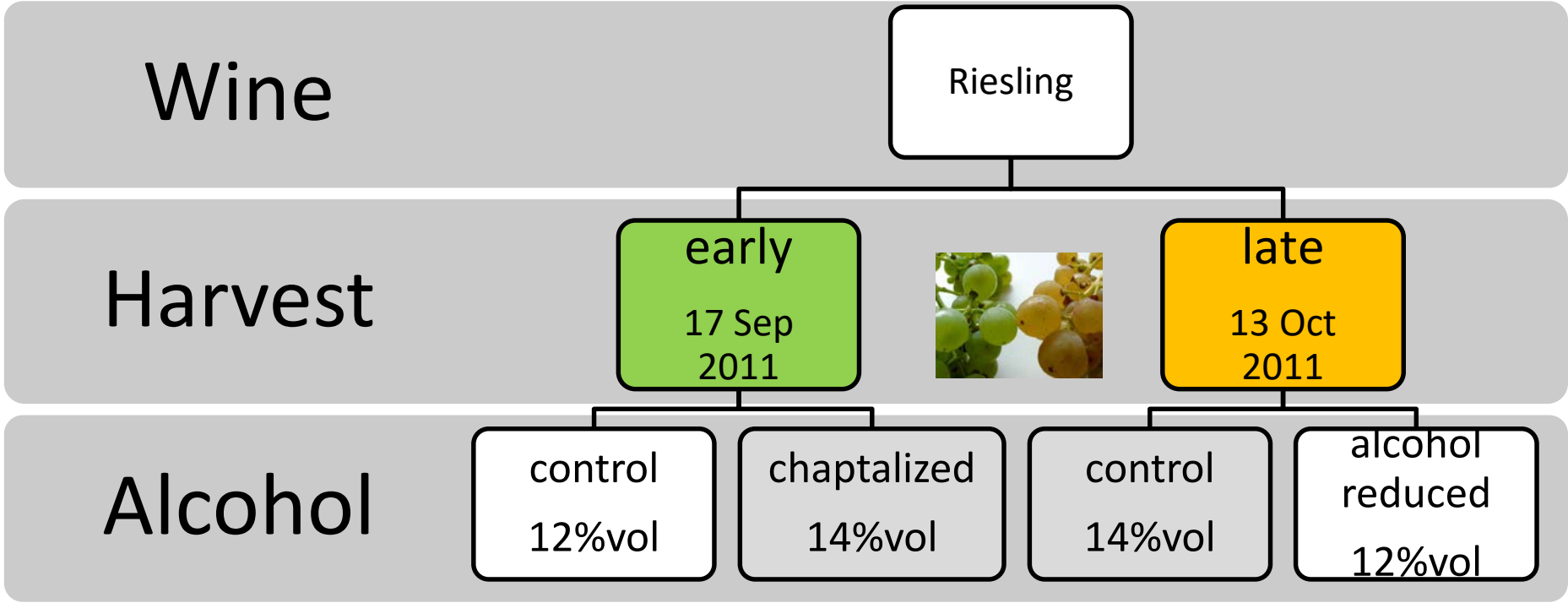
Partial Dealcoholization of Wine
Comparing Methods for dealcoholization

Dr. Andreas Blank

Wine Eng Conference

K+H process tec GmbH

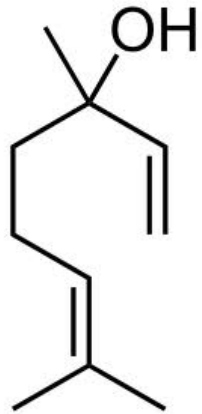
Why are we talking about alcohol reduction?

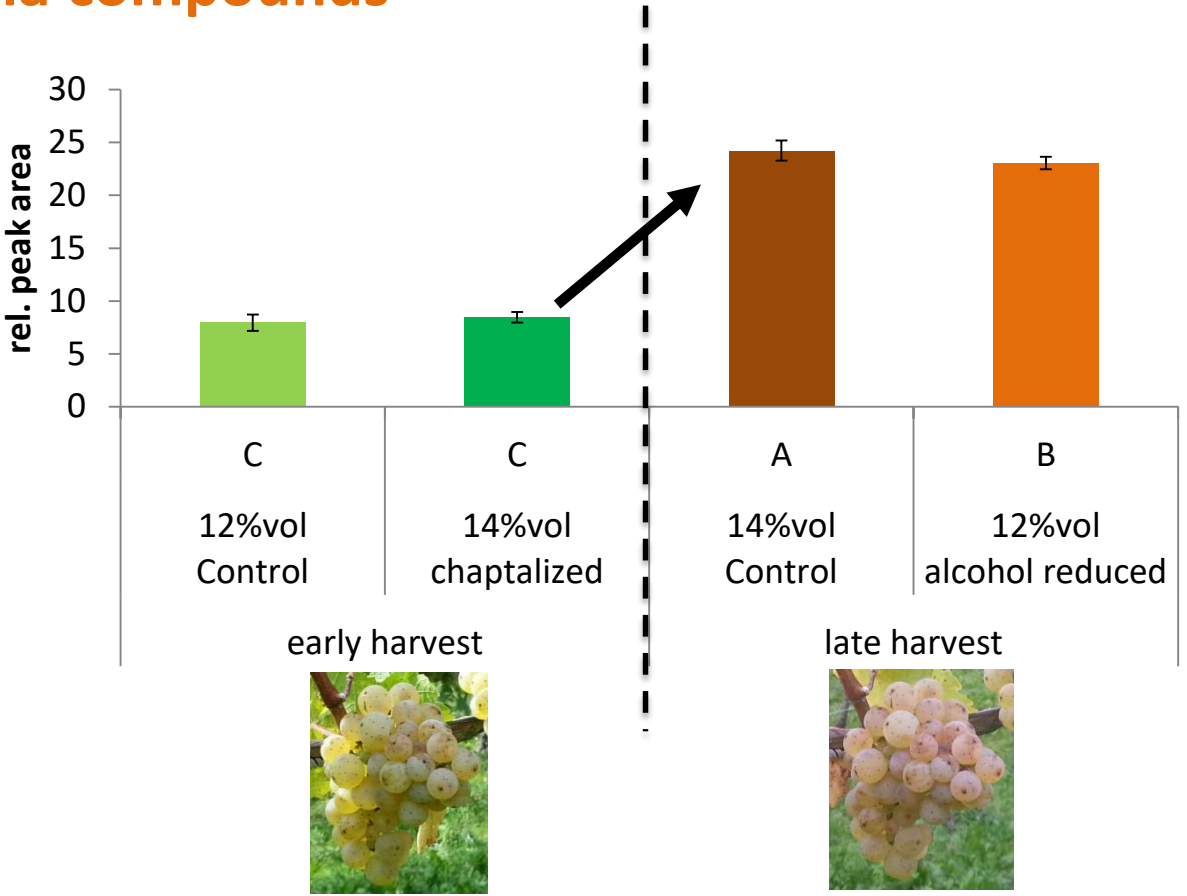


GC-Analysis aroma compounds

Riesling
2011

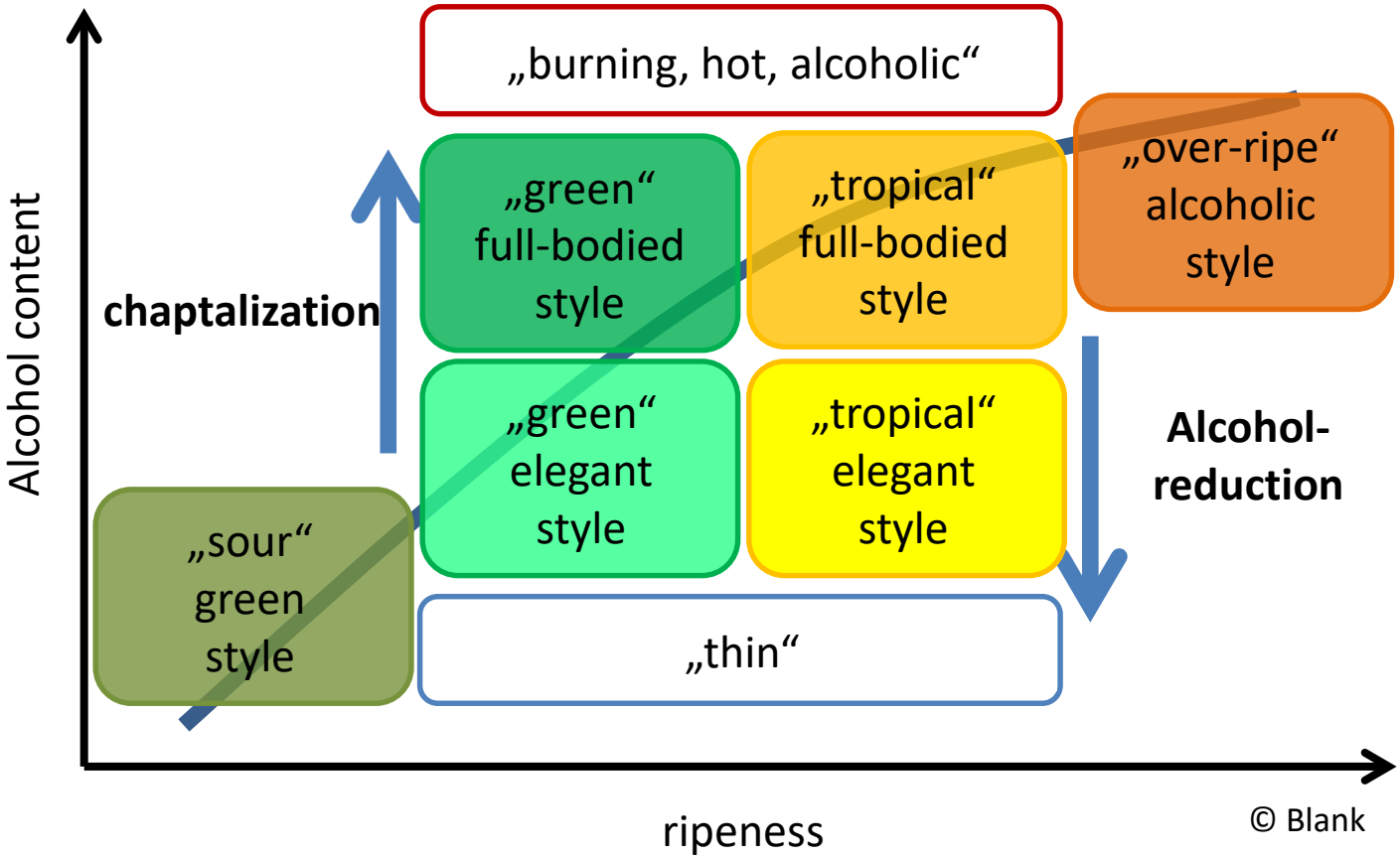
Linalool





Post hoc
Fisher LSD (5%)

Sensorial effect of ethanol wine



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Technologies: Overview

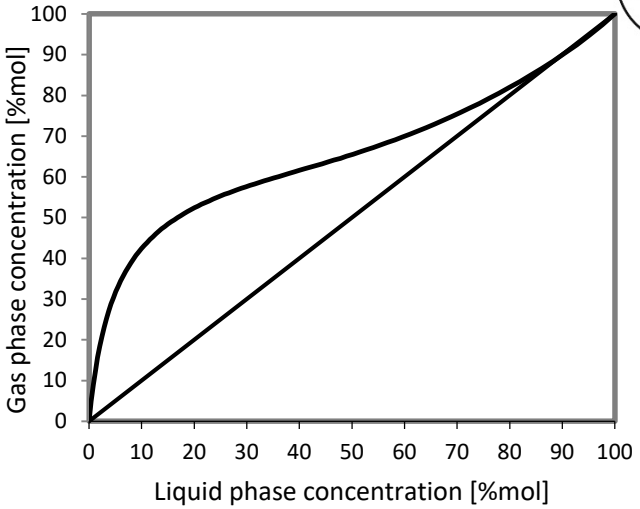
- Technical dealcoholization
  - Distillation / Spinning Cone Column
  - Membrane contactor
  - Reverse Osmosis
  - Combinations

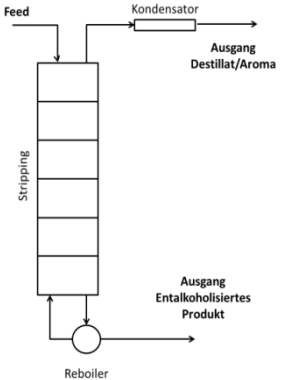
Distillation

Ethanol is more volatile than water

→ Distillation separates

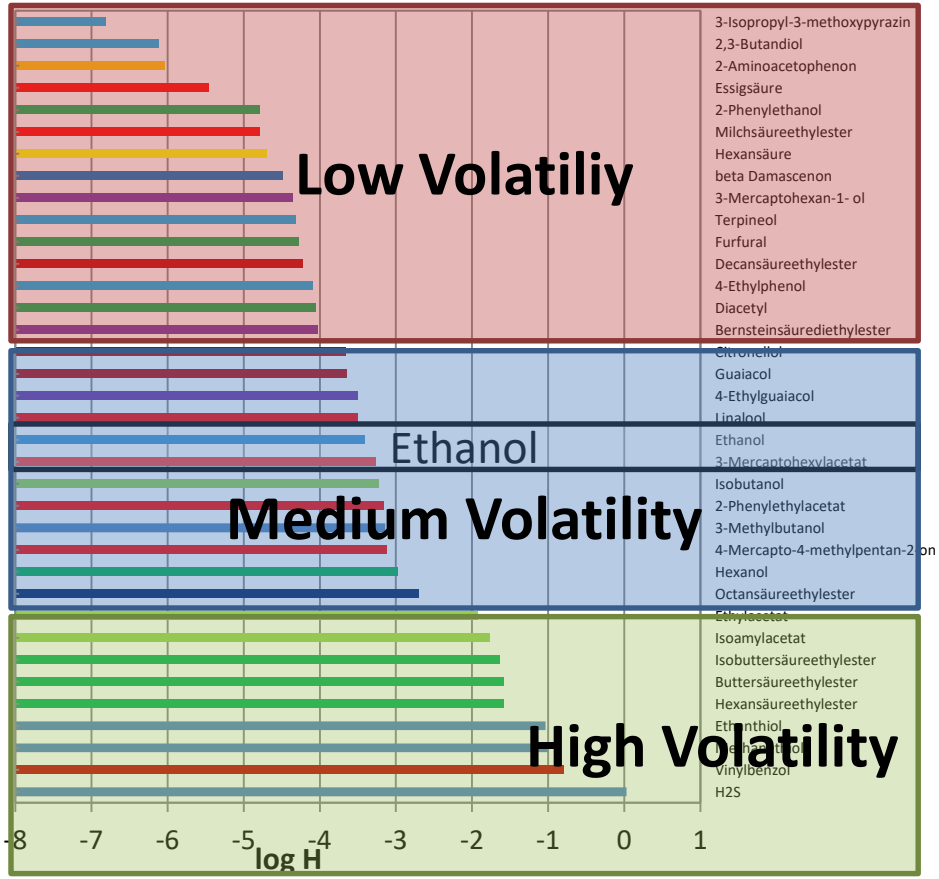
What about aroma compounds?





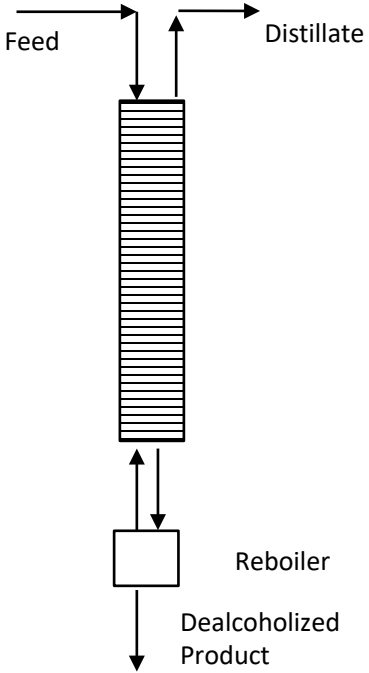
z.B. MCCABE und THIELE, 1925

Volatility of Aroma Compounds

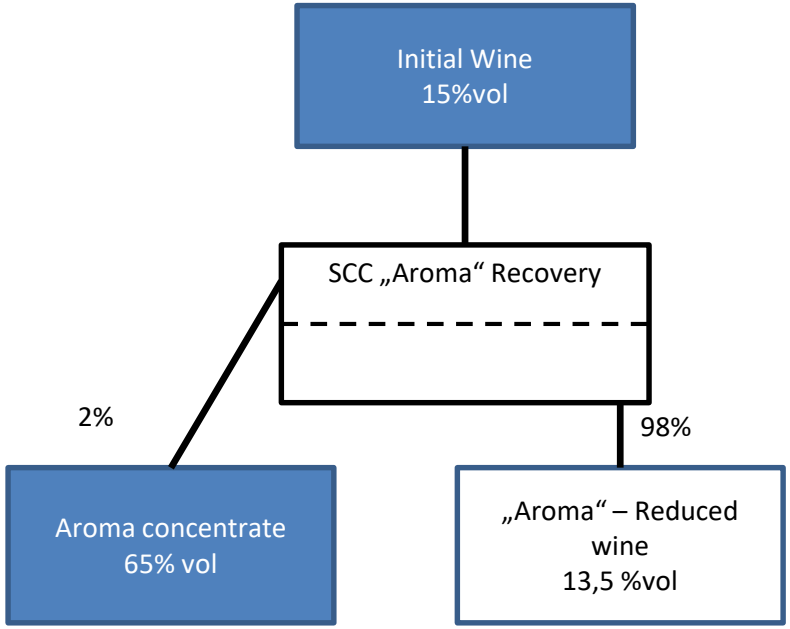


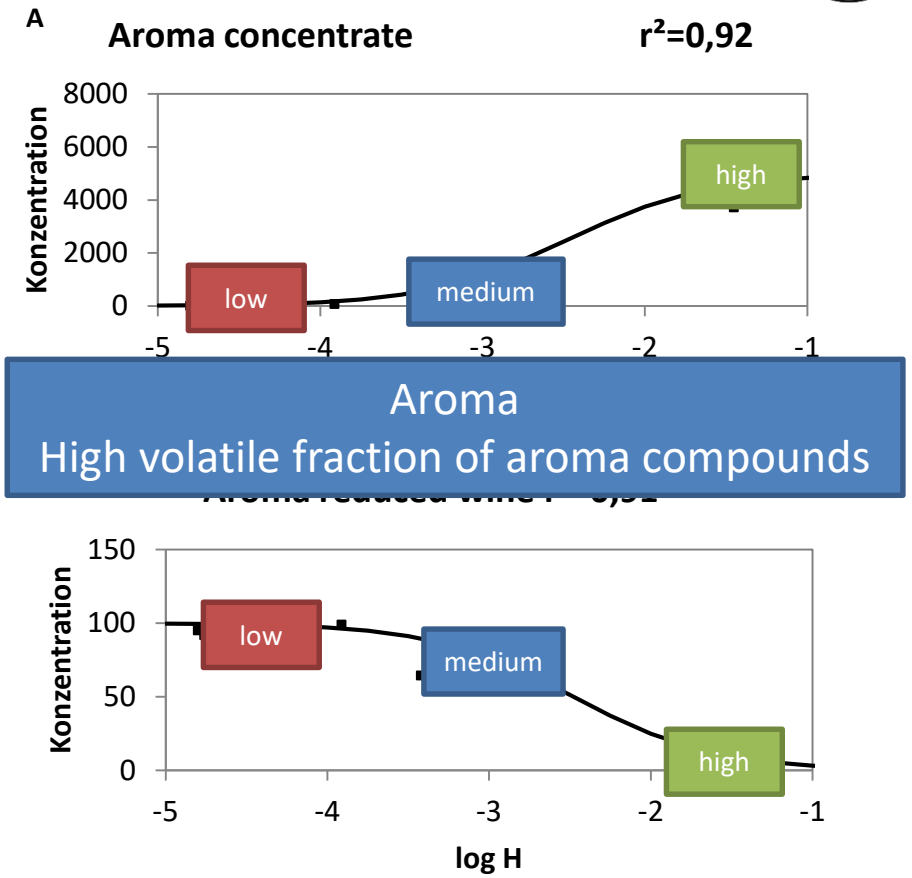
Blank, 2015
Own Data calculated from Archemcalc.org 2012
IKARI und KUBO, 1976

Spinning Cone Column Distillation



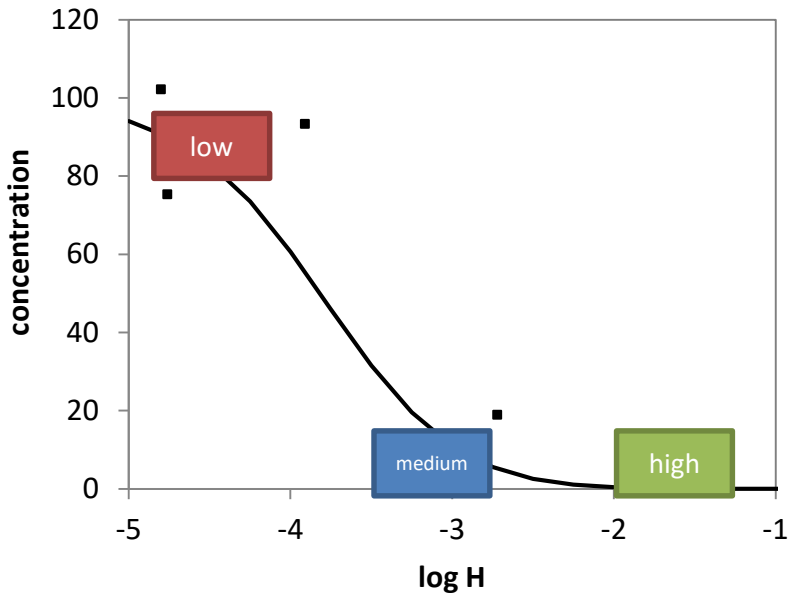
Spinning Cone Column





Fraction analysis

C



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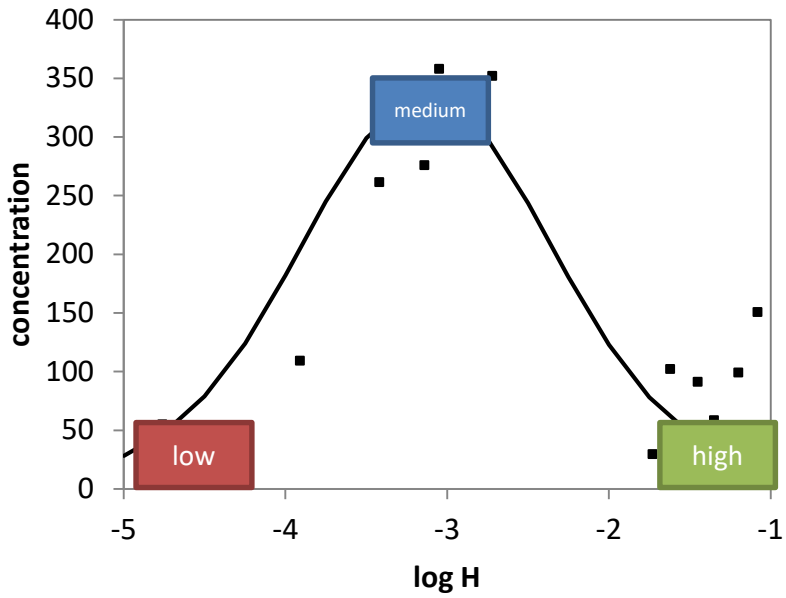
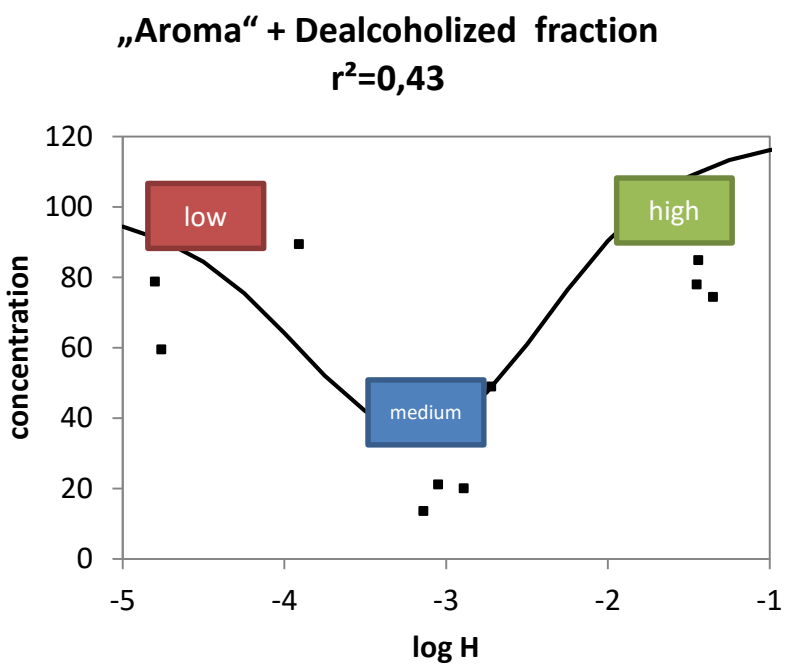


Recombination in Spinning Cone Column

E

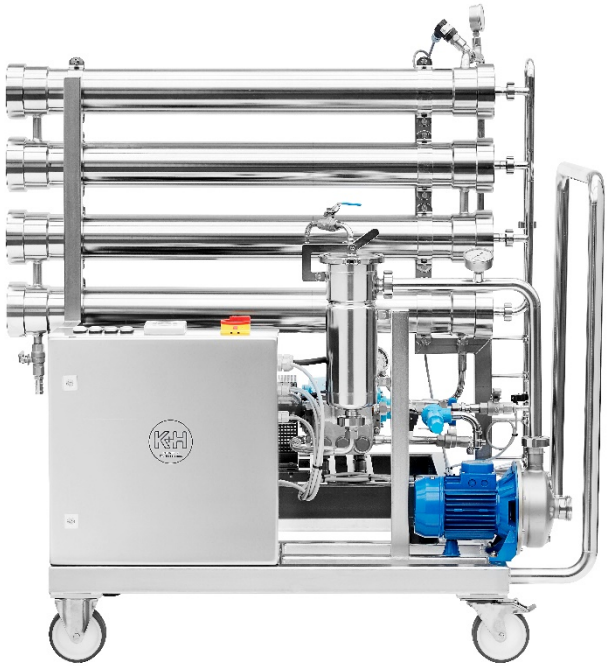


- Low and high volatile aroma compounds are completely recovered
- Medium volatile aroma compounds are lost equal to ethanol reduction.

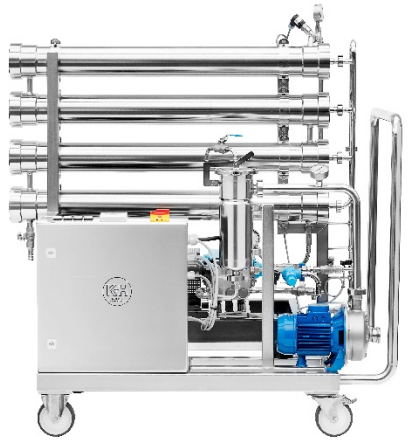
- Problem:

Many aroma compounds have similar volatitlity compared to Ethanol

Reverse Osmosis Alcohol Reduction



Reverse Osmosis
Alcohol Reduction



Initial Wine
15%vol

Reverse Osmosis

85%

15%

Concentrate
15,2%vol

Permeate
14,8%vol

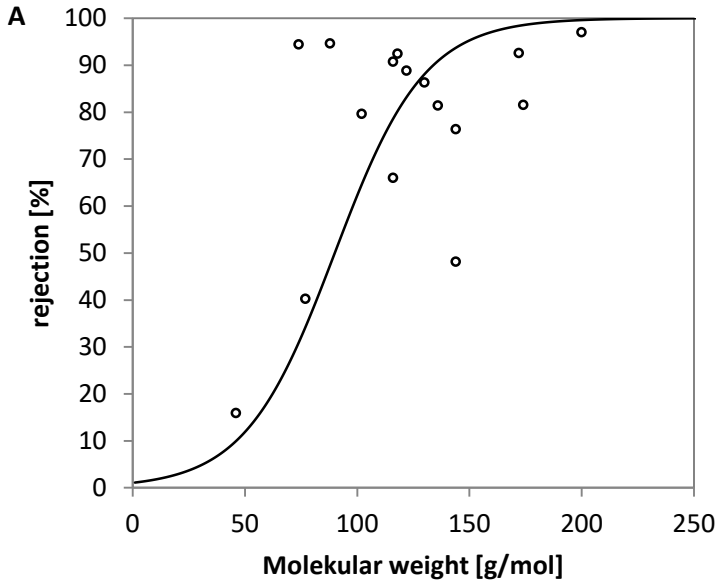
Reduced alcohol
wine
13,0%vol

Water
15%

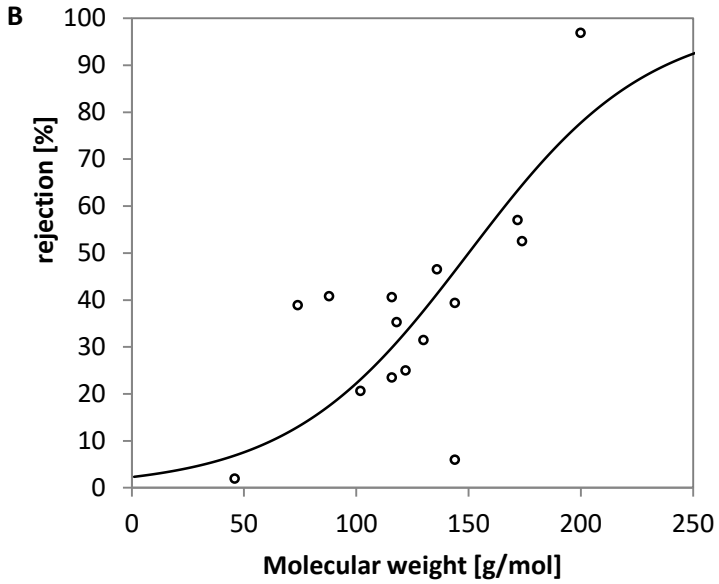
Separation by molecular size

RO Separation

Membrane A „tight RO“

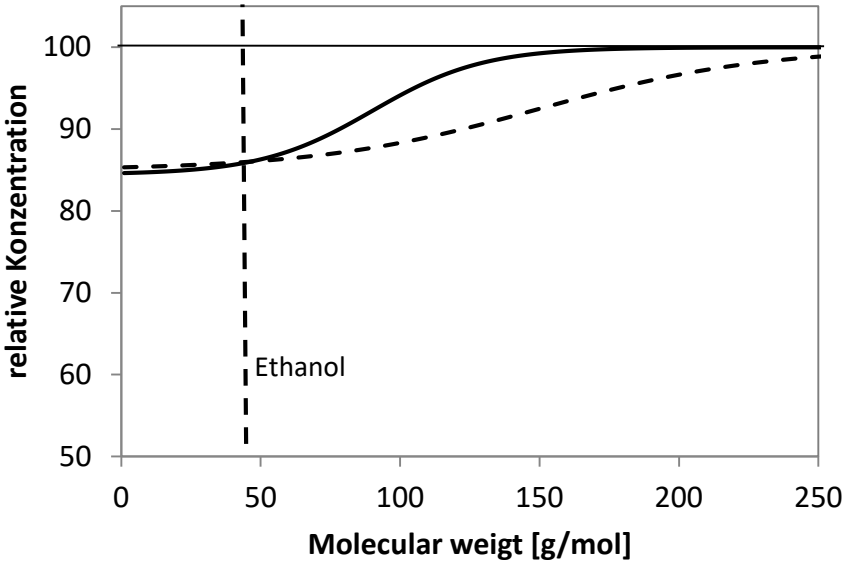


Membrane B „loose RO“



RO Separation – Take home

- RO Alcohol reduction



Separation as molecular sieve

Extract compounds can be lost

Membrane contactor – Osmotic Distillation

Porous Membrane of Polypropylen

hydrophobic

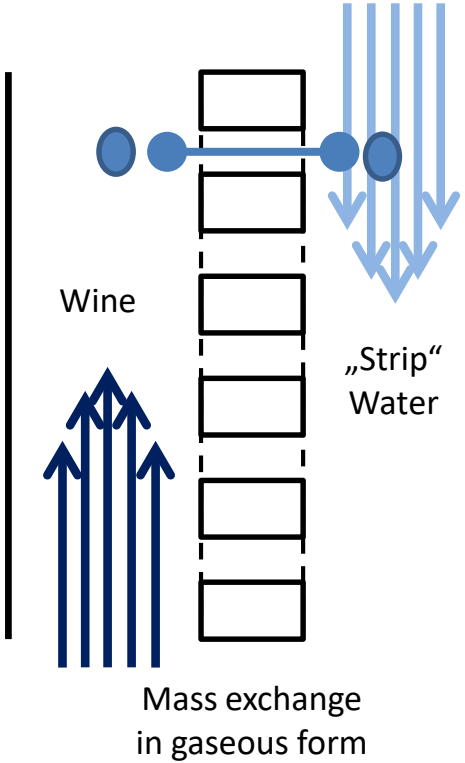
Main Purpose(CO_2/O_2)

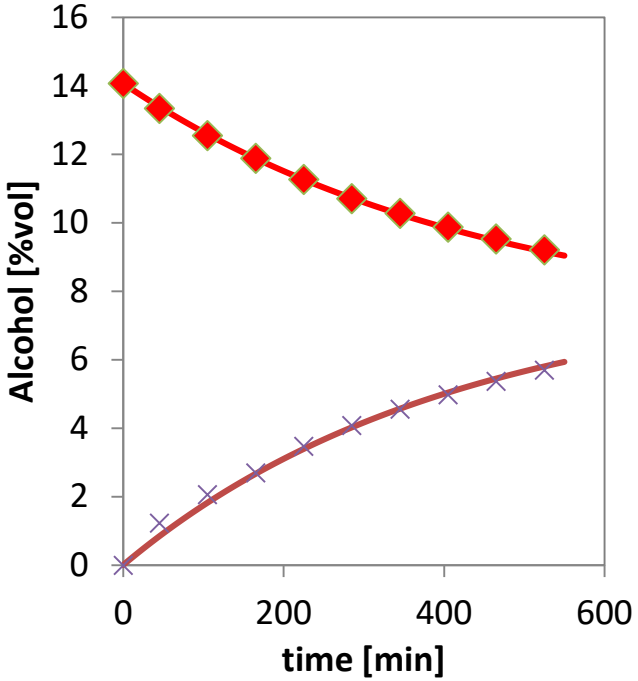
Application in Alcohol Management

Osmotic distillation

GABELMANN und HWANG, 1998
SENGUPTA *et al.*, 1998
BLANK *et al.*, 2013

Alcoholmanagement using Membrane Contactors





Osmotic Distillation: Analytical changes

Alcohol reduction by 2%vol.

Total number of wines:
n=15

2 factoral ANOVA
Treatment x Wine

No significant Interactions

Shown is significance of treatment

Analyse	control mean	reduced mean	Sign. p
Ethanol g/L	114,5	99,1	***
Sugar g/L	3,9	4,1	**
Sugar free Extrakt g/L	21,0	21,4	*
Residual extrakt g/L	10,6	11,0	**
Volatile acidity g/L	0,38	0,39	ns
Potassium mg/L	764	815	**
Magnesium mg/L	85,0	86,7	ns
SO_2 free mg/L	47,6	39,5	**
CO_2 g/L	1,0	0,8	*
E 420 nm/1cm	0,5	0,5	ns
Total phenols mg/L	678	687	ns

Ethanol is removed

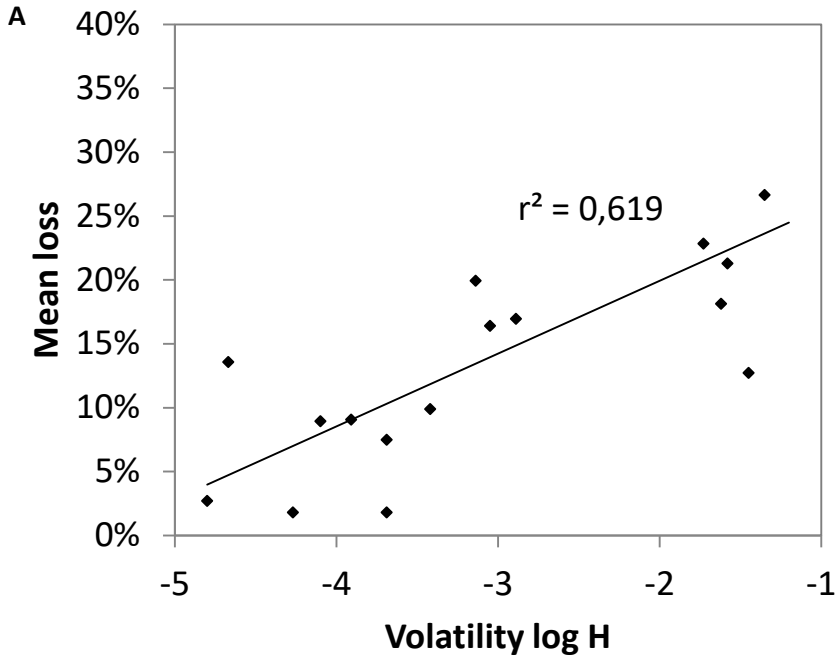
→ Concentration of Non-volatiles. 2-4 %

→ Loss of Gase SO_2 und CO_2

ns=non significant
** p=0,05*
*** p=0,01*
**** p=0,001*

Aroma compounds

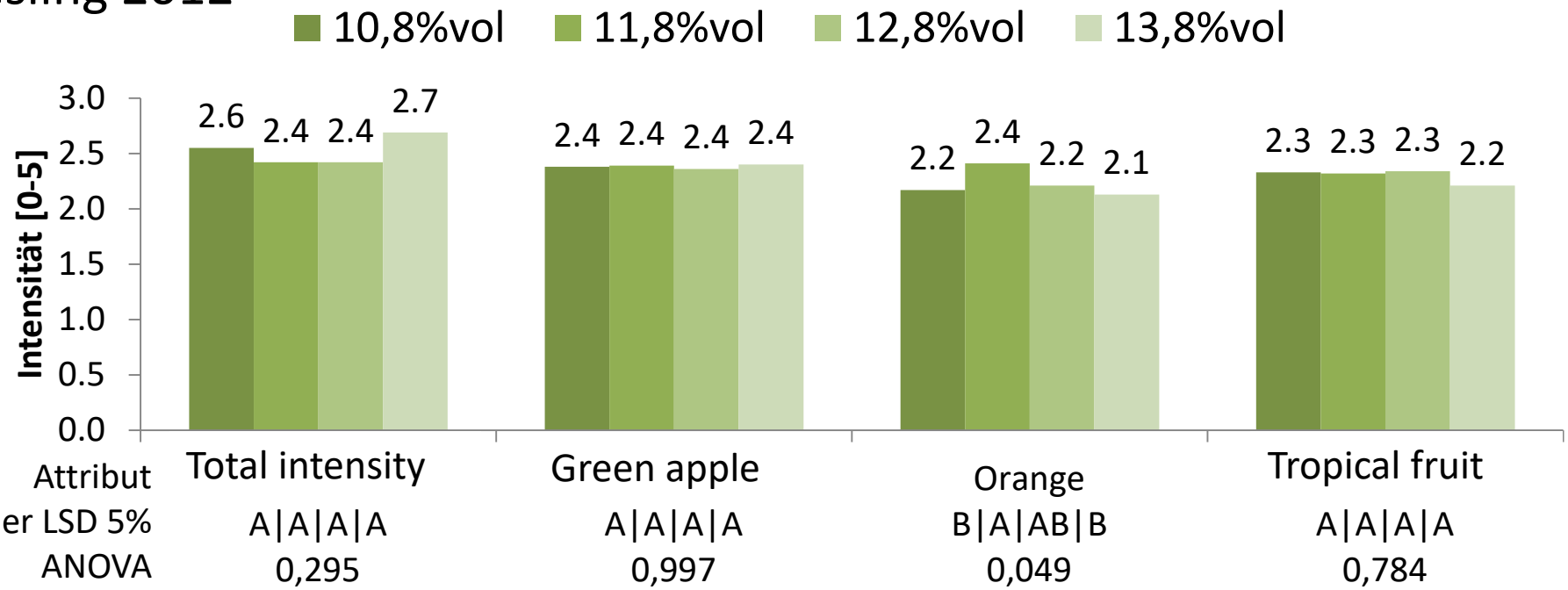
Compounds	Mean loss
Hexyl acetate	26%
Isoamyl acetate	23%
Buttersäureethylester	21%
Isobutanol	20%
Isobuttersäureethylester	18%
Phenylethylacetat	18%
Hexanol	17%
3-Methylbutanol	16%
Ethyl acetate	13%
Linalool	9%
Bernsteinsäurediethylester	9%
4-Vinylphenol	9%
Geraniol	8%
Nerol	3%
2-Phenylethanol	3%
Terpineol	2%



Reduzierung um 2%vol.
n=12 different wines
Flüchtigkeit log H berechnet mit archemcalc.com

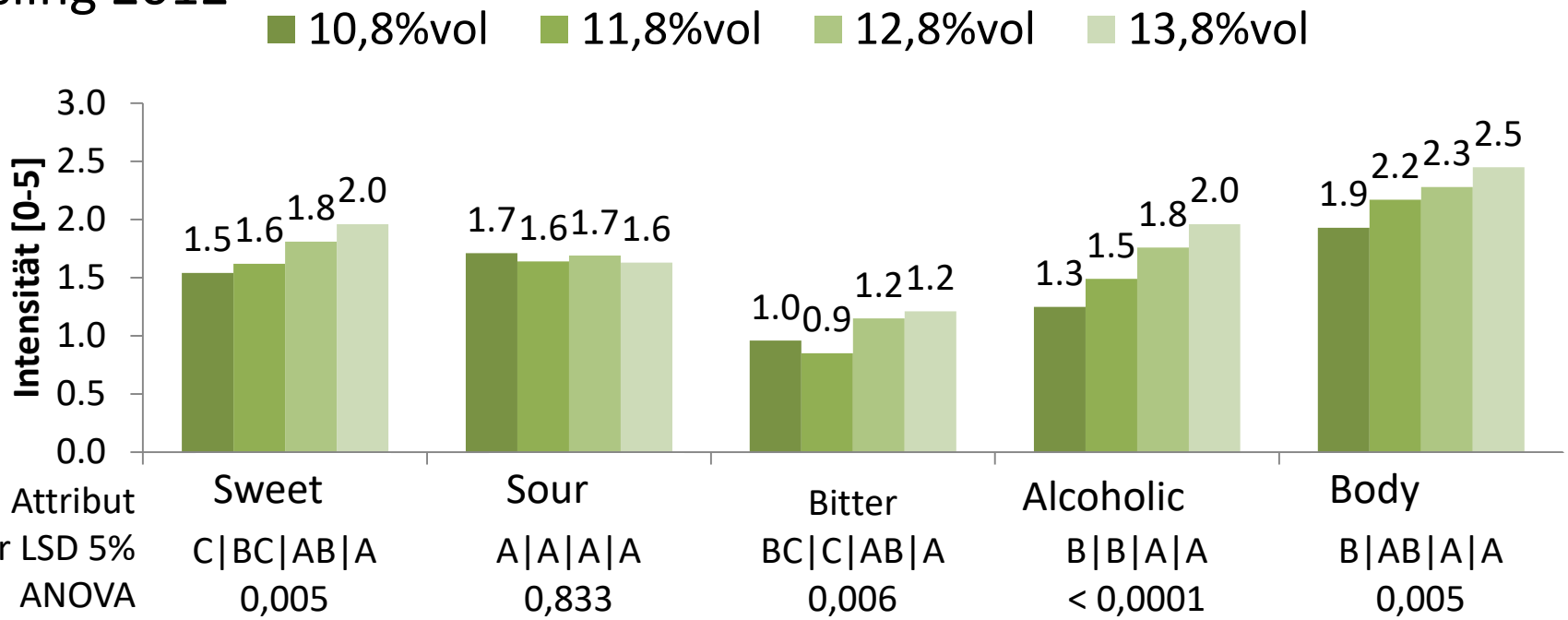
Alcohol reduction / Aroma attributes

Riesling 2012



Alkoholreduzierung:
Taste Attributes

Riesling 2012



Osmotic Distillation:
Take home

- Machine fo disolved gas management

Alcohol reduction:

- Treat small volume strong – blend back to desired content
- No significant difference in Wine Sensory
- Aroma loss dependant on volatility
- Concentration of extract compounds
- Losses of SO_2 and CO_2 have to be replaced

Comparing Technologies

- Technologies
  - Distillation
    - Distillation
    - Membrane contactor
  - Reverse Osmosis
    - Combination RO + MC
    - Reverse Osmosis RO+H2O

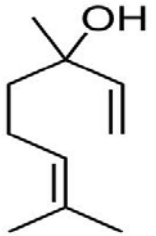
2 wines each in Duplicate

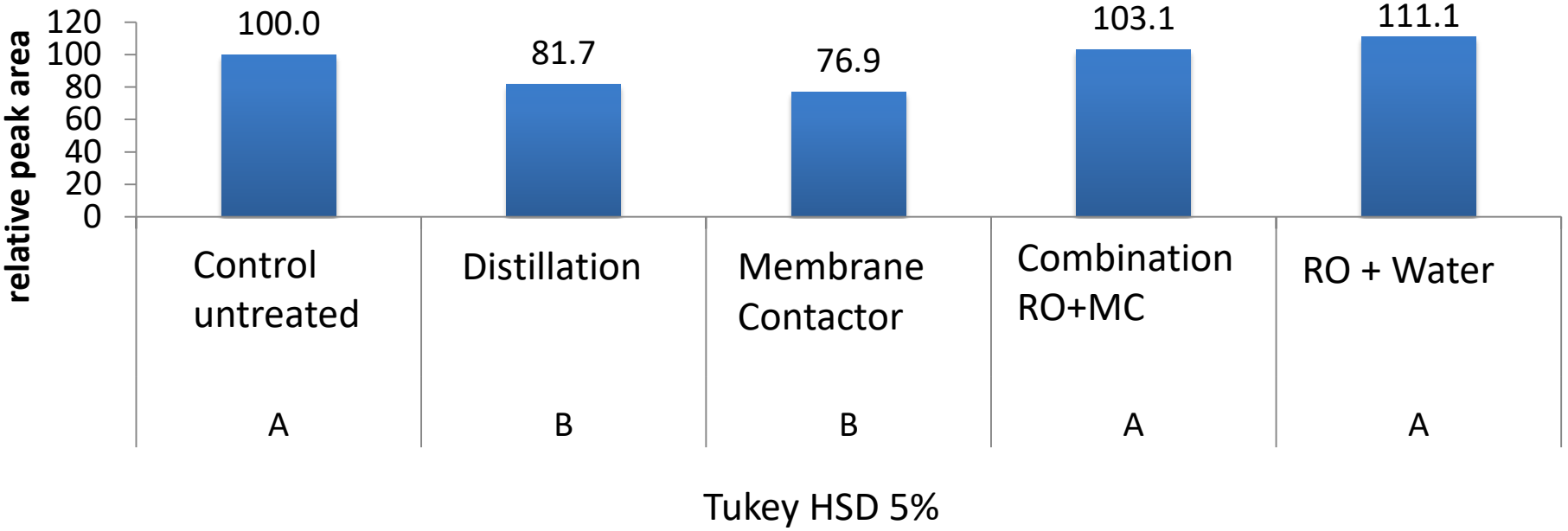
Linalool: Alcohol reduction by 2%vol.

Linalool: Terpen

Molecular weight: medium - large 154 g/mol

Volatility similar to Ethanol





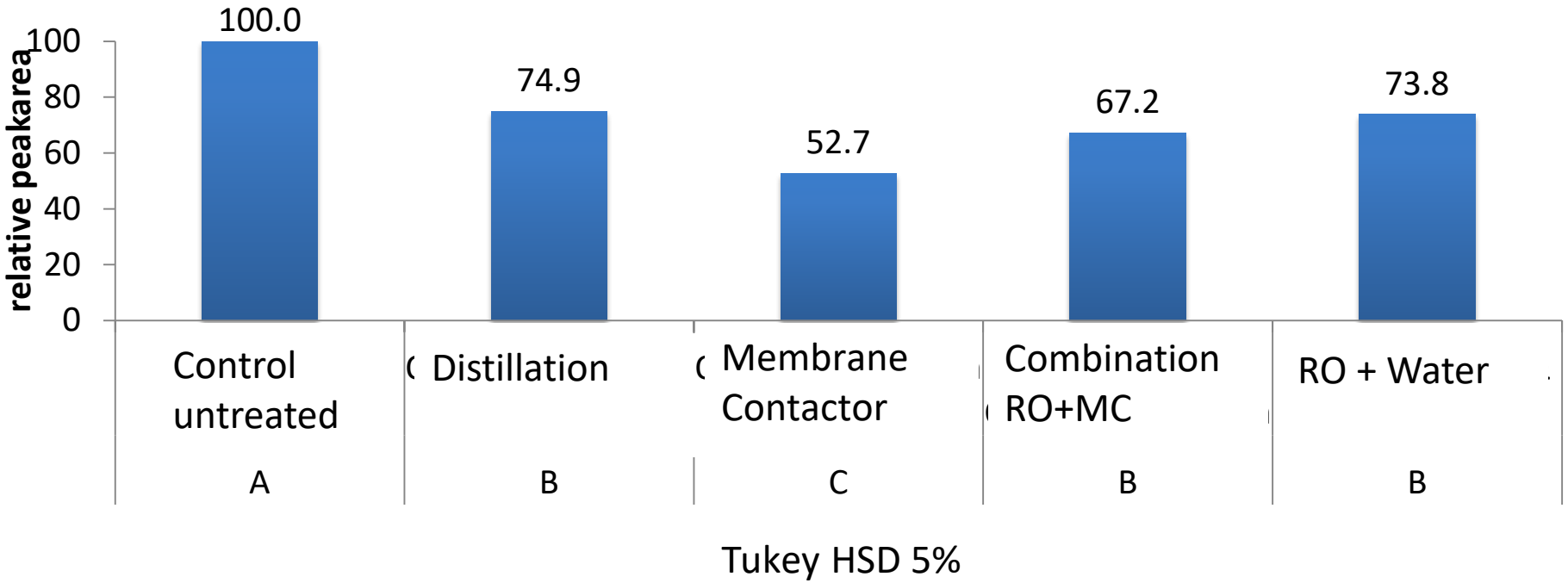
Tukey HSD 5%

Isoamyl acetate: Alcohol reduction by 2%vol

Isoamyl acetate:

Molecular weight: medium 130 g/mol

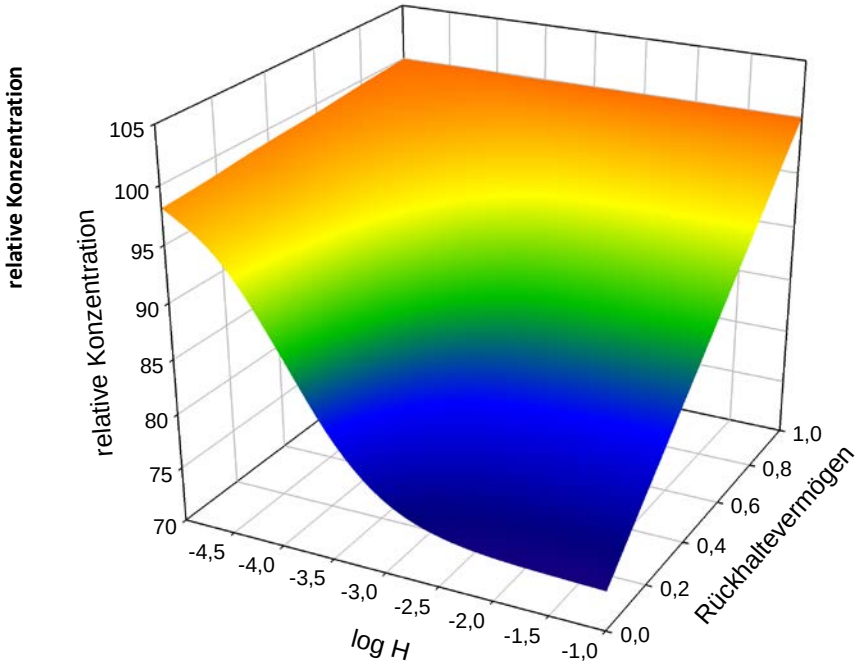
High volatile aroma compound



Comparing technologies

- Alcoholreduction always comes together with loss of some volatiles
- Strong treatment of small volume reduces aroma loss to an acceptable minimum

- Distillation (vacuum and membrane MC)
  - Separtation by volatility
- Reverse Osmoses
  - Separation by molecular size
- Spinning Cone Column SCC
  - Double distillation
- Kombination RO + Distillaiton
  - Combined separation



K+H VinZero

- Inline alcohol adjustment:
- Automated alcohol adjustment using RO and Membrane contacter
- In line alcohol probe

Conclusion

Technologies for dealcoholization

- Positive for wine style modification
- Separation by physical means
  - Volatility
  - Molecular sieve
  - Combination
- In-Line Alcohol reduction
  - Combi

VinZero

CO_2 Membrane System

Contact

K+H process tec GmbH

blank@kh-tec.de

Einordnung der Alkoholreduzierung:
Reifeentwicklung Riesling am Beispiel 2012

15.09
Unreif

30.09
„Frühe“ Lese

14.10
„Späte“ Lese

30.10
Überreif

Alkoholreduzierung:
Sensorischer Vergleich mit Lesezeitpunkt

Wein

Riesling

Vergleich Lesezeitpunkt
unabhängig vom Alkoholgehalt

Lesezeitpunkt

Früh
17.09.2011
84°Oe

Spät
13.10.2011
97°Oe

Alkohol

Kontrolle
12%vol

angereichert
14%vol

Kontrolle
14%vol

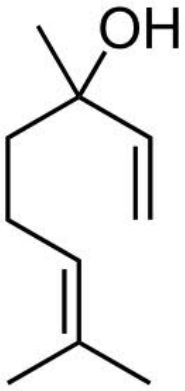
reduziert
12%vol

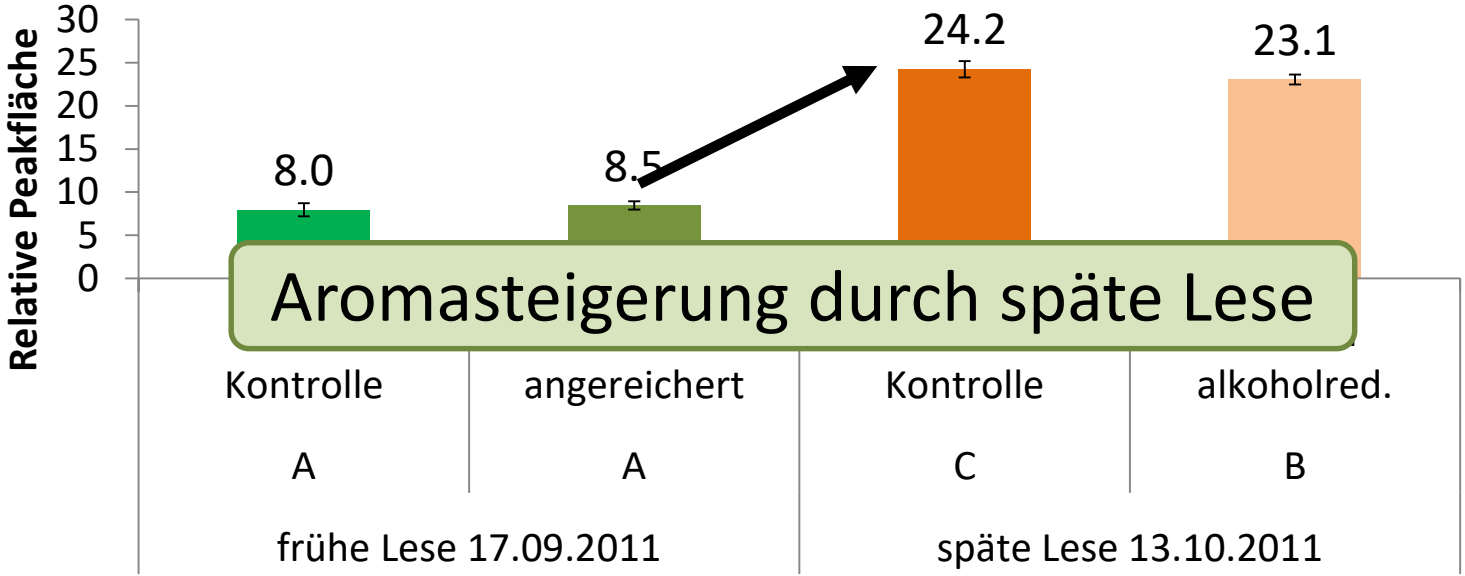
Blank *et al.*, 2013

Aromaintensität und Lesezeitpunkt

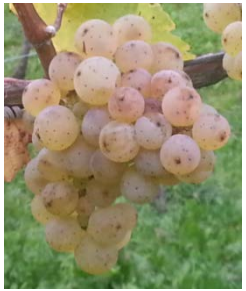
Riesling
2011

Linalool
Blumig

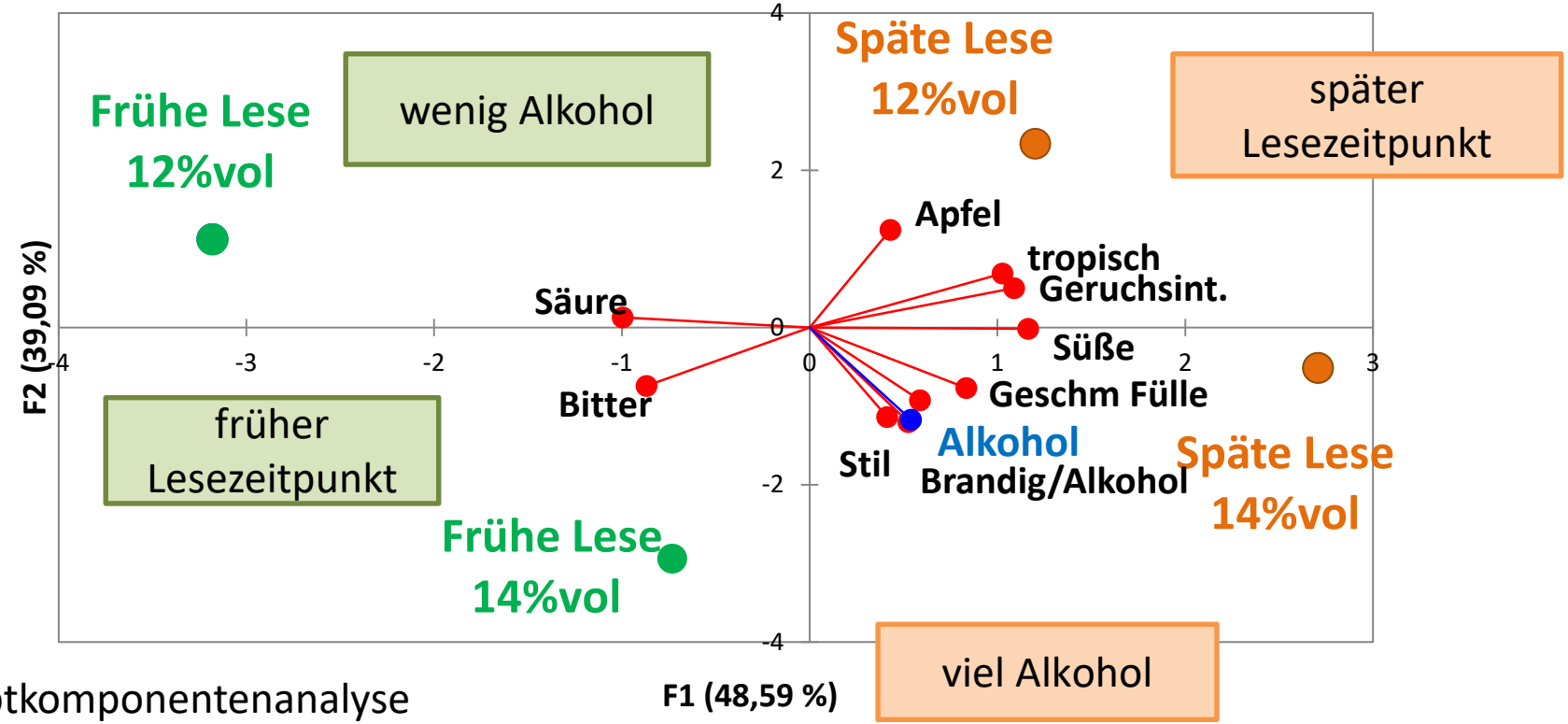




Paarweiser Vergleich
Fisher LSD (5%)



Alkoholreduzierung:
Sensorischer Vergleich mit Lesezeitpunkt



Hauptkomponentenanalyse
Riesling 2011
Sensorik: 23 Prüfer – 2 Wiederholungen

Zusammenfassung:
Teilweise Entalkoholisierung von Wein

- Technologische Verfahren
  - Osmotische Destillation gut geeignet für 2%vol. Reduzierung
  - Teilmengenverfahren vorteilhaft
- Modellierung
  - Inhaltsstoffveränderung lässt sich vorhersagen
- Sensorik
  - Geringer Einfluss der Verfahrenstechnik auf den Geruch
  - Geschmacksstilistik wird verändert
- Einordnung im Vergleich zum Lesezeitpunkt

Danksagung

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Projektträger BLE

Ganz besonders:

meine Familie

und Magali

Projektträger Bundesanstalt für Landwirtschaft und Ernährung

Gefördert durch:

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Eigene Veröffentlichungen/ Vorträge

Publikationen

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Blank, A.; Blankenhorn, D.; Sigler, J.; Amann, R.; Schmidt, O. 23.04.2013. *Membranverfahren zur teilweisen Reduzierung von Alkohol in Wein: Ein Verfahrensvergleich*. 61. Deutscher Weinbaukongress, Stuttgart

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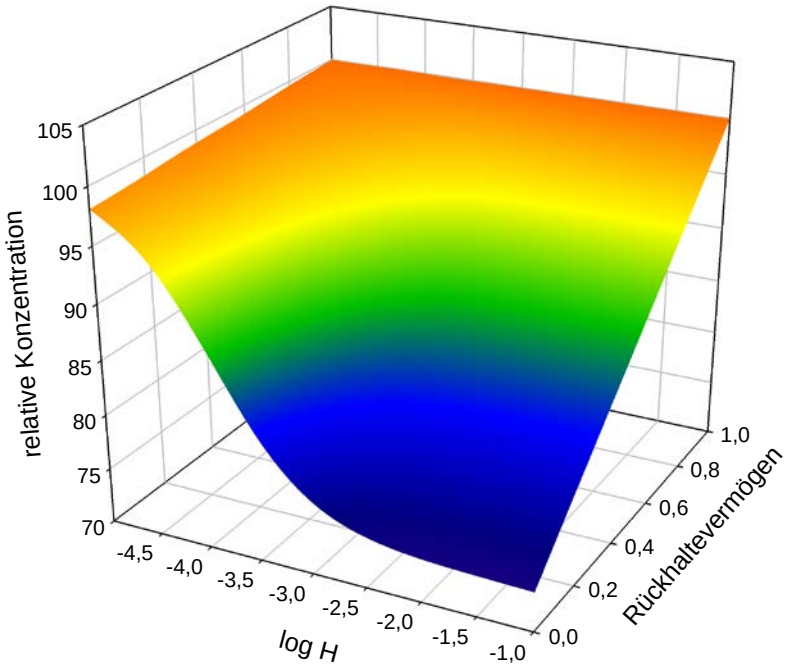
Zusammenfassung:
Teilweise Entalkoholisierung von Wein

- Technologische Verfahren
  - Osmotische Destillation gut geeignet für 2%vol. Reduzierung
  - Teilmengenverfahren vorteilhaft
  - Vergleichbar bei Spinning Cone Column, Umkehrosmose & Kombinationsverfahren
- Modellierung
  - Inhaltsstoffveränderung lässt sich vorhersagen
- Sensorik
  - Geringer Einfluss der Verfahrenstechnik auf den Geruch
  - Geschmacksstilistik wird verändert

Bewertung technische Verfahren: Verfahrensvergleich

- Alkoholreduzierung ist immer mit Aromaverlust verbunden!
- Teilmengenverfahren reduziert Aromaverluste auf ein Minimum.

- Osmotische Destillation OD
  - Trennung nach Flüchtigkeit
- Umkehrosmose UO
  - Trennung nach Molekulargewicht
- Spinning Cone Column SCC
  - Doppelte Destillation
- Kombination UO + OD
  - Kombination Destillation + Molekulargewicht



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Gil, M., Estévez, S., Kontoudakis, N., Fort, F., Canals, J.M., und Zamora, F. 2013. Influence of partial dealcoholization by reverse osmosis on red wine composition and sensory characteristics. European Food Research and Technology: 1-8.

Ikari, A., und Kubo, R. 1974. Behavior of various impurities in simple distillation of aquaeous solution of ethanol. Journal of Chemical Engineering of Japan 8 (4): 294-299.

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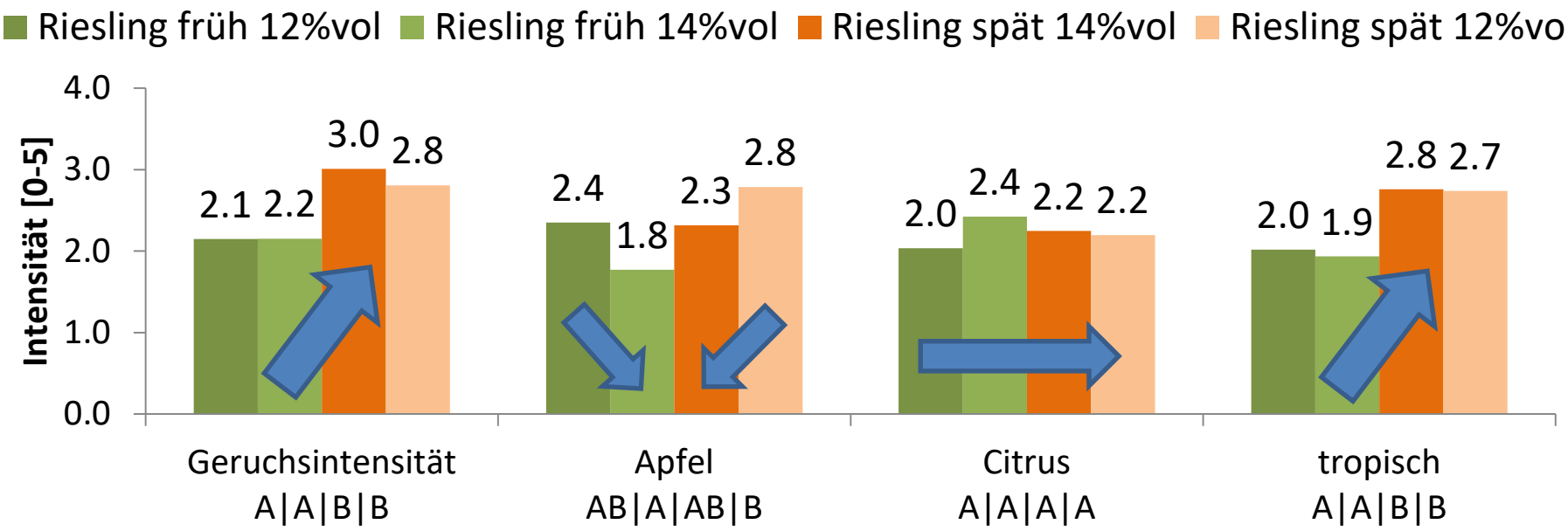
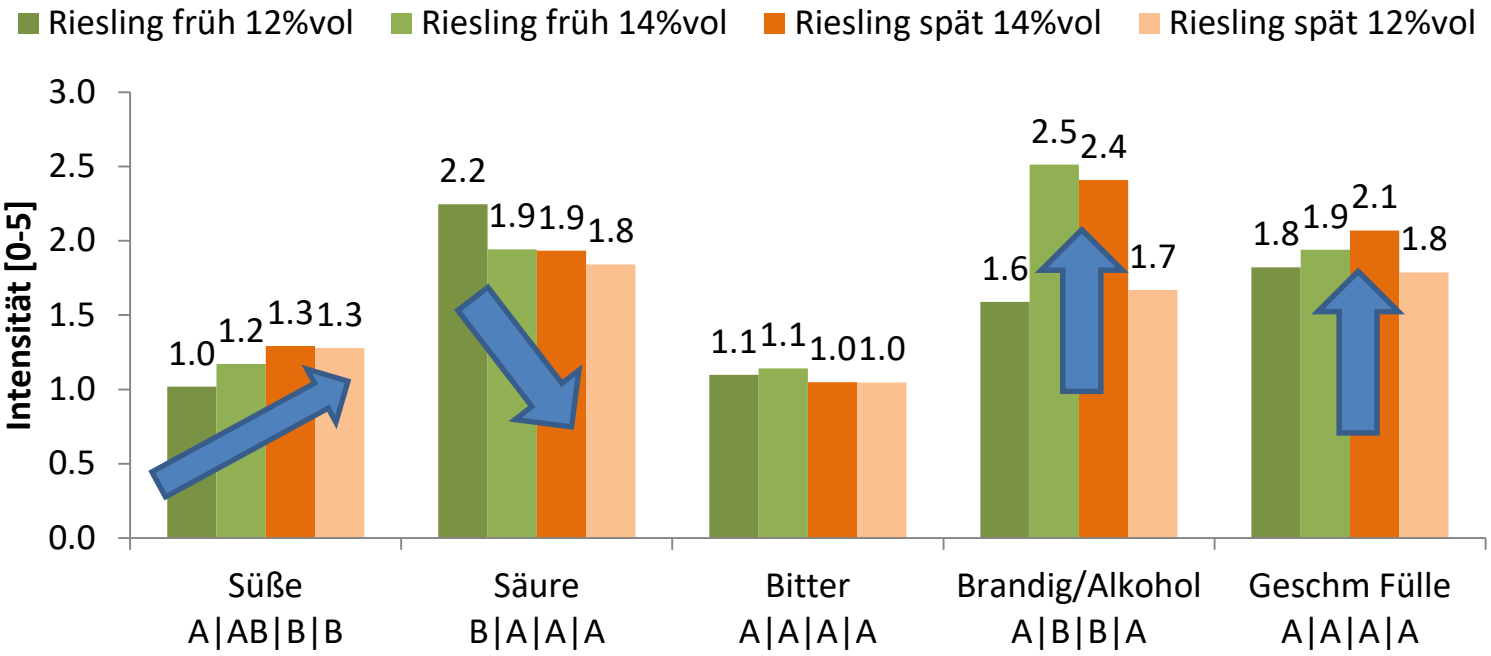
Änderung der Geruchsstilistik



Lesezeitpunkt > Alkohol

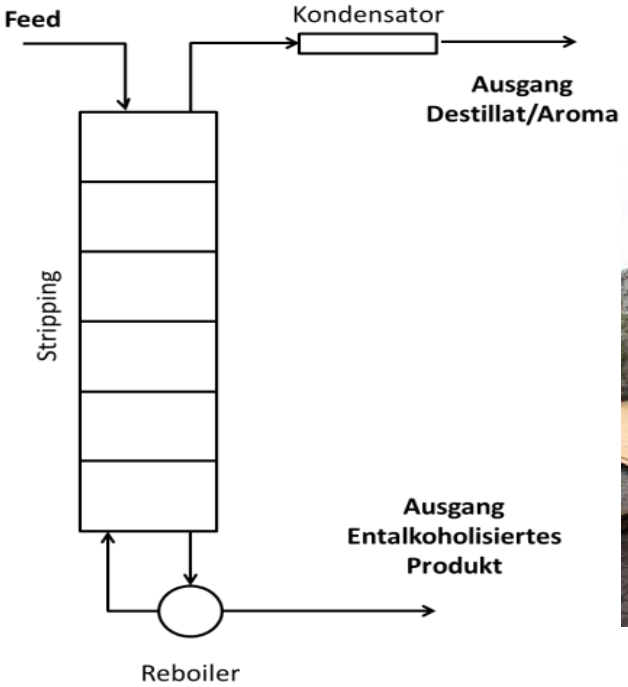
Paarweiser Vergleich
Fisher LSD 5%

Änderung der Geschmacksstilistik



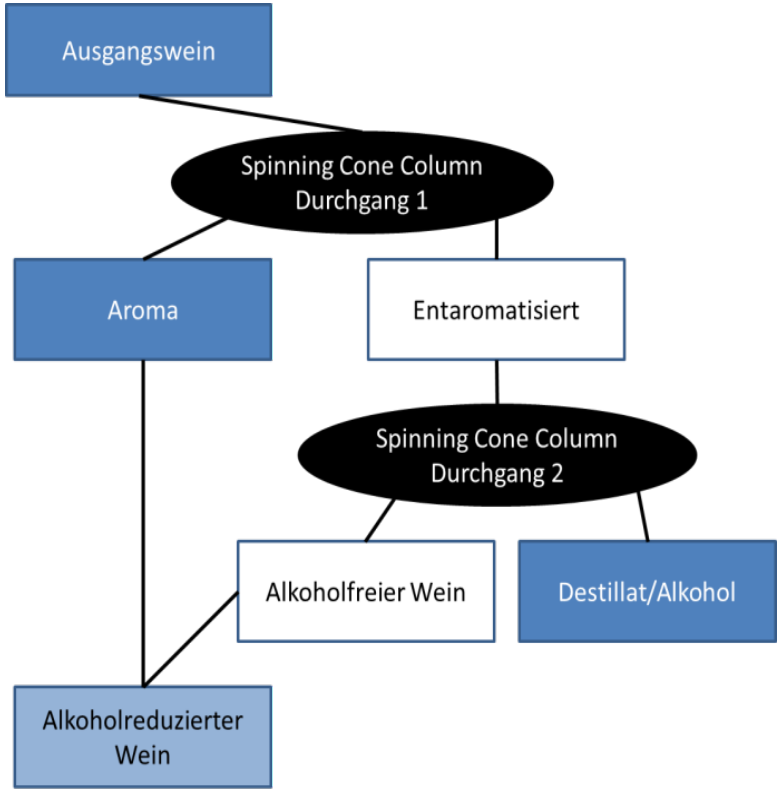
Paarweiser Vergleich Fisher
LSD 5%

Spinning Cone Column



- Stripping Kolonne
- Destillatives Verfahren
- Sehr guter Transfer der Inhaltsstoffe

Spinning Cone Column



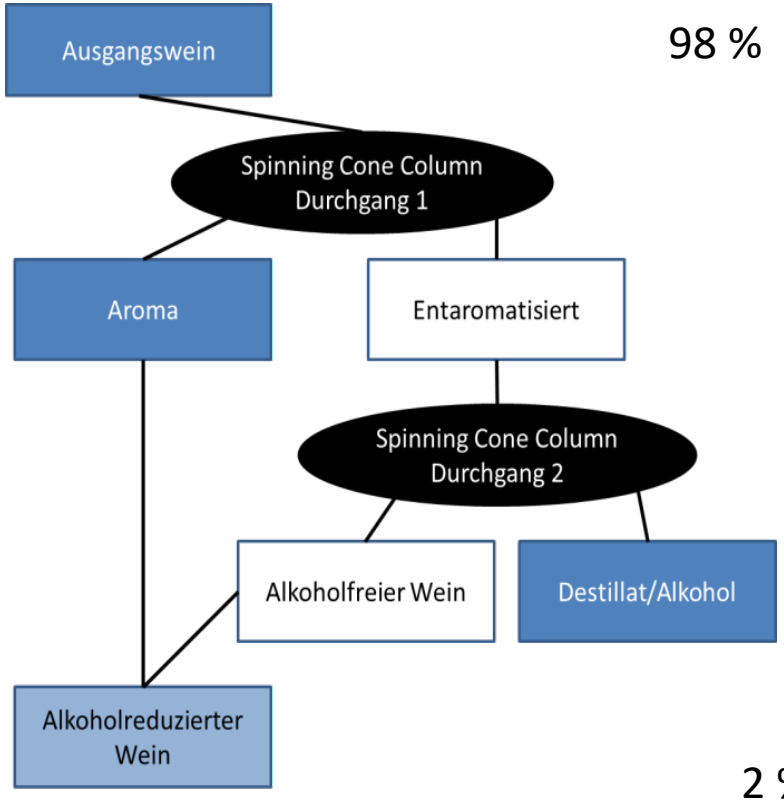
- 2 Stufen
- Abtrennung von Aroma
  ca. 2% der Masse
- Abtrennung von Alkohol
  ca. 20 % der Masse
- Rückverschnitt mit Ausgangswein

Versuche mit 5 verschiedenen Ausgangsweine

Ziel: Reduktion um 2%vol.

Versuche mit Modellierung

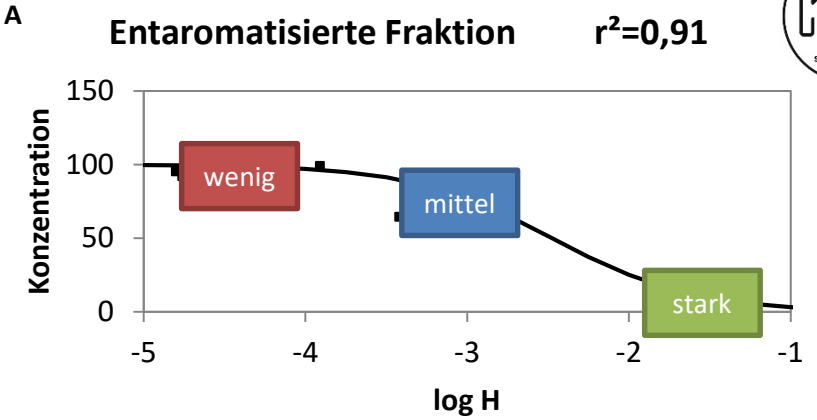
SCC


Ausgangswein
Spinning Cone Column Durchgang 1
Aroma
Entaromatisiert
Spinning Cone Column Durchgang 2
Alkoholfreier Wein
Destillat/Alkohol
Alkoholreduzierter Wein

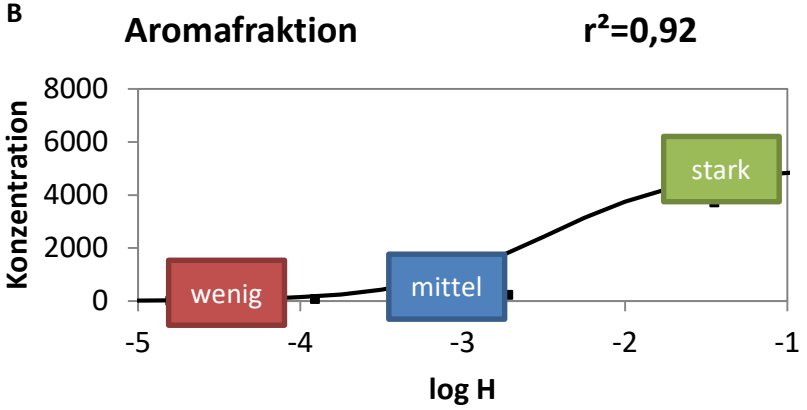
98 %

2 %

A

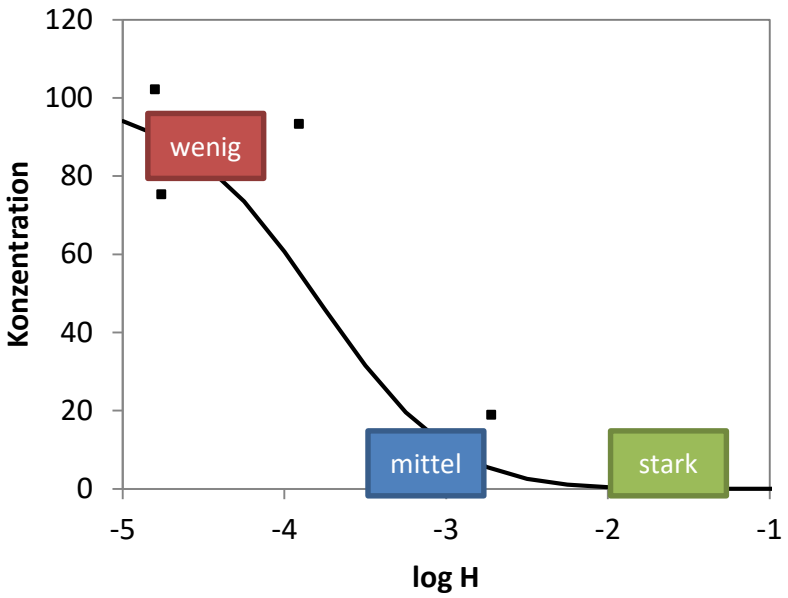

Entaromatisierte Fraktion
r²=0,91
Konzentration
150
100
50
0
wenig
mittel
stark
-5
-4
-3
-2
-1
log H

B

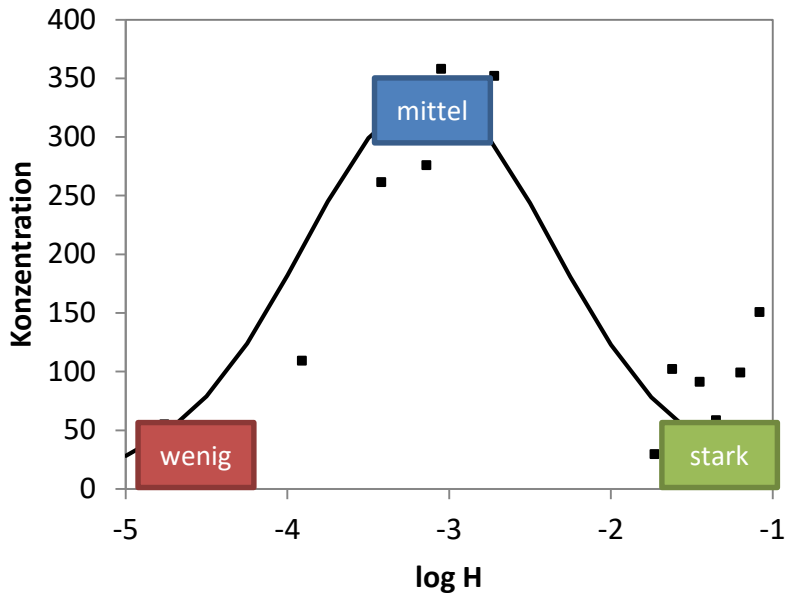

Aromafraktion
r²=0,92
Konzentration
8000
6000
4000
2000
0
wenig
mittel
stark
-5
-4
-3
-2
-1
log H

Alkohol vs. Entalkoholisiert

C

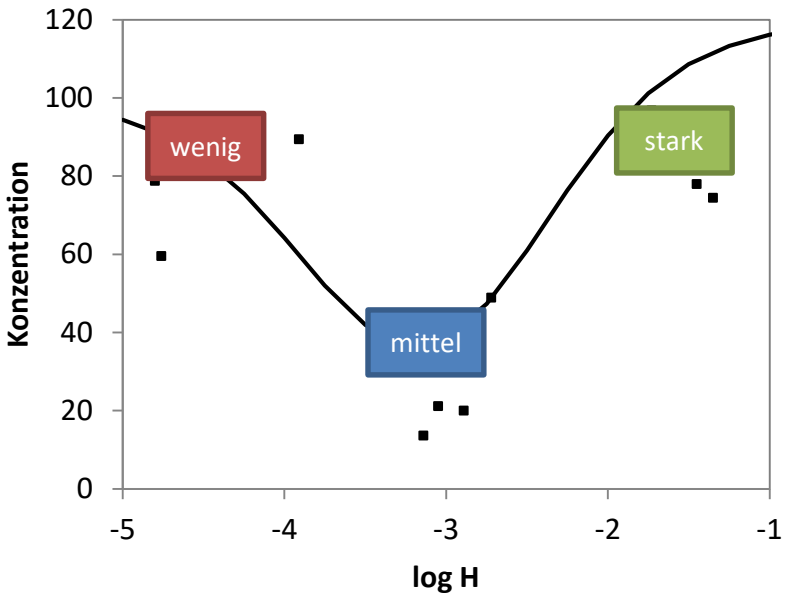


D

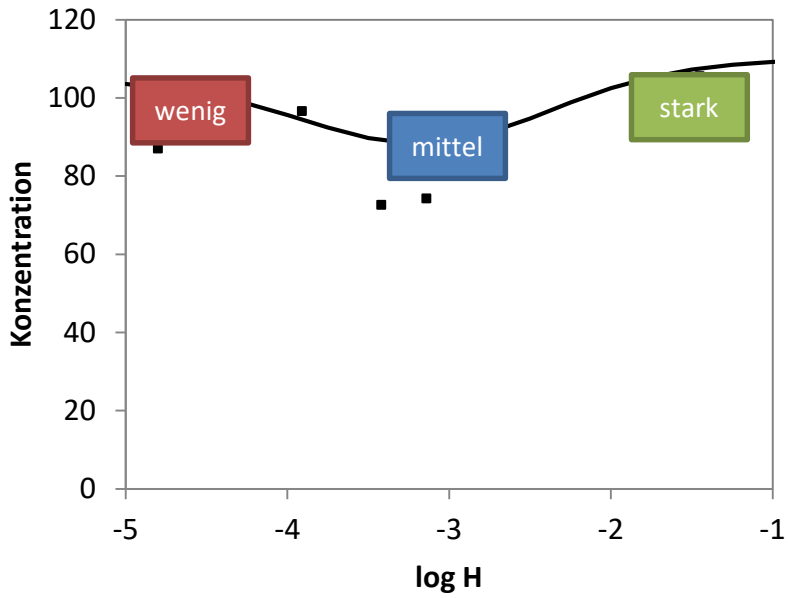


Alkoholfrei vs Rekombiniert

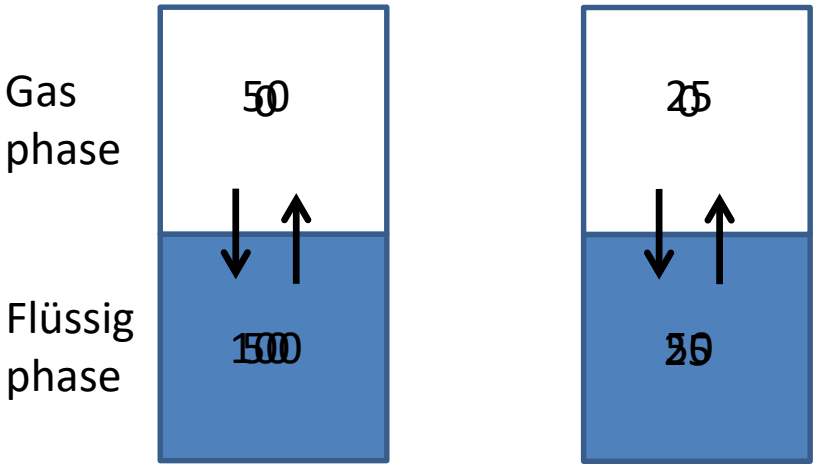
E



F



Modellierung auf Basis der Diffusion



Transfer ist abhängig von:

Ficksche Gesetz
F=k x A x delta c

Henry‘s Gesetz