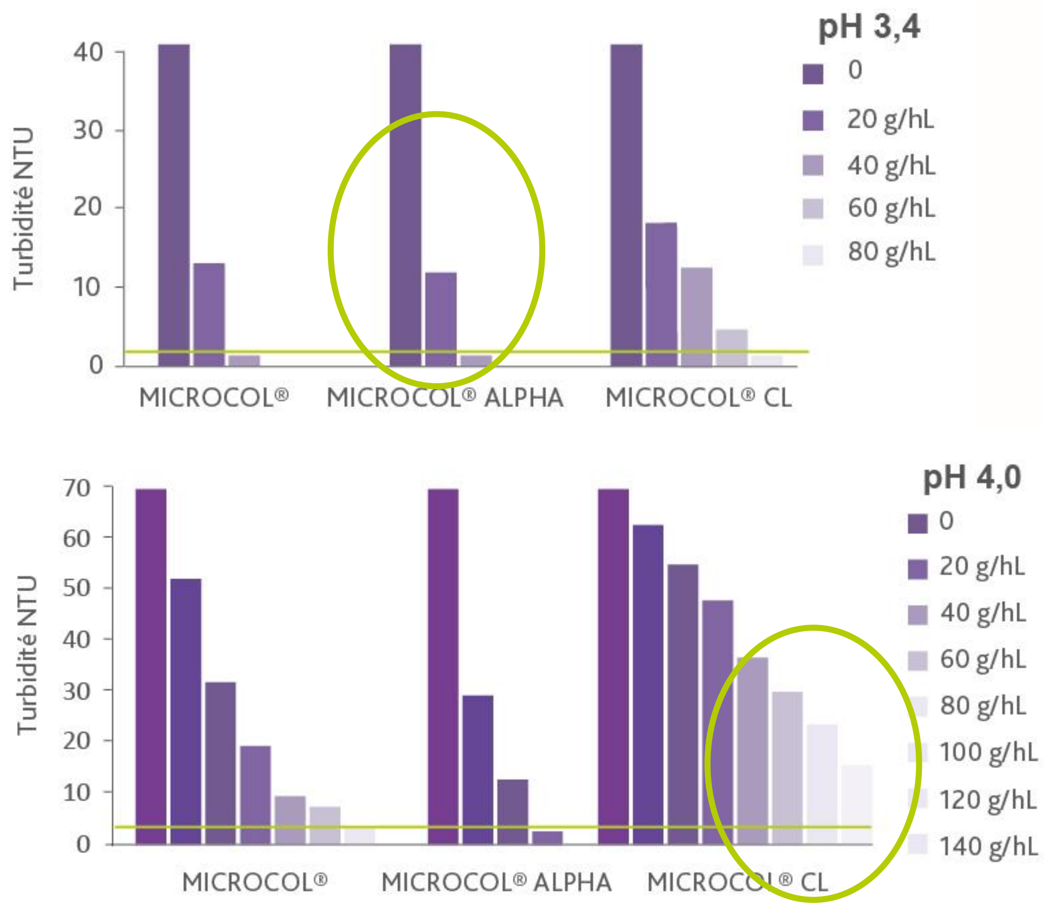


Moine-Ledoux, 2006

D. What are the parameters influencing stability?

pH effect on bentonite action:



Bentonite type, wine ***pH***, but also **bentonite preparation** influence ***protein stabilization***!

D. What are the parameters influencing stability?

2. Pre-fermentation operations impact stabilization

- ✓ In the case of a ripe Sauvignon or Semillon, skin contact will double the instability!
- ✓ SO_2 addition on grapes during maceration enhances protein extraction.
- ✓ Press juices are more unstable than free run juices.

3. Lees aging improves protein stability

Results (ΔNTU)	Wine post fermentation	Wine aged 4 months on lees	Wine aged 10 months on lees
HEAT TEST	45	34	17

Moine-Ledoux, 2006

D. What are the parameters influencing stability?

4. A LYSOZYM® treatment increases wine instability

	Control	Wine treated with **LYSOZYM®**
Heat test results (in Δ NTU)	12	57
Bentonite dose required to stabilise (mg/L)	400	1200

D. What are the parameters influencing stability?

5. Grape varietal, terroir and vintage also have an influence on the protein content of must and wines

6. A clogging filtration can make the wine unstable

	CLOGGING FILTRATION	
	Beginning of bottling	End of bottling
Heat test results (in Δ NTU)	0.5	4

The clogging filtration made the wine unstable by ***retaining protective colloids.***

E. How do I carry out the treatment in the cellar?

When to treat: on must? on wine?

On must

- ✓ For early release wine (no barrel aging, no lees aging).
- ✓ For *Botrytis* affected fruit.
  - ✓ Select a calcium bentonite: more clarifying than stabilizing.
  - ✓ Quickly rack the wine after fermentation.
  - ✓ ***No lees aging possible.***

On wine

- ✓ For all other wines.
- ✓ Select a sodium bentonite: high protein removal power, respectful of the wine aromas.

In all cases, **the bentonite treatment effectiveness** is directly linked to ***its preparation conditions.***

The Laffort bentonite range

MICROCOL® ALPHA

Natural sodium bentonite, microgranulated, with high protein removal power. For stabilization and clarification of must and wine over a large pH spectrum.

MICROCOL® CL

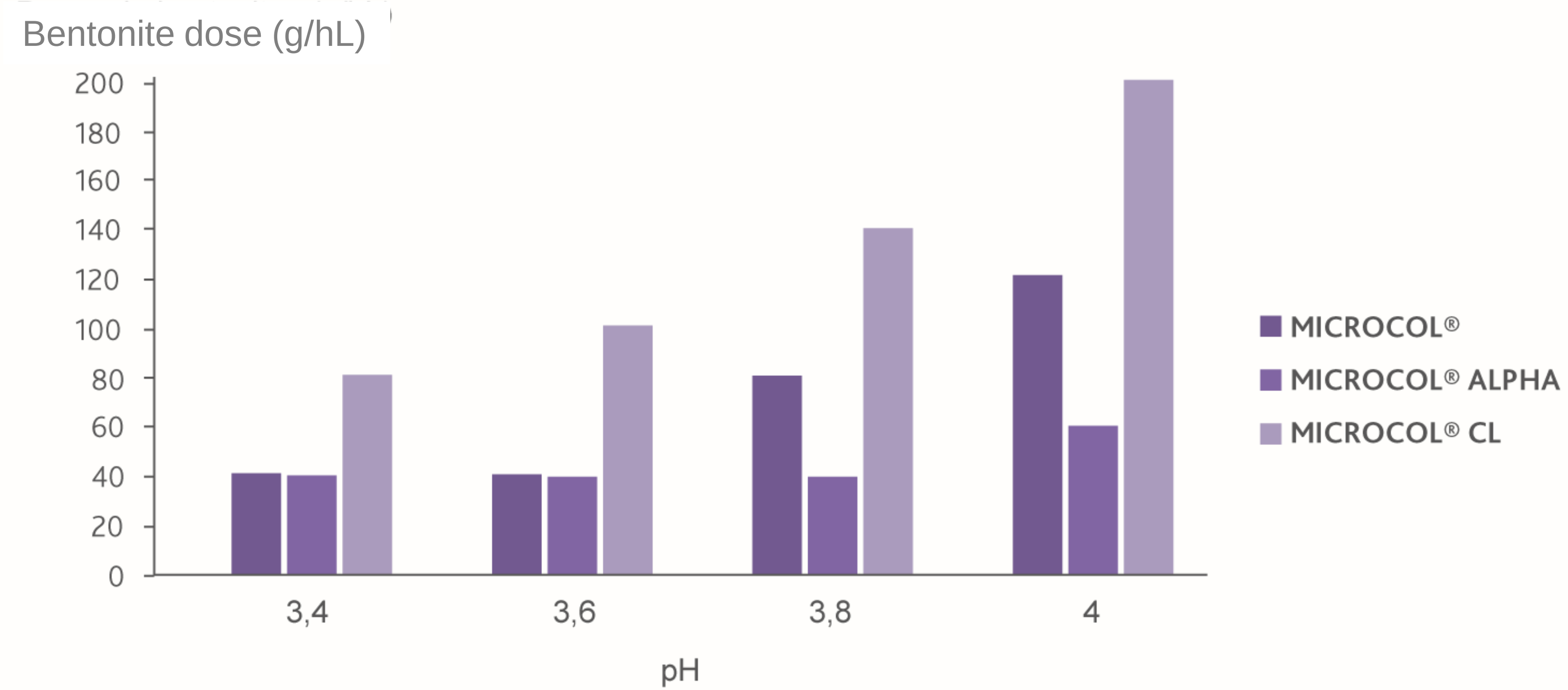
Natural calcium bentonite, powder. For clarification and stabilization of must and wine.

MICROCOL® FT

Calcium sodium bentonite. For stabilization during cross flow filtration.

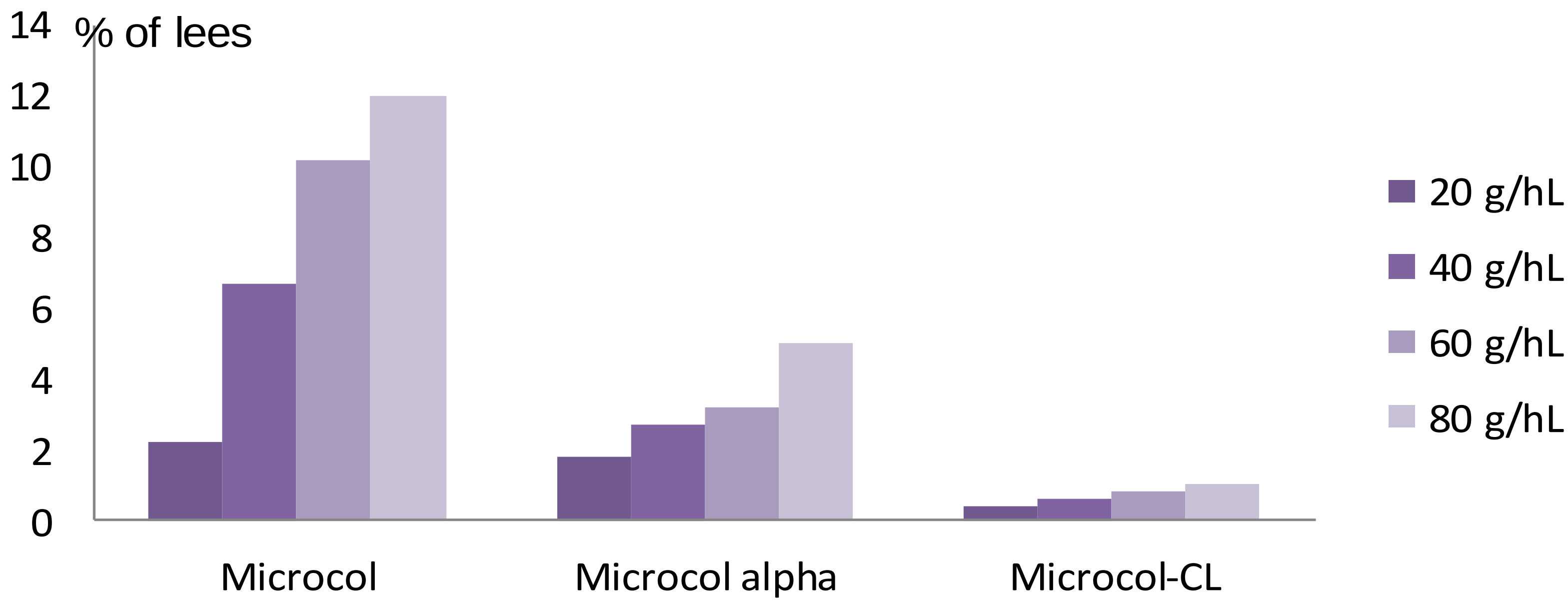
Bentonite specificities

Wine pH effect on adsorption power



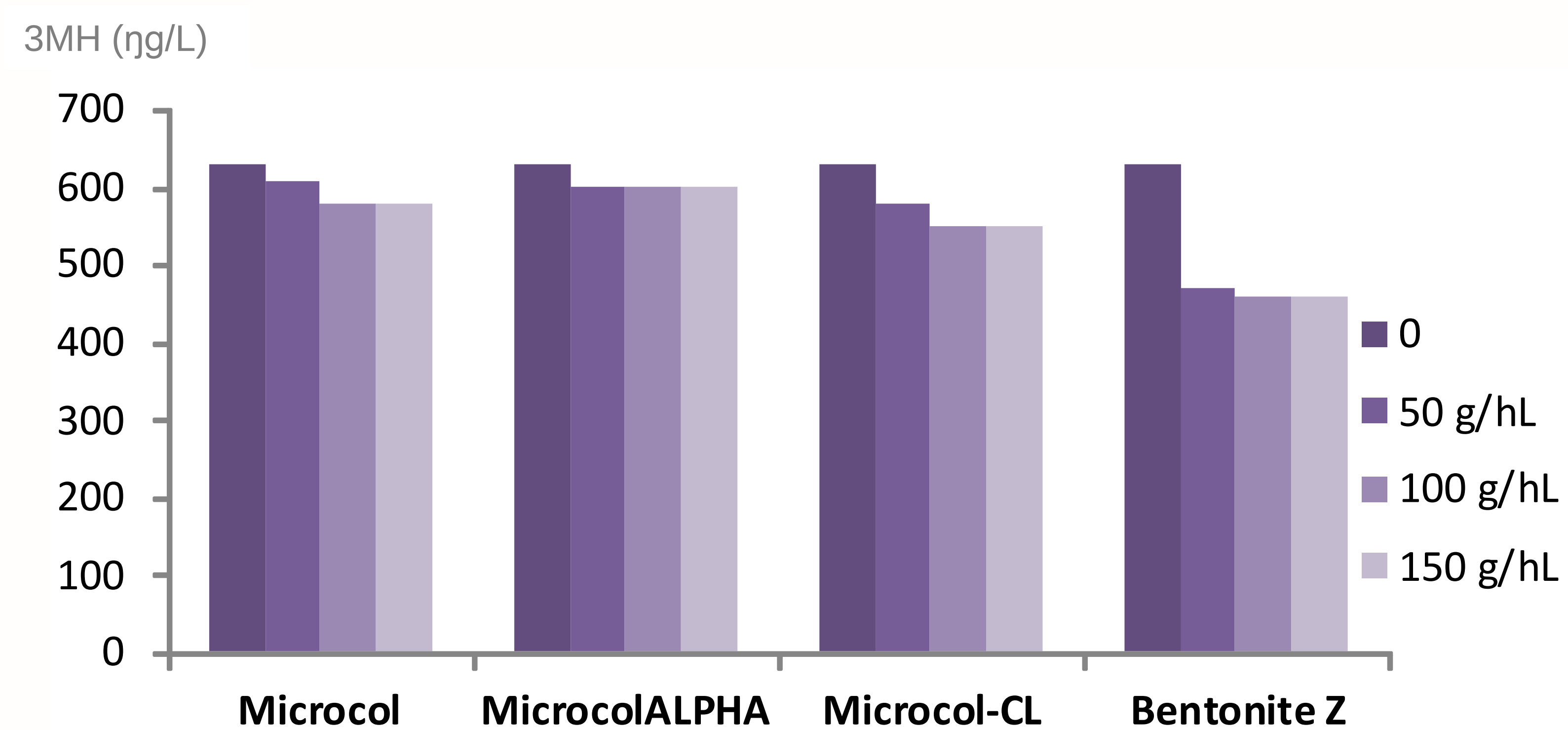
Bentonite specificities

Clarifying power of different bentonites



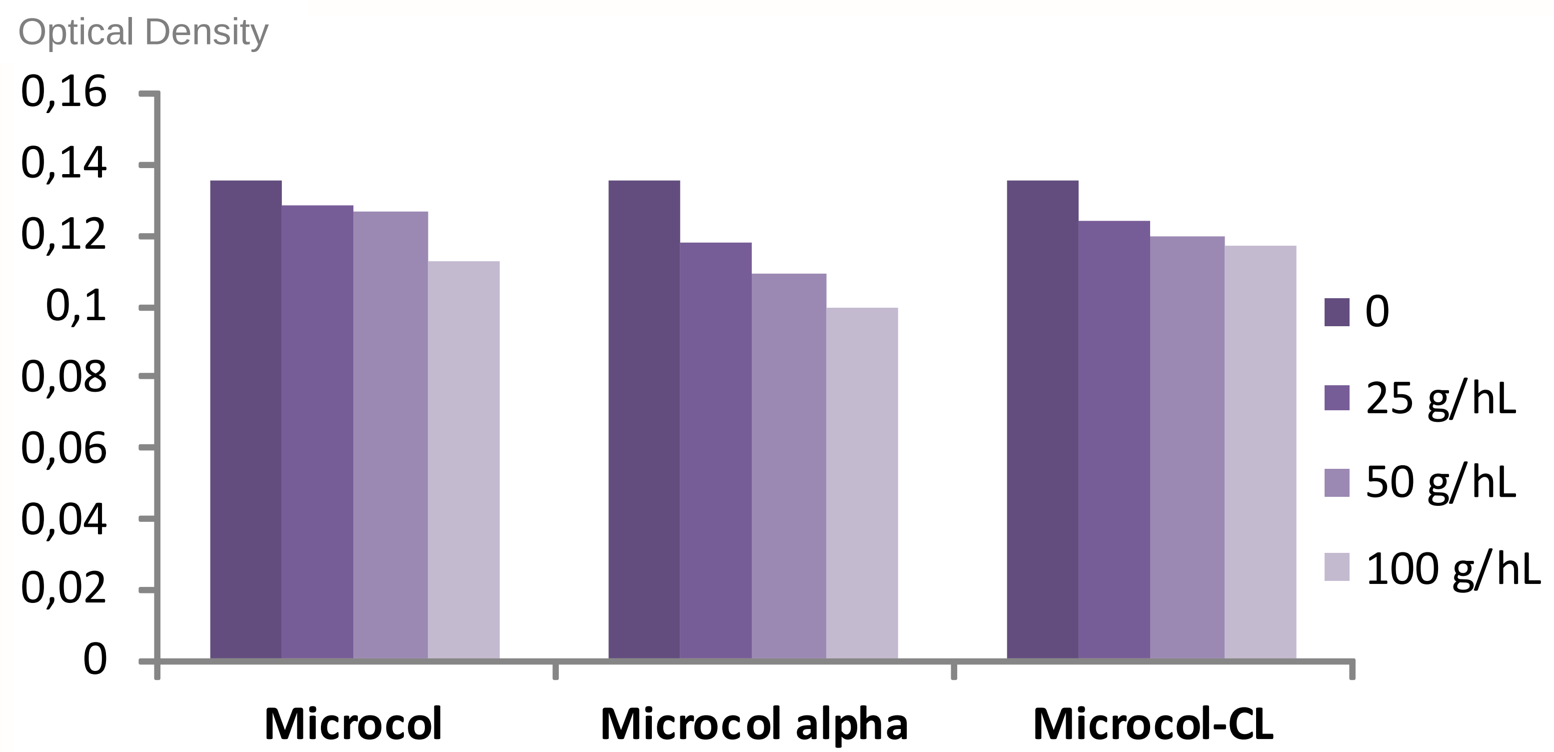
Bentonite specificities

Effect on aromatic compounds

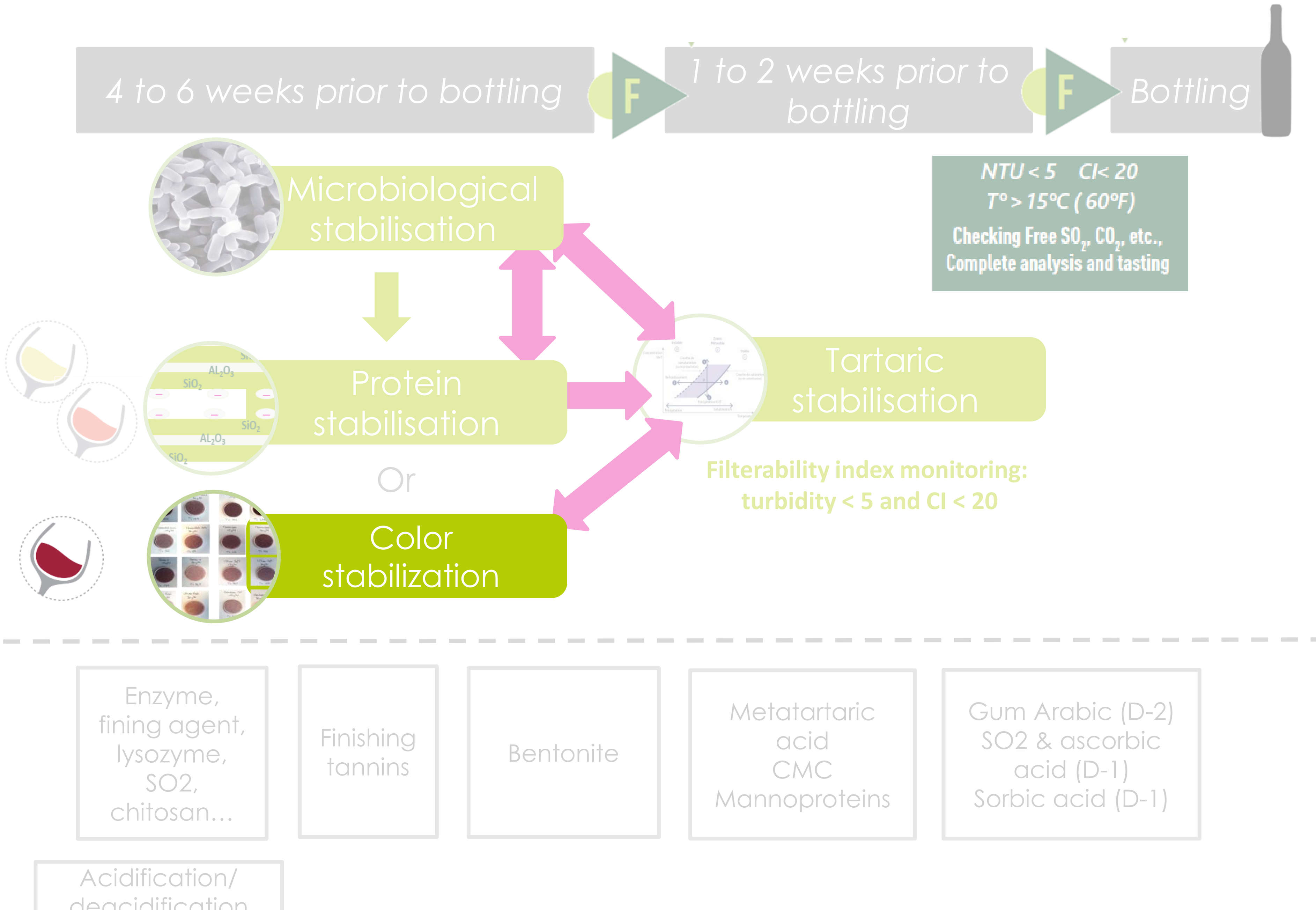


Bentonite specificities

Effect on rose color protection



Stabilisation roadmap: change in mindset



Color Stabilization

Risks linked to absence of stability

COLORING MATTER PRECIPITATION	Part of the colouring material in red wines is in a colloidal state; this fraction can potentially precipitate.
TYPES OF WINE CONCERNED	**Red wines:** ***red coloured aggregate often associated with crystals***
PHENOMENON FAVORED BY: 	- Bottling filtration quality: wine stable before bottling becoming unstable through ***retention of protective colloids in the case of a clogging filtration*** - ***Tartaric instability*** - Wine storage conditions: ***exposure to cold***

A. Is my wine stable in terms of coloring matter?

COLD TEST:

Stability is **estimated** by measuring the turbidity before and after cold storage in the following conditions:

- ✓ Filter 30 mL of wine on a 0.65 µm membrane (+ prefilter).
- ✓ Measure the turbidity of the sample: $\textbf{NTU}_{\text{before cold.}}$
- ✓ Place the sample at ***4°C for 48 hours.***
- ✓ Take out of the cold and, ***after 15 min at room temperature,*** measure the turbidity $\textbf{NTU}_{\text{after cold.}}$

∆ NTU = NTU after cold – NTU before cold

A. Is my wine stable in terms of coloring matter?

Cold test:

Stability is **estimated** by measuring the turbidity before and after cold storage.

Δ turb (NTU)	**< 5 NTU**	**Stable**
Δ turb (NTU)	5-10 NTU	Very slight instability
Δ turb (NTU)	10-20 NTU	Slight instability
Δ turb (NTU)	20-50 NTU	Usual Medium instability
Δ turb (NTU)	> 50 NTU	Strong instability

Coloring matter stabilization

«Amorphous red precipitate»

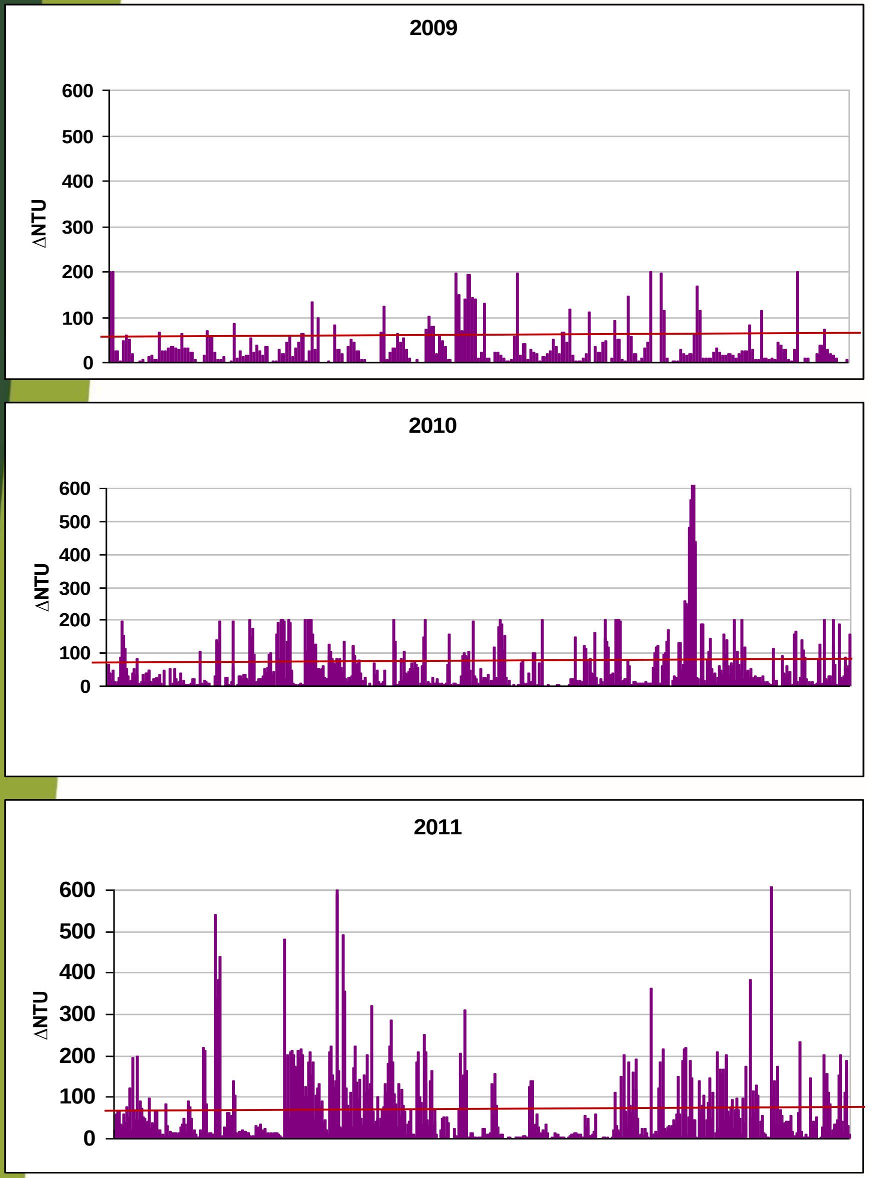
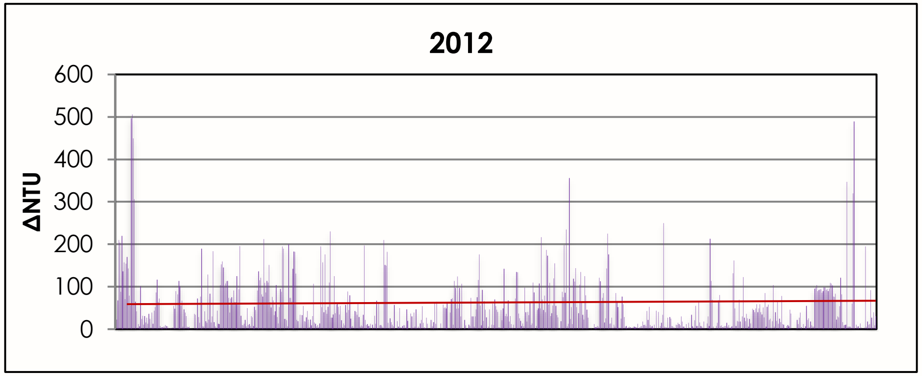


Illustration of the rise of the colouring matter instability



- Reduction of fining dosage mainly driven by organoleptic objective
- Early bottling and cellar thermoregulation preventing stabilization of the colouring matter in a natural way.
- New winemaking practices (thermovinification and flash pasteurisation) which extract more unstable compounds.

Coloring matter

«Amorphous red precipitate»

Identification of coloring matter precipitate

Project MATCOL

Institute of Chemistry and Biology of Membranes and Nano-objects

- *Chemical composition of coloring matter precipitation.*
- *Identify differences among wines from different varieties.*
- *Influence of temperature (+4 and -4 °C) on coloring matter formation*

Stabilization mechanism by Gum Arabic

Project VINARABIC

INRA SPO Montpellier

- *Which macromolecules (fractions) from **Acacia senegal gum** are responsible of stability properties?*
- *Which molecular mechanisms play a role in stabilisation reaction?*

Coloring matter stabilization

«Amorphous red precipitate»

Knowledges dating back from 1979

Phenolic compounds by themselves do not precipitate but they are sensitive to be adsorbed by a colloidal support (polysaccharide or protein).
Glories Y., 1979

Colouring matter could be eliminated by dialysis however it reassembles consequently, more or less fast, according to wine conservation.
Feuillets Œnologiques, 1979

Cold storage of a limpid red wine (filtered or centrifuged) could produce a red amorphous precipitate after two days. Glories Y., 1979

2016 new scientific insights:

Identification of colouring matter precipitate

Project MATCOL

Institute of Chemistry and Biology of Membranes and Nano-objects

- *Chemical composition of coloring matter precipitation: **81% of the precipitate has been identified***
- *Identify differences among wines from different varieties.*
- *Influence of temperature (+4 and -4 °C) on colouring matter formation*

- *Analyse by Nuclear Magnetic Resonance (**NMR**)*

Identification of compounds in coloring matter precipitate

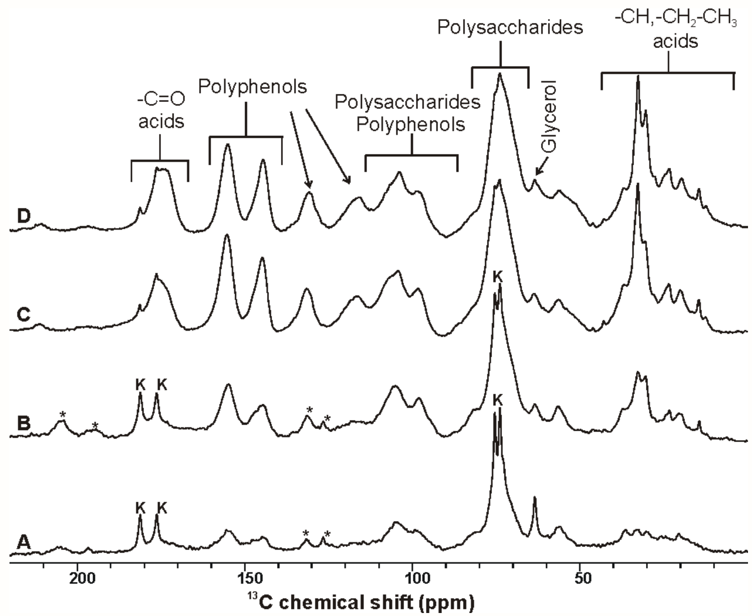
Analysis of coloring matter precipitate obtained after 2 days at 4°C by solid ^{13}C NMR

Cabernet Sauvignon
After 4 months barrel aging

Merlot
After 4 months barrel aging

Cabernet Sauvignon
After 1 month barrel aging

Merlot
After 1 month barrel aging



Precipitates profiles from Cabernet Sauvignon and Merlot show the **same family compounds.**

Barrel aging does not modify the involved compounds in the color matter precipitate.

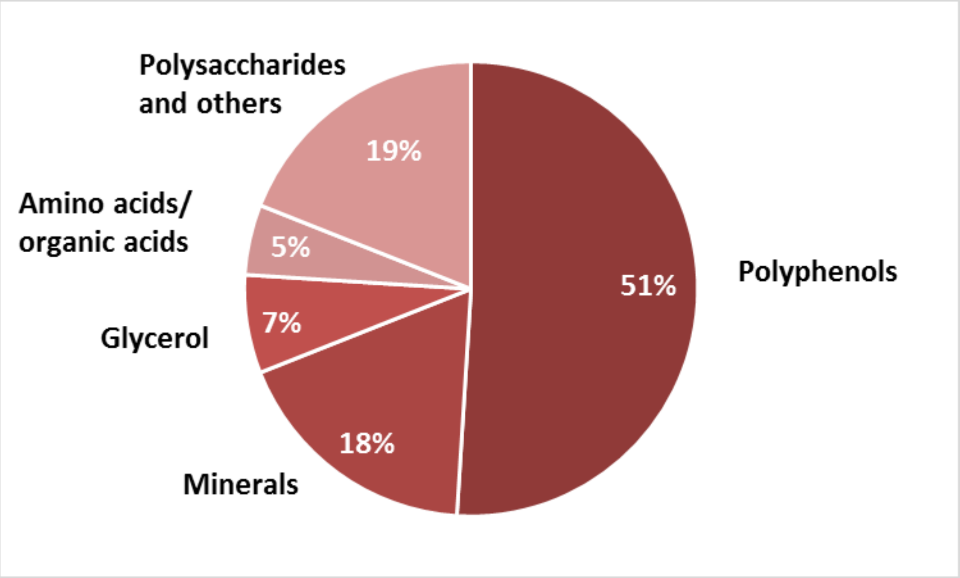
R&D PROJECT: MATCOL

Shipra Prakash, Axelle Grelard & Erick Dufourc (CBMN)

Identification of compounds in coloring matter precipitate

Main goals of this project:

- Identification 81% of precipitate.
- Identification of minerals, meanly potassium, calcium and iron.



Polyphenol fraction (procyanidins and anthocyanins) is in higher amount in the coloring matter precipitate from **Cabernet Sauvignon** than in **Merlot**.

R&D PROJECT: MATCOL
Shipra Prakash, Axelle Grelard & Erick Dufourc (CBMN)

Gum Arabic and properties

- Natural exudate from trees Acacia *senegal* & Acacia *seyal*
- Functional properties: interfacial, stability agent, surface agent.
- In wine, Acacia senegal gum **is responsible of colouring matter stabilisation** and Acacia seyal gum of **organoleptic quality enhancement.**

- Complex hetero-polyoside, charged and hyper branched (AGP family).
- *Macromolecules continuum*
  - *Molecular masse, hydrophobicity (protein concentration) and charge.*
    - **PROJECT VINARABIC : Acacia *senegal gum***

R&D PROJECT VINARABIC
Michaël Nigen, Thierry Doco & Christian Sanchez (INRA SPO Montpellier)

B. What are my options in terms of treatment?

Fining

Cold treatment

- Fining and cold treatment lead to coloring matter precipitation.
- Different effectiveness on stabilization depending of fining agents and wine.
- It reassembles in colloidal state during aging.

Arabic gum

- Stabilization of coloring matter.
- Limited effectiveness over time.

Stabilization mechanism by Arabic gum
Project VINARABIC
INRA SPO Montpellier

UMR SPO
Unité mixte de recherche
Sciences pour l'œnologie

UMR iAte
Ingénierie des Agropolymères & technologies émergentes

- *Which macromolecules (fractions) from* ***Senegal gum*** *are responsible of stability properties?*
- *Which molecular mechanisms play a role in stabilization reaction?*

B. What are my options in terms of treatment?

Fining

Cold treatment

- Fining and cold treatment lead to coloring matter precipitation.
- Different effectiveness on stabilization depending of fining agents and wine.
- It reassembles in colloidal state during aging.

Arabic gum

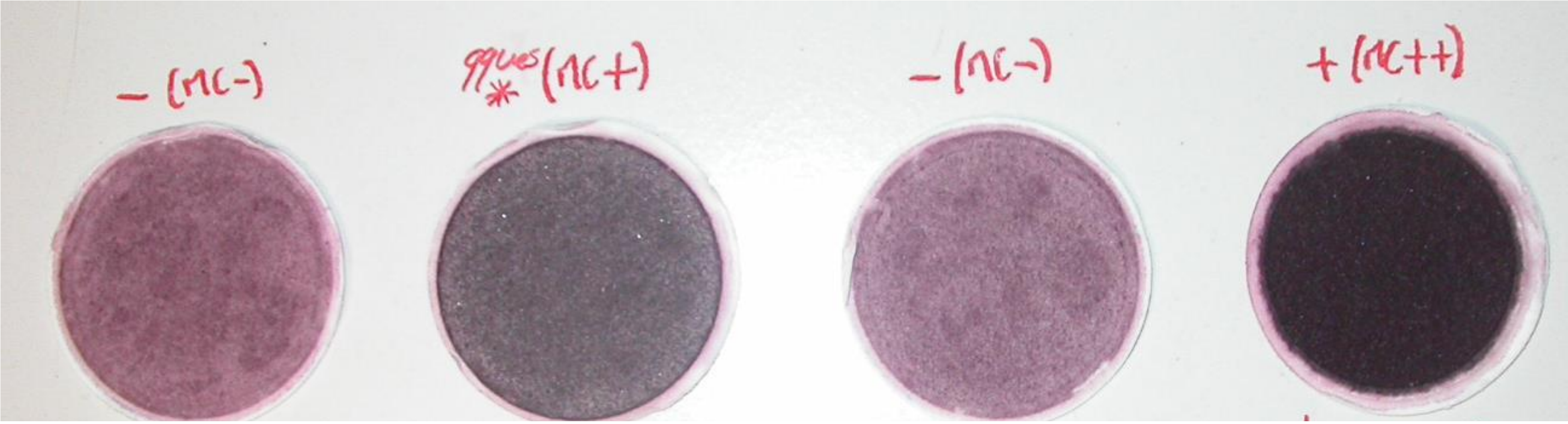
- Stabilization of coloring matter.
- Limited effectiveness over time.

Mannoproteins

- Stabilization of coloring matter.

B. What are my options in terms of treatment?

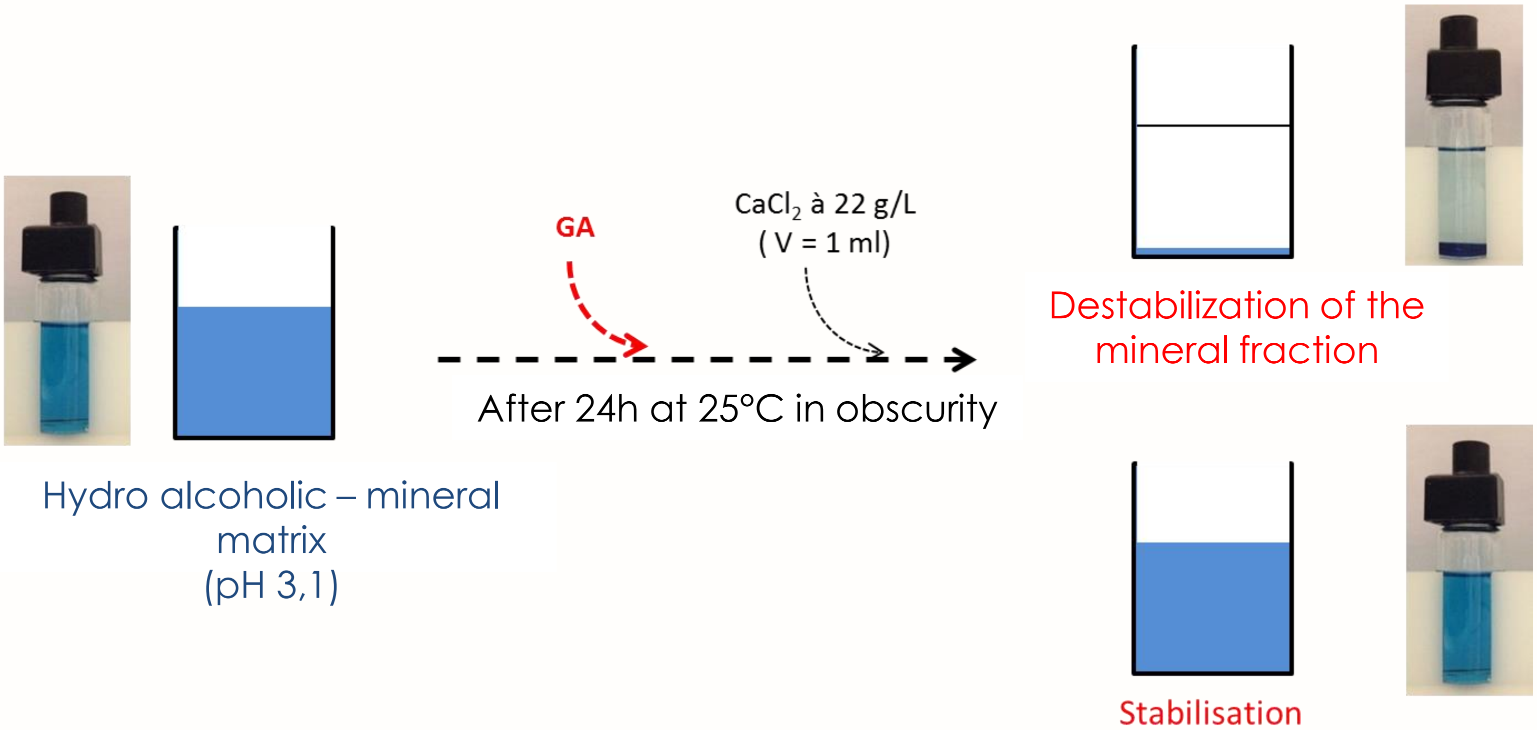
Observations since 2009: general improvement of red wine stability treated with mannoproteins → combined effect on tartaric and coloring matter instability



St Estèphe 2007	**St Estèphe 2007**	**St Julien 2007**	**St Julien 2007**
Treated 15 g/hL	Not treated	Treated 15 g/hL	Not treated

B. What are my options in terms of treatment?

- Methodology for Gum Arabic efficacy evaluation according to the Oenological Codex applied to mannoprotein products



Stabilization Index

Efficacy Test reference for gum Arabic accordingly to OIV COEI-1-GOMARA:2000

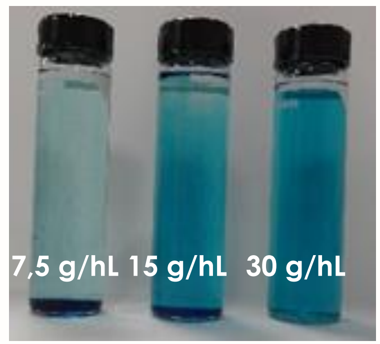
B. What are my options in terms of treatment?

- Methodology for Gum Arabic efficacy evaluation according to the Oenological Codex applied to mannoprotein products

Mannoprotein A

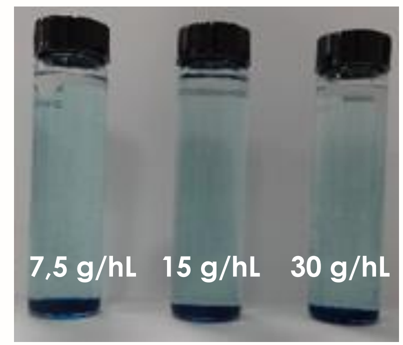
9

Mannoprotein B



6

Mannoprotein C

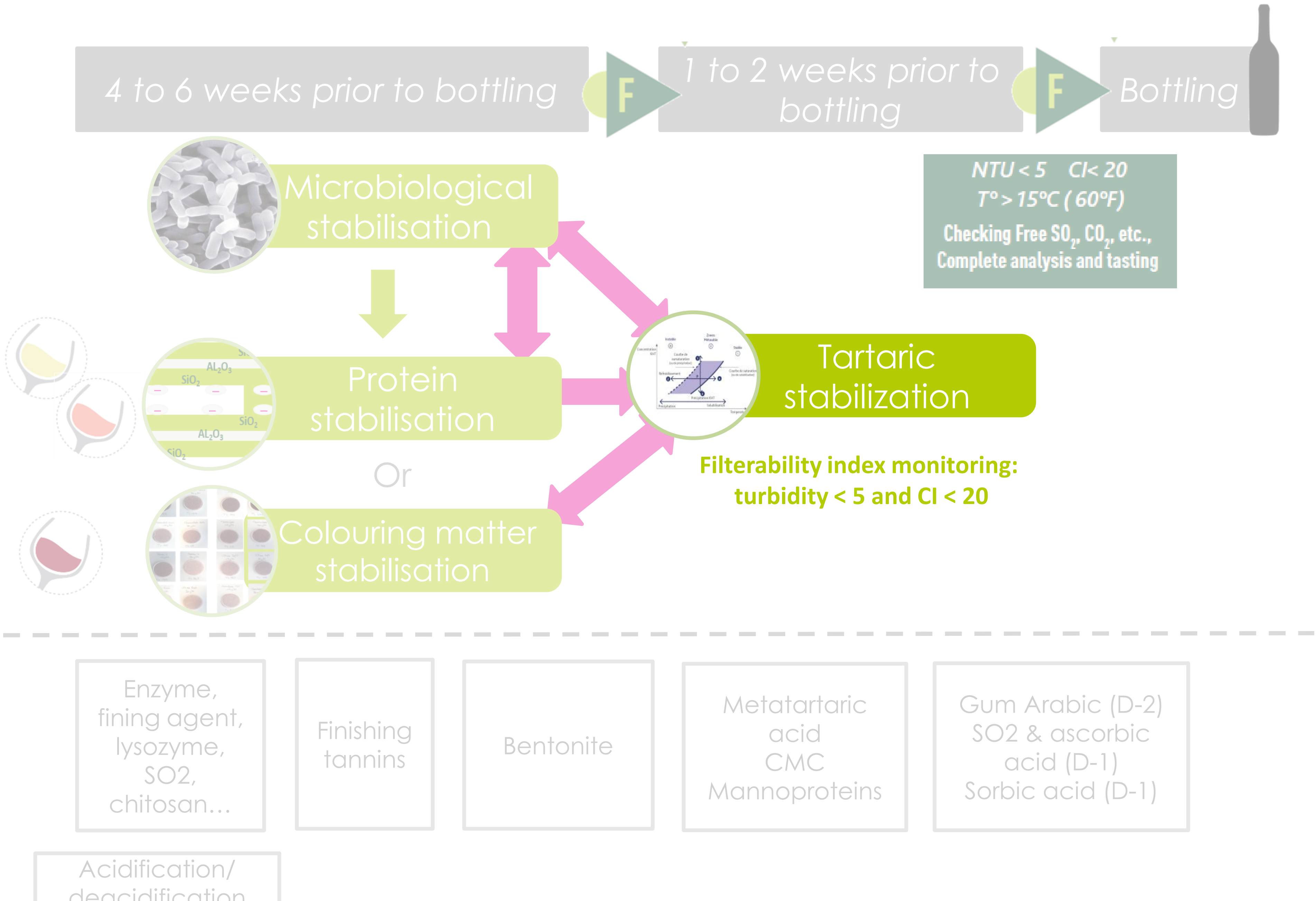


2

Stabilisation of a mineral solution with mannoprotein products at 7.5 g/hL, 15 g/hL and 30 g/hL.

Quantification of the stabilizing power (value 9 is equivalent to the stabilizing power of a Verek or Senegal gum)

Stabilisation roadmap: change in mindset



Tartrate Stabilization

Risks linked to absence of stability

TARTRATE PRECIPITATIONS	At a specific temperature, tartaric acid salts become super-saturated: their concentration is higher than the quantity theoretically soluble. Under cooler conditions this state leads to the formation of crystals .
TYPES OF WINE CONCERNED	**All types of wines: crystal formation**
PRECIPITATION FAVORED BY	- Bottling filtration quality: wine stable before bottling becoming unstable by ***retention of protective colloids due to clogging.*** - Coloring matter instability (blending with younger vintages). - De-acidification treatments before bottling. - Wine storage conditions: ***wine exposure to cold.***

ANALYSES OF WINES SAMPLED ON SUPERMARKET SHELVES:

In 2010

66%

Red 56% / white 72 % / rosé 90 %

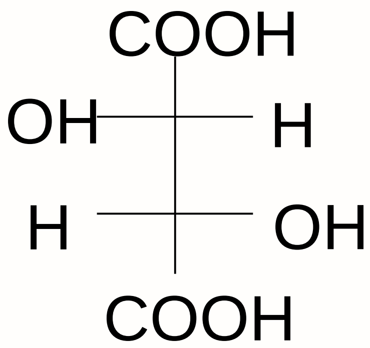
In 2016

59%

Red 72% / white 41 % / rosé 60%

Tartaric acid:

- ✓ Not very commonly found in nature.
- ✓ Acid the most important in grapes, must and wines.



$pKa_1 = 2.97$
$pKa_2 = 4.05$

	Concentration of tartaric acid
Verjus	15 g/L
Must from cooler areas	> 6 g/L
Must from warmer areas	2 to 3 g/L

Tartaric acid and its salts

At wine pH and given the presence of potassium and calcium cations, tartaric acid finds itself mainly in a salt state under 5 forms:

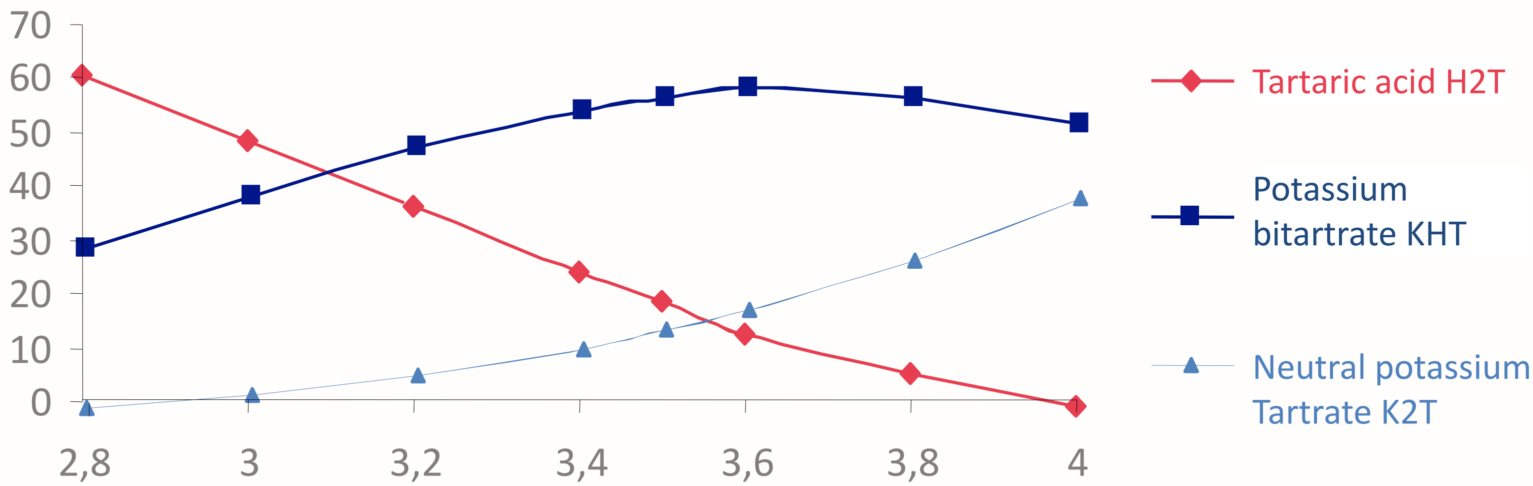
- ✓ Potassium hydrogen tartrate (KHT) (referred to in below table as Potassium bitartrate)
- ✓ Neutral potassium tartrate (K2T)
- ✓ Neutral calcium tartrate (CaT)
- ✓ Potassium and calcium tartrate double salt
- ✓ Mixed salt potassium and calcium tartromalate

Solubility of some tartaric acid salts	In water at 20°C / 68°F	In 10% alcohol at 20°C / 68°F
Tartaric acid	4.9 g/L	-
Potassium bitartrate KHT	5.7 g/L	2.9 g/L
Neutral calcium tartrate CaT	0.53 g/L	-

Tartrate precipitation and pH modification

Free forms of tartaric acid, bitartrate and tartrate percentage, depending on pH:

$$H_2T + K \rightleftharpoons KHT \rightleftharpoons K_2T$$



Remarkable pH (Négre, 1953): 3.59 for wines with 12% v/v alcohol

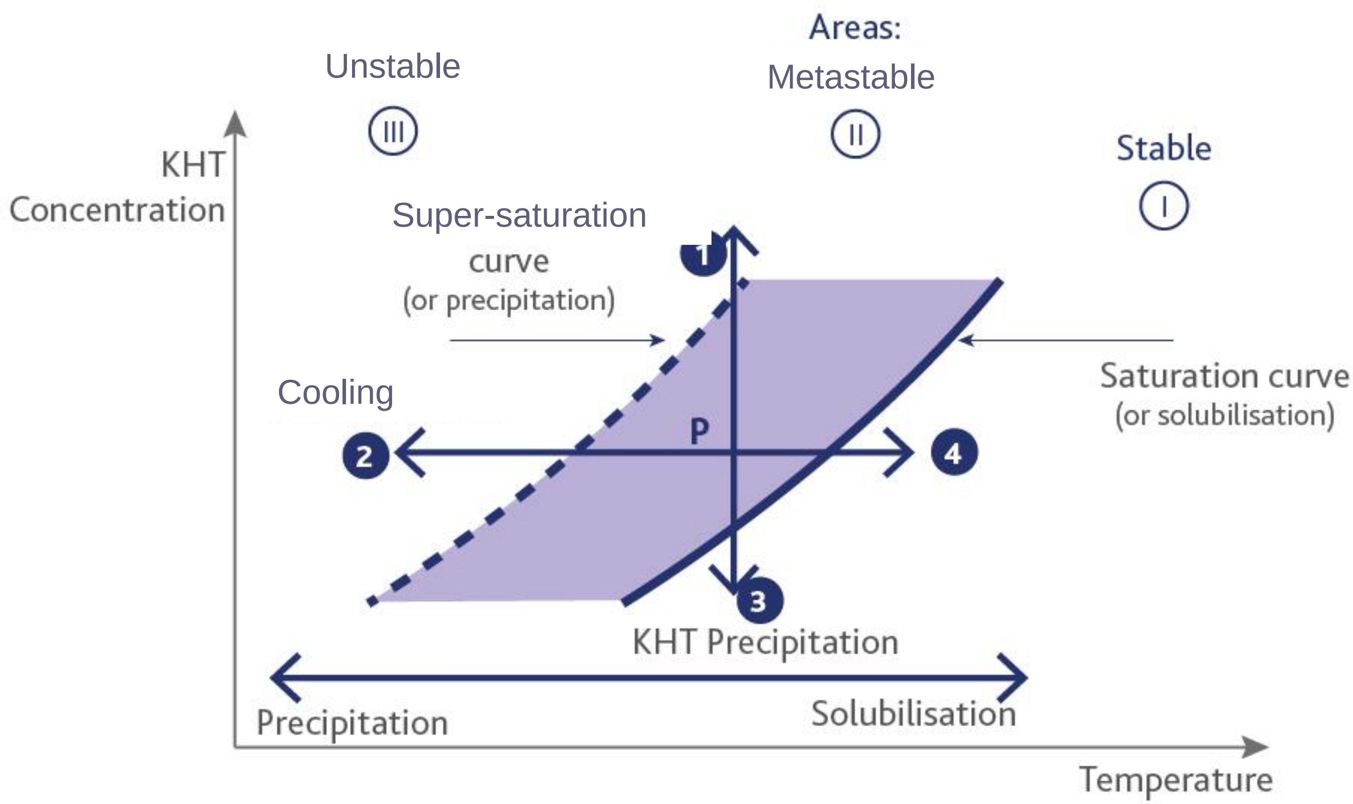
3.53 for wines with 10% v/v alcohol

pH 3.6: KHT precipitation fast and abundant

pH < 3.6: KHT precipitation lowers pH

pH > 3.6: KHT precipitation increases pH

Tartaric salt state diagram



Potassium bitartrate states in wine

Tartaric salt supersaturation range

Protection effect of some molecules towards tartaric acid salts crystallisation
(after Maujean et al., 1985)

	Super-saturation window SaturationTemp- Supersaturation Temp
Control	20.80
Bentonite (30 g/hL)	18.20
Decolorizing carbon (30 g/hL)	19.75
Gum arabic (10 g/hL)	20.60
Metatartaric acid (6.6g/hL)	>23.00
Membrane filtration 10000 Da	14.05

A. What are my options in terms of treatment?

Subtractive methods: physical techniques

Removing constituents responsible for precipitation

Cold treatment *(with or without seeding)*:
Preventive KHT precipitation

Electrodialysis: selective removal of K+ ions

Inhibition methods

Inhibition of KHT crystal nucleation and/or growth phase

Additives:

- ✓ Sodium Carboxymethylcellulose (CMC): **CELSTAB®**

Ingredient naturally occurring in wine:

- ✓ Yeast mannoproteins- **MANNOSTAB®**

B. Is my wine stable from a tartaric standpoint?

Review of tartaric stability tests

1. Reference test: Crystallization test

- ✓ Filter 250 mL of wine on a 0.65µm membrane.
- ✓ Place the wine at -4°C / 25°F for 6 days.
- ✓ Visual reading after 6 days:
  - ***White wine:***
    - Absence of crystals (stable wine)
    - Presence of crystals: perform chemical identification tests
  - ***Red wine:***
    - Filter the wine and look for the presence of crystals and/or coloring matter

✓ The *crystallization test* models the natural phenomenon of tartaric precipitations in bottle (takes into account the matrix and the presence of protective colloids). Instability factors: temperature, filtration.

B. Is my wine stable from a tartaric standpoint?

Review of tartaric stability tests

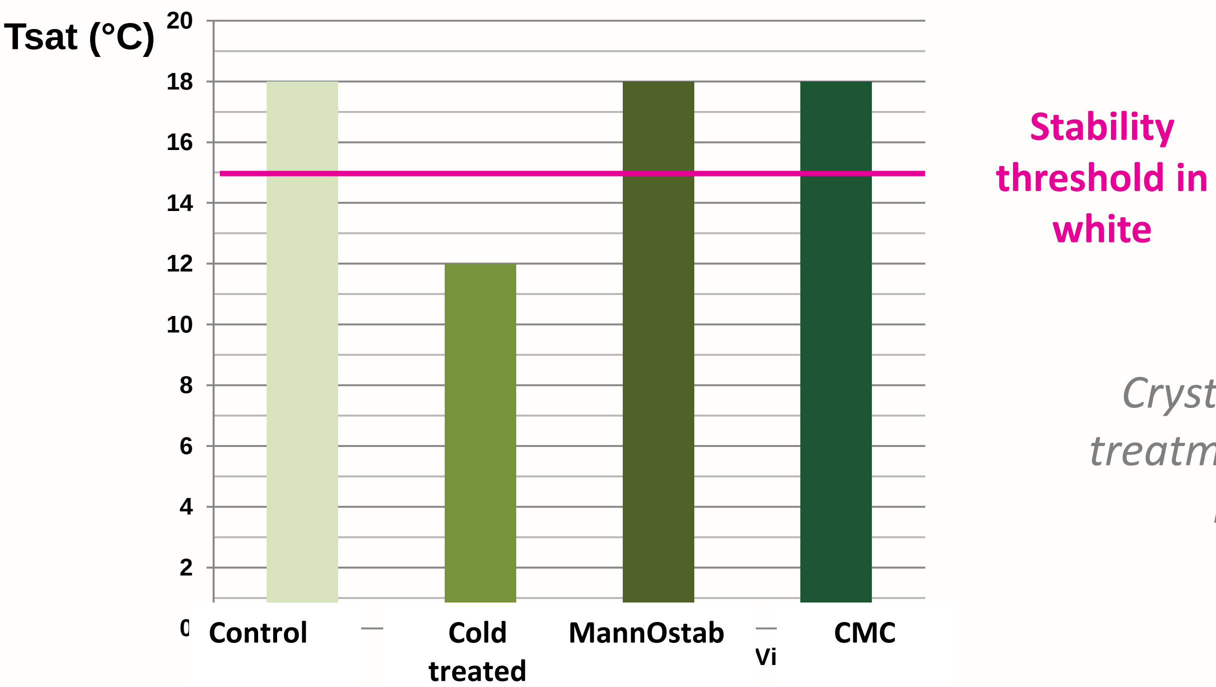
2. Saturation temperature (Tsat)

Temperature at which KHT can be dissolved in wine

- White or rosé wine: stable if ------ Tsat < 15° C
- Red wine: stable if ------ Tsat < 21° C

✓ *Measures the wine state and its potential tartaric stability level*

✓ *Not appropriate to validate a treatment with crystallization inhibition techniques*



*Crystallization inhibition treatments **do not increase KHT solubility.***

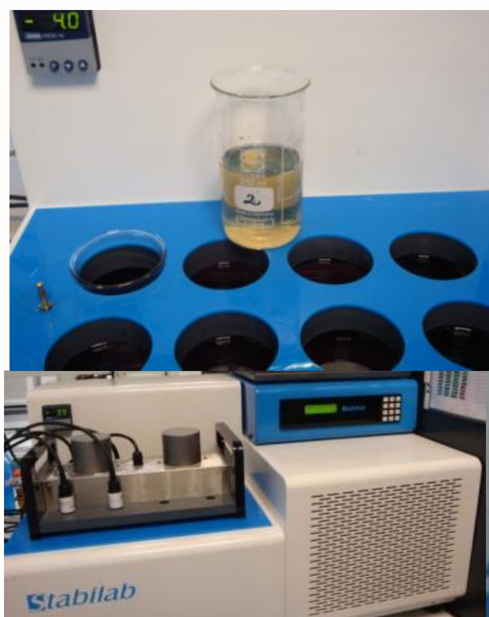
B. Is my wine stable from a tartaric standpoint?

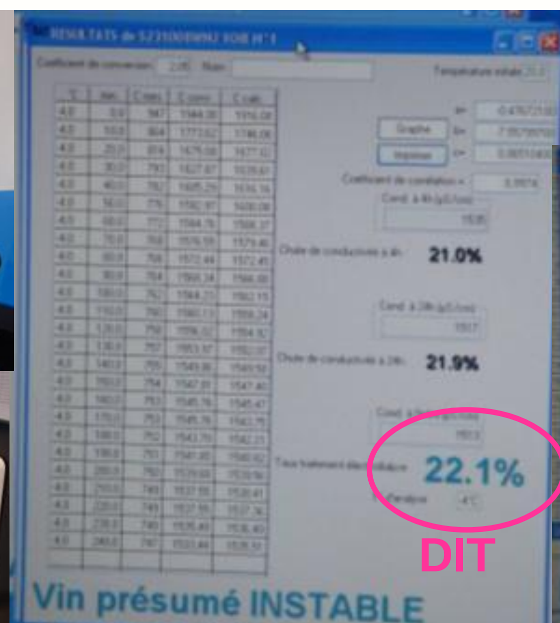
Review of tartaric stability tests

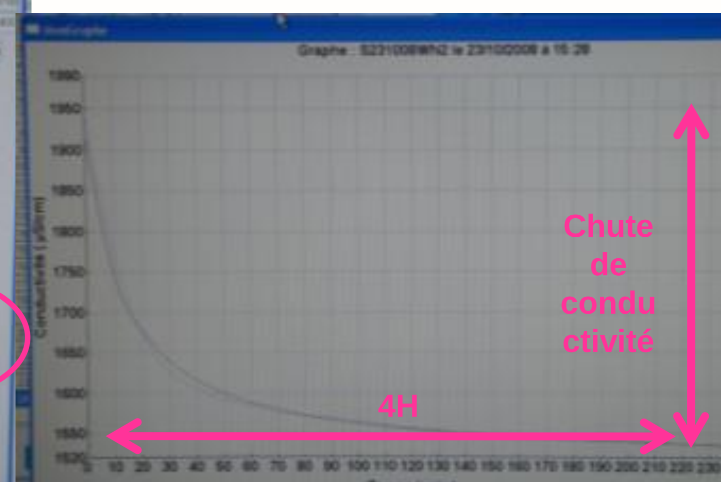
Quantify the tartaric instability: *conductivity test*

3. DIT measure: Degree of Tartaric Instability (DIT%)
equivalent to a mini-contact test at 4hrs, -4°C / 25°F, + 4 g/L KHT

Definition of a de-ionisation rate to ensure stability (0 to 30%)
(Measurement device **STABILAB®** - Eurodia/INRA Patent)







B. Is my wine stable from a tartaric standpoint?

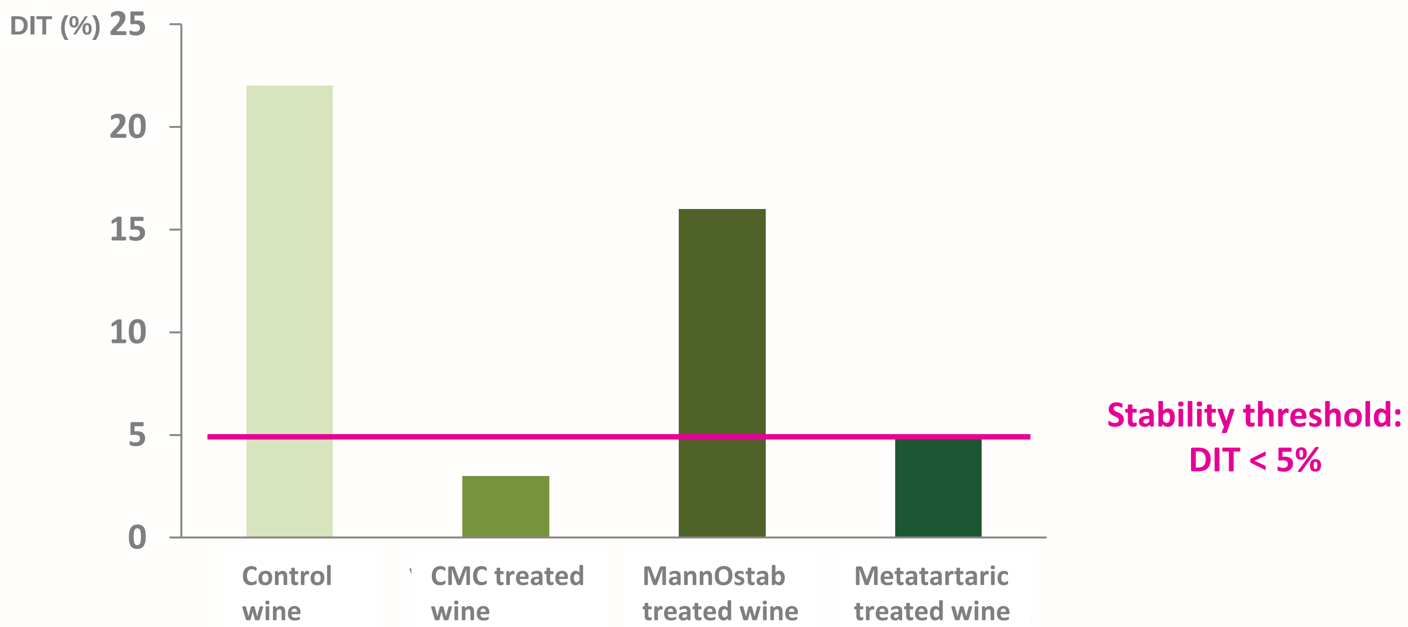
Review of tartaric stability tests

3. DIT (Degree of Tartaric Instability): *STABILAB®* *Eurodia/Inra Patent*

Measure of the conductivity drop to infinity, after addition of cream of tartar in excess

Test -4˚C/ 25˚F, 4g/L cream of tartar, 4 hours

- ✓ *Measures the wine state and its potential tartaric stability level*
- ✓ ***Detects the presence of crystallization inhibitors***
- ✓ *BUT cannot validate an inhibition treatment, especially if instability is high.*



B. Is my wine stable from a tartaric standpoint?

Review of tartaric stability tests

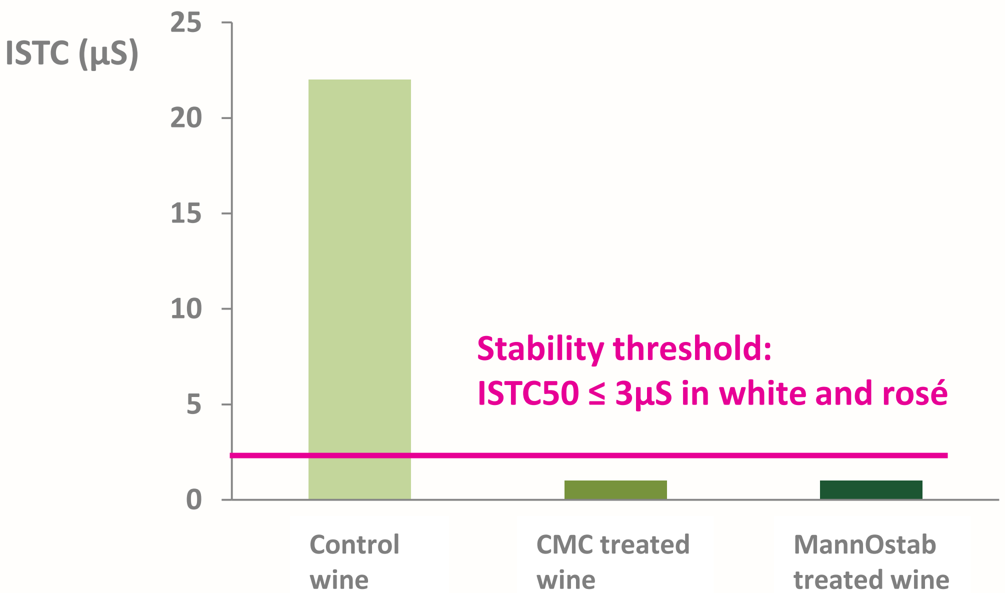
4. ISTC50 (Critical Tartaric Stability Index): *STABILAB®* *Eurodia/Inra Patent*

*Measurement of the conductivity variation on a wine **considered stable**:*

- Dissolve 0.5g/L ultra-purified cream of tartar at 37˚C/ 99˚F in the wine
- Measure conductivity at -4˚C/ 25˚F,
- Then, measure conductivity over 4 hours
- (2 critical stabilization factors : saturation of cream of tartar and temperature)

✓ ***Validates a crystallization inhibition treatment for white and rosé wines***

✓ ***Equivalent to crystallization test 6 days / -4˚C (white wine)***



<u>In red wines</u>: the crystallization is the appropriate test due to the possible interaction between coloring matter stability and tartaric stability.

C. How do I choose the appropriate treatment?

Tartaric instability level & type of wine

Potential tartaric stability state: DIT (%)

Stability threshold (white, rosé, red wine): < 5 %
(under our lab measurement conditions)

DIT Value (%)	> 20	> 20	< 20
Category of wine	Basic / premium Early consumption	Basic / premium Early consumption	Super Premium – ageing wines (6 months minimum)
Recommended treatment	**POLYTARTRYL®**	**CELSTAB®**	**MANNOSTAB®**
Maximum legal dosage (g/hL)	10	10	-
Treatment dosage (g/hL)	10	10	10 - 30
White wines	Direct treatment	Direct treatment	Natural stabilisation of white, rosé and red wines
Red & rosés wines	Direct treatment	Risk of interaction with colouring matter: haze and/or crystal formation	

D. How do I check the efficacy of the treatment?

Did the elected treatment stabilize my wine?

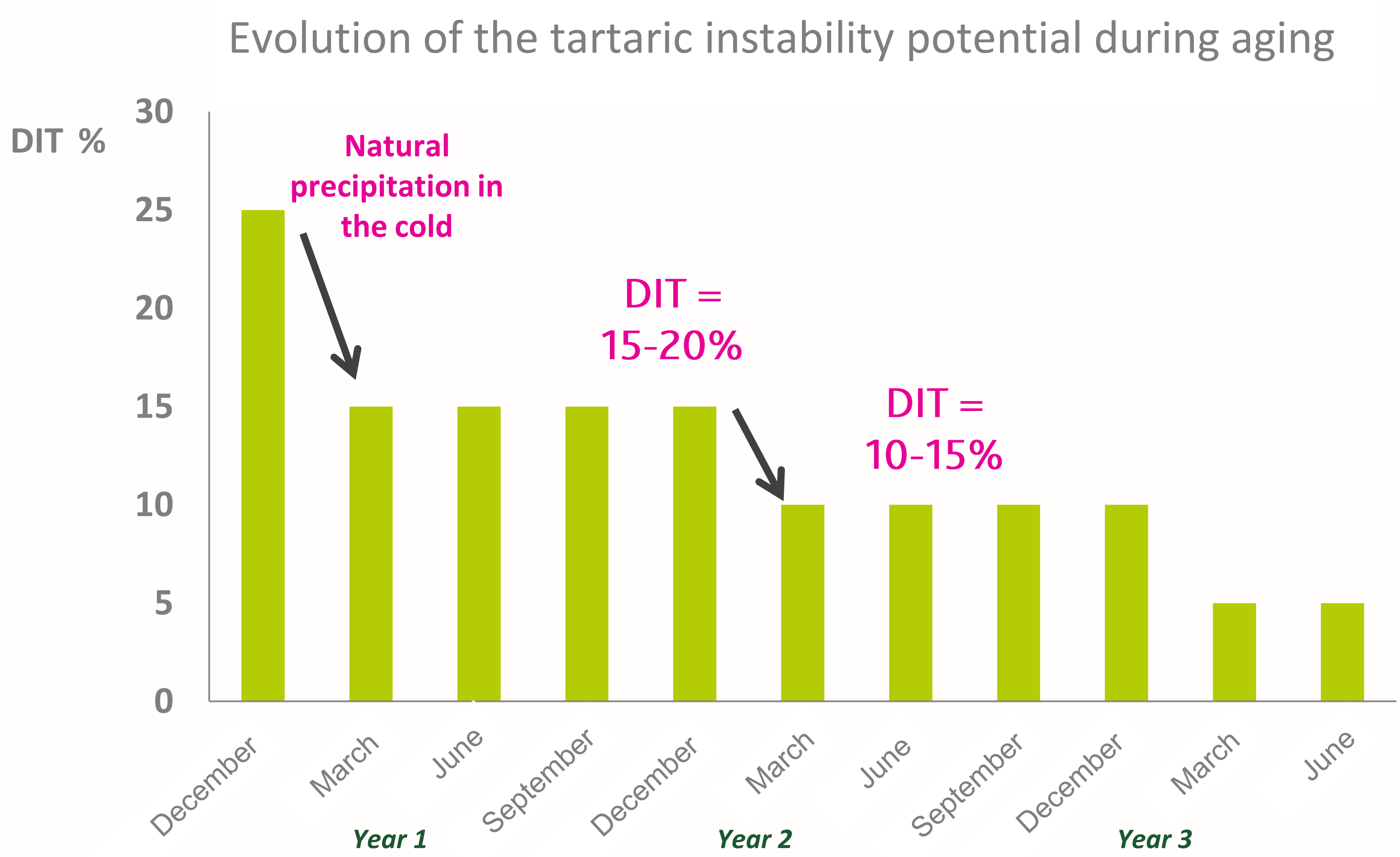
DIT Value (%)	> 20	> 20	< 20
Category of wines	Basic / premium Early consumption	Basic / premium Early consumption	Super Premium – ageing wines
Treatment to validate	**POLYTARTRYL®**	**CELSTAB®**	**MANNOSTAB®**
White wines	**ISTC50 / CHECKSTAB**		
Rose wines	**ISTC50 / CHECKSTAB**	**Crystallisation test***	**ISTC50 / CHECKSTAB**
Red wines	**Crystallisation test***	Not recommended	**Crystallisation test***

CHECKSTAB validation : we have observed an overestimation of the mannoprotein dose necessary for the stabilization

***Tartaric stability is linked to colouring matter stability. The crystallisation test is the only test taking into account the potential interaction between both stabilities.**

E. What are the parameters influencing tartaric stability?

In the case of non temperature controlled cellars, the winter cold leads to spontaneous tartaric precipitations reducing therefore the instability potential of aging wines.



E. What are the parameters influencing tartaric stability?

Ageing on lees improves tartaric stability

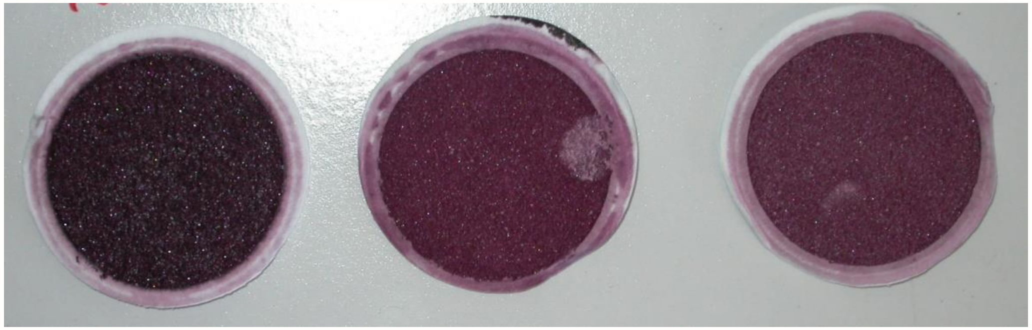
Graves white wine	Before treatment	After cold storage	After 10 months on lees
Tsat	21	16	18
Crystallization (visually)	**	0	0
mini contact	120	30	20
Tcs	-6	-18	-24

After Moine-Ledoux, 1996

E. What are the parameters influencing tartaric stability?

Coloring material stability (red wines)

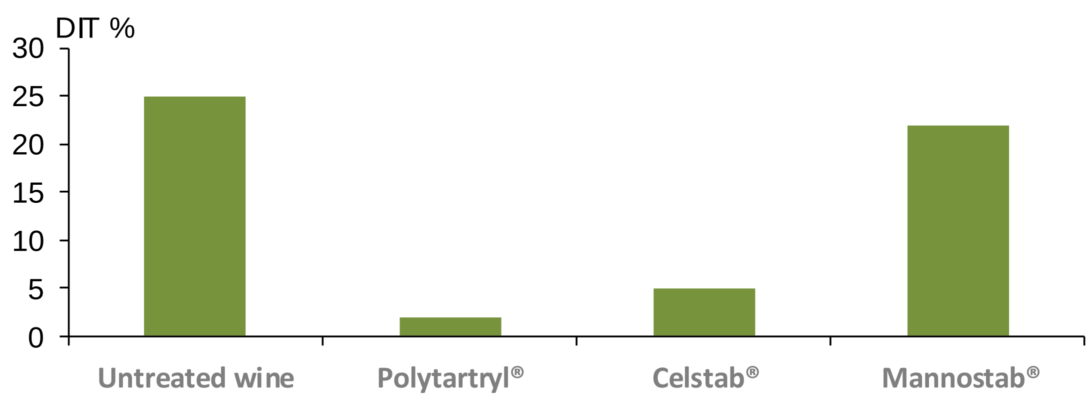
If the wine shows a very high coloring matter instability (young wines, wine blended with younger vintages, inappropriate fining), there is a risk of tartaric precipitation, caused by the precipitation of colloidal coloring matter.



Wine	2008 fined wine	2008 wine + 10 g/hL **MICROCOL® ALPHA** bentonite
DIT %	19.1 %	9.7%
MANNOSTAB® Dose	No stabilization	15 g/hL

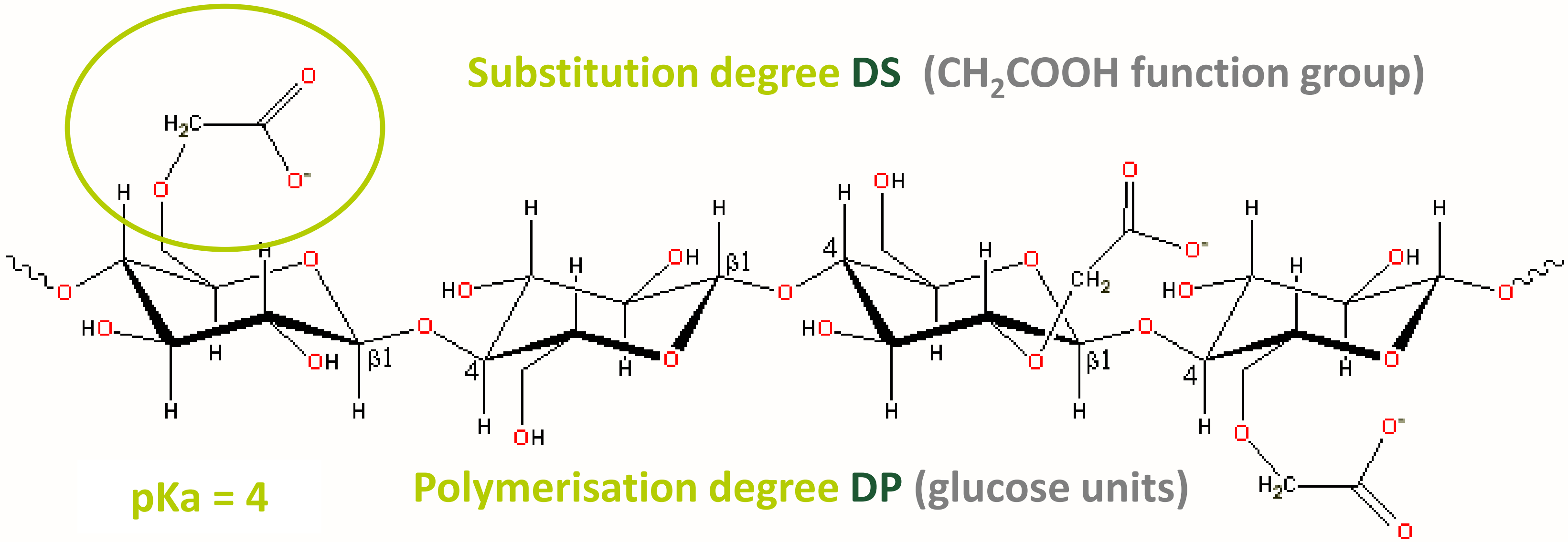
Metatartaric acid: POLYTARTRYL®

- ✓ Metatartaric acid has been ***authorised since 1956.***
- ✓ At this time, the LAFFORT® company has developed its production in collaboration with Emile Peynaud.
- ✓ This tartaric acid polyester is the strongest inhibitor of potassium bitartrate.
- ✓ It hydrolyses over time; the higher the temperature, the faster the hydrolysis.



E466 Cellulose gums (CMC): CELSTAB®

Substitution degree **DS** (CH_2COOH function group)

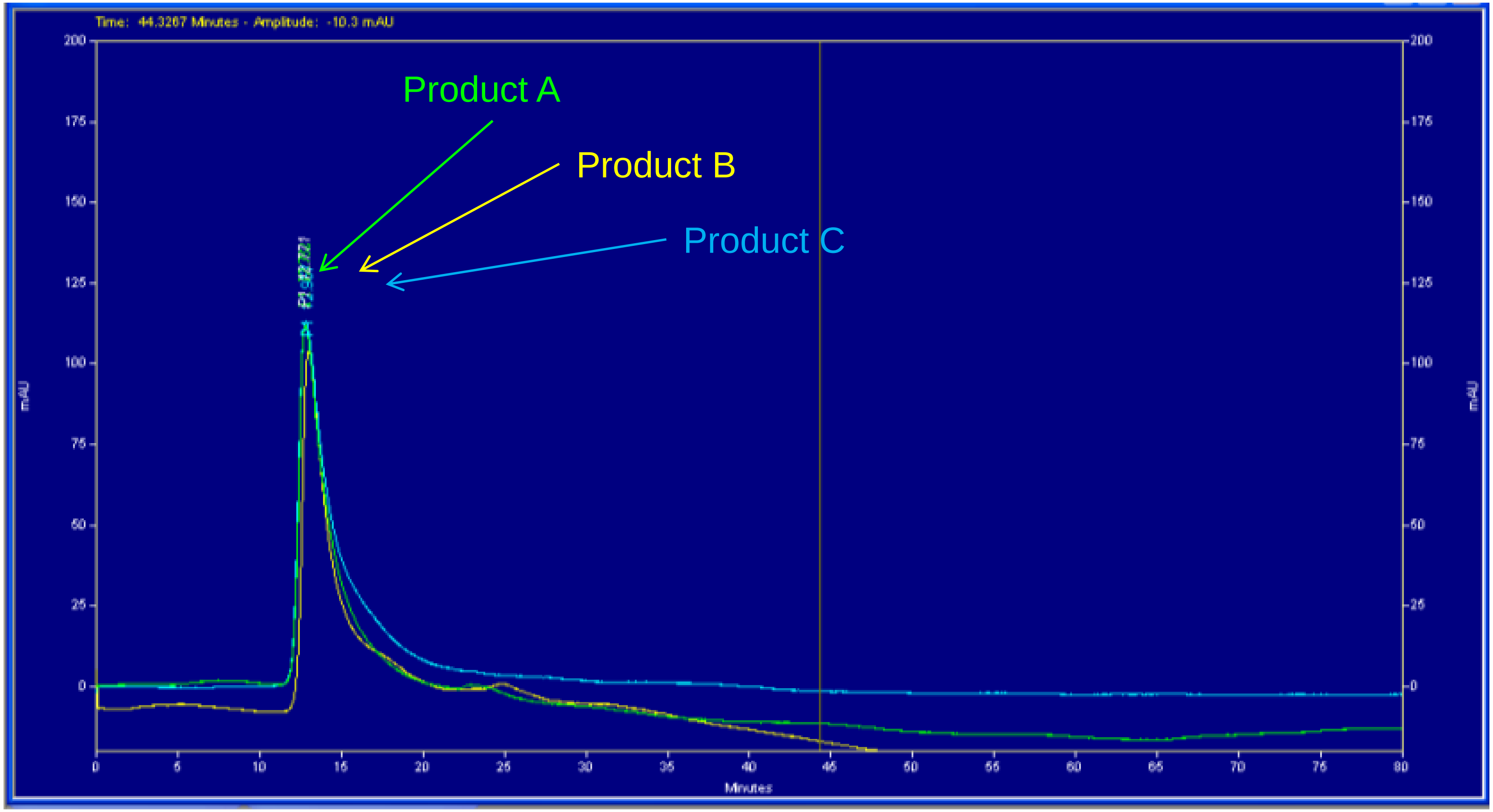


pKa = 4

Polymerisation degree **DP** (glucose units)

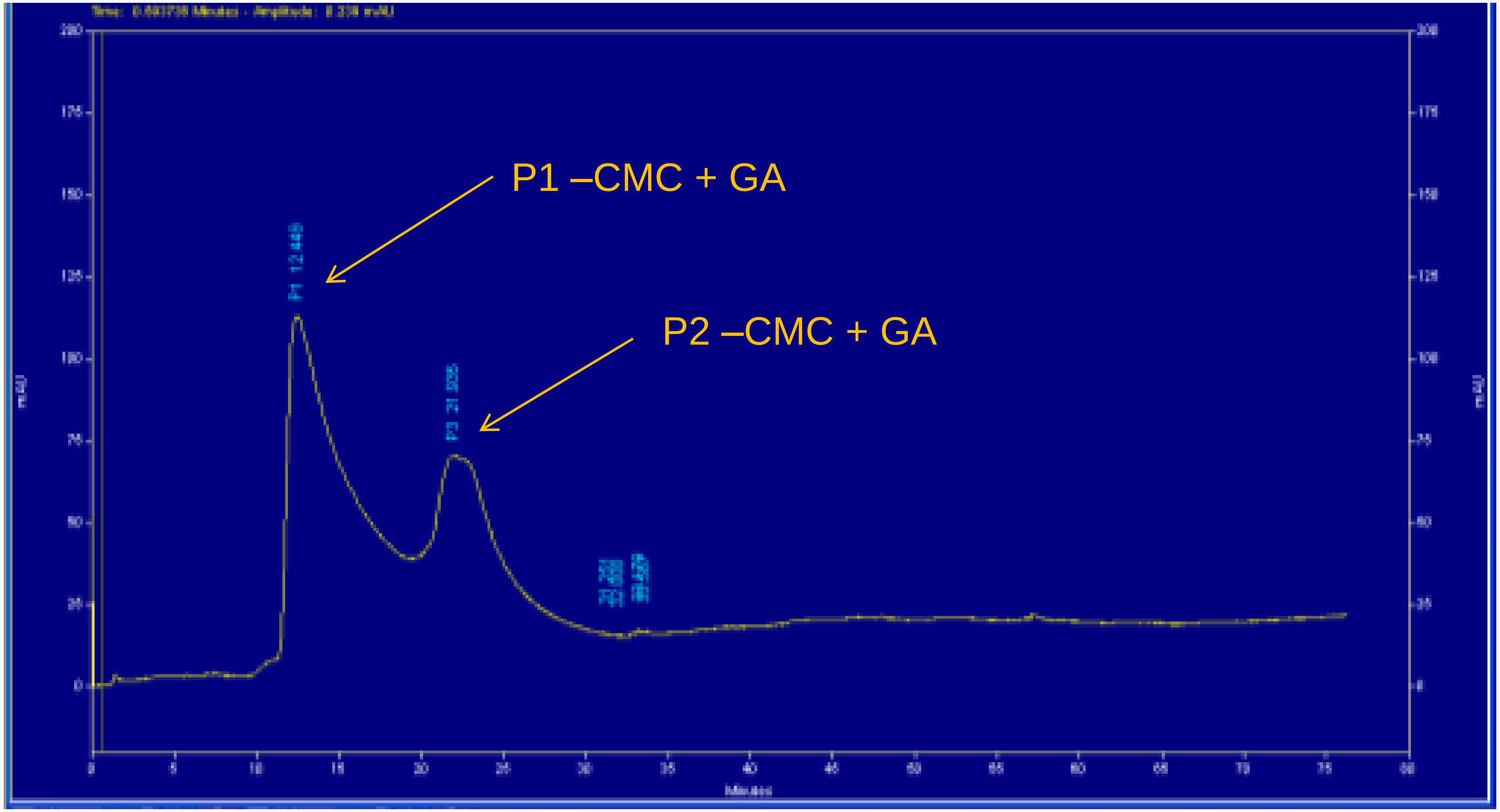
- ✓ Coupled with Na^+ ion: “Sodium CMC”
- ✓ Characterised by:
  - *DS = substitution degree (carboxymethyl groups)*
    ➔ ***EFFICACY***
  - *DP = polymerisation degree (glucose units)*
    ➔ ***VISCOSITY***

E466 CMC HPLC profile
One single peak confirms the product’s purity

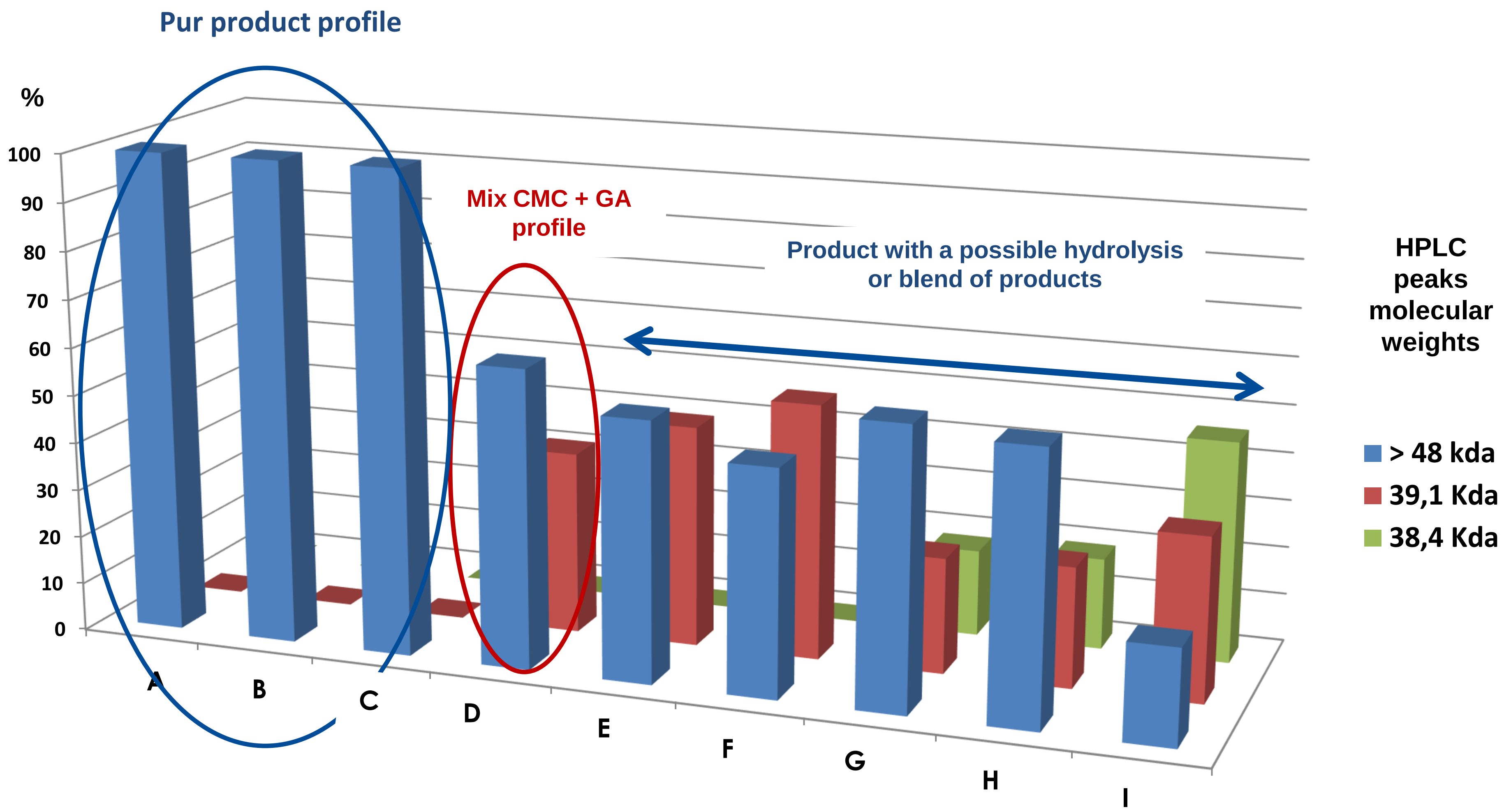


CMC + GA HPLC profile
2 peaks = 2 products

P1 > 48 Kda
P2 = 39 Kda



Benchmark – CMC market product HPLC profiles



F. How do I perform the treatment in the cellar? CELSTAB

Celstab® Treatment:

Before final bottling filtration on a wine fined and clarified (Clogging Index < 20, Turbidity < 5 NTU)

Dosage: 1 mL/L (CELSTAB® is a 10% solution)

Implementation:

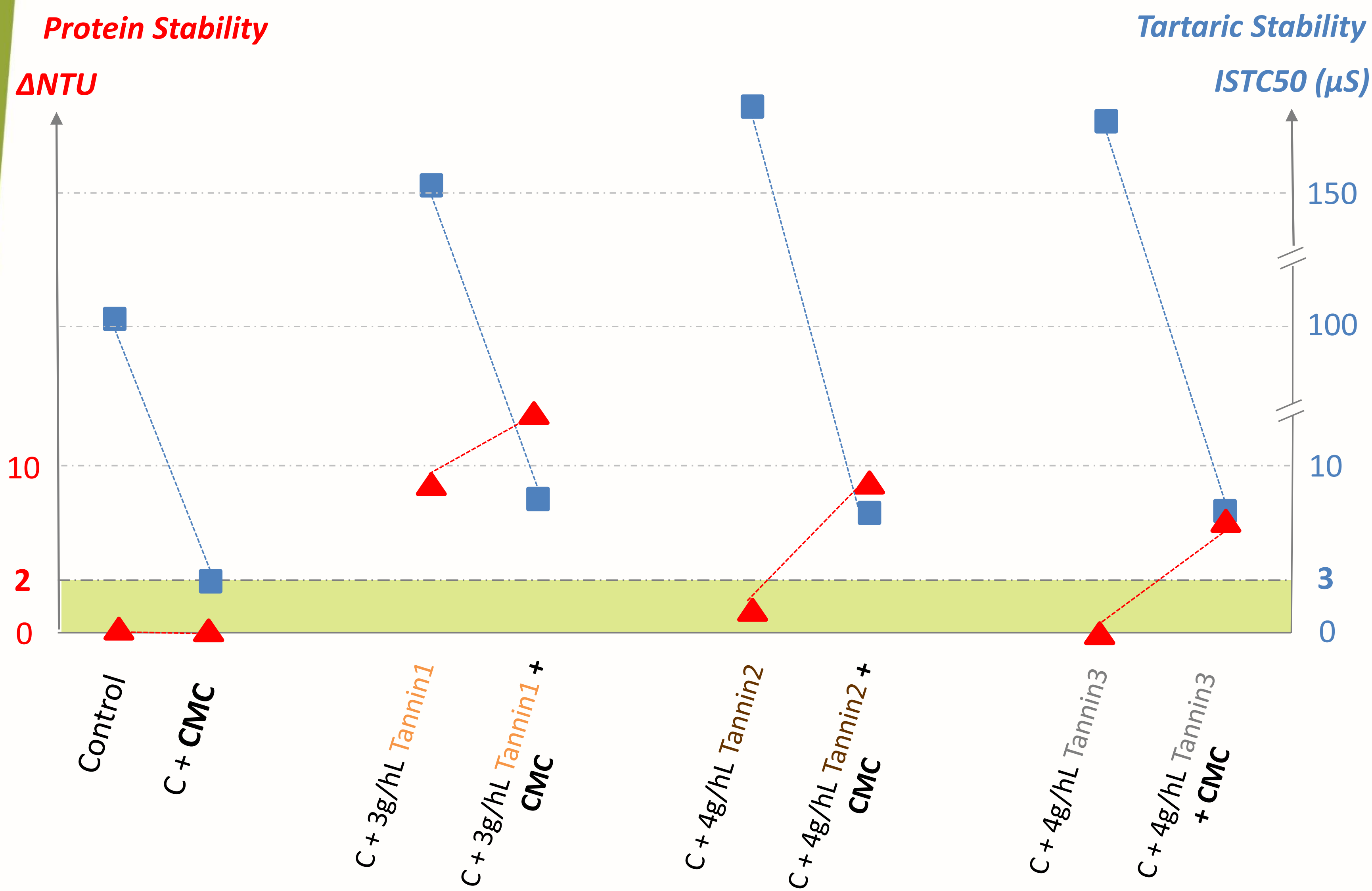
Dilute the solution in twice its volume of wine.

- **Still wines:** incorporation using a dosage pump or an Oenodoseur 48 hours before bottling.
- **Sparkling wines**: incorporation at tirage

Enological conditions:

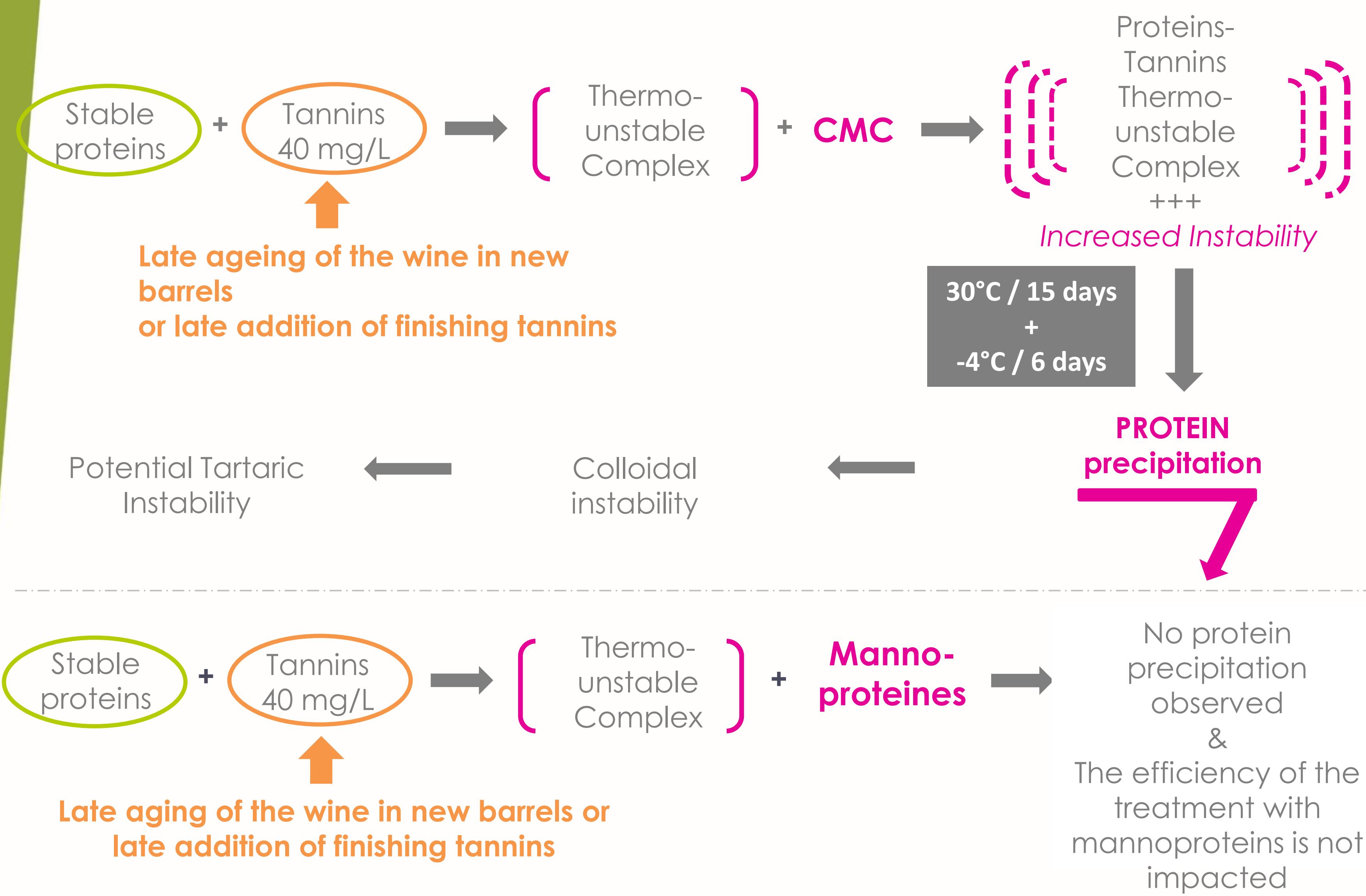
- ✓ Use **CMC** on **protein stable wines.**
- ✓ CMC forms a **haze on wines treated with LYSOZYM®.**
- ✓ CMC forms a **haze with tannins.**

Impact of the addition of tannins on a wine treated with bentonite regarding protein and tartaric stabilities



Addition of CMC = 1 mL/L

Impact of the addition of CMC or Mannoproteins on wines treated with tannins during tartaric stabilization



Impact of CMC implementation on clogging index and coloring matter

STABILIZATION

> Tartrate Stabilization

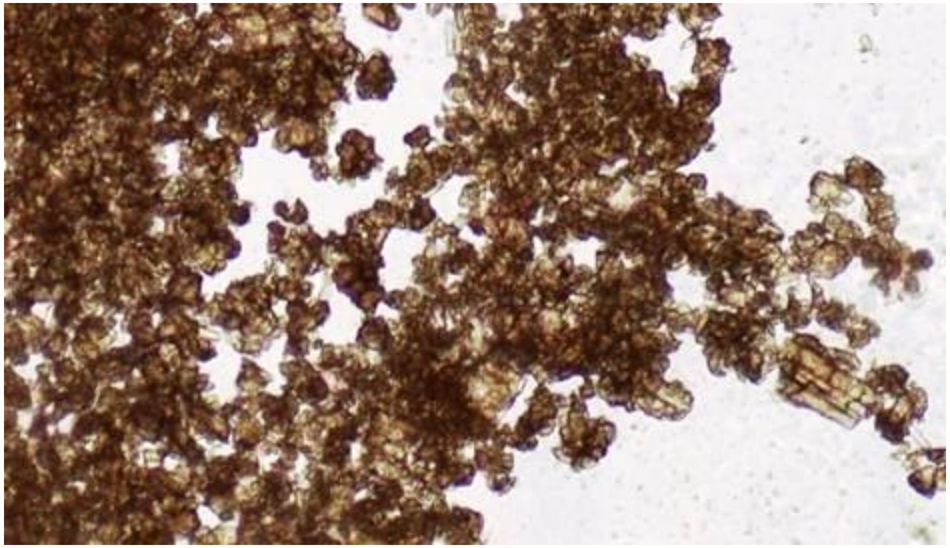
CELSTAB®: Results on rose wines

Wine	Parameter	Control	CELSTAB® 10 cL/hL	CELSTAB® 10 cL/hL STABIVIN® 2.5 cL/hL	CELSTAB® 10 cL/hL STABIVIN® 5 cL/hL	CELSTAB® 10 cL/hL STABIVIN® 10 cL/hL
Wine 1: Mesterrieux Coop (rosé)	Turbidity	0.3	0.3		0.3	0.3
	Clogging index	7	7	8	8	8
	DIT	18.50%	3.70%	3.70%	3.30%	2.90%
	Crystallisation	+ (mc -)	- (mc-)	- (mc-)	- (mc-)	- (mc-)
	Protein stability	Made stable (45g/hl)	Stable	Stable	Stable	Stable
	DO280	10.62	10.56	10.42	10.64	10.54
	ICM	0.667	0.760	0.764	0.734	0.804
Wine 2: Lafon (rosé)	Turbidity	1.6	1.6	1.6	5.7	5.7
	Clogging index	27	27	32	Clogging	Clogging
	DIT	21.80%	3.10%	2.60%	1.40%	0.80%
	Crystallisation	+ (mc-)	- (mc-)	- (mc-)	**- (haze)**	**- (haze)**
	Protein stability	Stable	Stable	Stable	Stable	Stable
	DO280	**13.42**	13.04	12.74	13.44	13.00
	ICM	0.604	0.622	0.606	0.795	0.775
Wine 3: Sauveterre Coop (rosé)	Turbidity	0.2	0.5	0.5	0.6	0.7
	Clogging index	7	7	7	7	7
	DIT	20.40%	5.30%	5.20%	4.20%	4.10%
	Crystallisation	+ (mc-)	**- (haze)**	**- (haze)**	**- (haze)**	**- (haze)**
	Protein stability	Stable	Stable	Stable	Stable	Stable
	DO280	**13.62**	13.08	13.02	13.02	13.00
	ICM	0.628	0.746	0.728	0.729	0.721

CELSTAB®

Results on rosé wines

Identification of haze:



Crystals of a very particular shape, brown colour.

Presence of K, Ca and tannins.

CELSTAB® on rosé wines

On 15 rosé wines from different origins:

- ✓ 7 can be stabilized with CMC (46.6%).
- ✓ 8 cannot be stabilized with CMC (53.3%).
- ✓ The wines not advised for CMC treatment show a high polyphenol index (IPT) or a weak clogging index.
- ✓ After treatment they all show a good DIT or ISTC50, but they all have the same haze with the crystallisation test (6 days at -4°C).
- ✓ The addition of stabilizing arabic gum has no effect on this haze.
- ✓ No increase of protein instability is noticed.
- ✓ When the clogging index is good, it remains good after addition of **CELSTAB®** and **STABIVIN®**.

Results of these trials confirm that ***a feasability test in the lab*** is necessary on rosé wine before a **CELSTAB®** ***treatment.***

Trials on red wines

Coloring matter interaction risk

Crystallization test results (6 days at -4°C):

Bordeaux Sup Rouge 2008 + 10g/hL CMC A, B and C

Reading on pre-filters after 6 days:

- Absence of crystals: stable wine from KHT point of view
- But: significant sediment of coloring matter – reaction under cold conditions

Reading on pre-filter / test 6d -4°C
wine treated with 10g/hL CMC
Highlights the reaction CMC – colouring matter in the cold (-4°C)

Reading room temperature controls on pre-filter
Wine treated with 10 g/hL CMC
Absence of coloring matter sediment

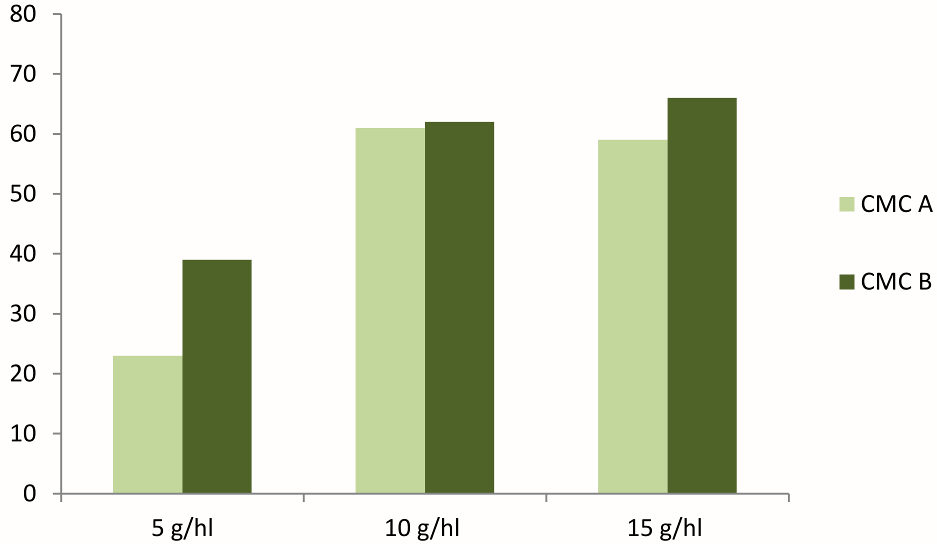
Trials on red wines

Coloring matter interaction risk

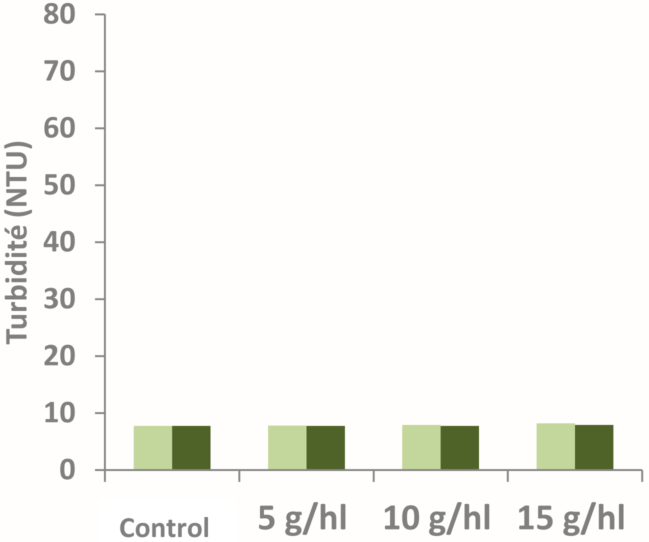
Results of crystallisation tests 6 days -4°C:

Bordeaux Sup Rouge 2008 + dose 5, 10 and 15 g/hL CMC

Highlighting the formation of a haze at -4°C / 25°F



Turbidity after 6 days at -4°C / 25°F according to CMC dose



Turbidity at room temperature according to CMC dose

Cold temperatures catalyse haze formation "CMC – coloring matter"
Trials beforehand are necessary to evaluate the risk of interaction with coloring matter, prior to any CMC treatment on red and rose wines

F. How do I perform the treatment in the cellar? MANNOSTAB

MANNOSTAB® TREATMENT: **Before final bottling filtration on a wine fined and clarified (Clogging Index < 20, Turbidity < 5 NTU)**

Dosage: **100 – 300 mg/L** according to a test, or according to the aging period

Implementation: *(check out the video!)*

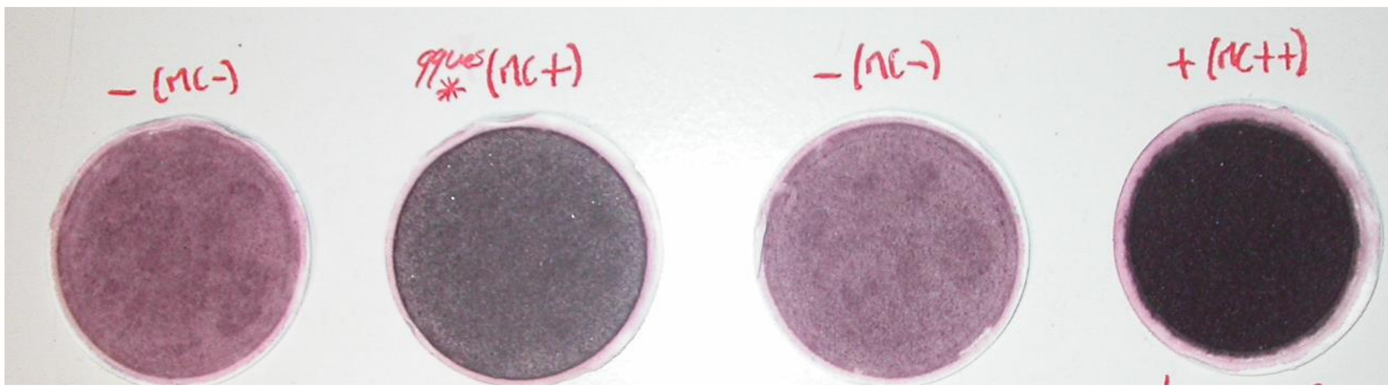
- ✓ Dissolve **MANNOSTAB®** in 10 times its weight in warm water (10% solution)
- ✓ Let it rest a few minutes and add during a racking or a pump-over
- ✓ Homogenize with a pump-over for at least 1.5 times the tank volume

Enological conditions:

- ✓ Treatment of a wine aged for a minimum of 6 months
- ✓ Any clogging filtration can lead to a loss of colloids and/or **MANNOSTAB®** and therefore renders the treatment partially or completely ineffective
- ✓ Homogenise with a pump-over for at least 1.5 times the volume of the tank
- ✓ Avoid any thermal shock > 5°C in the 72h following bottling

MANNOSTAB® treatment
in red wines

Observations since 2009: general improvement of red wine stability
→ combined effect on tartaric and coloring matter instability



St Estèphe 2007	St Estèphe 2007	St Julien 2007	St Julien 2007
Treated 15 g/hL	Not treated	Treated 15 g/hL	Not treated

Calcium Stabilization : Risks linked to absence of stability

PRECIPITATION OF NEUTRAL CALCIUM TARTRATE	Neutral calcium tartrate is a low solubility salt, 10 times less than KHT. In case of super-saturation (high level in Ca + high pH), there is a risk of CaT precipitation.
TYPES OF WINE CONCERNED	**All types of wine:** **crystal formation**
PRECIPITATION FAVORED BY: 	- ***Wine calcium content*** - Bottling filtration quality: wine stable prior to bottling becoming unstable through the ***retention of protective colloids in the case of a clogging filtration.***

Calcium Stabilisation

***White, rosé and red wines:* risk if [Ca] > 60 mg/L**

We recommend testing juice as early as possible, during fermentation if necessary.

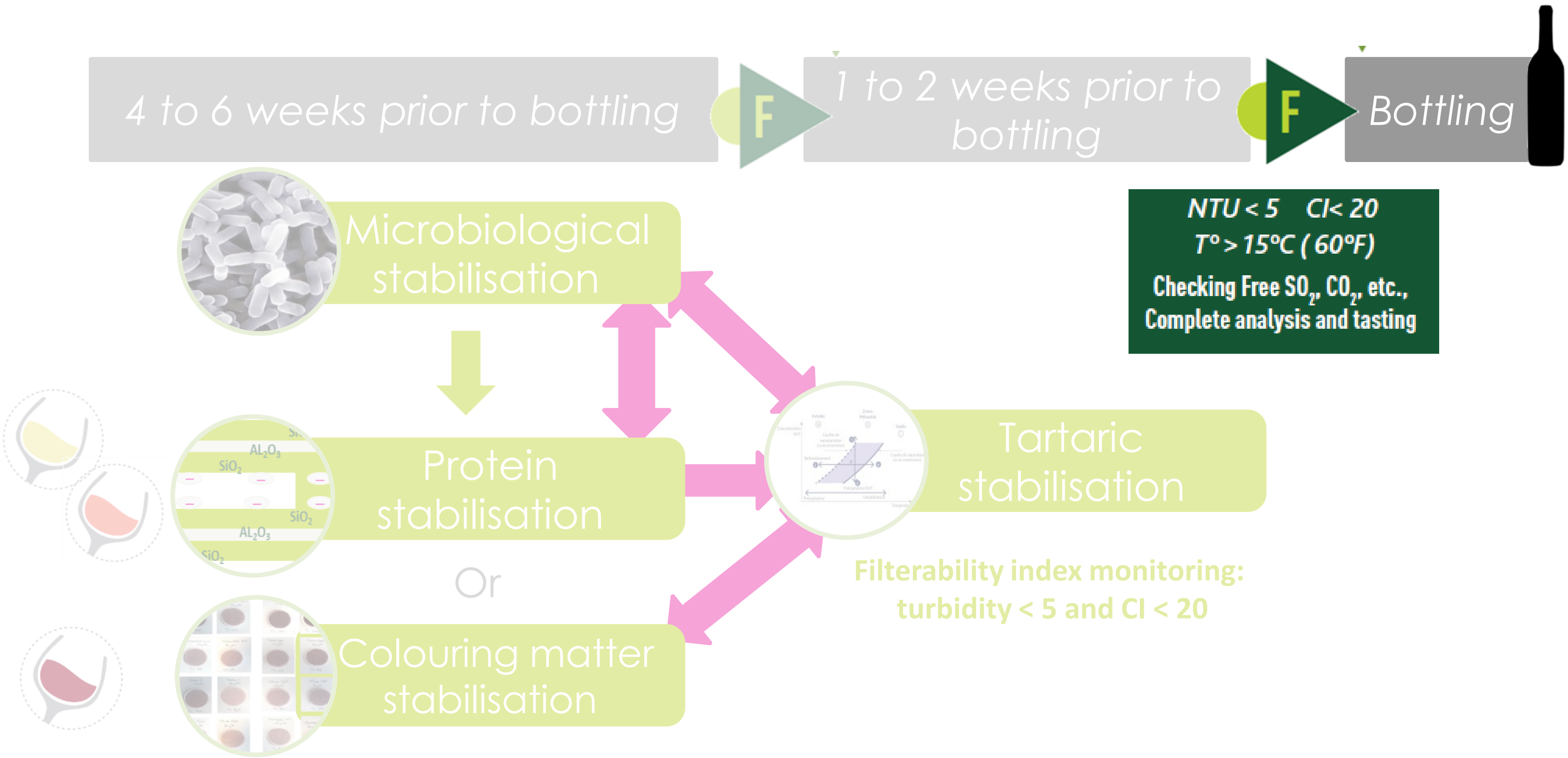
Wine de-acidification with calcium carbonate can elevate calcium levels above 60 mg/L ppm, test treated wines.

Elevated calcium levels can cause calcium tartrate precipitation, and inhibition methods are efficient in preventing only KHT precipitation.

Treatment options:

- **Cold:** although the CaT solubility is little sensitive to temperature, it seems part of CaT may be precipitated when the wine is cooled down?
- **CMC?**
- **Treatment with calcium racemate**: not easy to implement; addition at the juice phase is preferential

Stabilisation roadmap: change in mindset



Enzyme, fining agent, lysozyme, SO2, chitosan…

Finishing tannins

Bentonite

Metatartaric acid
CMC
Mannoproteins

Gum Arabic (D-2)
SO2 & ascorbic acid (D-1)
Sorbic acid (D-1)

Acidification/
deacidification

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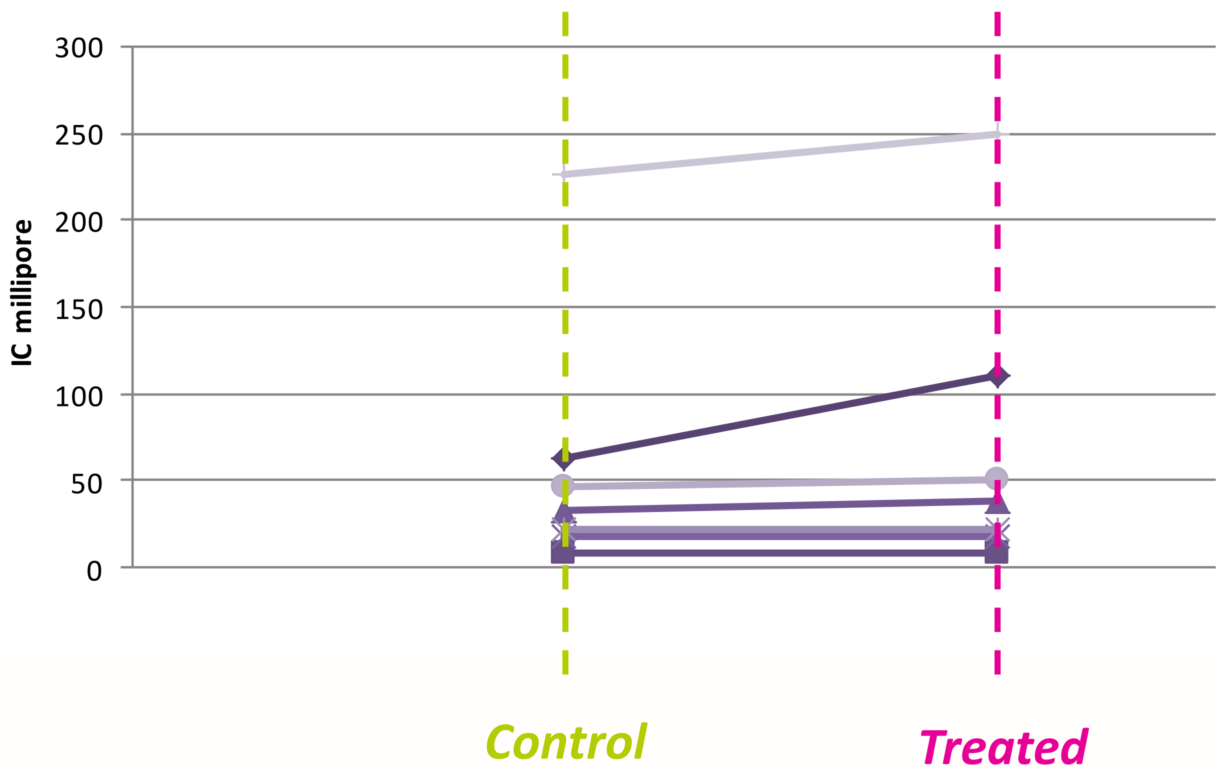
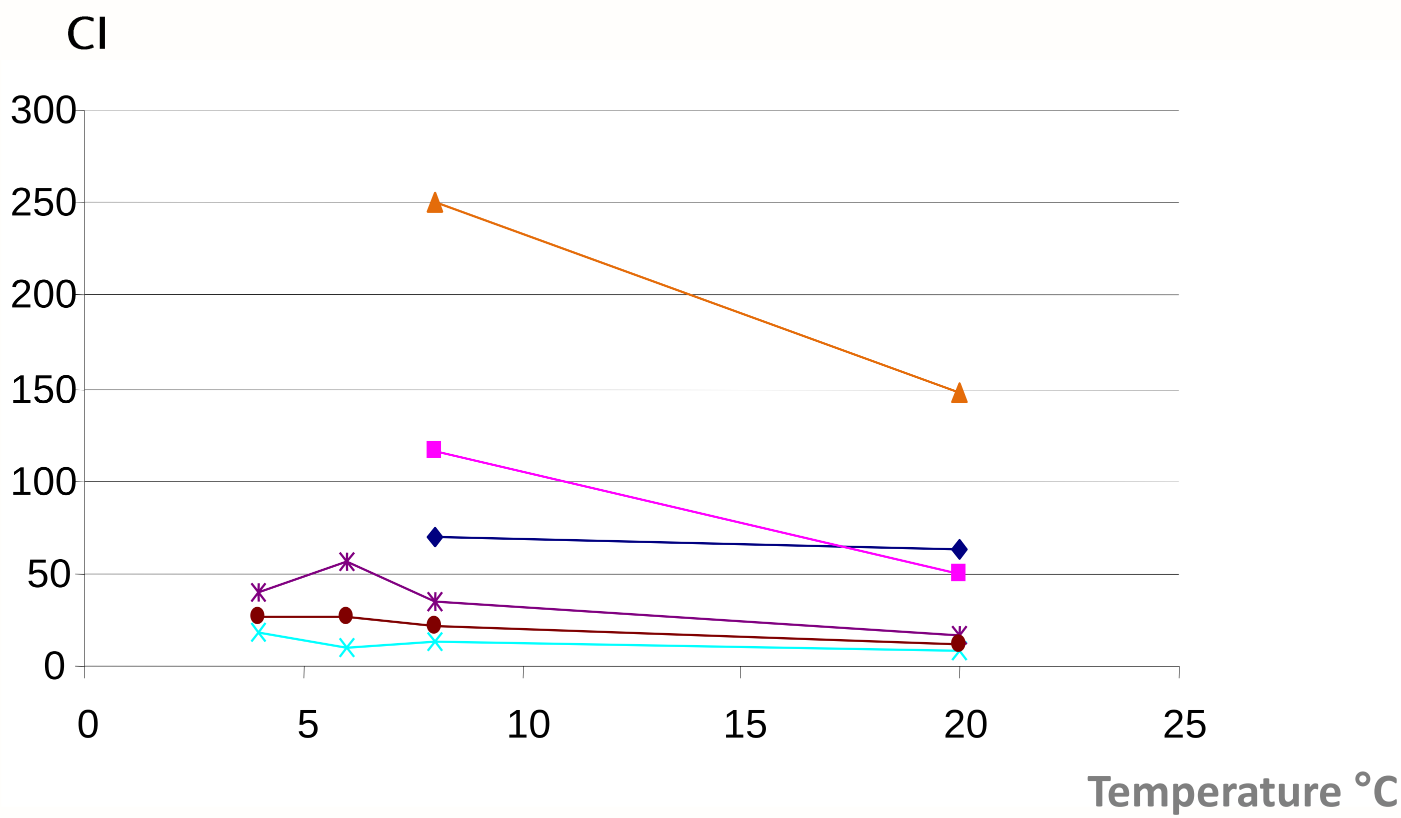
Filtration & Filterability

Effect of colloids on filterability



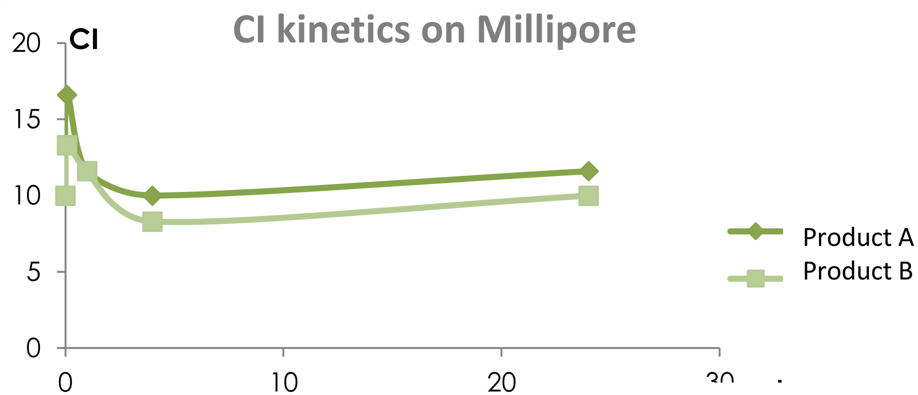
If wines are well prepared (CI < 50):
no effect of colloid addition

Effect of temperature on filterability

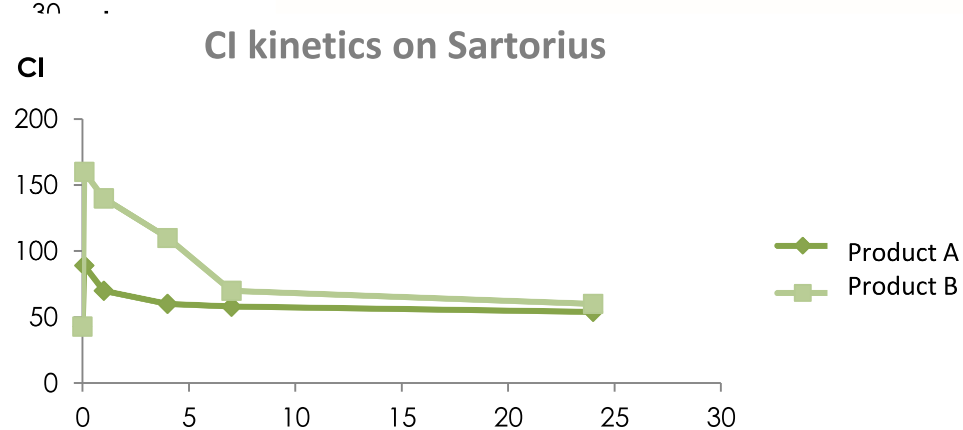


Below 15°C / 60°F, wines is more susceptible to clogging!

Effect of membrane nature on filterability

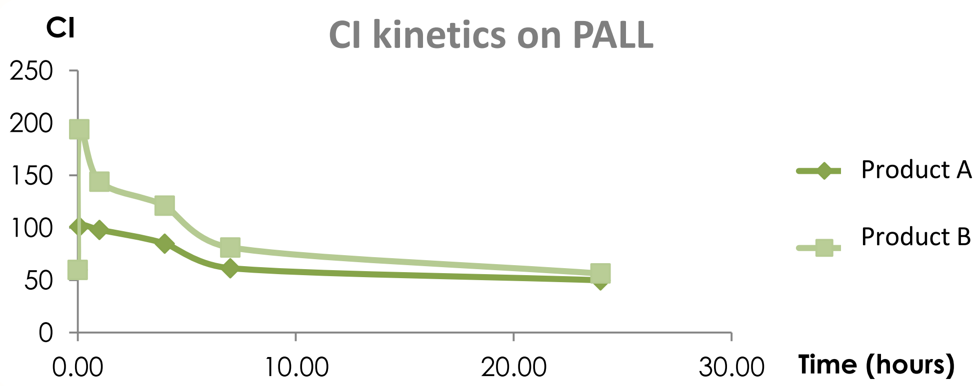


Millipore: cellulose acetate / nitrate



Sartorius: cellulose acetate

Pall: Nylon



CI very different depending on membranes!

Preparing the wines for filtration

Wine **filterability** is therefore paramount, depending (amongst others) on:

- temperature
- membrane on which the clogging index is tested,

There is no correlation between filterability and wine turbidity; a clear wine can clog filters! It is essential to assess both parameters when preparing the wine for filtration.

☑ POSITIVE FACTORS TO IMPROVE FILTERABILITY

- **ENZYME Addition** ——— ***action on filterability***

Ensures pectin and/or glucan chains breakdown, to improve settling (racking)

- PECTINASES
- β. GLUCANASES

- **FINING** ——— ***decreases the load***

Ensures settling of particles in suspension (colloids) present in the wine

- **RACKING** ——— ***decreases the load***

Lees removal

- **DEGASSING** ———

Reduction of the CO_2 load ensures minimal degradation of the cake during DE filtration.

- **APPROPRIATE ADDITION OF ENOLOGICAL PRODUCTS**

Every enological product has its own solubilization properties. A solubilization in water OR in wine prior to the final addition to the wine will facilitate a better filterability of treated wines

Autolees preparation and its impact on filterability

Turbidity and clogging index of a red wine treated directly with AUTOLEES at 150 and 300 mg/L are much more important than those of the red wine treated with a prior dissolution in water and this even after 72h of contact.

		Solubilisation in water at 10%		**Solubilisation in wine**	
	Control (red wine)	15 g/hl	30 g/hl	15 g/hl	30 g/hl
Turbidity 1h	0,8	1,8	3	6,9	15,8
Clogging Index (PALL) 1h	42	53	66	93	227
Turbidity 72h	0,8	1,3	2	4,6	9,4
Clogging Index (PALL) 72h	42	51	66	95	189

➡ **Autolees must first be dissolved in WATER (at 10%) before its addition to the wine for a better filterability of treated wines.**

Tannins preparation and its impact on filterability

- Tannins must be dissolved directly in WINE, and not in water, for a better filterability of treated wines. They can be prepared from 1% up to 10%.
- The turbidity of tannins or wine solutions is not correlated to the filterability of the treated wine.

		Solubilisation in water (30 min)		Solubilisation in wine (30 min)	
	Control	wine + 10 g/hl tannin	wine + 20 g/hl tannin	wine + 10 g/hl tannin	wine + 20 g/hl tannin
Turbidity	0,4	4,6	7,6	2,7	4,4
Clogging Index 1h	32	45	45	33	32

☒ NEGATIVE FACTORS FOR FILTERABILITY

- **TEMPERATURE**

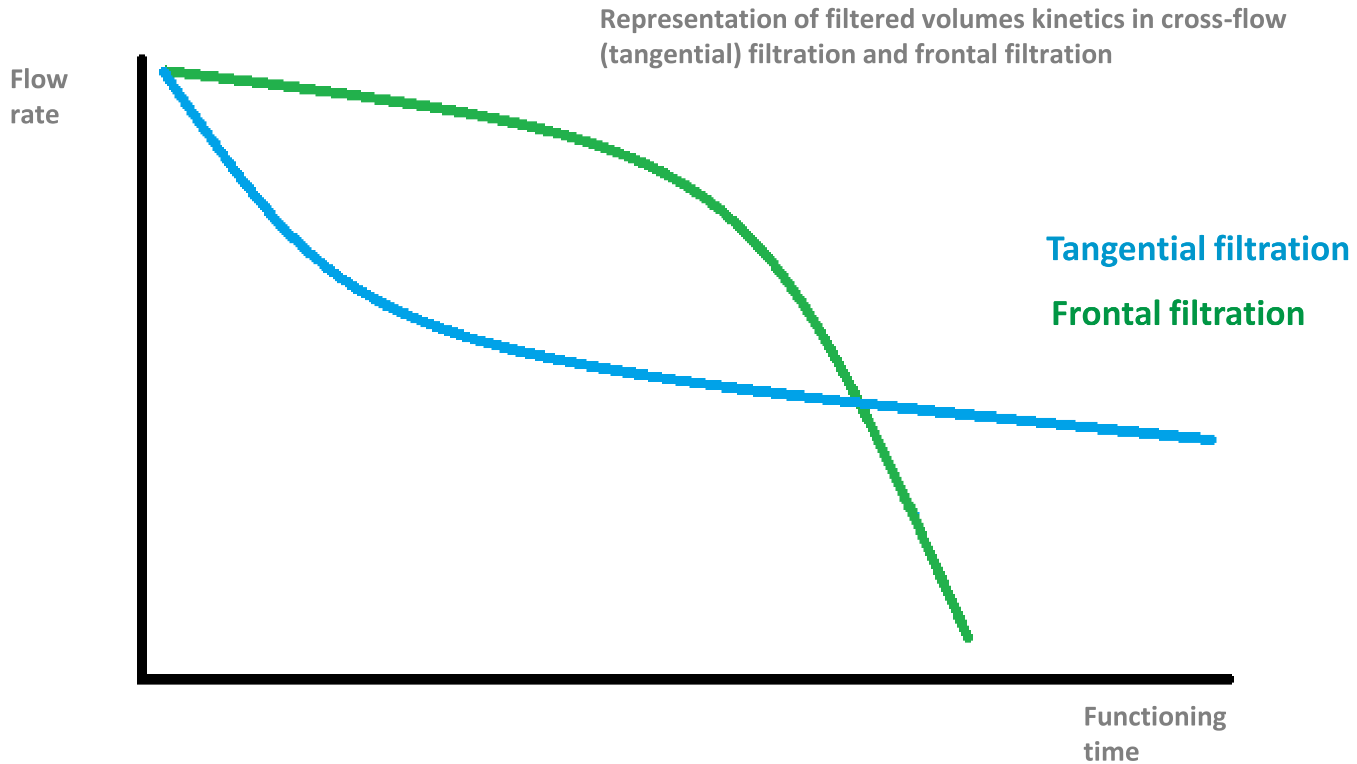
  The lower the temperature, the poorer the filterability (viscosity increases). Greater propensity for oxygen to dissolve.

  1°C = 2% flow rate

- **CHARGE**

Evaluate wine filterability by controlling the clogging index (minimum one week before bottling).

Cross-flow filtration / pad or DE filtration



Cross-flow filtration and enological treatments

Enological products compatible with cross-flow filtration:

Organic fining agents:
Gelatine, isinglass, casein, albumin

Vegetal fining agents:
Pea and potato proteins (**VEGECOLL®**) – ***rinse with cold water!***

Mineral fining agents:
Bentonite (**MICROCOL® FT**)
Silica gel and Carbon – ***PROHIBITED, too abrasive***

Synthetic fining agents:
PVPP – do the treatment 7 days prior to cross-flow

Cross-flow filtration and enological treatments

Oenological products compatible with cross-flow filtration: (i.e. Bucher Vaslin recommendations)

Tartaric stabilisation:
Mannoproteins, CMC (5 days ahead)

Other products:
Tannins, Concentrated must, SO_2 – prior to filtration
Gum arabic, N_2/CO_2 – post filtration

Note: each cross-flow supplier may have different recommendations

Cross-flow filtration and enological treatments

In line additions compatible with Cross-Flow Filtration (Bucher Vaslin):

- Rectified concentrated must
- Specific bentonite (Microcol FT)
- Vegecoll
- SO_2 solution

- N_2/CO_2 (to adjust CO_2 levels)

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Bottling timeline

Our recommendations at bottling:

SO_2:	24 hours prior to bottling
Ascorbic acid:	24 hours prior to bottling
Sorbic acid: *(with a SO2 addition)*	24 hours prior to bottling
Gum Arabic:	48 hours prior to bottling
Cellulose gum:	48 hours prior to bottling
Mannoproteins:	48 hours prior to bottling

The addition of these different products must be combined with an effective homogenisation to avoid changes in quality at bottling.

Last additions prior to bottling

Incompatibilities:

- CMC is incompatible with Lysozyme.

WHITE WINE CHECK-LIST

We recommend complete the final blend prior to initiating any stabilisation process

BOTTLING LAFFORT

4 to 6 weeks prior to bottling	1 to 2 weeks prior to botting	D day

Finishing tannins (dissolve at 10% in wine)

OENOLEES® MP CI<50 (dissolve at 10% in water)

Stabivin® SP or Œnogom® Instant (D -2)
SO_2 and ascorbic acid (D -1)
sorbic acid (D -1)

#1 MICROBIOLOGICAL STABILITY

Microbial load determination

Complete assessment (Yeasts, Acetic Bacteria, Lactic Bacteria)

TREATMENT

Treatment options to reduce the microbial load:
SO_2
Enzyme addition
LYSOZYM
Fining
Physical treatments

Double check microbiological stability

#2 PROTEIN STABILITY

Protein instability determination

Heat test
30 min. at 80°C
(Refer to the detailed protocol)

If ΔNTU < 2: stable wine *If ΔNTU > 2: unstable wine*

TREATMENT

Bentonite dose determination.
Bentonite treatment: same bentonite in lab and in cellar.
Importance of product preparation and implementation.

LAFFORT YouTube

IMPLEMENTATION OF MICROCOL®ALPHA

LAFFORT® expert's advice
+ 20 mL/hL SILIGEL & 20 mL/hL gelatine for a fast sedimentation

Double check the stabilisation status prior to racking or filtration

F

#3 TARTARIC STABILITY

Tartaric instability determination

Crystallisation test
6 days at -4°C

If no crystals or DIT < 5% = stable wine

DIT Test
4 hrs at -4°C
+4 g/L cream of tartar

If presence of crystals or DIT > 5% = unstable wine

TREATMENT

LAFFORT® expert's advice
We recommend to remove Carbon Dioxide in the wine prior to any tartaric stabilisation treatment

- Physical processes
- Inhibitory methods

POLYTARTRYL®
CELSTAB®
MANNOSTAB®

Filterability index monitoring: turbidity < 5 and CI < 20
Importance of mixing the tank

Double check stabilisation
ISTC50 measure:
(test 4hrs at -4°C + 0.5g/L cream of tartar)
ISTC50 ≤ 3µS = stable wine

F

NTU < 5 CI < 20
T° > 15°C (60°F)
Checking Free SO_2, CO_2, etc., Complete analysis and tasting

#4 BOTTLING

Complete wine analysis and tasting (D+4)

THE KEYS TO A SUCCESSFUL BOTTLING

- 1 stable batch + 1 stable batch ≠ 1 stable blend.
- Use CMC on protein stable wines.
- CMC and metatartaric acid form a haze on wines treated with lysosyme.
- CMC forms a haze with tannins.
- Metatartaric acid used on a cold wine creates a reversible haze.

DIT: Degree of Tartaric Instability
CI: Clogging Index

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F The quality of the filtration is essential to prevent the retention of protective colloids which might cause a new instability in the wine. Below 15°C / 60°F, wine flow decreases / - 1°C = 2% flow rate loss

RED WINE CHECK-LIST

We recommend complete the final blend prior to initiating any stabilisation process

BOTTLING LAFFORT

4 to 6 weeks prior to bottling	1 to 2 weeks prior to botting	D day

Finishing tannins (dissolve at 10% in wine)

OENOLEES® MP CI<50 (dissolve at 10% in water)

Stabivin® range or Œnogom® range (D-2) SO_2 and sorbic acid (D-1)

#1 MICROBIOLOGICAL STABILITY

Microbial load determination

Complete assessment (Yeasts, Acetic Bacteria, Lactic Bacteria)

TREATMENT

Treatment options to reduce the microbial load:
- SO_2 addition
- Enzyme addition
- LYSOZYM
- Fining
- OENOBRETT®
- Physical treatments

Double check microbiological stability

#2 COLOURING MATTER STABILITY

Instability determination

Cold test (4°C for 48h)

Δturb (NTU)	< 5 NTU	Stable
Δturb (NTU)	5-10 NTU	Slight instability
Δturb (NTU)	10-20 NTU	Medium instability
Δturb (NTU)	20-50 NTU	Typical instability
Δturb (NTU)	> 50 NTU	Strong instability

FINING TRIALS
at least 2 fining agents at 2 different doses

TREATMENT

Fining:
- Gelatin
- Albumin
- OENOLEES®
- VEGECOLL®

LAFFORT® expert's advice

BENTONITE: a 5 to 10 g/hL dose following the addition of the proteic fining agent will enhance the coloring matter stability and optimise the use of the fining agent

ENZYME (EXTRALYSE®) used in the end of vinification or ageing will favor the stabilisation of colouring matter.

F

#3 TARTARIC STABILITY

Instability determination

Crystallisation test 6 days at -4°C

If no crystals or DIT < 5% = stable wine

DIT Measure test 4 hrs at -4°C + 4g/L cream of tartar

If presence of crystals or DIT > 5% = unstable wine

TREATMENT

- Physical processes
- Inhibitory methods:
  - POLYTARTRYL®
  - MANNOSTAB®

Filterability index monitoring: turbidity < 5 and CI < 20
Importance of homogenisation

Double check stabilisation
➩ crystallisation test

F

NTU < 5 CI < 20
T° > 15°C (60°F)
Checking Free SO_2, CO_2, etc., Complete analysis and tasting

#4 BOTTLING

Complete wine analysis and tasting (D+4)

THE KEYS TO A SUCCESSFUL BOTTLING

- 1 stable batch + 1 stable batch ≠ 1 stable blend.
- Metatartaric acid may form a haze in wines treated with lysozyme.
- Metatartaric acid used on a cold wine creates a reversible haze.
- The use of arabic gum will improve the colouring matter stability.

DIT: Degree of Tartaric Instability
CI: Clogging Index

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F The quality of the filtration is essential to prevent the retention of protective colloids which might cause a new instability in the wine. Below 15°C / 60°F, wine flow decreases / - 1°C = 2% flow rate loss

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Preparing your wines for your next bottling?

Let's talk…

Thank you for your attention!