STABILITY OF BELGIAN BEER: A VISION FOR ALL BREWERS

Philippe Cario, AEB Group
The purpose of this presentation is to give an overview of some new methods that have allowed to improve the protection of the final beer according to the evolution of the process and the regulation, through the use of technological auxiliaries SiO$_2$ and PVPP.

The research is focused on the optimal time and place within the brewery to add these stabilisers. Qualitative and economical aspects have been studied, considering the various equipments available in the Belgian breweries.
The idea to work recently on stabilisation improvement came from a legal requirement about flavor stability: legal SO$_2$ level $< 10$ppm.

Concept to inhibit the LOX from the mash tun to partly avoid interaction between fatty acid hydroperoxides and aldehydes in order to preserve beer freshness.
Brewhouse process

Fermentation - maturation process
- Traditional stabiliser: SiO\textsubscript{2}/Isinglass

Filtration – BBT – Bottling process
- Traditional antioxidant: Ascorbic Acid / SO\textsubscript{2}
- Traditional colloidal stabiliser: PVPP, SiO\textsubscript{2}, Enzymes
Brewhouse process

Recent technology: Antioxidant from the mashing
Tannins / traditionnal antioxidant

ANTIOXIN® SBT

Fermentation - maturation process

Selected yeast extract with high glutathione content

FERMOPLUS® GSH

Filtration – BBT – Bottling process

Limit or avoid any antioxidant treatment
ITT, Tbar method and triangular tests to measure the impact on flavour stability
Innovate, improve

As we achieved for flavour stability, is it possible for brewers to partly or fully anticipate the risk of colloidal instability from the brewhouse?

Many publications have related the relationship between flavour and colloidal stability from polyphenols:

► XIII Chair De Clerck was dedicated on the « Polyphenol Paradox in Alcoholic Beverages »
► D. Callemien and S. Collin:
  - Involvement of Flavanoids in Beer Color Instability during Storage. J. Agric. Food Che. (2007);
  - Structure, organoleptic properties, quantification methods, and stability of phenolic compounds in beer. A review. Food Reviews International (2010).
Objective: remove or inhibit the negative components coming from raw materials (malt, hop, water) before starting mechanical and physical actions during the beer process, such as boiling, oxygen addition...
Main problem in colloidal stabilisation

To increase their efficiency, most of the big brewers have drastically increased their °P from 12 to 15, up to 20:

- Colloidal stabilisation during maturation and kieselguhr filtration might be insufficient or expensive.
- Wort composition has changed while the yeast remains the same (stress, viability, vitality, floculation/time of maturation...).
- Seasonality of production = variation in the post fermentation process.
- Importance to act both on proteins and polyphenols to preserve a great equilibrium between flavour and colloidal stability.
Belgium special beers: a multitude of process, difficult stabilisation in practice

- High gravity wort.
- Diversity in the brewery size and equipments.
- Large number of malt varieties, lot numbers, regular change of protein and polyphenol content.
- Belgium beer is exported worldwide = obligation to be stable until the beer has reached the final consumer.
Haze formation: mode of combination between proteins and polyphenols

- **Chill haze** \((0.1 - 1 \mu)\): reversible association between small polymerised polyphenols and proteinaceous material. This haze may increase with beer ageing.

- **Permanent haze** \((1 - 10 \mu)\): active precursors in haze formation leading to the formation of polymerised polyphenols (oxidation of proanthocyanidins in particular).
The flavonoids which have a strong role in precipitating colloidal haze:

Monomeric flavanoids

- (+)-Catechin

Dimeric flavanoids

- Prodelphinidin B3
- Procyanidin B3

Siebert and Lynn (J.Am.Soc.Brew., 1997) also described the combination between proline molecule from proteins and polyphenols leading to haze.
PVPP – Synthetic insoluble polymer, polyvinylpolypirrrolidion and polyphenols binding involves hydrogen bonding and hydrophobic stacking, corresponding to the same mechanism as protein (Polyproline) and polyphenols binding. P. Aron and T. Shelhammer, J. Inst. Brew. 2010.
Silica is a processing aid able to achieve a selective removal of haze sensitive proteins.

- **Silica gels**
  - Silica gel under humidity controlled (xero, hydro)

- **Colloidal silica**
  - Silica sol under surface area controlled
Silica Sol Process Overview

- Water
- Sodium Silicate
- Process Water
- Steam
- Condensate
- Particle Growth
- Acid
- Caustic
- Concentration
- Quality Assurance
- Water (Recycled)
- QA Tank
- Reactor
- Particle Growth
- Modified Products
- QA Tank
- Reactor
- Chemical Upgrade/Optimization
- Finished Storage
- SPINDASOL® SB1 (wort Clarification)
- SPINDASOL® SB3 (Beer Maturation, RBT)
- EBC Glasgow 2011
High gravity: Important charge in the cold wort

Hot wort from whirlpool to heat exchanger <10EBC

Cold wort after heat exchanger to the fermenter >80EBC
High charge of the wort

- Difficult propagation, attenuation
- Low floculation
- Short DE or CF filtration cycle
- High stabilisation regime to achieve the proper shelf-life
- Possible problem of organoleptic equilibrium

Qualitative risks
Cost inconvenient (yield)
Clarifying the wort is a technique widely developed since 15 years.

Clarifying and stabilising the wort in the same time represents a recent innovative action which brings many advantages to the brewery, especially to the special beer producers.

The sooner will be the addition of stabiliser within the brewhouse, the better adsorption will be observed on the polyphenol and protein content.
End of boiling: comparative use of clarifying vs pre-stabilising agent

<table>
<thead>
<tr>
<th>Belgium beer 16°C</th>
<th>Control</th>
<th>Clarifier 1</th>
<th>Clarifier 2</th>
<th>Stabiliser 1</th>
</tr>
</thead>
<tbody>
<tr>
<td>Industrial application</td>
<td>No addition</td>
<td>Carageen 4g/hL</td>
<td>SiO₂ Silica sol 20 g/hL</td>
<td>PVPP + SiO₂ 20 g/hL</td>
</tr>
<tr>
<td>Average haze whirlpool to fermenter (cold wort)</td>
<td>43,4 EBC</td>
<td>36,9 EBC</td>
<td>27,9 EBC</td>
<td>16,8 EBC</td>
</tr>
<tr>
<td>Turbidity after forcing test*</td>
<td>9,4 EBC</td>
<td>5,0 EBC</td>
<td>5,0 EBC</td>
<td>3,4 EBC</td>
</tr>
</tbody>
</table>

- 3 weeks of maturation (-1 – 0°C)
- Forcing test after bottling: 6 days at 60°C + 1 day at 0°C
Comparative turbidity of 2 different malt seasons treated with PVPP-SiO$_2$, in the same brewery

<table>
<thead>
<tr>
<th></th>
<th>Control</th>
<th>POLYGEL®BH 20g/Hl</th>
<th>POLYGEL®BH 30g/Hl</th>
</tr>
</thead>
<tbody>
<tr>
<td>Haze (EBC90°)</td>
<td>44,5</td>
<td>31,8 (-29,5%)</td>
<td>26,4 (-40%)</td>
</tr>
<tr>
<td>2010 winter malt</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Haze (EBC90°)</td>
<td>38</td>
<td>16 (-58%)</td>
<td>13,6 (-63%)</td>
</tr>
<tr>
<td>2011 winter malt</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

haze sampled every 30hL
Wort composition, trub compaction: many benefits to anticipate the colloidal stabilisation
Difference of trub = reduction of stabilising action before beer filtration

Without treatment

With stabiliser

Industrial application
Any risk to reach a low haze due to any excess of clarification or pre-stabilisation with PVPP and/or SiO₂?

Haze follow up of the cold wort transferred to the fermenter

Impact on yield

Turbidity (EBC 90°)

Time (min.)
Follow up of the yeast population during fermentation

Yeast population (M cells/ml)

- **vitality**
- **viability**

**Yeast population**

- **Control**
- **POLYGEL BH**

Days

0 2 4 6 8 10 12
Follow up of the fermentation

Density during fermentation

- Control
- POLYGEL BH
Analysis of the lipids

Analysis of the lipids at the beginning of boiling

- Palmitic acid
- Stearic acid
- Oleic acid
- Linoleic acid
- Linolenic acid

Average of the worts treated with POLYGEL BH
Control wort
Analysis of the lipids

Analysis of the lipids at the end of boiling

- Palmitic acid
- Stearic acid
- Oleic acid
- Linoleic acid
- Linolenic Acid

- Average of the worts treated with POLYGEL BH
- Control wort
Advantages to clarify and pre-stabilise the wort

- Wort is clarified and standardised as well as all the steps post-brewing
- Yeast is less stressful (propagation, linearity between generation)
- Flocculation is faster
- Performance of kieselguhr filtration is improved (dP, g/hL, hL/cycle):
  - No increase of PVPP or SIO₂
  - No loss of admissible volume in the KG filter
- Quality and cost of stability are under control, the most effective in global (Stab + Kies.)
The large number of applications and the feedback from Prof. Maudoux (UCL) and chief brewers allowed to define a more efficient injection point.

The addition of the PVPP and SiO$_2$ complex is ideally added right after the saccharification of the wort.

Iodine test before adding POLYGEL® BH
### 17°P Wort composition
Malt 70% - Gritz 30%

<table>
<thead>
<tr>
<th>Polyphenols measurement</th>
<th>Control</th>
<th>Addition of POLGEL® BH</th>
<th>Total reduction</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dosage during boiling</td>
<td>230 ppm</td>
<td>187 ppm</td>
<td>-18.7%</td>
</tr>
<tr>
<td>Dosage at the end of saccharification</td>
<td>230 ppm</td>
<td>151 ppm</td>
<td>-34.3%</td>
</tr>
</tbody>
</table>
It has been demonstrated that $\text{SiO}_2$ has an action on sensible proteins and does not impact the foam stability.

**Impact on proteins**

(measurment on cold wort)

**wort composition**

70% malt 30% gritz
Follow up of maturation

After 2 days of maturation (same yeast, same generation), we can measure 4M cells/ml with PVPP-SiO$_2$ complex (on right), 8M cells/ml for the control (left).

Major impact on filter run in case the brewery is not equipped with centrifuge.
Check the potential stability of the green beer before DE or CF filtration and bottling

- Tannic acid (EBC Analytical 9.40)
- Chapon Method (EBC Analytical 9.41)

During filtration, once added PVPP-SiO₂ complex during bottling, a single addition of silica gel will be sufficient to insure a stable ageing: between 10 and 20g/hL according to the final gravity of beer for the years 2010, 2011 and 2012.
Comparing the shelf life from 2 treatments

100% Malt 17°P
ANTIOXIN ® SBT

CASE 1

Pre-stabilising treatment – End of Boiling
Polygel® BH

30 g/hL

Settlement in the whirlpool (trub)

Fermentation Maturation

15 g/hL Siligel®S
Security Stabilisation

CASE 2

0 g/hL

PVPP + SiO₂
20/40 g/hL

Filtration Bottling

Final Stabilisation
Qualitative results and impact on the process

<table>
<thead>
<tr>
<th></th>
<th>Turbidity final beer (EBC 90°)</th>
<th>Turbidity Final beer (EBC 25°)</th>
<th>Turbidity after forcing test 1 year (EBC 90°)</th>
<th>Polypeh-nols Final beer (ppm)</th>
<th>Polypeh-nols after forcing test (ppm)</th>
<th>Sensible proteins on final beer (EBC 9.40)</th>
<th>Sensible proteins after forcing test (EBC 9.40)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Case 1</td>
<td>0,54</td>
<td>0,17</td>
<td>1,71</td>
<td>155</td>
<td>157</td>
<td>1,2</td>
<td>1,7</td>
</tr>
<tr>
<td>Case 2</td>
<td>0,57</td>
<td>0,18</td>
<td>1,65</td>
<td>159</td>
<td>162</td>
<td>1,1</td>
<td>1,8</td>
</tr>
</tbody>
</table>

**DE filtration**

<table>
<thead>
<tr>
<th></th>
<th>Case 1</th>
<th>Case 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Av. dosage 17°P beer (pre-coat included)</td>
<td>120 g/hL</td>
<td>180 g/hL</td>
</tr>
</tbody>
</table>

**Yield**

<table>
<thead>
<tr>
<th></th>
<th>Case 1</th>
<th>Case 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Av. total process (brewhouse + fermentation + Mat &amp; filtration)</td>
<td>4,3%</td>
<td>5,1%</td>
</tr>
</tbody>
</table>
Possible stabilisation according to the brewer’s process: Belgium cases
Brewhouse process
Antioxidant from the mashing
Main stabilisation after saccharification

Fermentation - maturation process
Selected yeast extract with high glutathione content

Filtration – BBT – Bottling process
Limitate stabiliser addition
Avoid any antioxidant treatment
Case 1: Traditional technology

Brewhouse:
- Mashing
- Press filter or lautet tun
- Wort kettle
- Whirlpool or decanter

Ferm.:
- Wort

Mat.:
- SiO₂ – Silica gel
- SILIGEL® S
- Optimise contact time

Filtration:
- Stable beer!

12-14 Sept. 2012
XIV De Clerck Chair
Case 2: Traditional technology with a PVPP filter

**CLARIFICATION**

**Brewhouse**
- Mashing
- Press filter or lauter tun
- Wort kettle
- Whirlpool or decantor

**Ferm.**
- Wort

**Mat.**
- SiO₂ – Silica gel
  - SPINDASOL® SB1
  - Optimise contact time
- 1

**Filtration**
- PVPP reg.
- 2

Stable beer!

12-14 Sept. 2012
XIV De Clerck Chair
Case 3: No DE filtration

In the case of refermentation, white/wheat beer, the troubles in the bottle will come exclusively from the yeast = better flavour and colloidal stability.

Brewhouse
- Mashing
- Press filter or lauter tun
- Wort kettle
- Whirlpool or decantor

Ferm.
- wort

Mat.
- trub

Possible refermentation

SiO₂ – Silica sol
SPINDASOL® SB3
Optimise contact time

Stable beer!

PVPP-SiO₂
POLYGEL® BH

12-14 Sept.2012
XIV De Clerck Chair
Case 4: Unitank technology

In the case of unitank, the addition of SiO₂ under liquid form will allow to stabilise, speed up floculation, without recirculation and dangerous movement within the vessel.

Brewhouse

Mashing
Press filter or lautering tun
Wort kettle
Whirlpool or decantor

Combined Ferm. & mat.

SiO₂ – Silica sol
SPINDASOL® SB3
Through the CIP of the tank (spray ball)

Possible refermentation

Stable beer!

Purge yeast + SiO₂

trub

wort
Belgium cases 1 to 4: reproducible in all type of brewery

Experience has shown that these stabilisation scenarios could be adapted from a micro up to an industrial brewery.
CONCLUSION

It is possible to both improve the freshness and the stabilisation of the beer from the brewhouse.

The use of antioxidant from the mash tun inhibits the LOX and brings a better beer ageing (ASBC 2007).

Clarification and stabilisation from the brewhouse allows a better standardisation of the process and a better shelf life of beer.

Quality and the yield during the process can be improved.

Both antioxidant and « pre »-stabilisers are fully eliminated during production.
Acknowledgements

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- UCL
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Thank you for your attention

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