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Louvain Institute of Biomolecular Science and Technology (LIBST)

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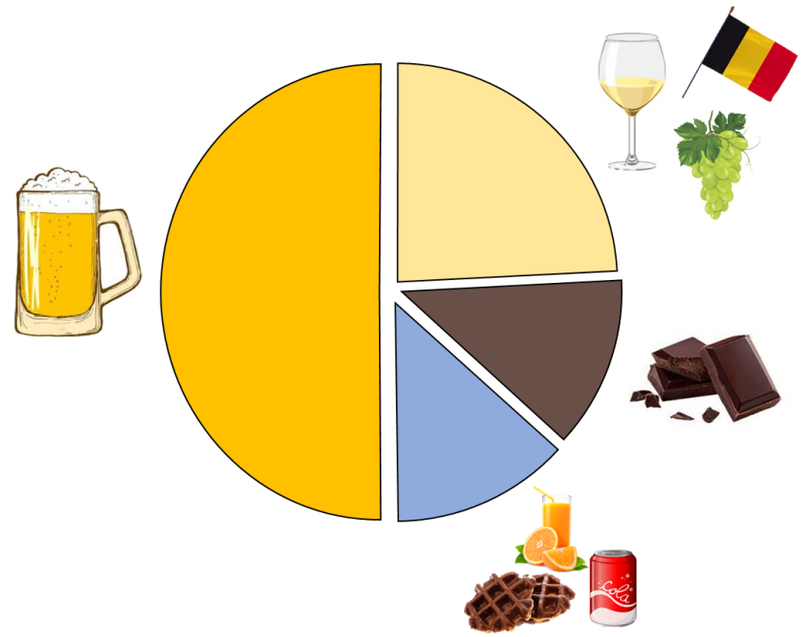
Flavor research

INBr

Unité de brasserie et des industries alimentaires

30 ans de recherche et de savoir-faire sur les flaveurs d'aliments fermentés



4 axes de recherche :

- R1. Qualité organoleptique et stabilité au cours du vieillissement
- R2. Les polyphénols et le pouvoir antioxydant
- R3. Découverte de nouvelles molécules dans le houblon
- R4. Enjeux environnementaux

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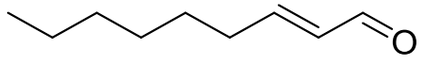
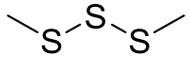
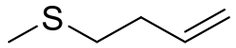
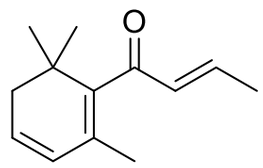
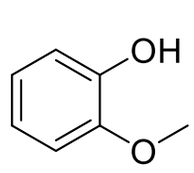
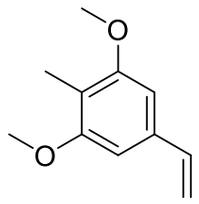
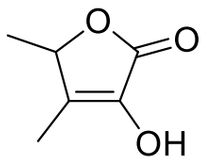
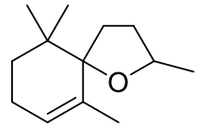
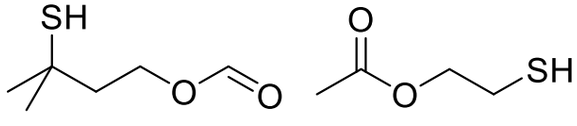
R_1 Qualité organoleptique et stabilité au cours du vieillissement

Élucidation des voies menant aux défauts de vieillissement des bières

les principales contributions internationales de notre laboratoire depuis 1990

11 thèses de doctorat (PhD):

- ⇨ trans-2-Nonénal (gout de carton) 
  S. Noël (98) G. Lermusieau (02)
  C. Liégeois (04)
- ⇨ Diméthyltrisulfure (choux) 
  L. Gijs (03)
- ⇨ Méthional (pomme de terre) 
  Ph. Perpète (00)
- ⇨ bêta-Damascénone (fruits rouges) 
- ⇨ Guaiacol, 4MP, 4-Vinylsyringol (fumé, vieux)  
  D. Callemien (07)
- ⇨ Sotolon (Madeire) 
  C. Scholtes (15)
- ⇨ Composés dérivés de théaspirane (Sauternes, fruits sec) 
  C. Vermeulen (05), J. Gros (12), Tran T.T.H. (14)
- ⇨ Thiols polyfunctional (oignon, empyreumatique, ribes, argumes, ..)
  C. Silva (22)



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R_1 ⇨ trans-2-Nonénal (goût de carton)

Composés de vieillissement des lagers

J. Inst. Brew., 105 (1999), 269-274

The use of Oxygen 18 in appraising the impact of oxidation process during beer storage

by Sophie Noël, Nadine Metais, Sabine Bonte, Etienne Bodart, Fabrice Peladan, Stéphane Dupire and Sonia Collin

Unité de Brasserie et des Industries Alimer
2/Bte 7, B-1348 Louvain-la-Neuve, Belgiu

Received 11th June 1998

To appraise the incidence of oxygen in
$18O_2$ was injected into the headspace
bombardment and isotopic mass spe
Although oxygen did cause considerabl
it was not incorporated into the carbon
is not due to lipid oxidation but to wort
to be a good indicator of beer flavou
(addition of polyvinyl pyrolidone pow
investigated.

J. Am. Soc. Brew. Chem. 1999, 57(1), 29-33.

Nonoxidative Mechanism for Development of *trans*-2-Nonenal in Beer

G. Lermusieau, S. Noël, C. Liégeois, and S. Collin,[1] *Unité de Brasserie et des Industries Alimentaires, Université Catholique de Louvain, Place Croix du sud 2/Bte 7, B-1348 Louvain-la-Neuve, Belgium*

ABSTRACT

J. Am. Soc. Brew. Chem. 57(1):29-33, 1999

Oxidation is usually recognized as the major cause of development o
a stale flavor in beer. However, no significant difference in *trans*-2-nonena
concentration has been observed between oxygen-receiving and oxygen
free beers after aging. Moreover, $^{18}O_2$ was not incorporated into the carbony
fraction, indicating that the cardboard flavor in beer is not due to lipi
oxidation in the bottle. As shown by adding deuterated nonenol to the pitchin
wort, nonenal oxidation and sulfitic adduct degradation were also inefficien
pathways of *trans*-2-nonenal synthesis. On the other hand, wort nonena
linked to amino acids and proteins revealed to be able to release nonena
at the beer pH. The measured nonenal potential proved a good indicato
of beer staling; therefore, UV spectroscopy was used to find which experi
mental conditions destabilize *trans*-2-nonenal precursors in wort. Laboratory
scale experiments confirmed that SO_2 can reduce both lipid autooxidatio
and the nonenal potential rise while the wort is boiling.

Keywords: Aging, Oxidation, Schiff base, Stability

oxidized character, but the chief flavor change was to the sweet, caramelized note which is quite different from the cardboard character usually associated with beer staling. It is well known that the major contributor to this stale flavor is *trans*-2-nonenal (8,21),

J. Agric. Food Chem. 1999, 47(10), 4323-4326.

J. Agric. Food Chem. **1999**, *47*, 4323–4326

Release of Deuterated Nonenal during Beer Aging from Labeled Precursors Synthesized in the Boiling Kettle

Sophie Noël, Catherine Liégeois, Guillaume Lermusieau, Etienne Bodart, Camille Badot, and Sonia Collin*

Unité de Brasserie et des Industries Alimentaires, Université Catholique de Louvain, Place Croix du Sud 2/Bte 7, B-1348 Louvain-la-Neuve, Belgium

The use of labeled nonenal enabled the demonstration that the appearance of the cardboard flavor in finished beer comes from lipid auto-oxidation during wort boiling and not from lipoxygenasic activity during mashing. Free *trans*-2-nonenal produced by linoleic acid auto-oxidation in the kettle disappears, owing to retention by wort amino acids and proteins. This binding linkage protects *trans*-2-nonenal from yeast reduction but is reversible, allowing release of the compound at lower pH during aging. Labeled *trans*-2-nonenal is detected after aging when deuterated precursors form in the boiling kettle. The amount of alkenal released correlates with the concentration of reversible associations in the pitching wort. This work brings new illumination to the formation of *trans*-2-nonenal and overturns many previous hypotheses. It also explains why a reduction in the beer pH intensifies the cardboard flavor.

R_1 ⇨ Diméthyltrisulfure (chou)

⇨ Méthional (défaut de moût)

J. Agric. Food Chem. 2000, 48(12), 6196-6199.

3-Methylthiopropionaldehyde as Precursor of Dimethyl Trisulfide in Aged Beers

Laurence Gijs, Philippe Perpète, Aurore Timmermans, and Sonia Collin*

Unité de Brasserie et des
Université catholique de Lo

J. Am. Soc. Brew. Chem. 2002, 60(2), 68-70.

Effect of the Reducing Power of a Beer on Dimethyltrisulfide Production During Aging

Laurence Gijs and Sonia Collin,[1] *Unité de Brasserie et des Industries Alimentaires, Faculté d'Ingénierie biologique, agronomique et environnementale, Université catholique de Louvain, Croix du Sud, 2 bte 7, B-1348 Louvain-la-Neuve, Belgium*

ABSTRACT

J. Am. Soc. Brew. Chem. 60(2):68-70, 2002

In synthetic matrices, degradation of 3-methylthiopropionaldehyde, the main precursor of dimethyltrisulfide in aged beer, is influenced by the presence of anti- and prooxidants. Surprisingly, the reducing power of the beer proved not to be a key determinant of dimethyltrisulfide production during beer aging. However, other properties of anti- and prooxidants must be taken into account, such as the ability to bind 3-methylthiopropionaldehyde (sulfitic adducts) or methanethiol (copper complexation). This binding leads, respectively, to a higher or lower dimethyltrisulfide level.

Keywords: Beer aging, Methional

Chemicals

Ethylmethy
chased from
ascorbic acid
ethanol p.a.
potassium me

Beer Produc

A control b
other product
added during

Composés de vieillissement des lagers

⇨ bêta-Damascénone (fruits rouges)

J. Agric. Food Chem. 2002, 50(20), 5612-5616.

How Low pH Can Intensify β-Damascenone and Dimethyl Trisulfide Production through Beer Aging

LAURENCE GIJS, FABIENNE CHEVANCE, VESNA JERKOVIC, AND SONIA COLLIN*

Unité de Brasserie et des Industries Alimentaires, Faculté d'Ingénierie biologique, agronomique et environnementale, Université catholique de Louvain, Croix du Sud, 2 bte 7, B-1348 Louvain-la-Neuve, Belgium

Flavor quality is of major importance to the consumer, but the flavor characteristics of beer appear to deteriorate greatly with time, at a rate depending on the composition of the beer and its storage conditions (notably pH). Prior to identifying the influence of pH on the development of the most intense staling flavors found in aged lager beers, the corresponding key flavor compounds were determined by aroma extract dilution analysis. In addition to *trans*-2-nonenal, β-damascenone seems at least as important in the flavor of aged beer. Ethyl butyrate, dimethyl trisulfide, 2-acetylpyrazine, 3-(methylthio)-propionaldehyde, 2-methoxypyrazine, maltol, γ-nonalactone, and ethyl cinnamate are also relevant to the sensory profile of aged beer. Upon aging, a beer having a higher pH produces less β-damascenone, because acid-catalyzed glycoside hydrolysis is decreased. On the other hand, it produces more 3-(methylthio)propionaldehyde, owing to Strecker degradation of methionine. Raising the beer pH additionally causes the release of 3-(methylthio)propionaldehyde from sulfitic adducts. These adducts, more stable at a lower pH, protect the aldehyde against premature oxidation to 3-(methylthio)propionic acid, thus making it available for dimethyl trisulfide formation during aging.

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R_1 ⇨ 4-Vinylsyringol (fumé, vieux)

J. Agric. Food Chem. 2006, 54(4), 1409-1413.

Identification of a Stale-Beer-like Odorant in Extracts of Naturally Aged Beer

DELPHINE CALLEMIEN, SÉBASTIEN DASNOY, AND SONIA COLLIN*

Unité de Brasserie et des Industries Alimentaires, Université catholique de Louvain, Croix du Sud, 2/bte 7, B-1348 Louvain-la-Neuve, Belgium

For a long time, beer staling has been a prime concern in brewery research. Yet, to improve flavor stability, better knowledge of all chemicals involved is still needed. From our aroma extract dilution analyses (AEDA) applied to naturally aged lager beers emerged an old-beer-like odorant at $RI_{CP-SIL\ 5\ CB} = 1532$ and $RI_{FFAP} = 2809$, with a FD value close to that of *trans*-2-nonenal (the well-known cardboard off-flavor found in aged beers). Specific phenol extraction, GC cold trapping, and mass spectrometry (electron impact and chemical ionization) Although already mentioned in some fresh beers, this co involved in the aging process of lager beers.

Composés de vieillissement des lagers

⇨ Guaiacol (torréfié), 4-methylphénol

J. Agric. Food Chem. 2014 62(39), 9522-9528.

Guaiacol and 4-Methylphenol as Specific Markers of Torrefied Malts. Fate of Volatile Phenols in Special Beers through Aging

Caroline Scholtes, Sabrina Nizet, and Sonia Collin*

Unité de Brasserie et des Industries Alimentaires, Earth and Life Institute (ELIM), Université catholique de Louvain, Croix du Sud 2, Box L7.05.07, B-1348 Louvain-la-Neuve, Belgium

ABSTRACT: Phenol-specific extracts of 12 Belgian special beers were analyzed by gas chromatography hyphenated to olfactometry (AEDA procedure) and mass spectrometry (single ion monitoring mode). As guaiacol and 4-methylphenol were revealed to be more concentrated in brown beers (>3.5 and >1.1 μg/L, respectively), they are proposed as specific markers of the utilization of dark malts. Analysis of five differently colored malts (5, 50, 500, 900, and 1500 °EBC) allowed confirmation of high levels of guaiacol (>180 μg/L; values given in wort, for 100% specialty malt) and 4-methylphenol (>7 μg/L) for chocolate and black malts only (versus respectively <3 μg/L and undetected in all other worts). Monitoring of beer aging highlighted major differences between phenols. Guaiacol and 4- aging, reaching levels not far from their sens gradually degraded in POF(+)-yeast-derived b L, the vanillin concentration increased over a Beers initially showing an above-threshold le

KEYWORDS: *special malts, phenols, brown b*

Composés de vieillissement des ales

⇨ Sotolon (Madeire)

J. Agric. Food Chem. 2015, *63*(11), 2886-2892.

How Sotolon Can Impart a Madeira Off-Flavor to Aged Beers

Caroline Scholtes, Sabrina Nizet, and Sonia Collin*

Unité de Brasserie et des Industries Alimentaires, Earth and Life Institute ELIM, Faculté d'Ingénierie biologique, agronomique et environnementale, Université catholique de Louvain, Croix du Sud, 2 box L7.05.07, B-1348 Louvain-la-Neuve, Belgium

ABSTRACT: 4,5-Dimethyl-3-hydroxy-2(5*H*)-furanone or sotolon is known to impart powerful Madeira-oxidized−curry−walnut notes to various alcoholic beverages. It has been much studied in oxidized Jura flor-sherry wines, aged Roussillon sweet wines, and old Port wines, in which it contributes to the characteristic "Madeira-oxidized" aroma of these beverages. No scientific paper describes how sotolon might be involved in the Madeira off-flavor found in aged beers. The specific extraction procedure applied here allowed us to quantify this lactone in 7 special beers, at levels sometimes well above its threshold (from 5 to 42 μg/L after 6, 12, 18, and 24 months of natural aging, while unquantifiable in fresh beer). Investigation of spiked beers led us to highlight the key role of pro-oxidants and acetaldehyde. Addition of ascorbic acid without sulfites should be avoided by brewers, as the former would intensify sotolon synthesis. Acetoin, a beer fermentation byproduct, also emerged as possible precursor in beer when combined with serine.

KEYWORDS: *beer aroma, aging, sotolon, oxidation*

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R_1 ⇨ Composés dérivés du théaspirane (Sauternes, fruits secs)

J. Agric. Food Chem. 2015 63(37), 8247-8253.

Occurrence of Theaspirane and its Odorant Degradation Products in Hop and Beer

Caroline Scholtes,[†] Sabrina Nizet,[†] Hadrien Massart,[†] Pascal Gerbaux,[‡] and Sonia Collin*[,†]

[†]Unité de Brasserie et des Industries Alimentaires, Earth and Life Institute ELIM, Faculté d'Ingénierie biologique, agronomique et environnementale, Université catholique de Louvain, Croix du Sud, 2 box L7.05.07, B-1348 Louvain-la-Neuve, Belgium

[‡]Organic Chemistry Laboratory, Mass Spectrometry Center, University of Mons, U-MONS, Place du Parc, 23, B-7000 Mons, Belgium

ABSTRACT: In model oxidized media, six theaspirane-derived compounds were identified by gas chromatography–high resolution mass spectrometry: 4-hydroxy-7,8-dihydro-β-ionone, 6-hydroxy-7,8-dihydro-α-ionone, dihydrodehydro-β-ionone, two monoepoxides, and a derived alcohol. Only 4-hydroxy-7,8-dihydro-β-ionone and dihydrodehydro-β-ionone have been described previously in the literature. Investigation of hop revealed five of these compounds in free form together with theaspirane (especially in the Mosaic variety), while the Citra and Amarillo hop varieties emerged as very interesting for the release of theaspirane, 4-hydroxy-7,8-dihydro-β-ionone, and dihydrodehydro-β-ionone from glucoside precursors. For the first time, theaspirane, 4-hydroxy-7,8-dihydro-β-ionone, 6-hydroxy-7,8-dihydro-α-ionone, and both monoepoxides were found in a fresh commercial top fermentation beer (only theaspirane, 4-hydroxy-7,8-dihydro-β-ionone, and dihydrodehydro-β-ionone have recently been mentioned as Gueuze constituents).

KEYWORDS: *theaspirane degradation, hop aroma, beer aging*

Composés de vieillissement des ales

⇨ Thiols polyfunctionnels (oignon, empyreumatique, ribes, agrumes, ..)

Flavour Fragr. J. 2013, 28, 174-179.

Occurrence of the ribes odorant 3-sulfanyl-3-methylbutyl formate in aged beers

Thi Thu Hang Tran, Sabrina Nizet, Jacques Gros and Sonia Collin*

ABSTRACT: 2-Sulfanyl-3-methylbutyl formate and acetate were synthesized without purification steps, quantified with a pulsed flame photometric equimolar detector, and characterized by comparison with commercially available 3-sulfanyl-3-methylbutyl formate and acetate (retention indexes, mass spectra, odour descriptors, and intensities). Both formates exhibited a typical ribes flavour, in contrast to both acetates, which were much more piquant. The sensorial threshold of 3-sulfanyl-3-methylbutyl formate was much lower (57 ng/l in beer, BE-GC-LoADS = 0.0006 ng) than those measured for the three other esters. Only 3-sulfanyl-3-methylbutyl formate was perceived at the sniffing port in beer extracts. Concentrations up to 1230 ng/l were measured in pilot beers after 1 month at 20°C, although the compounds are rarely detected in commercial beers with highly oxygen-protected bottling. Accelerated ageing in the presence

Wiley & Sons, Ltd.

J. Inst. Brew. 2012,118, 198-204.

Fate of 2-sulphanylethyl acetate and 3-sulphanylpropyl acetate through beer aging

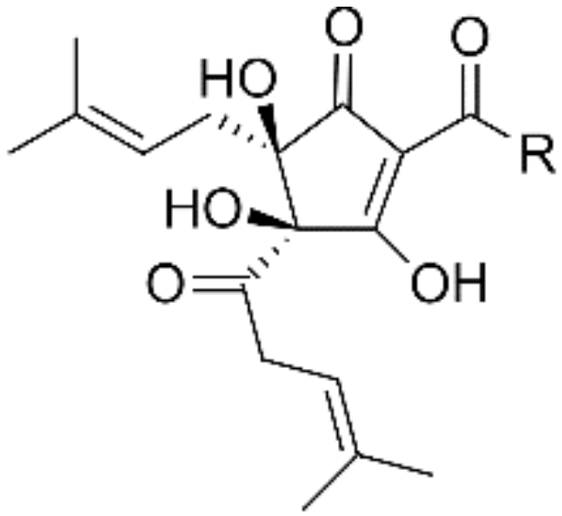
Thi Thu Hang Tran, Jacques Gros, Sabine Bailly, Sabrina Nizet and Sonia Collin*

Sulphanylalkyl alcohols and their corresponding acetates were investigated in 14 commercial Belgian beers. Although it was the major peak at the pulsed-flame photometric detector, the empyreumatic 2-sulphanylethyl acetate was found at concentrations below its individual odour threshold, estimated at 40 μg/L (0–4 μg/L in most fresh beers, 5–12 μg/L in three fresh high-bitter beers). Both the Ehrlich pathway and hop constituents contribute to this content. In 11 of the investigated samples, synthesis of 2SE-A and 3-sulphanylpropyl acetate (roasted, burned) continued during the first three months of storage. Although below their individual thresholds, these compounds might interact by synergy with other aged flavours. As yeast was absent from most of the investigated bottles, chemical degradation of precursors is suspected. Copyright © 2012 The Institute of Brewing & Distilling

R_1 La stabilité organoleptique des bières houblonnées à cru

Les publications récentes

⇨ L'amertume



J. Am. Soc. Brew. Chem., 76 (2018), 236-246.

Why Humulinones are Key Bitter Constituents Only After Dry Hopping: Comparison With Other Belgian Styles

Carlos Silva Ferreira, Eloi Thibault de Chanvalon, Etienne Bodart, and Sonia Collin

Unité de Brasserie et des Industries Alimentaires, Earth and Life Institute ELIM, Faculté des Bioingénieurs, Université catholique de Louvain, Louvain-la-Neuve, B-1348, Belgium

ABSTRACT

Although long renowned worldwide for its unique dry-hopped (DH) Trappist beer, Belgium did not develop this process for other brands until the last decade. Twenty-one commercial Belgian DH beers were investigated and compared with a few other typical Belgian beers whose production involves either late hopping or aged hop addition (Gueuze). Bitterness was determined by spectrophotometric measurements (isooctane extraction) and by reversed phase high performance liquid chromatographic with UV detector (RP-HPLC-UV) (simultaneous quantitation of humulones, *cis*-/*trans*-isohumulones, reduced isohumulones, humulinones, and hulupones). In dry-hopped Belgian beers, humulinones (found at concentrations up to 13.3 mg/L) were estimated to be responsible for up to 28% of their bitterness. As humulinones revealed to be gradually lost through boiling (22%), clarification (5%), and fermentation (14%), non-dry-hopped (NDH) beers often displayed levels below 1.7 mg/L. Even in Gueuze beers for which old, humulinone-containing hops are used, no humulinone was found. Contrary to humulones, which were detected up to 7.2 mg/L in DH beers, hulupones were found at less than 3 mg/L in all Belgian beer styles. Humulinones were not produced in the boiling wort from humulones (in contrast to hulupones, readily synthesized from lupulones) but were significantly solubilized from hop thanks to their hydrophilicity. Yet, while the co-form accounted for about 50% of the humulones, the n-form prevailed for humulinones. Some humulinone degradation products were evidenced by RP-HPLC-MS/MS, and as suggested by their retention time (RT), should be more polar than their precursors. Bottle refermentation emerged as an additional critical step of humulinone loss, explaining the low levels found even in some strongly DH beers.

KEYWORDS
Bitterness; bottle refermentation; dry-hopped beer; hop; humulinone

J. Am. Soc. Brew. Chem., 78 (2020), 103-113.

Fate of Bitter Compounds through Dry-Hopped Beer Aging. Why *cis*-Humulinones Should be as Feared as *trans*-Isohumulones?

Carlos Silva Ferreira and Sonia Collin

Unité de Brasserie et des Industries Alimentaires, Louvain Institute of Biomolecular Science and Technology LIBST, Faculté des Bioingénieurs, Université catholique de Louvain, Louvain-la-Neuve, Belgium

ABSTRACT

The evolution of isohumulones, main bitter compound of beers, through storage and its bitter impact has been studied for many years. With the uprising of highly hopped and, especially, dry-hopped beers during the last decade, other compounds such as humulones, *cis*-humulinones and hulupones have been shown to participate in both the sensory and measured bitterness of these beers. However, the fate of these compounds through aging is still unclear. The present work evaluated, by means of spectrophotometric and RP-HPLC-UV analyses, the evolution of bitterness units (BU), *cis*- and *trans*-isohumulones, humulones, *cis*-humulinones, hulupones, and tetrahydro-isohumulones in 21 Belgian dry-hopped beers after two years of storage. Besides the expected degradation of *trans*-isohumulones, it was observed an average decrease of 91% of humulones and 73% of *cis*-humulinones. Together, these results indicate a decrease of 18 – 43% of the calculated bitterness intensity of dry-hopped Belgian beers. The losses of *cis*-humulinones in dry-hopped beers bring seven new oxidation compounds, some of them very similar to those issued from *trans*-isohumulones. The bitterness impact of these compounds is yet to be determined.

Abbreviations used: DH: dry-hopped; RP-HPLC-UV: Reversed phase high pressure liquid chromatography with UV detector; RT: Retention time; HRMS: High Resolution Mass Spectrometer

KEYWORDS
Beer aging; bitterness; bottle refermentation; dry-hopped beer; humulinone

R_1 ⇨ Le profil aromatique

J. Am. Soc. Brew. Chem., 79 (2021), 259-271.

Fate of Hop and Fermentation Odorants in Commercial Belgian Dry-Hopped Beers over 2 Years of Bottle Storage: Key-Role of Oxidation and Hop Esterases

Carlos Silva Ferreira and Sonia Collin

Unité de Brasserie et des Industries Alimentaires, Louvain Institute of Biomolecular Science and Technology (LIBST), Faculté des Bioingénieurs, Université catholique de Louvain, Louvain-la-Neuve, Belgium

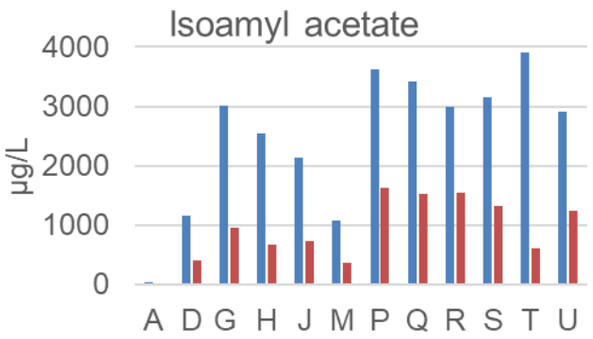
ABSTRACT

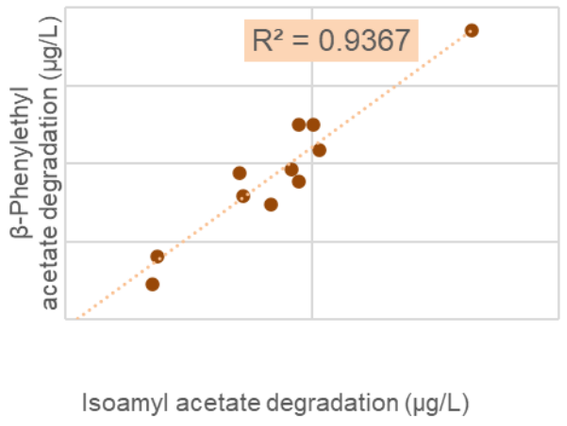
The aim of the present work was to compare levels of short chain fatty acids, esters, terpenoids and polyfunctional thiols in (mostly bottle-refermented) commercial Belgian dry-hopped beers before and after 2 years of storage at 20 °C (the usual best-before date in Belgium). Among the hop-derived volatiles, the terpenoids linalool and geraniol, the polyfunctional thiols 3SHol, 3SHA and 3S4MPol, and the esters ethyl isobutyrate, ethyl isovalerate and ethyl heptanoate (up to 499, 53, 0.2, 2, 3, 84, 63, and 19 µg/L, respectively) were found above their sensory thresholds in most fresh dry-hopped beers. The fermentation-derived esters reached concentrations similar to those previously reported for non-dry-hopped beers, with ethyl hexanoate and isoamyl acetate (up to 0.4 and 3.9 mg/L, respectively) often above their sensory thresholds. Except ethyl isovalerate (more than 85% still present), most hop odorants and fermentation esters showed degradation over the 2-year storage period: only 45%–70% of linalool, geraniol, and ethyl hexanoate and even less than 40% for polyfunctional thiols, ethyl isobutyrate, and ethyl heptanoate initial concentrations were detected after storage. How the dry-hopping process affects this degradation was further investigated in model media. Fermentation esters proved to be more strongly impacted in dry-hopped than in non-dry-hopped beers because of hop esterase activity. In addition to being aware of the need to avoid hop esterases, craft brewers are here advised to use bottle refermentation for its ability to regenerate some flavors and consume packaged oxygen. No deleterious effect of yeast, such as short chain fatty acid excretion, was evidenced.

Abbreviations: SAFE: Solvent assisted flavor evaporation; pHMB: 4-(hydroxymercuri)benzoic acid sodium salt; DHB: dry-hopped beer; EST: external standard; IST: internal standard

KEYWORDS

Beer flavor; bottle refermentation; dry hopping; hop enzymes; SAFE; staling





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R_1 La stabilité organoleptique des NABLABs

Les publications récentes (et moins récentes)

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JOURNAL OF THE AMERICAN SOCIETY OF BREWING CHEMISTS
https://doi.org/10.1080/03610470.2022.2142756

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Flavor Defects of Fresh and Aged NABLABs: New Challenges Against Oxidation

Margaux Simon, Gaël Vuylsteke and Sonia Collin

Unité de Brasserie et des Industries Alimentaires, Louvain Institute of Biomolecular Science and Technology (LIBST), Faculté des Bioingénieurs, Université Catholique de Louvain, Louvain-la-Neuve, Belgium

ABSTRACT

At present, non-alcoholic (NAB) and low-alcoholic beers (LAB) exhibit major staling defects even when fresh, partly due to absence of ethanol as an antioxidant. In the present work, the aroma stability of eleven commercial NABLABs available on the Belgian market, issued from different technological processes, was assessed. NABLABs were investigated, both when fresh and after one year of storage at 20°C in the dark. Six stale-odorant compounds were found above their perception threshold in aged NABLABs: sotolon, abhexon, methional, phenylacetaldehyde, dimethyltrisulfide and β-damascenone. Based on the chemical structure of the first four, it can be concluded that oxidation is the main issue for NABLABs aging. Yet, five of these usual staling defects of a six-month lager beer were already key-odorants in fresh NABLABs: dimethyltrisulfide, methional, and β-damascenone were above the thresholds in all samples, phenylacetaldehyde in 10 out of 11, and sotolon in 7. In co… NABLABs acceptability.

KEYWORDS

Beer aging; methional; NABLABs; oxidation; sotolon; stale odorants

beverages MDPI

Article

Why Oxidation Should Be Still More Feared in NABLABs: Fate of Polyphenols and Bitter Compounds

Margaux Simon and Sonia Collin *

Unité de Brasserie et des Industries Alimentaires, Louvain Institute of Biomolecular Science and Technology (LIBST), Faculté des Bioingénieurs, Université catholique de Louvain, Croix du Sud, 2 box L7.05.07, B-1348 Louvain-la-Neuve, Belgium
* Correspondence: sonia.collin@uclouvain.be

Abstract: Nowadays, non-alcoholic (NAB) and low-alcoholic beers (LAB) still significantly suffer from staling defects when fresh, partially due to absence of ethanol as antioxidant. In the current work, the fate of flavan-3-ols (monomers, dimers, and trimers) and bitter compounds (isohumulones, humulinones, etc.) of 11 commercial NABLABs available on the Belgian market was monitored through one year of aging at 20 °C in the dark. Fresh NABLABs contained variable flavan-3-ols and bitter compounds levels (between 3.0–10.0 mg/L and 8.0–39.0 mg/L, respectively), depending on different technological processes used. Chill haze and color were also investigated as potential oxidation markers of fresh and aged beers. Surprisingly, contrary to conventional beers, the oligomers' concentration (dimer and trimer procyanidins) exhibited a strong correlation ($R^2 = 0.95$) with chill haze before aging, suggesting prematured oxidation of the samples. After a year of storage, significant degradation occurred as for regular dry hopped beers (process very sensitive to oxidation), only 27% remaining for flavan-3-ol dimers and an average 16% for *trans*-isohumulones. Oxidation risk appears here as the main weakness of NABLABs, which could be probably improved by spiking very efficient antioxidants.

Keywords: NABLABs; flavan-3-ols; isohumulones; oxidation; color; chill haze

Citation: Simon, M.; Collin, S. Why Oxidation Should Be Still More Feared in NABLABs: Fate of

2000

2374 J. Agric. Food Chem. 1999, 47, 2374–2378

Contribution of 3-Methylthiopropionaldehyde to the Worty Flavor of Alcohol-Free Beers

Philippe Perpète and Sonia Collin*

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…sually criticized for two major defects: a lack of fruity aroma and a strong …butanal and 2-methylbutanal are described in the literature as being …ble for the worty taste. Although detected in large amounts in most malt …ompounds have proven unable to confer worty taste to beers. In this work, …om wort with a Likens−Nickerson microextractor. The resulting extract …a. Following GC/MS and GC/olfactometry analysis, 3-methylthiopropional- …he key feature since it remained the most organoleptically active compound …. In vitro assays showed that 3-methylthiopropionaldehyde is a substrate …*ces cerevisiae* reducing enzymes. Screening of various strains led us to …the 3-methylbutanal reductase activity, the higher the 3-methylthiopro- …activity. Monitoring of 3-methylthiopropionaldehyde concentration through …rocess has also been undertaken.

…*ohol-free beer; reductase activity; worty aroma; methional*

ELSEVIER Food Chemistry 66 (1999) 359–363 Food Chemistry www.elsevier.com/locate/foodchem

Fate of the worty flavours in a cold contact fermentation

Philippe Perpète, Sonia Collin*

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Received 22 December 1998; accepted 8 February 1999

Abstract

3-Methylbutanal, 2-methylbutanal and 3-methylthiopropionaldehyde are described in the literature as preponderantly responsible for the worty taste of alcohol-free beers. Even in a cold contact process, such aldehydes are reduced through fermentation. The chemical removal of aldehydes by amino acids or proteins does not exceed 20% of the initial concentration, although such mechanisms appear much more effective at 28°C. The role of *Saccharomyces cerevisiae* brewer's yeast, in the reduction of wort aldehydes, is confirmed here. Only viable yeasts allow a significant decrease in carbonyl level. Unfortunately, residual concentrations are higher for Strecker aldehydes among which 3-methylthiopropionaldehyde is characterized by a very low flavour threshold. Effects of yeast strain, pitching rate and inhibitors have been assessed. © 1999 Elsevier Science Ltd. All rights reserved.

Keywords: Alcohol-free beers; Cold contact process; Methional; Enzymatic reduction; Worty taste

Food Chemistry 70 (2000) 457–462 Food Chemistry www.elsevier.com/locate/foodchem

How to improve the enzymatic worty flavour reduction in a cold contact fermentation

Philippe Perpète, Sonia Collin *

Laboratoire de Brasserie et des Industries Alimentaires, Université Catholique de Louvain, Place Croix du Sud, 2 bte 7, B-1348, Louvain-la-Neuve, Belgium

Received 30 November 1999; received in revised form 30 November 1999; accepted 17 February 2000

Abstract

Although much more efficient at 28°C than at low temperature, enzymatic removal of Strecker aldehydes by brewer's yeast, …ed to 60–85% of the initial concentration, whatever the fermentation conditions. This …sidual concentrations imparting the well-known unpleasant worty taste to alcohol-free …s shown to hinder more complete enzymatic reduction in the cold contact fermentation …rights reserved.

…cess; Methional; Polyphenols; Reduction

ELSEVIER Food Chemistry 71 (2000) 379–385 Food Chemistry www.elsevier.com/locate/foodchem

Influence of beer ethanol content on the wort flavour perception

Philippe Perpète, Sonia Collin *

Laboratoire de Brasserie et des Industries Alimentaires, Université Catholique de Louvain, … Louvain-la-Neuve, Belgium

Received 4 January 2000; received in revised form 1 May 2000; accep…

Abstract

The sensorial threshold value of 3-methylthiopropionaldehyde appears to be influenced … in typical lager beers, ethanol significantly increases aldehyde retention, leading to low… alcohol-free beers, both the absence of ethanol and the higher level of mono- and disacch… flavours. As improving Strecker aldehyde reduction by yeast seems difficult for alcohol-f… cally modified yeasts or adding steps in the process, another interesting approach could be… flavour thresholds. © 2000 Elsevier Science Ltd. All rights reserved.

Keywords: Alcohol-free beers; Cold contact process; Strecker aldehydes; Flavour perception; Ethano…

2384 J. Agric. Food Chem. 2000, 48, 2384–2386

Evidence of Strecker Aldehyde Excretion by Yeast in Cold Contact Fermentations

Philippe Perpète and Sonia Collin*

Laboratoire de Brasserie et des Industries Alimentaires, Université Catholique de Louvain, Place Croix du Sud, 2 bte 7, B-1348 Louvain-la-Neuve, Belgium

A medium containing labeled leucine-d_{10} has been used to show that *Saccharomyces cerevisiae* was able to produce deuterated 3-methylbutanal in a cold contact fermentation. Whereas residual unreduced Strecker aldehydes bound to polyphenols were considered until now as the main defect of alcohol-free beers, yeast emerges from this work as an additional source of worty flavor. When the same experiment was conducted without leucine, 3-methylbutanal excretion also occurred, indicating that valine could be used by yeast as precursor.

Keywords: *Strecker aldehyde; Saccharomyces cerevisiae; amino acid; excretion; cold contact process*

La qualité organoleptique des bières spéciales

R_1 Impact des malts spéciaux, des *Brettanomyces* et du vieillissement en fût

J. Am. Soc. Brew. Chem., 80 (2022), 1-9.

JOURNAL OF THE AMERICAN SOCIETY OF BREWING CHEMISTS
https://doi.org/10.1080/03610470.2022.2109380

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Comparative Investigation of Flavors in Red and Brown Flemish Beers: Key-Role of *Brettanomyces* and Torrefied Malts in Ethylphenols Occurrence

Alexandre Dusart, Jean-Paul Ryckaert and Sonia Collin

Unit of Brewery and Food Sciences, Louvain Institute of Biomolecular Science and Technology (LIBST), Faculty of Bioscience Engineering, Université Catholique de Louvain, Louvain-la-Neuve, Belgium

ABSTRACT

Red and brown Flemish sour beers form a distinct class of Belgian beers obtained by mixed (yeast/lactic bacteria) microbial fermentation and often resulting from blending a 1-to-2-year-old beer with a younger one to obtain a balance between acidic character and sweetness. A detailed composition in volatiles (phenols, lactones, esters, alcohols, acids, …) of three beers representative of the red and brown subcategories is presented. GC data were obtained after different extraction procedures, including solvent-assisted flavor evaporation (SAFE) and headspace. The first results showed the influence of *Brettanomyces* yeast on the phenol and ester contents. An efficient *Brettanomyces* activity in the red sour beers (especially in Rodenbach Vintage) was observed, favored by long maturation in wooden casks. This was organoleptically perceived by the horsey flavors brought by 4-ethylguaiacol and 4-ethylphenol, and the solvent-like ethyl acetate through esterase activity. The brown Flemish sour beer (produced in stainless steel fermenters) showed significantly more unreduced 4-vinylguaiacol and 4-vinylphenol, although traces of 4-ethylguaiacol and 4-ethylphenol were also detected (most probably here issued from torrefied malts, as suggested by the opposite substituted phenol/guaiacol ratio).

KEYWORDS

Beer volatiles; *Brettanomyces*; red and brown Flemish beers; SAFE extraction

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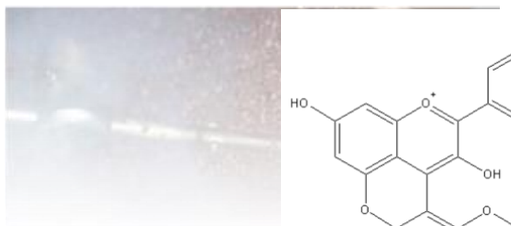
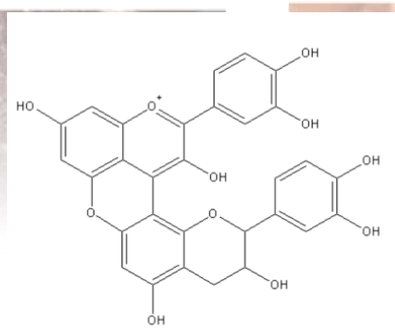
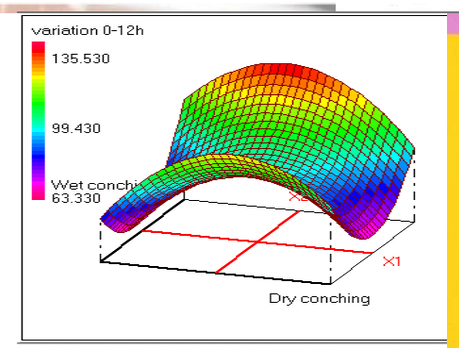
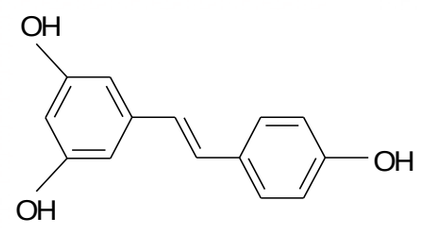
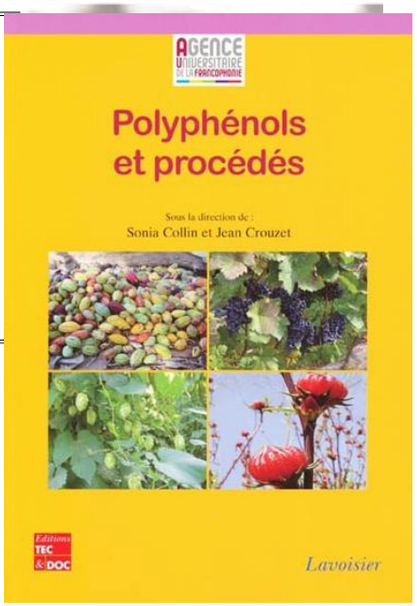
R_2 Les polyphénols du malt, du houblon, du cacao, … et leur devenir au travers des procédés de fabrication







6 thèses de doctorat (PhD) :

C. Counet (04), V. Jerkovic (07),
D. Callemien (07), J. Eyamo (16)
C. De Taeye (17), C. Silva (22)

R_2 Développement de nouvelles méthodes pour la détermination du profil polyphénolique et de l'activité antioxydante

9066 J. Agric. Food Chem. 2007, 55, 9066–9073

JOURNAL OF AGRICULTURAL AND FOOD CHEMISTRY

Involvement of Flavanoids in Beer Color Instability during Storage

DELPHINE CALLEMIEN AND SONIA COLLIN*

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Besides Maillard reactions, structural rearrangements of flavan-3-ol monomers cause color changes in beer during storage. Acetone/water-soluble fractions (70/30, v/v) of three lager beers of the same batch, differently stabilized before bottling in glass or poly(ethylene terephthalate) (PET) bottles, were monitored by normal-phase HPLC-ESI(−)-MS/MS over a 1-year period of storage at 20 °C. In parallel, beer color was monitored by the European Brewery Convention assay. The evolution of color was similar in the silica gel-filtered beer to that in identically bottled and stored poly(vinylpolypyrrolidone)-treated samples, despite the high flavanoid dimers content of the former. On the other hand, color evolved more rapidly in the PET bottle, suggesting a key role of oxygen. The kinetics was still increased in model media containing (+)-catechin, while no color was detected when normal-phase HPLC-fractionated dimers or trimers were investigated. (+)-Catechin emerged as the precursor of less polar products, characterized by a yellow-brown color. MS/MS enabled us to identify these products as issued from the oxidation and intramolecular additions of dehydrodicatechin B4. Similar structures were found in aged beers spiked with (+)-catechin. Beer storage in the absence of oxygen and at low temperature is recommended so as to minimize the synthesis of such pigments.

KEYWORDS: Beer; proanthocyanidins; flavanoids; color aging; storage; stability

J. Agric. Food Chem. **2000,** *48,* 1129–1134 **1129**

Measuring Antioxidant Efficiency of Wort, Malt, and Hops against the 2,2′-Azobis(2-amidinopropane) Dihydrochloride-Induced Oxidation of an Aqueous Dispersion of Linoleic Acid

Catherine Liégeois, Guillaume Lermusieau, and Sonia Collin*

Université Catholique de Louvain, Unité de Brasserie et des Industries Alimentaires, Place Croix du Sud, 2/Bte 7, B-1348 Louvain-la-Neuve, Belgium

This paper presents a simple, convenient method for determining the efficiency of antioxidants in aqueous systems. Production of conjugated diene hydroperoxide by oxidation of linoleic acid in an aqueous dispersion is monitored at 234 nm. 2,2′-Azobis(2-amidinopropane) dihydrochloride is used as a free radical initiator. Among 12 antioxidants tested, phenolic compounds proved to be the most efficient, both kinetically and in terms of the inhibition time (T_{inh}). Applied to wort, malt, and hops, the method confirmed a significant antioxidant activity in such products, especially hops. This assay can be used to follow oxidative changes throughout the brewing process and to understand the contribution of each raw material.

Keywords: *Antioxidant; lipid oxidation; AAPH; beer*

Use of RP-HPLC-ESI(–)-MS/MS to Differentiate Various Proanthocyanidin Isomers in Lager Beer Extracts

Delphine Callemien and Sonia Collin,[1] *Unité de Brasserie et des Industries Alimentaires, Université catholique de Louvain, Louvain-la-Neuve, Belgium*

ABSTRACT

Normal-phase high-performance liquid chromatography electrospray ionization(–) tandem mass spectrometry (NP-HPLC-ESI(–)-MS/MS) and thioacidolysis analyses recently have revealed the presence of (+)-catechin, (–)-epicatechin, procyanidin dimers, and prodelphinidin trimers in acetone/water (70/30, vol/vol) LH-20 extracts of lager beers. In this study, detailed structures were determined by reversed-phase (RP) HPLC-ESI(–)-MS/MS. Four dimers were identified: three procyanidins (B1, B3, and B4) and one prodelphinidin (B3). Previously detected in hop or malt, three trimers (the procyanidin C-4α-8-C-4α-8-C and two prodelphinidins, GC-4α-8-C-4α-8-C and GC-4α-8-GC-4α-8-C) were distinguished for the first time in beer. As expected based on previous thioacidolysis data, most beer proanthocyanidins carried a catechin as the terminal unit.

Keywords: Beer, Flavan-3-ols, HPLC-ESI(–)-MS/MS, Proanthocyanidins

In a previous work, normal-phase high-performance liquid chromatography electrospray ionization(–) tandem mass spectrometry (NP-HPLC-ESI(–)-MS/MS) analyses of an acetone/water (70/30, vol/vol) LH-20 beer extract enabled us to detect up to trimers of proanthocyanidins (5,6). To obtain more structural information, thioacidolysis also was applied to isolated dimer and trimer fractions. Our results indicated that catechin forms the main terminal unit in dimers and trimers, whereas both catechin and gallocatechin (especially in trimers) are the major constituents of the extension units.

The aim of this study was to establish detailed structures of beer proanthocyanidins. The acetone/water (70/30, vol/vol) LH-20 extract was analyzed by both NP- and reversed-phase (RP) HPLC-ESI(–)-MS/MS. Tandem mass spectrometry analysis was chosen

R_2 L'étude du profil polyphénolique des bières houblonnées à cru

L'importance des flavan-3-ols dans les bières dry-hoppées

JOURNAL OF THE AMERICAN SOCIETY OF BREWING CHEMISTS
https://doi.org/10.1080/03610470.2022.2062156

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Why Catechin and Epicatechin from Early Hopping Impact the Color of Aged Dry-Hopped Beers while Flavan-3-ol Oligomers from Late and Dry Hopping Increase Colloidal Instability

Carlos Silva Ferreira, Margaux Simon and Sonia Collin

Unité de Brasserie et des Industries Alimentaires, Louvain Institute of Biomolecular Science and Technology (LIBST), Faculté des Bioingénieurs, Université catholique de Louvain, Louvain-la-Neuve, Belgium

ABSTRACT

Dry hopping imparts distinct aromas but also a series of non-volatile compounds suspected of causing flavor and physical instability during beer storage. In this work, color, chill haze, total polyphenols, total flavanoids, and flavan-3-ol monomers (catechin and epicatechin) and oligomers (procyanidin dimers and trimers) were monitored in five commercial pale-colored Belgian dry-hopped beers over 24 months of storage at 20 °C in the dark. Fresh dry-hopped beers contained unusually high levels of flavan-3-ol monomers (up to 6.6 mg/L) and oligomers (up to 14.1 and 10.2 mg/L dimers and trimers, respectively). The increase in color intensity during storage (up to 6.4°EBC) correlated with fresh beer monomer levels, while the oligomer content correlated with chill haze formation (up to 25.7°EBC). The evolution of these two physical attributes also correlated with the level of total polyphenols in the fresh beers. In a pilot-scale production, kettle hopping was shown to impart either monomers (early) or oligomers (late), while dry hopping promoted efficient extraction of both monomers and dimers (extraction yields of 62 and 74%, respectively). Dry hopping thus plays an important role in color and chill haze increase.

Abbreviations: RP-HPLC-ESI(-)-MS/MS: Reversed phase high pressure liquid chromatography with electrospray ionization in negative mode and mass spectrometer detector operating in MS/MS mode; DH: dry-hopped; NDH: non-dry-hopped

KEYWORDS

Beer aging; beer color; beer haze; dry-hopped beer; flavan-3-ol; procyanidin

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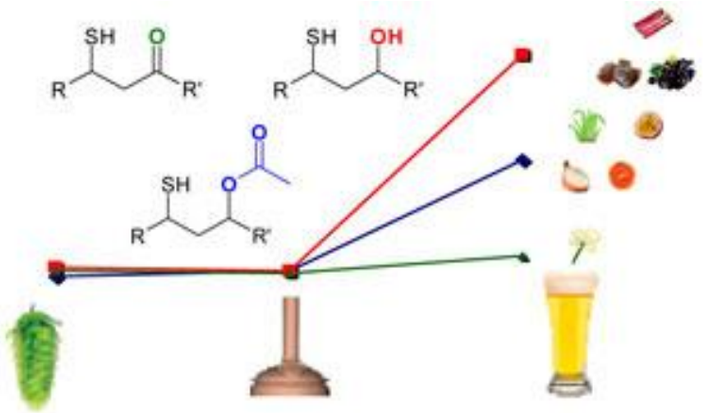
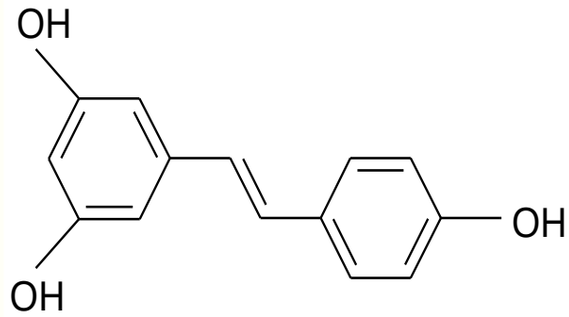
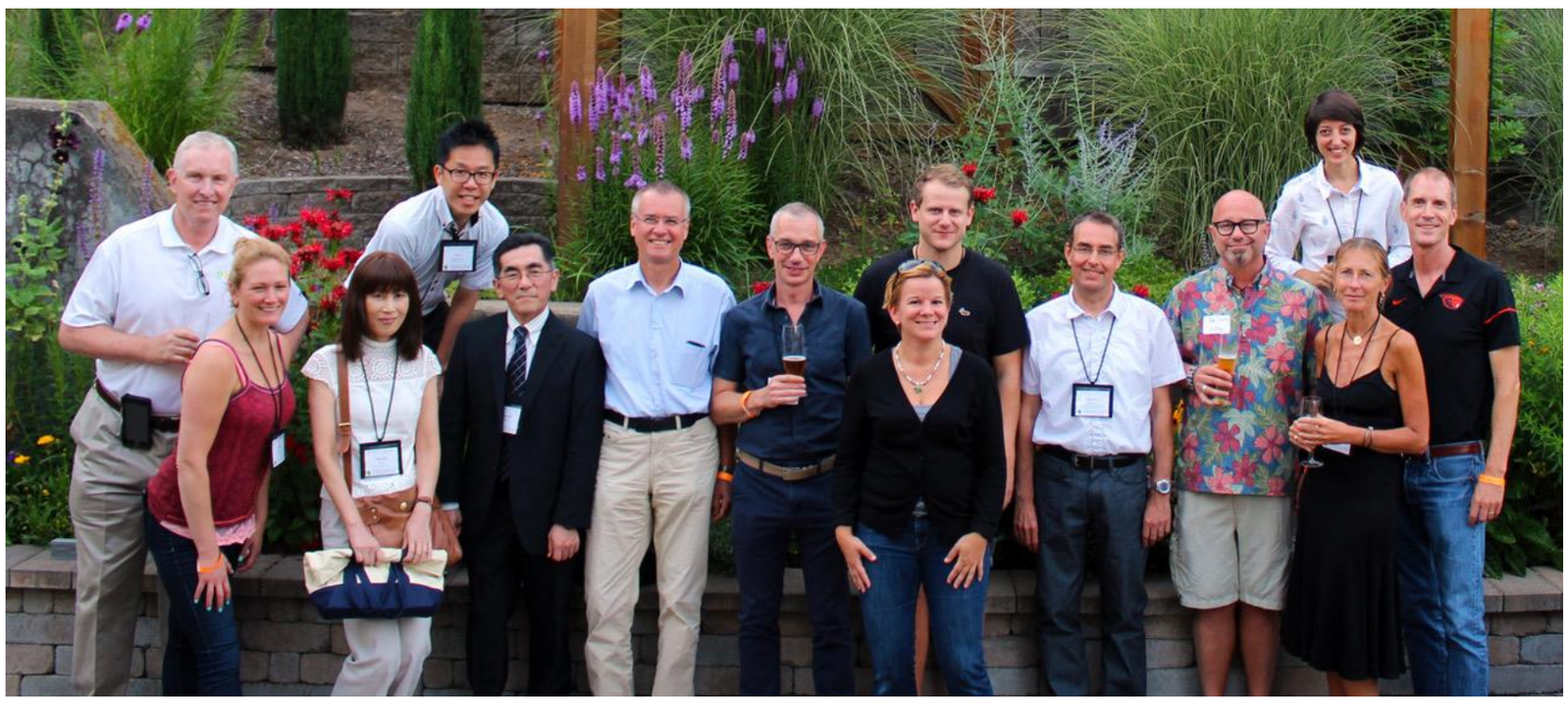
R_3 Découverte de nouvelles molecules dans le houblon : resvératrol, thiols et précurseurs, estérases ..

9 thèses de doctorat (PhD) :

G. Lermusieau (02)
V. Jerkovic (07)
D. Callemien (07)
C. Vermeulen (05)
J. Gros (12)
C. Scholtes (15)
M.L. Kankolongo (17)
C. Chenot (22)
C. Silva (22)







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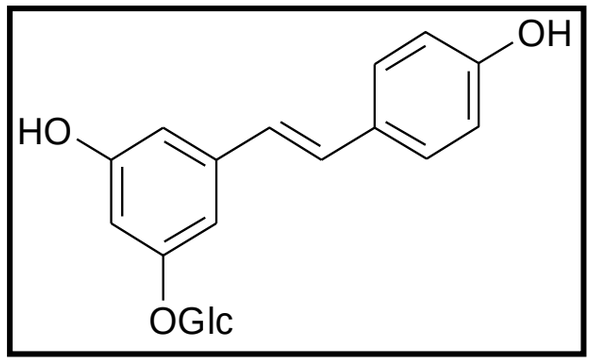
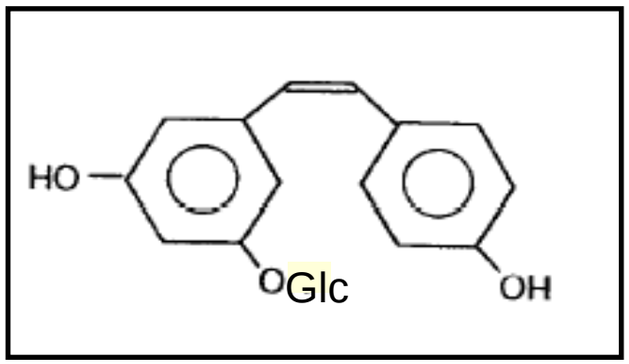
R_3 Le resvératrol et ses précurseurs

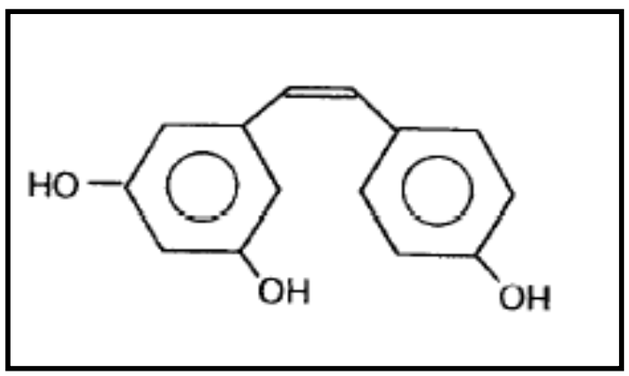
trans-Piceid



cis-Piceid



trans-Resveratrol



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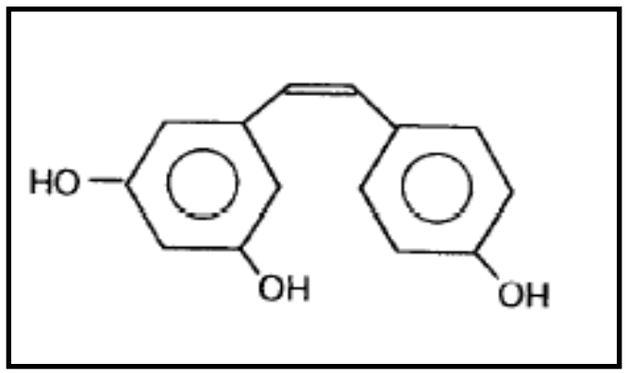
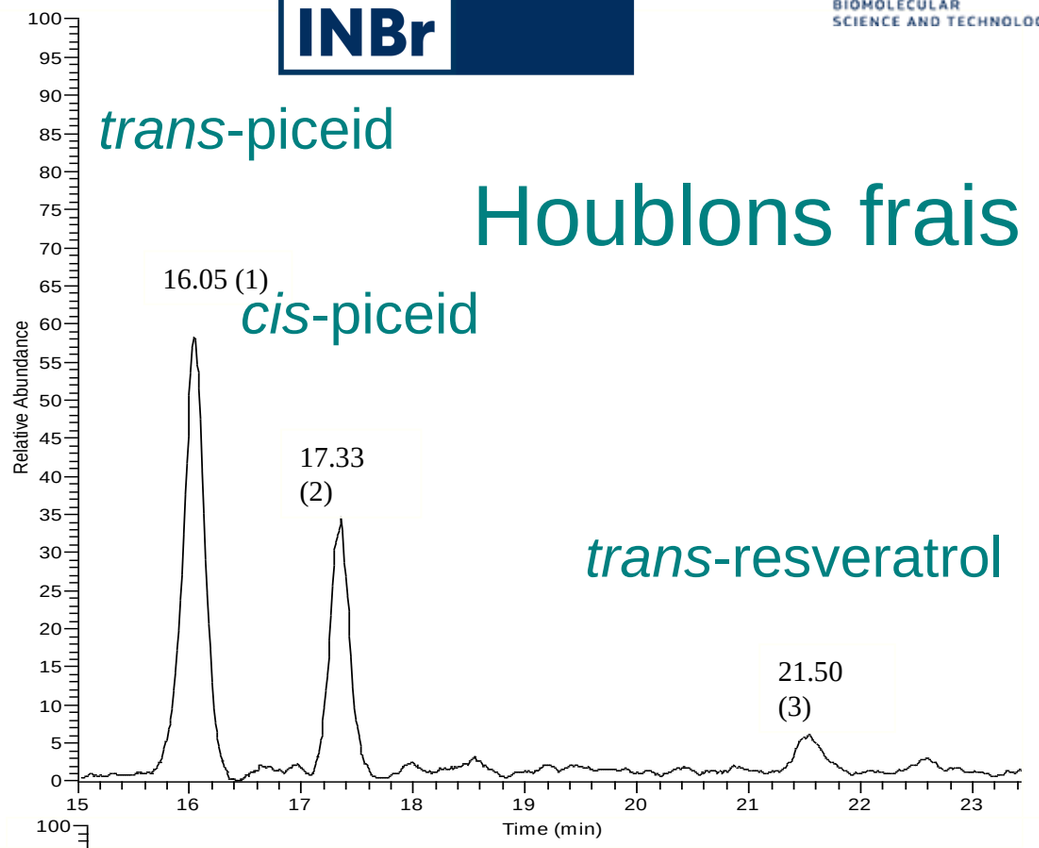
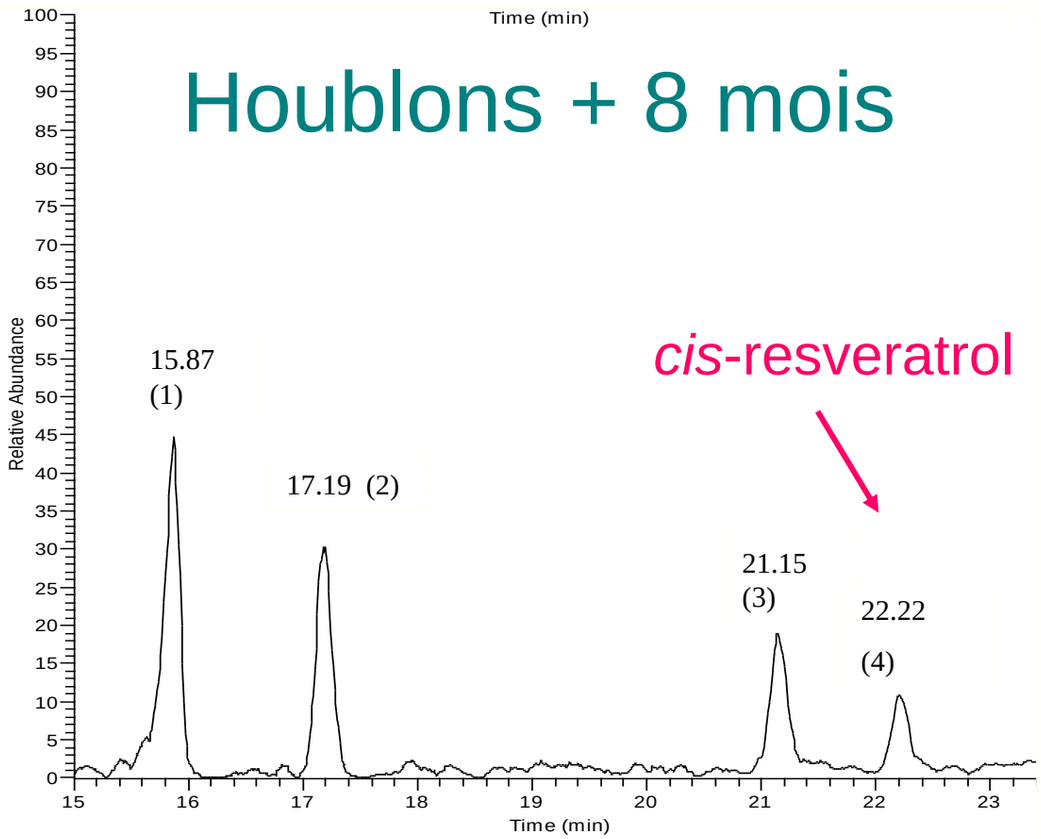


trans-piceid

Houblons frais



Houblons + 8 mois



cis-resveratrol

V. Jerkovic & S. Collin. *J. Agric. Food Chem.*, 55 (2007), 8754-8758.

V. Jerkovic, F. Nguyen, S. Nizet & S. Collin. *Rapid Communications in Mass Spectrometry*, 21 (2007), 2456-2466.

V. Jerkovic & S. Collin. *J. Agric. Food Chem.*, 56 (2008), 584-590.

UCLouvain

R_3 Le resvératrol et ses précurseurs

4202 J. Agric. Food Chem. 2005, 53, 4202–4206

JOURNAL OF AGRICULTURAL AND FOOD CHEMISTRY

Determination of Stilbenes in Hop Pellets from Different Cultivars

VESNA JERKOVIC, DELPHINE CALLEMIEN, AND SONIA COLLIN*

Unité de Brasserie et des Industries Alimentaires, Faculté d'Ingénierie biologique, agronomique et environnementale, Université catholique de Louvain, Croix du Sud, 2 bte 7, B-1348 Louvain-la-Neuve, Belgium

About 30% of the polyphenols in wort and beer derive from hop, but little is yet known about their nutritional impact. The recent discovery of *trans*-resveratrol and piceid isomers in hop opens new doors to understanding beer health benefits. In the present work, resveratrol was quantified by HPLC–APCI–MS/MS in pellets from 9 different cultivars. Concentrations ranging from 4 to 9 mg/kg *trans*-piceid, from 2 to 6 mg/kg *cis*-piceid, and up to 1 mg/kg *trans*-resveratrol were detected. As previously shown for total polyphenols and flavonoids, the lower the α-acid content, the higher the total stilbene content.

KEYWORDS: Resveratrol; stilbene; polyphenols; hop cultivars; beer

584 J. Agric. Food Chem. 2008, 56, 584–590

JOURNAL OF AGRICULTURAL AND FOOD CHEMISTRY

Fate of Resveratrol and Piceid through Different Hop Processings and Storage Times

VESNA JERKOVIC AND SONIA COLLIN*

Unité de Brasserie et des Industries Alimentaires, Faculté d'Ingénierie Biologique, Agronomique et Environnementale, Université catholique de Louvain, Croix du Sud, 2 bte 7, B-1348 Louvain-la-Neuve, Belgium

trans-Piceid and *trans*-resveratrol contents of hop cones, hop pellets, CO_2 extracts, and spent hop from American varieties (harvest 2004) were determined by reverse-phase high-performance liquid chromatography–atmospheric pressure chemical ionization–tandem mass spectrometry [RP-HPLC–APCI(+)–MS/MS]. Pelletization induced strong stilbene degradation in some cultivars. Similarly, 1 year of storage at 4 °C led to a huge loss of *trans*-piceid, especially in the case of hop cones (much faster than in model media, although well protected from light and oxygen). Therefore, after 8 months of storage, the overall stilbene content was in the same range whatever the conditioned form. Absent in fresh hop cones or pellets, *cis*-resveratrol was released from *cis*-piceid in all stored samples. On the other hand, no δ-viniferin was detected despite it is present in light-protected model media spiked with *trans*-piceid. Because supercritical carbon dioxide proved inefficient for recovering resveratrol and piceid from pellets, spent hop emerged as the most interesting material for subsequent specific stilbene extraction.

KEYWORDS: Resveratrol; stilbene; polyphenols; hop conditioning; hop storage; beer; resveratrol stability

Hop as an Interesting Source of Resveratrol for Brewers: Optimization of the Extraction and Quantitative Study by Liquid Chromatography/Atmospheric Pressure Chemical Ionization Tandem Mass Spectrometry

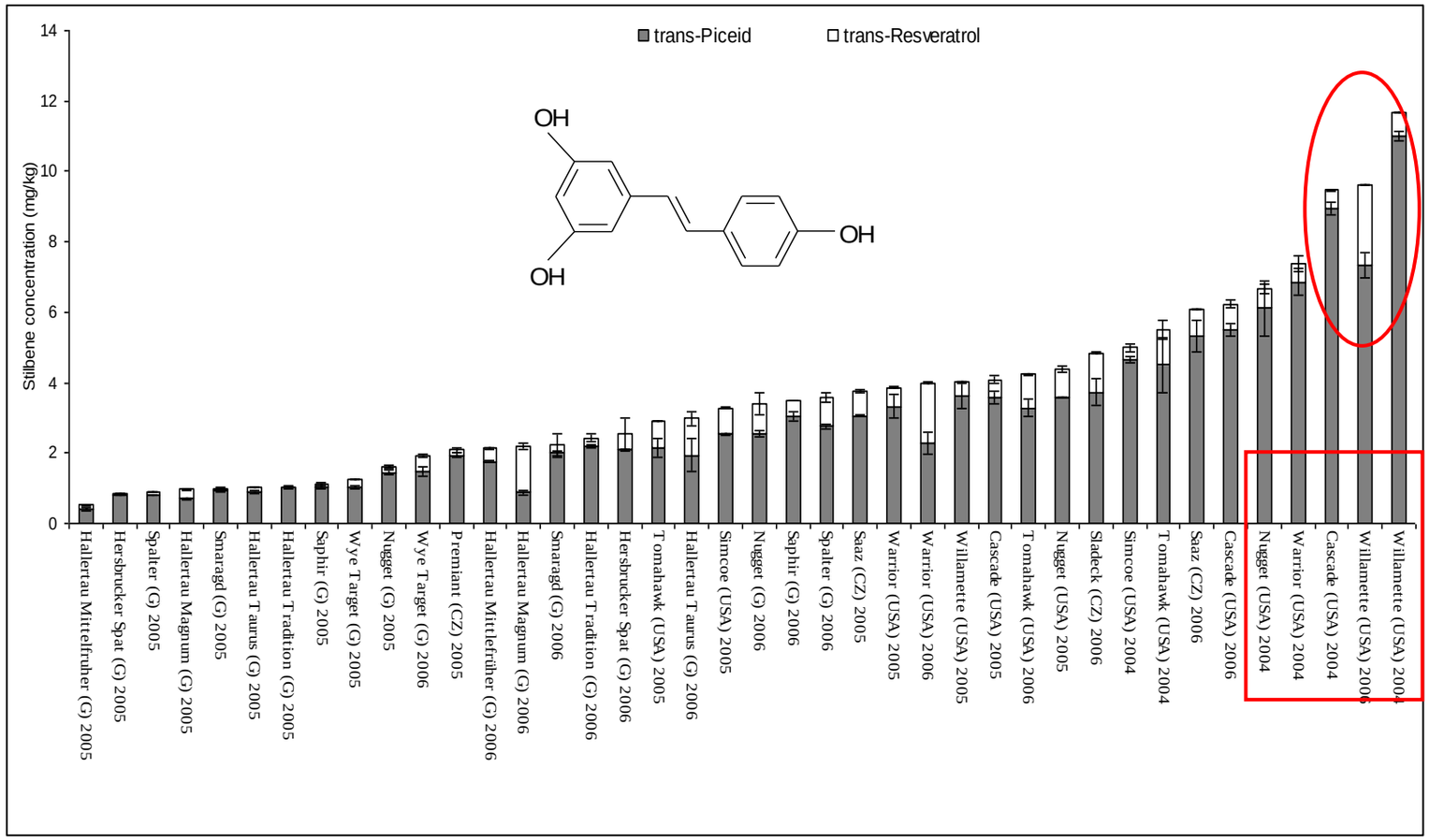
DELPHINE CALLEMIEN,† VESNA JERKOVIC,† RAOUL ROZENBERG,‡ AND SONIA COLLIN*,†

Unité de Brasserie et des Industries Alimentaires, Université Catholique de Louvain, Croix du Sud, 2/bte 7, B-1348 Louvain-la-Neuve, Belgium, and Laboratoire de Spectrométrie de Masse, Université Catholique de Louvain, Place Louis Pasteur 1, B-1348 Louvain-la-Neuve, Belgium

Nowadays, hop is used almost exclusively by brewers for bitterness and flavor. Although hop polyphenols have been widely studied in the past decade for their antioxidant activity in the boiling kettle, very little is known about their real impact on health. The discovery of resveratrol in hop pellets highlights the potential health-promoting effect of moderate beer consumption. Here, we have optimized a quantitative extraction procedure for resveratrol in hop pellets. Preliminary removal of hydrophobic bitter compounds with toluene and cyclohexane at room temperature allows 99% *trans*-resveratrol recovery by ethanol:water (75:25, v/v) solid/liquid extraction at 60 °C. Reverse phase liquid chromatography proves an excellent means of separating isomers. In addition, we have compared two mass spectrometry ionization methods—atmospheric pressure chemical ionization (APCI) and electrospray ionization (ESI)—in both the positive and the negative modes. On the basis of standard additions applied with the optimized extraction procedure and reverse phase high-performance liquid cromatography–APCI(+)–tandem mass spectrometry, it appears that Tomahawk hop pellets (T90, harvest 2002) contain 0.5 ppm *trans*-resveratrol, 2 ppm *trans*-piceid, no *cis*-resveratrol, and 0.9 ppm *cis*-piceid.

KEYWORDS: Hop; extraction; resveratrol; polyphenols; health; HPLC-MS/MS; APCI; ESI

J. Agric. Food Chem., 53 (2005) 424-429.



Patent with ABInBev for the use of spent hops

Spent hop products, their content in stilbenes and their use as antioxidant for comestible production. WO2007EP63558, EP20060125587, WO2008068344, US2010/0166926A1

(19) United States
(12) Patent Application Publication
Jerkovic et al.
(10) Pub. No.: US 2010/0166926 A1
(43) Pub. Date: Jul. 1, 2010

(54) SPENT HOP PRODUCTS, THEIR CONTENT IN STILBENES AND THEIR USE AS ANTIOXIDANT FOR COMESTIBLE PRODUCTION

(76) Inventors: Vesna Jerkovic, Nivelles (BE); Sonia Colin, Suarlee (BE)

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Washington, DC 20036 (US)

(21) Appl. No.: 12/448,111
(22) PCT Filed: Dec. 7, 2007
(86) PCT No.: PCT/EP2007/063558
§ 371 (c)(1),
(2), (4) Date: Dec. 14, 2009

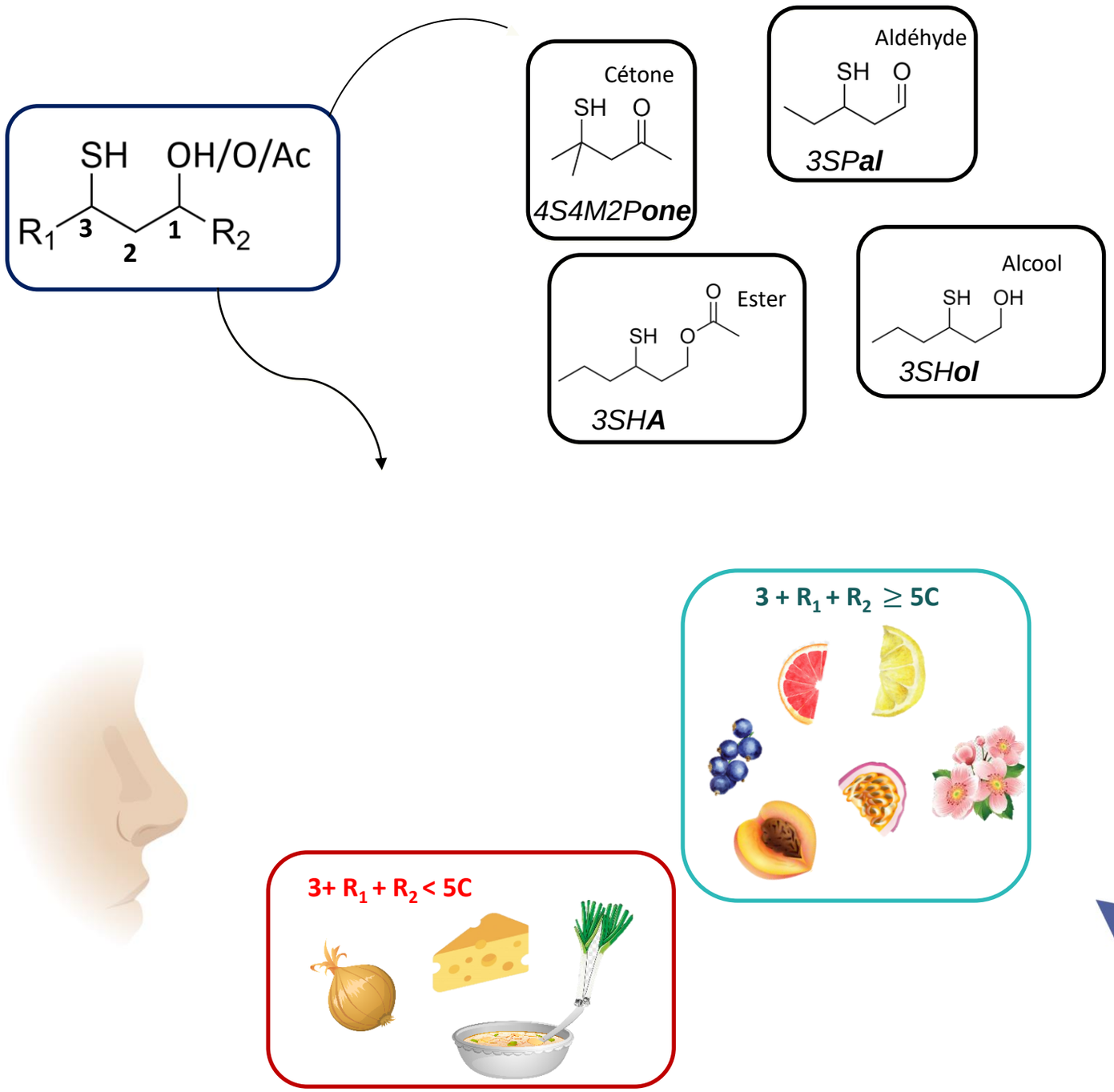
(30) Foreign Application Priority Data
Dec. 7, 2006 (EP) ..... EP 06125587.3

Publication Classification
(51) Int. Cl.
C12H 1/14 (2006.01)
(52) U.S. Cl. 426/271

(57) ABSTRACT

A method of producing a comestible by adding spent hop material during the production of the comestible, wherein the stilbenes and flavanoids contained in the spent hop material act as quenchers of oxygen resulting in an improved flavor stability of the comestible, and wherein the surviving stilbenes present in the comestible provide additional anti-oxidative properties to the comestible, leading to health benefits for the consumer.

R_3 Les thiols polyfonctionnels



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INBr Flavor research

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R_3 Les thiols polyfonctionnels

Synthèse par chimie combinatoire

Combinatorial Synthesis and Sensorial Properties of Polyfunctional Thiols

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Unité de Brasserie et des Industries Alimentaires, Faculté d'Ingénierie biologique, agronomique et environnementale, Université Catholique de Louvain, Croix du Sud, 2 bte 7, 1348 Louvain-la-Neuve, Belgium

Over the past few years, polyfunctional thiols present as trace components have been found to play a major role in many food flavors, due to their exceptionally low odor thresholds. Unfortunately, their presence in minute concentration (in ng/kg to a few μg/kg) and their high reactivity make it very difficult to extract and identify them. Furthermore, most of them are not yet commercially available. The aim of this work was to characterize the chromatographic and sensorial properties of 10 synthetic mercaptoketones and mercaptoalcohols. Combinatorial chemistry proved to be a very useful way to synthesize them rapidly. Sulfur-selective sulfur chemiluminescence detection chromatograms coupled with mass spectroscopy enabled the target compounds to be identified. Flavor profiles and best estimate gas chromatography lowest amount detected by sniffing (BE-GC-LOADS) values were further determined by GC−olfactometry. As expected, new, exceptionally odorant molecules (BE-GC-LOADS < 0.1 ng) were revealed by this unusual approach.

Keywords: *Combinatorial chemistry; polyfunctional thiols; flavors; BE-GC-LOADS; sulfur compounds*

Combinatorial Synthesis and Sensorial Properties of Mercapto Primary Alcohols and Analogues

CATHERINE VERMEULEN, CHRISTINE GUYOT-DECLERCK, AND SONIA COLLIN*

Unité de Brasserie et des Industries Alimentaires, Faculté d'Ingénierie biologique, agronomique et environnementale, Université catholique de Louvain, Croix du Sud, 2 bte 7, B-1348 Louvain-la-Neuve, Belgium

Combinatorial chemistry was used to extend the knowledge of beer thiols by synthesizing 13 mercapto primary alcohols and 6 other compounds (mercapto ketones and corresponding alcohols). Their respective mass spectra, retention indices on two columns, odor descriptors, and best estimated−gas chromatography−lowest amounts detected sniffing (BE-GC-LoADS) are reported. The descriptor "cheese", commonly associated with mercapto aldehydes and thioesters, was rarely used for mercapto alcohols or mercapto ketones. Polyfunctional thiols containing at least six carbon atoms usually gave a delicate odor of rhubarb/carrot, whereas most of the mercapto primary alcohols of intermediate size were described as onion-like, plastic-like, or pungent. Sensorial analysis of beers spiked with copper(II) ions allowed confirmation of the participation of thiols in the overall odor of fresh beer. The finding of 4-mercapto-4-methyl-2-pentanone and 3-mercaptohexanol in an XAD-2 Amberlite resin beer extract indicates a requirement for a more efficient thiol extraction procedure allowing identification of relevant polyfunctional thiols for brewing scientists.

KEYWORDS: Combinatorial synthesis; polyfunctional thiols; flavor; aroma; beer; sulfur compounds; BE-GC-LoADS

Synthesis and Sensorial Properties of Mercaptoaldehydes

CATHERINE VERMEULEN AND SONIA COLLIN*

Unité de Brasserie et des Industries Alimentaires, Faculté d'Ingénierie biologique, agronomique et environnementale, Université catholique de Louvain, Croix du Sud, 2 bte 7, B-1348 Louvain-la-Neuve, Belgium

Mercaptoalcohols and mercaptoketones have been described in the past decade as relevant character-impact compounds in various food products. On the other hand, very little is known of mercapto-aldehydes. Only 3-mercapto-2-methylpentanal and 3-mercaptohexanal have been found in onions and beef liver, respectively. The aim of this work was to synthesize 13 mercaptoaldehydes not commercially available by combinatorial chemistry. Chromatographic analyses of the mixture allowed the determination of their retention indices and mass spectroscopy fragments for further identification in foods. Gas chromatography−olfactometry demonstrated how some of them are characterized by unusual odor and very low perception threshold.

KEYWORDS: Combinatorial chemistry; polyfunctional thiols; flavors; BE-GC-LOADS; sulfur compounds

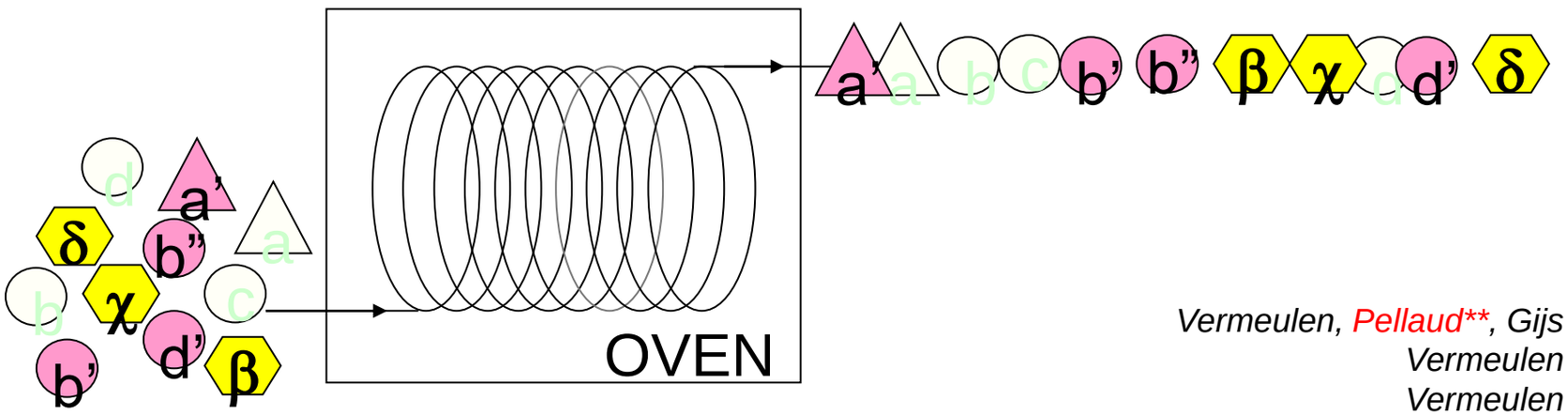
Combinatorial Synthesis and Sensorial Properties of 21 Mercapto Esters

CATHERINE VERMEULEN AND SONIA COLLIN*

Unité de Brasserie et des Industries Alimentaires, Faculté d'Ingénierie biologique, agronomique et environnementale, Université catholique de Louvain, Croix du Sud, 2 bte 7, B-1348 Louvain-la-Neuve, Belgium

The aim of this work was to determine the chromatographic and sensorial properties of 21 synthetic mercapto acetates, among which two—3-mercaptohexyl-acetate and 3-methyl-3-mercaptobutyl-acetate—have been previously detected in foods, especially passion fruit and wine. Combinatorial chemistry was used to synthesize them from α,β-unsaturated ketones and aldehydes in a very short time. GC-SCD, GC-MS, and GC−olfactometry proved to be very helpful techniques for analyzing them individually in the reaction mixture obtained. Compared to other polyfunctional thiols, mercapto … high BE-GC-LoADS values (best estimate−gas chromatography−lowest amount … with very diverse descriptors.

…natorial chemistry; polyfunctional thiols; flavors; BE-GC-LoADS; sulfur compounds

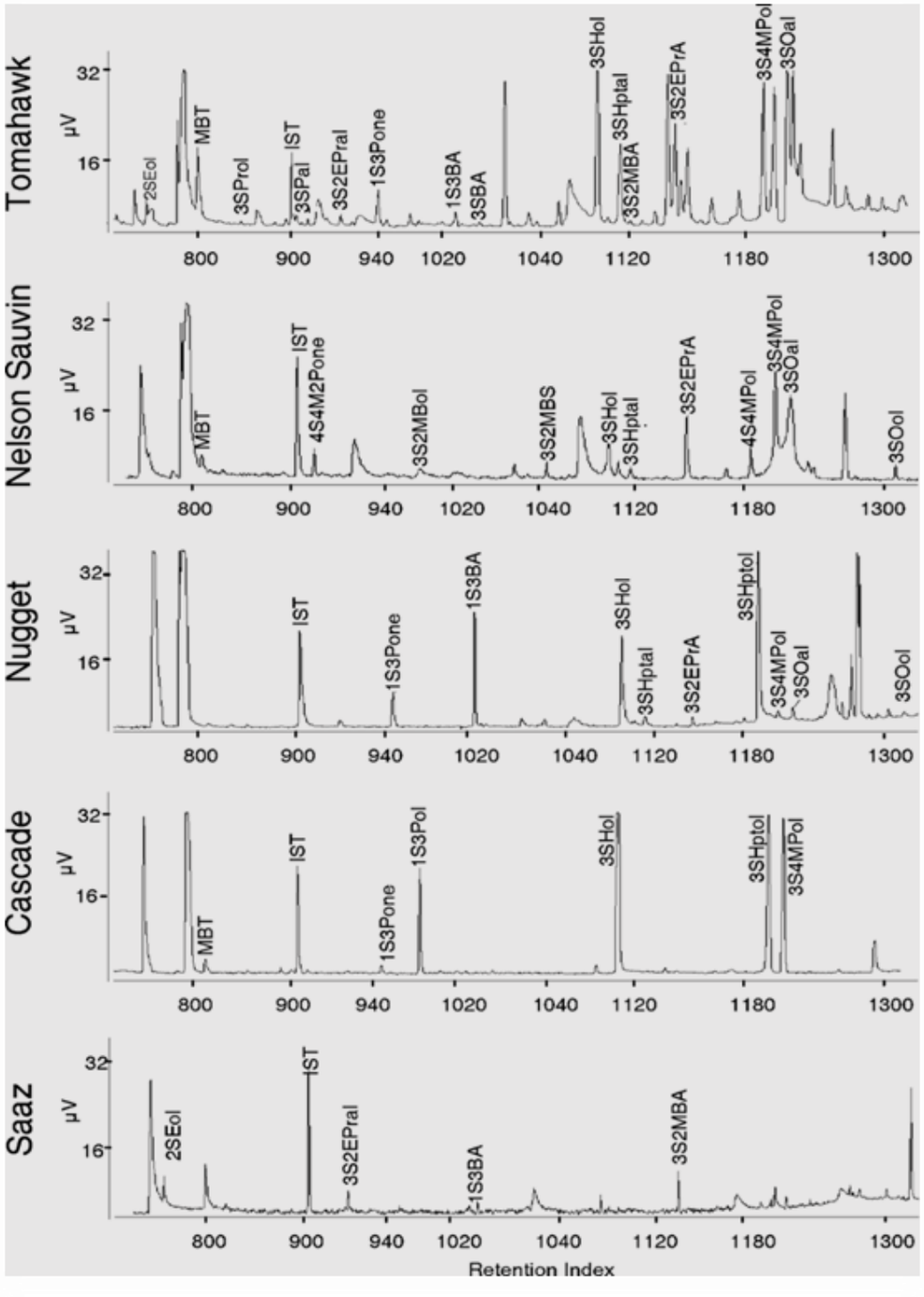


*Vermeulen, Pellaud**, Gijs & Collin, J. Agric. Food Chem., 49 (2001) 5445-5449.*
Vermeulen & Collin, J. Agric. Food Chem., 50 (2002) 5654-5659.
Vermeulen & Collin, J. Agric. Food Chem., 51 (2003) 3618-3622.
Vermeulen & Collin, J. Agric. Food Chem., 51 (2003) 3623-3628.
Vermeulen, Gijs & Collin, Food Rev. Intern., 21 (2005) 69-137.
Vermeulen & Collin, Combin. Chem. & High Throughput Screen., 9 (2006) 583-590.

UCLouvain

R_3 Les thiols polyfonctionnels

Découvertes de 41 structures dans le houblon



JOURNAL OF AGRICULTURAL AND FOOD CHEMISTRY
ARTICLE
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Occurrence of Odorant Polyfunctional Thiols in the Super Alpha Tomahawk Hop Cultivar. Comparison with the Thiol-rich Nelson Sauvin Bitter Variety

Jacques Gros, Sabrina Nizet, and Sonia Collin*

Unité de brasserie et des industries alimentaires, Earth and Life Institute ELIM, Faculté d'ingénierie biologique, agronomique et environnementale, Université catholique de Louvain, Croix du Sud, 2 bte 7, B-1348 Louvain-la-Neuve, Belgium

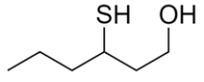
ABSTRACT: Tomahawk hop (*Humulus lupulus*) is a recently developed Super Alpha cultivar (14−18% α-acids w/w), already widely used by brewers to impart bitterness and a citrus-like aroma to beer. By comparison with two bitter varieties (Nelson Sauvin and Nugget) and two aromatic ones (Cascade and Saaz), the Tomahawk cultivar showed a very particular terpenoid profile, rich in both α- and β-selinenes (>600 mg/kg IST equiv in total), methyl geranate (>40 mg/kg IST equiv), and geraniol (>200 mg/kg). Tomahawk also proved to contain a wide variety of odorant polyfunctional thiols. The major β-sulfanyl acetate, 3-sulfanyl-2-ethylpropyl acetate, newly identified here, was found at similar levels in the famous Sauvignon-like Nelson Sauvin and Tomahawk varieties (15−44 μg/kg IST equiv). On the other hand, lower levels of total β-sulfanyl alcohols were measured in Tomahawk, although 3-sulfanylhexan-1-ol was found at a similar level and the 3-sulfanyl-4-methylpentan-1-ol previously claimed to be specific to the Nelson Sauvin variety was also evidenced in the Super Alpha cultivar (9−13 μg/kg IST equiv). As revealed by boiling and fermentation, Tomahawk hop also contains very interesting bound polyfunctional thiols that should be investigated for better use by brewers.

KEYWORDS: hop (*Humulus lupulus*), essential oil, flavor, polyfunctional thiols, cysteine adducts, Super Alpha variety

⇒ Chaque variété = **profil unique**

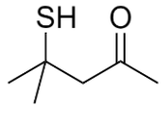
⇒ Certains thiols = **ubiquitaires**

ex: **3SHol**



⇒ D'autres = **propres à certaines variétés**

ex: **4S4M2Pone** (Nelson Sauvin)



⇒ Saaz = très pauvre vs variétés duals

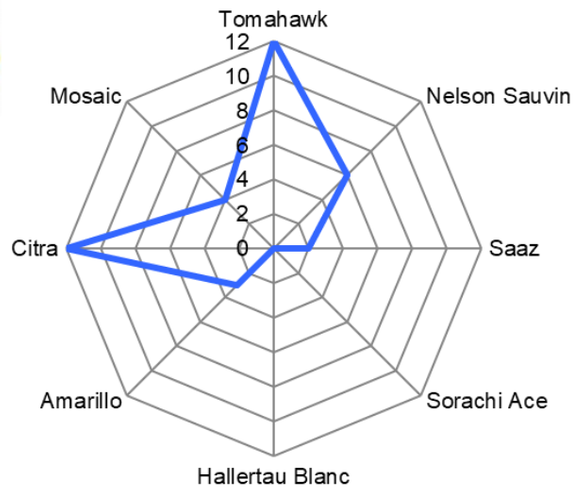
R_3 Les thiols polyfonctionnels

Analyses de houblons duals

Pamplemousse
Rhubarbe

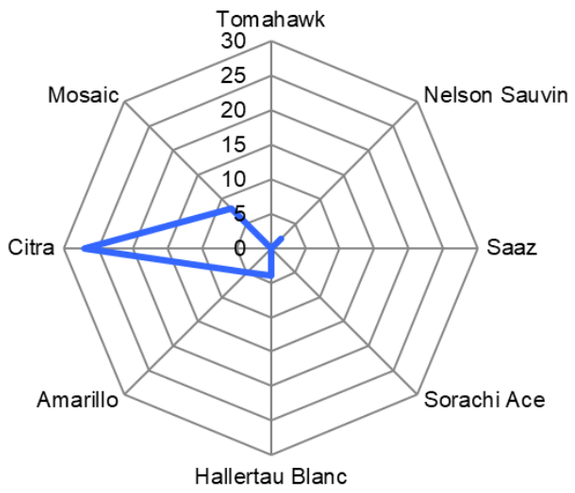
3SHol

Concentrations in µg/Kg



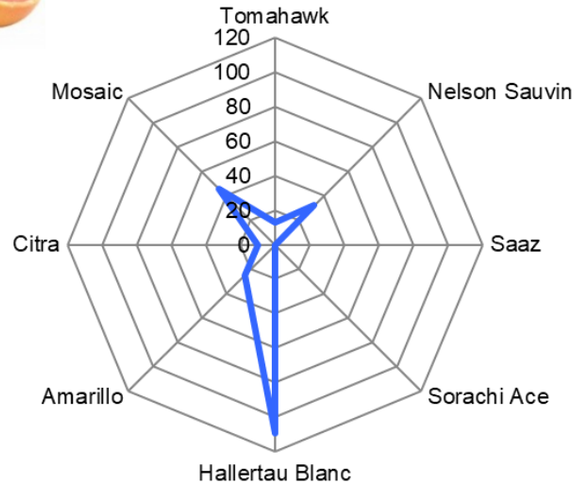
Fruit de la passion

3SHA



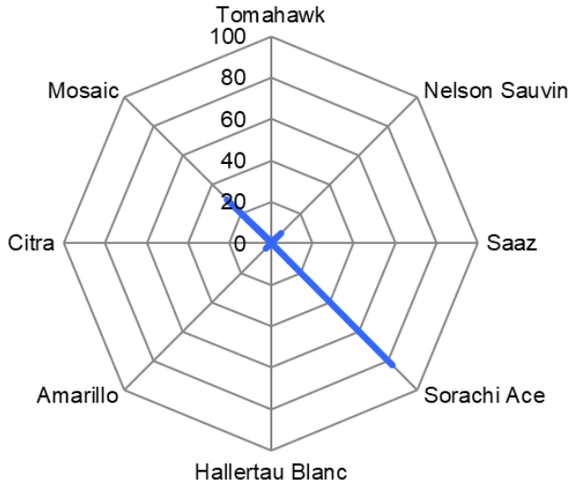
Pamplemousse

3S4MPol



Cassis

4S4M2Pone



Kankolongo, Gros, Nizet & Collin, J. Agric. Food Chem. 2015, 63, 3022−3030.

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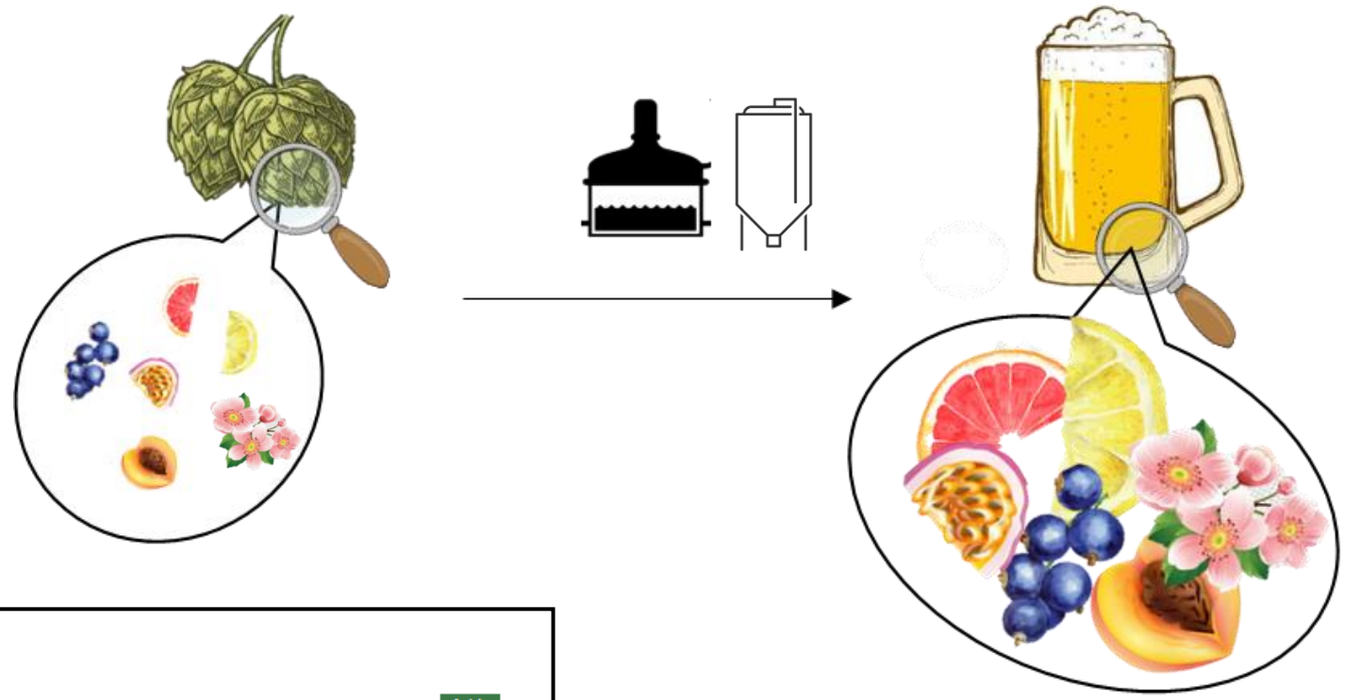
R_3 Les thiols polyfonctionnels

Evolution à l'ébullition et après fermentation



JOURNAL OF AGRICULTURAL AND FOOD CHEMISTRY

Article

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Occurrence of Odorant Polyfunctional Thiols in Beers Hopped with Different Cultivars. First Evidence of an *S*-Cysteine Conjugate in Hop (*Humulus lupulus* L.)

Jacques Gros, Florence Peeters, and Sonia Collin*

Unité de brasserie et des industries alimentaires, Earth and Life Institute ELIM, Faculté d'Ingénierie biologique, agronomique et environnementale, Université catholique de Louvain, Croix du Sud, 2 box L7.05.07, B-1348 Louvain-la-Neuve, Belgium

ABSTRACT: Forty-one thiols, mainly β-sulfanylalkyl acetates, β-sulfanylalkyl alcohols, and β-sulfanylalkyl carbonyls, were recently evidenced in hop. In a beer hopped with the Tomahawk cultivar, most of them were found at higher levels than expected. The aim of the present work was to investigate the polyfunctional thiols in beers hopped with different varieties. A few thiols proved not to come only from hop (mainly 2-sulfanylethyl acetate, μg/L levels, and 1-sulfanylpentan-3-one and 1-sulfanylpentan-3-ol, ng/L levels, internal standard (IST) equivalents). The thiol profile of Saaz-hopped beer proved similar to that of the reference beer produced without hop. A high level of 3-sulfanyloctan-1-ol emerged as an indicator of the use of Tomahawk hop (140 ng/L, IST equivalents; FD (flavor dilution) = 65536). In both Cascade- and Tomahawk-hopped beers, 3-sulfanylhexan-1-ol and 3-sulfanylheptan-1-ol were smelled at high flavor dilutions, although only for the latter, significant amounts of the unreduced 3-sulfanylheptanal were found in hop. As already claimed for hop authentication, 3-sulfanyl-4-methylpentan-1-ol remains a good marker of Nelson Sauvin-hopped beers (548 ng/L, IST equivalents; FD = 65536), together with 4-sulfanyl-4-methylpentan-2-one (128 ng/L, FD = 4096). As illustrated by the huge production occurring during fermentation, accurate prediction of hop varietal impact requires quantitating thiol adducts in hop. *S*-3-(1-Hydroxyhexyl)-cysteine was evidenced here for the first time in Cascade hop.

KEYWORDS: *beer, polyfunctional thiols, hop (Humulus lupulus), varietal aroma, S-cysteine conjugate*

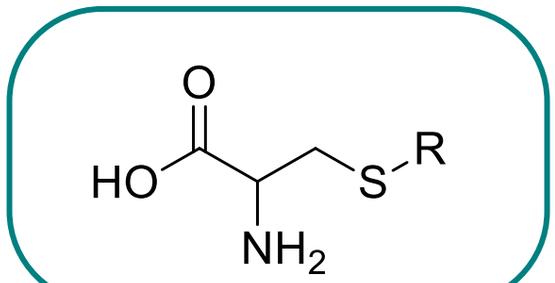
J. Agric. Food Chem., 60 (2012), 7805-7816

R_3 Les thiols polyfonctionnels

Mise en évidence de formes cystéinées dans le houblon

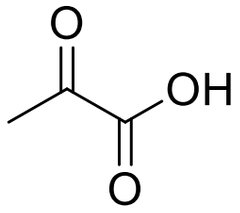
Méthode indirecte

Méthode directe



cysteine-S-conjugates

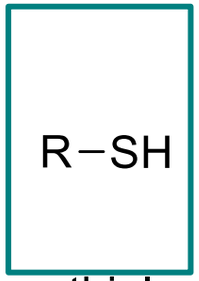
Apotryptophanase
(activité β-lyase)



pyruvic acid

+ NH_4OH +

ammonia (aq)



thiols

Chromatographie liquide
(HPLC-ESI(+)-MS/MS)

Gros, Peeters & Collin, J. Agric. Food Chem., 60 (2012) 7805-7816

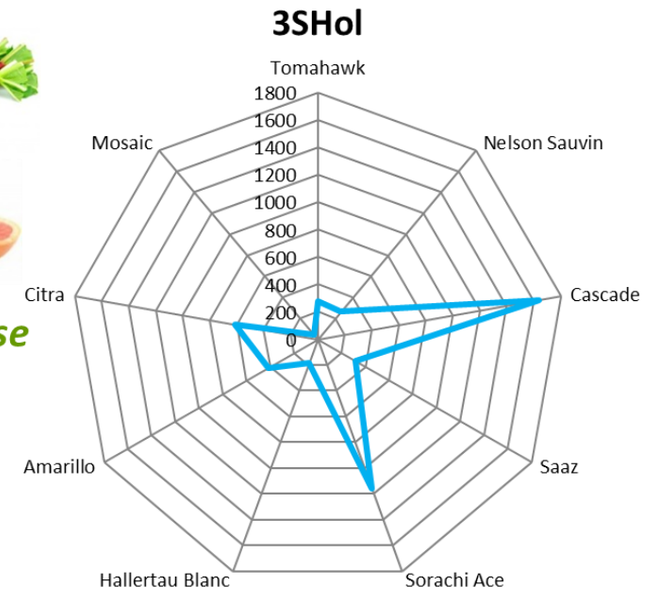
Chromatographie gazeuse
(GC-PFPD)

Gros, Tran & Collin, J. Inst. Brew., 119 (2013) 221-227

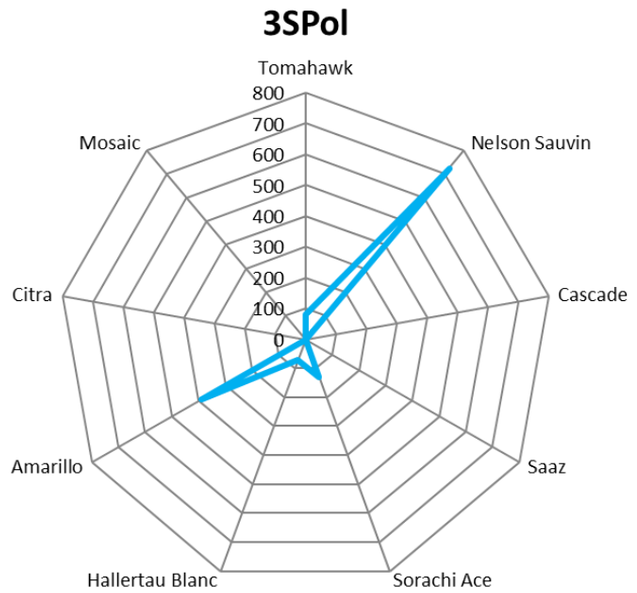
R_3 Les thiols polyfonctionnels

Les formes cystéinées dans les houblons duals

Pamplemousse Rhubarbe

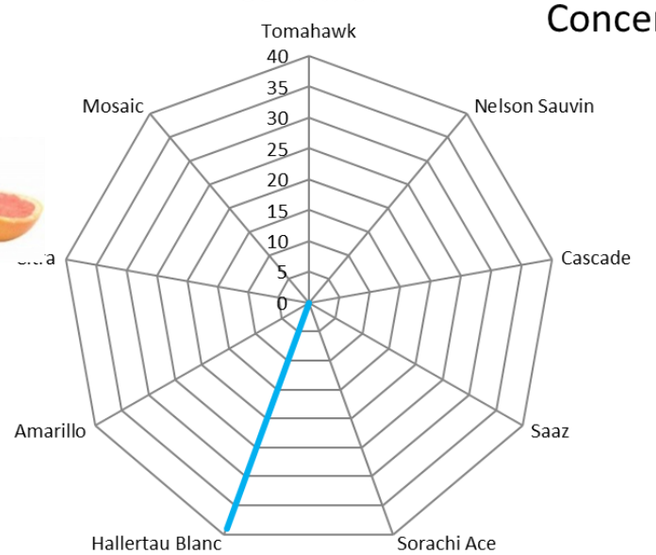


Citron

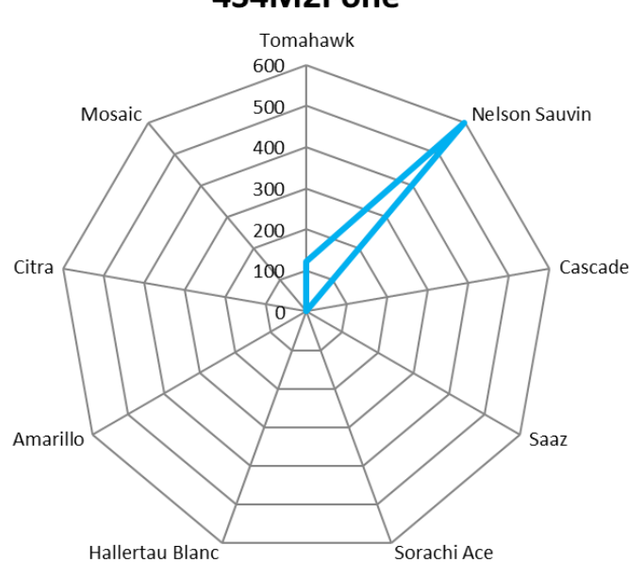


Concentrations in µg/Kg

Pamplemousse



Cassis



→ 3000 ppb (20-120 X > libres) = potentiel aromatique !

Kankolongo, Gros, Nizet & Collin, J. Agric. Food Chem. 2015, 63, 3022−3030.

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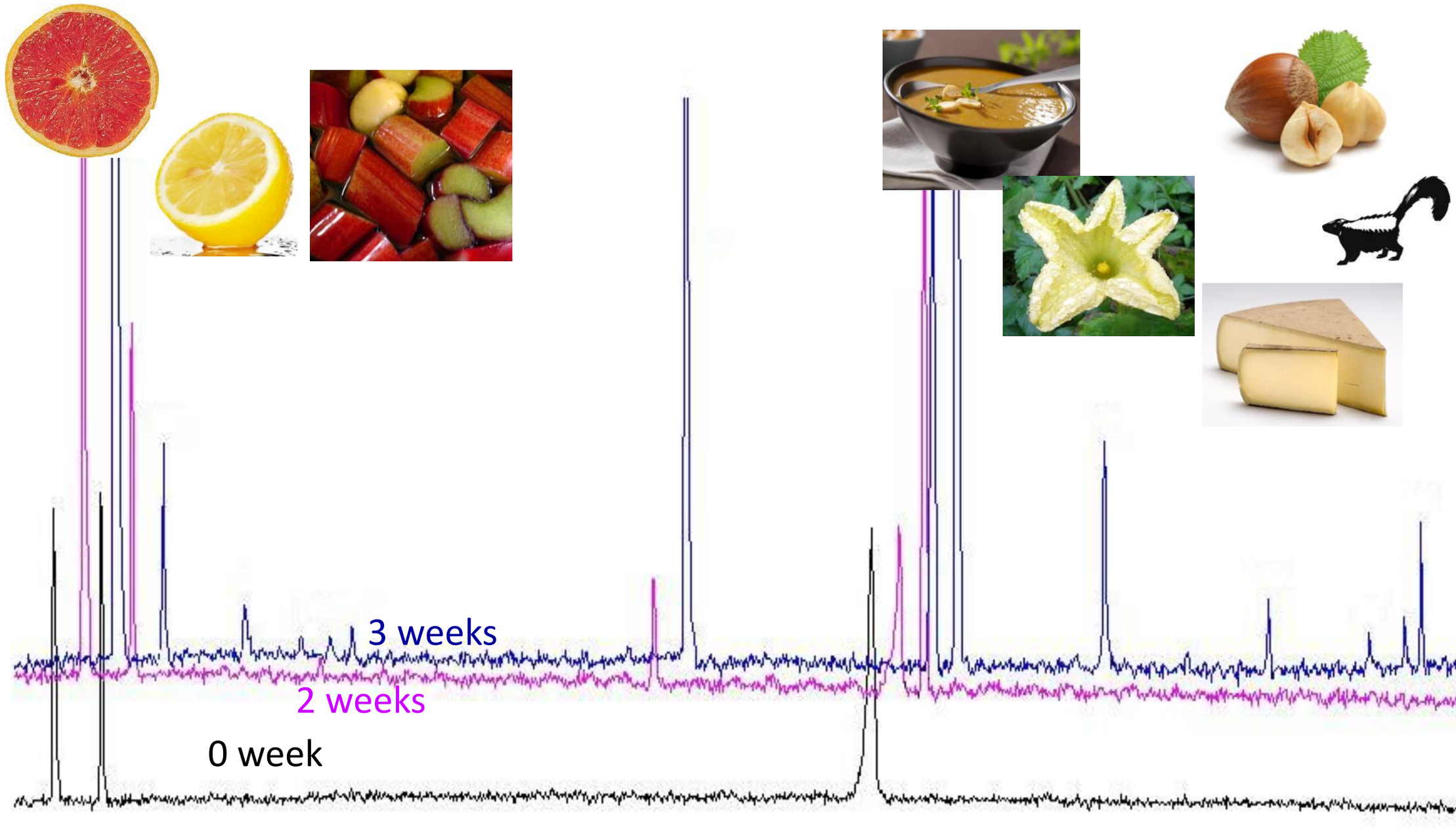
R_3 Les thiols polyfonctionnels

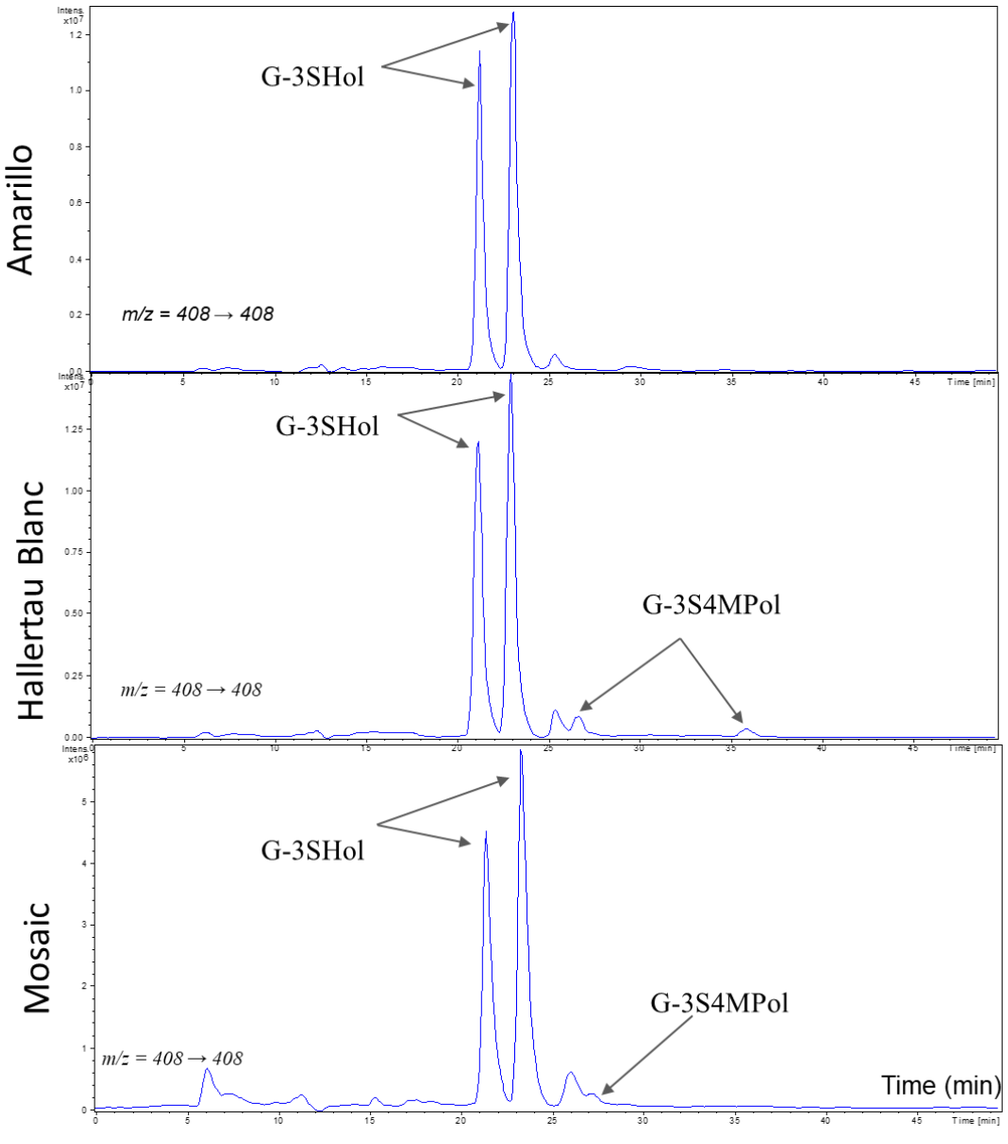
Relargage de 19 thiols durant la refermentation en bouteille



Nizet, Gros, Peeters, Chaumont, Robiette & Collin, J. ASBC, 71 (2013) 15-22.

R_3 Les thiols polyfonctionnels

Première mise en évidence de formes glutathionées dans les houblons

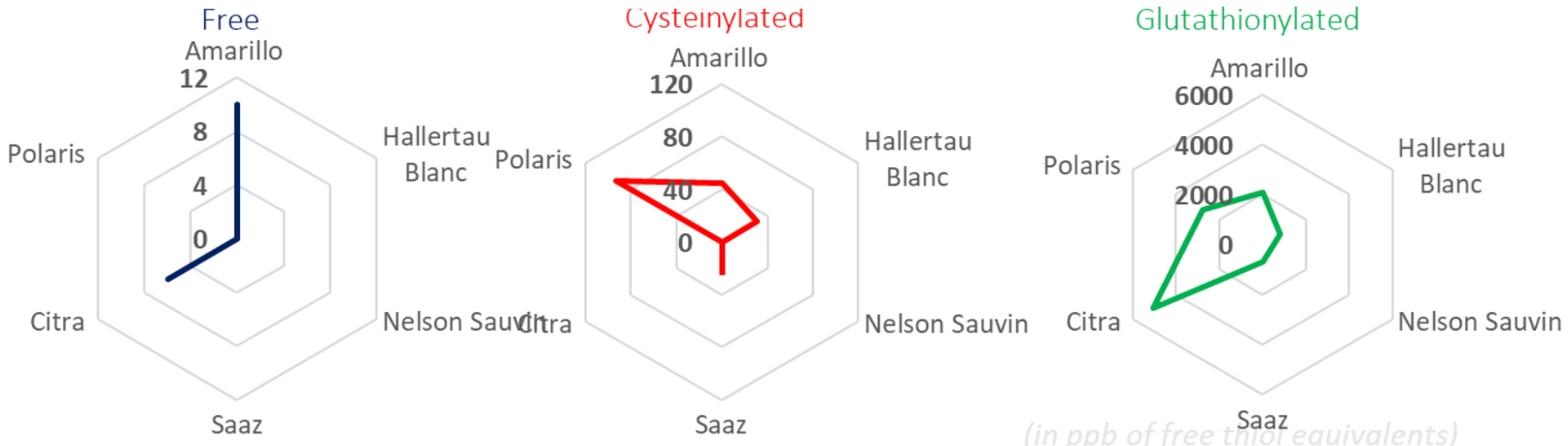


- \+ Riches : Sorachi Ace & Citra
- G-3SHol ubiquitaire dans les houblons → 100 ppm = énorme potentiel !
- 2 Diastéréoisomères
- G-3S4MPol uniquement dans l'Hallertau Blanc & le Polaris

Kankolongo, Decourrière, Alonso, Bodart, Robiette & Collin, J. Agric. Food Chem., 64 (2016) 8572−8582.

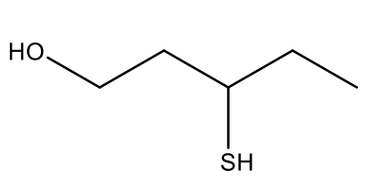
R_3 Les thiols polyfonctionnels

Mise en évidence de nouvelles formes glutathionées dans les houblons

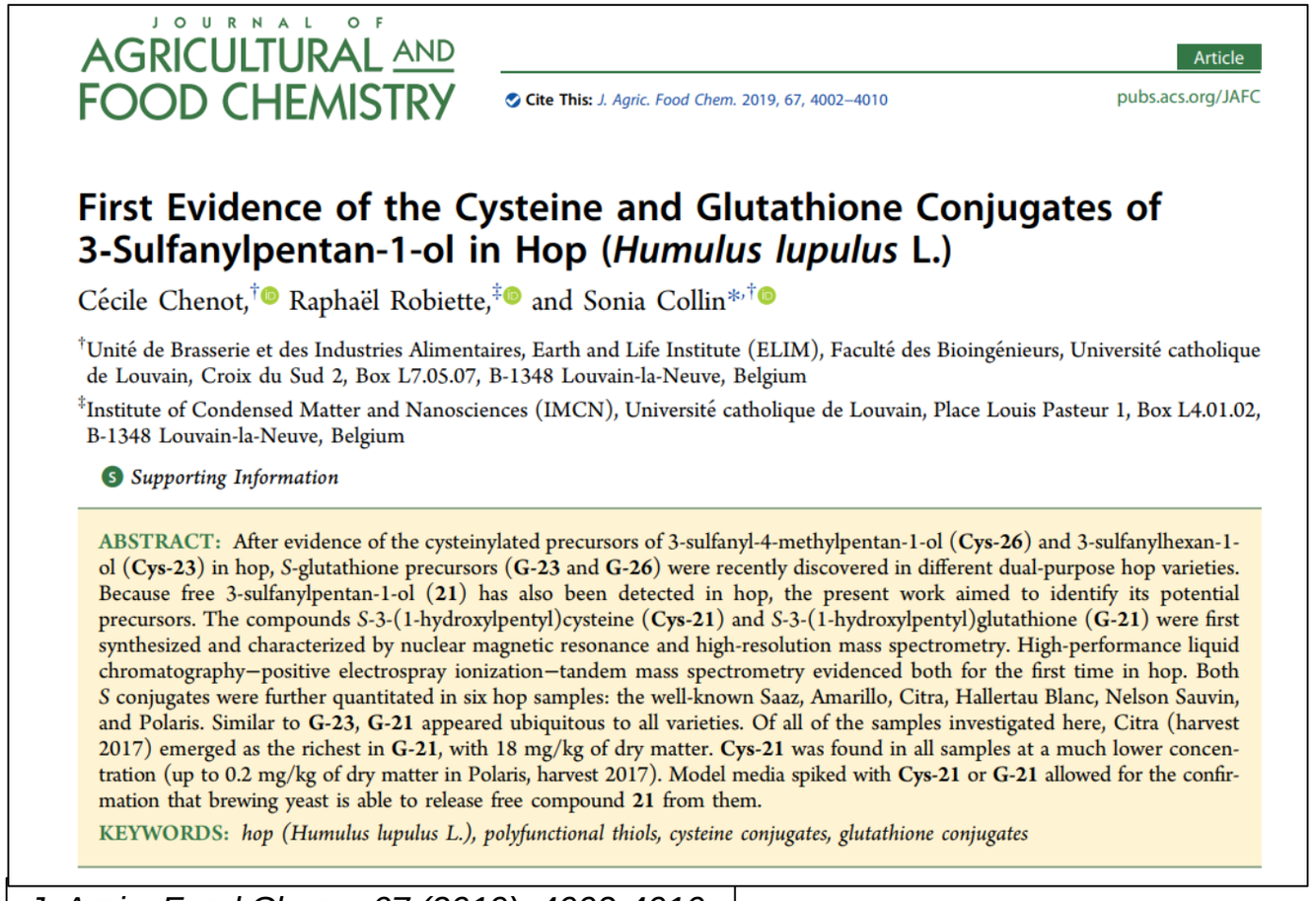


(in ppb of free thiol equivalents)

3-sulfanylpentanol



- **Ubiquitaire**
- **Pas de relation** entre forme libre (max: Amarillo), cysteinée (max: Polaris) et glutathionée (max: Citra)



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Article

Cite This: *J. Agric. Food Chem.* 2019, 67, 4002–4010

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First Evidence of the Cysteine and Glutathione Conjugates of 3-Sulfanylpentan-1-ol in Hop (*Humulus lupulus* L.)

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Supporting Information

ABSTRACT: After evidence of the cysteinylated precursors of 3-sulfanyl-4-methylpentan-1-ol (**Cys-26**) and 3-sulfanylhexan-1-ol (**Cys-23**) in hop, *S*-glutathione precursors (**G-23** and **G-26**) were recently discovered in different dual-purpose hop varieties. Because free 3-sulfanylpentan-1-ol (**21**) has also been detected in hop, the present work aimed to identify its potential precursors. The compounds *S*-3-(1-hydroxylpentyl)cysteine (**Cys-21**) and *S*-3-(1-hydroxylpentyl)glutathione (**G-21**) were first synthesized and characterized by nuclear magnetic resonance and high-resolution mass spectrometry. High-performance liquid chromatography−positive electrospray ionization−tandem mass spectrometry evidenced both for the first time in hop. Both *S* conjugates were further quantitated in six hop samples: the well-known Saaz, Amarillo, Citra, Hallertau Blanc, Nelson Sauvin, and Polaris. Similar to **G-23**, **G-21** appeared ubiquitous to all varieties. Of all of the samples investigated here, Citra (harvest 2017) emerged as the richest in **G-21**, with 18 mg/kg of dry matter. **Cys-21** was found in all samples at a much lower concentration (up to 0.2 mg/kg of dry matter in Polaris, harvest 2017). Model media spiked with **Cys-21** or **G-21** allowed for the confirmation that brewing yeast is able to release free compound **21** from them.

KEYWORDS: *hop (Humulus lupulus L.), polyfunctional thiols, cysteine conjugates, glutathione conjugates*

J. Agric. Food Chem., 67 (2019), 4002-4010.

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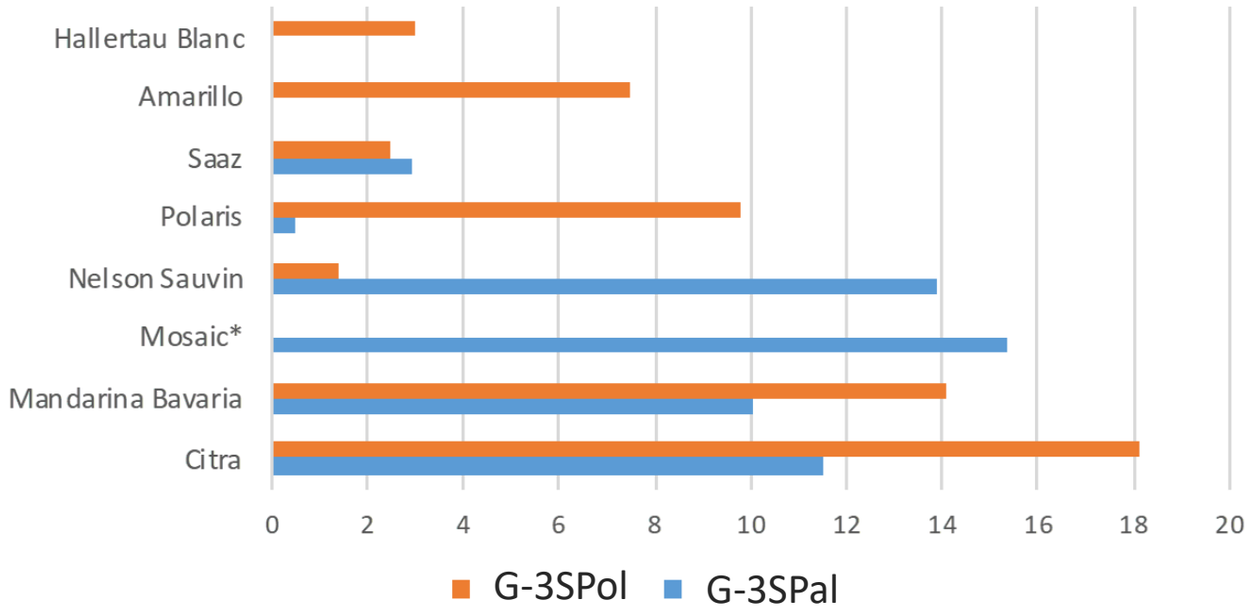
Mise en évidence de nouvelles formes glutathionées dans les houblons

3-sulfanyl -pentanal & -hexanal + 3 sulfanyl -pentyl & -hexyl acétate

5C



ppm

- **G-3SPal :** retrouvé dans 6 houblons
- Qté similaire à G-3SPol (excepté Nelson Sauvin)
- **Cys-3SPal** : nd
- **Cys-3SPA :** 4.2 ppm dans Mosaïc (detecté dans Citra & Nelson Sauvin)

6C

L

- **G-3SHal :** detecté dans les mêmes houblons que G-3SPal (excepté Citra)
- 10 à 100 x moins que G-3SHol
- **Cys-3SHal** : nd
- **Cys- et G- 3SHA** : nd

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https://doi.org/10.1080/03610470.2021.2015560

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Thiol S-Conjugate Profiles: A Comparative Investigation on Dual Hop and Grape Must with Focus on Sulfanylalkyl Aldehydes and Acetates Adducts

Cécile Chenot[a], Sébastien Haest[a], Raphaël Robiette[b] and Sonia Collin[a]

[a]Unité de Brasserie et des Industries Alimentaires, Louvain Institute of Biomolecular Science and Technology (LIBST), Faculté des Bioingénieurs, Université catholique de Louvain, Louvain-la-Neuve, Belgium; [b]Institute of Condensed Matter and Nanosciences (IMCN), Université catholique de Louvain, Place Louis Pasteur, Louvain-la-Neuve, Belgium

Chenot, Haest, Robiette, Collin. J. Am. Soc. Brew. Chem., 81 (2023), 23-32.

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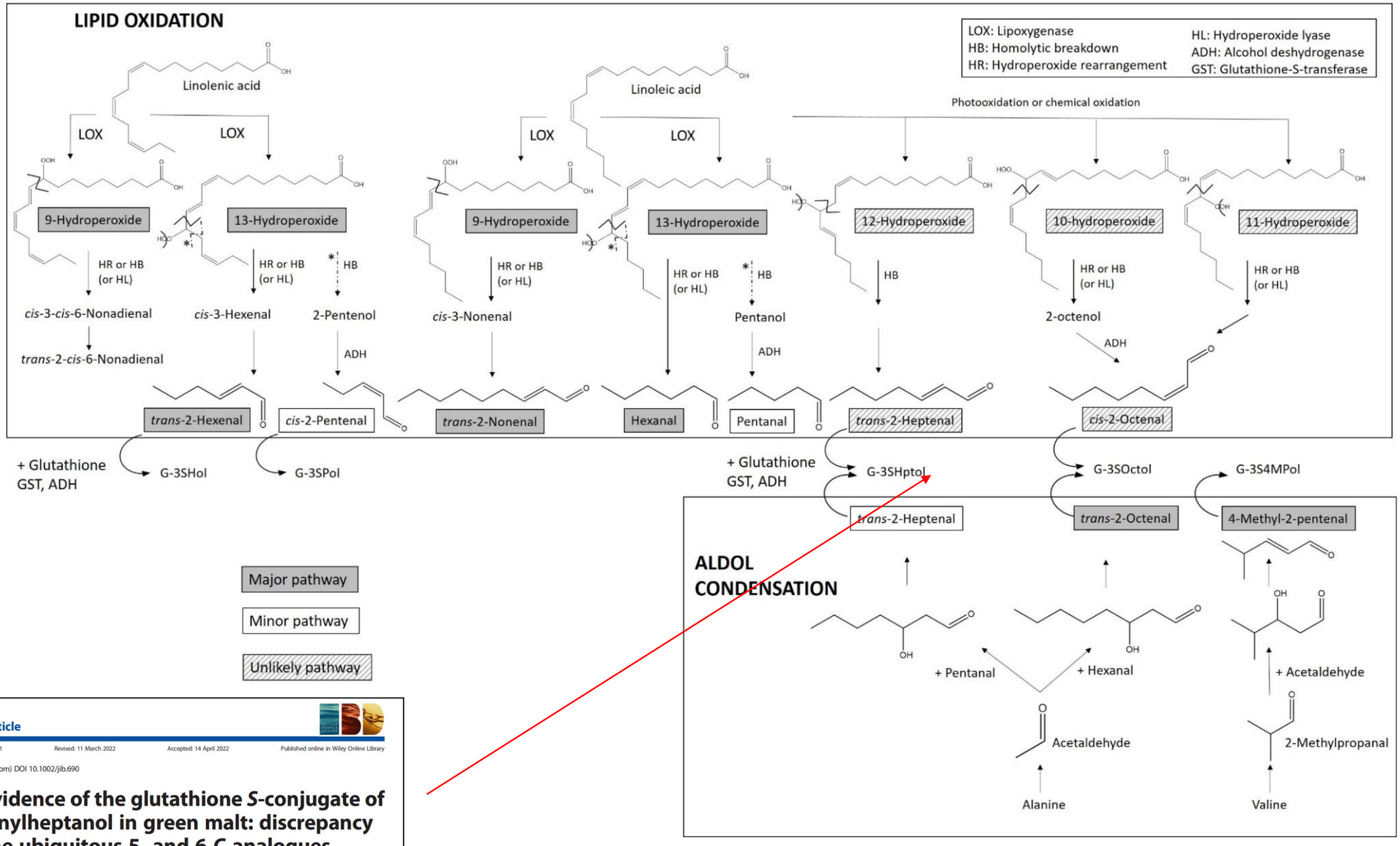
R_3 Les thiols polyfonctionnels

Mise en évidence de nouvelles formes glutathionées dans les houblons et malts



Research article

Received: 13 October 2021 Revised: 11 March 2022 Accepted: 14 April 2022 Published online in Wiley Online Library

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First evidence of the glutathione *S*-conjugate of 3-sulfanylheptanol in green malt: discrepancy with the ubiquitous 5- and 6-C analogues

Cécile Chenot,[1] Raphaël Robiette[2] and Sonia Collin[1]*

Chenot, Robiette, Collin. J. Inst. Brew., J. Inst. Brew., 128 (2022), 43-51.

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Relargage par levures hautes en fermentation primaire

JOURNAL OF AGRICULTURAL AND FOOD CHEMISTRY

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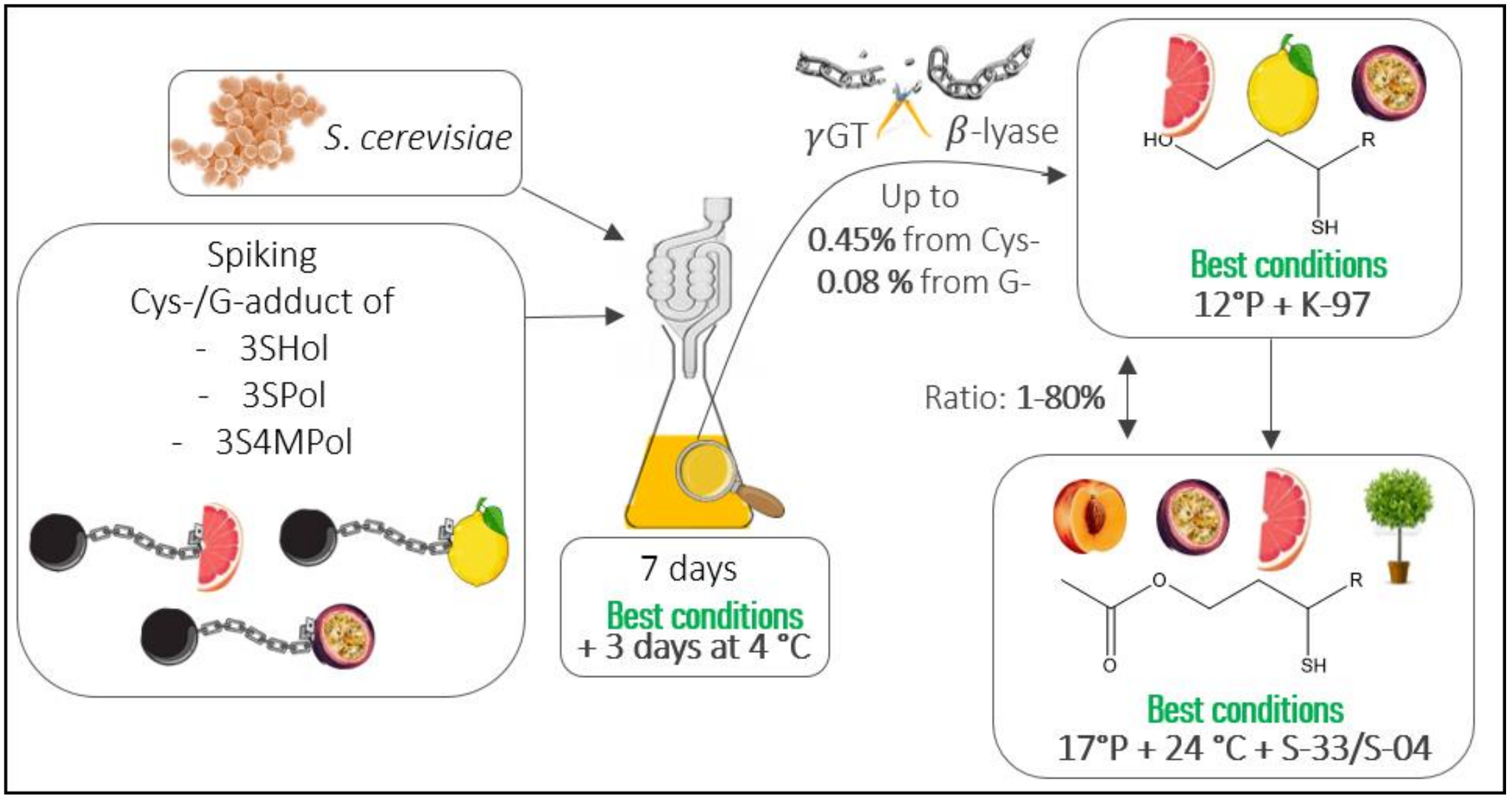
Modulation of the Sulfanylalkyl Acetate/Alcohol Ratio and Free Thiol Release from Cysteinylated and/or Glutathionylated Sulfanylalkyl Alcohols in Beer under Different Fermentation Conditions

Cécile Chenot, Eloi Thibault de Chanvalon, Philippe Janssens, and Sonia Collin*



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Relargage par levures basses en fermentation primaire

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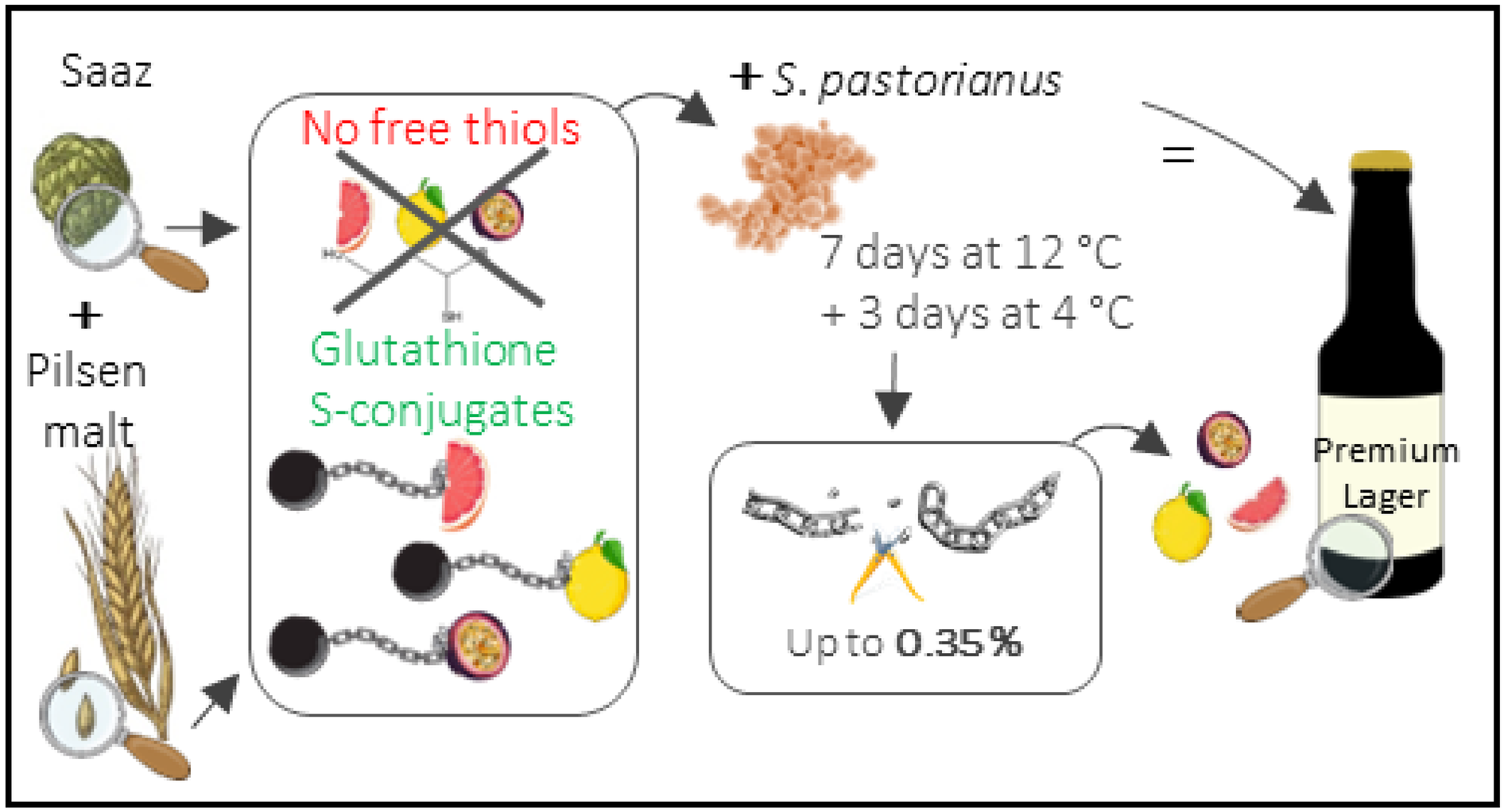
Malt and Hop as Sources of Thiol *S*-Conjugates: Thiol-Releasing Property of Lager Yeast during Fermentation

Cécile Chenot, William Donck, Philippe Janssens, and Sonia Collin*

Cite This: https://doi.org/10.1021/acs.jafc.1c07272 Read Online



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Les thiols polyfonctionnels

Relargage par enzymes exogènes

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https://doi.org/10.1080/03610470.2021.2021766

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Ability of Exogenous or Wort Endogenous Enzymes to Release Free Thiols from Hop Cysteinylated and Glutathionylated *S*-Conjugates

Cécile Chenot[a], Guillaume Willemart[a], Jacques Gros[b] and Sonia Collin[a]

[a]Unité de Brasserie et des Industries Alimentaires, Louvain Institute of Biomolecular Science and Technology (LIBST), Faculté des Bioingénieurs, Université Catholique de Louvain, Louvain-la-Neuve, Belgium; [b]Anheuser-Busch InBev nv/sa, Leuven, Belgium

ABSTRACT

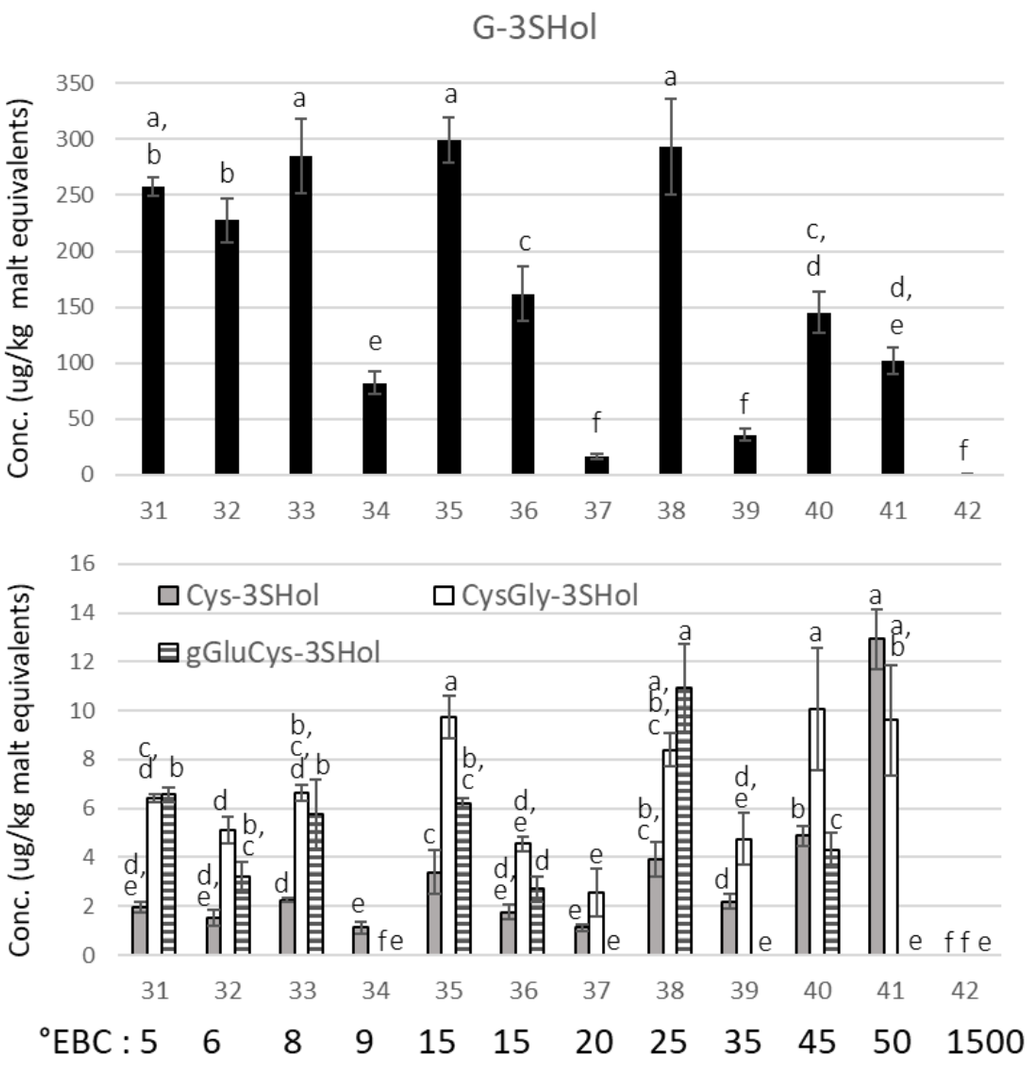
Following identification of the substantial pool of aromas represented by cysteinylated and glutathionylated precursors in hop, many studies have focused on developing methods for releasing volatile thiols based on chemical hydrolysis or yeast activity. The present work aimed to evaluate the release activity of either commercially available exogenous enzymes or endogenous wort enzymes (issued from hop or malt) on synthetic Cys- or G-precursors of 3SHol, 3SPol, 3SHptol, 3S4MPol and 4S4M2Pone. In a model medium at pH 7.7, both apotryptophanase and cystathionine β-lyase appeared very efficient on Cys-adducts (up to 77.9% release). Interestingly, a slight proportion of this release (up to 0.9%) appeared to result from chemical hydrolysis. In polyphenols rich media such as hop and beer, total release was much lower (under 0.2%). For release from G-adducts, a combination of γGT and apotryptophanase proved to be effective, highlighting for the first time the ability of β-lyase activity to release free PFTs also from CysGly-adducts. The best release rates (above 2% for all thiols) were obtained with a two-step incubation: 75 units γGT with synthetic glutathionylated precursors for 16 h at 37 °C in the presence of alanine followed by apotryptophanase for 7 h at room temperature. The alternative combination of γGT with *S. cerevisiae* was found to release free thiols more efficiently than yeast activity alone. Compared to commercially available enzymes, endogenous enzymes extracted from hop or malt displayed lower efficiency (below 0.3% for γGT activity and below 0.002% for β-lyase).

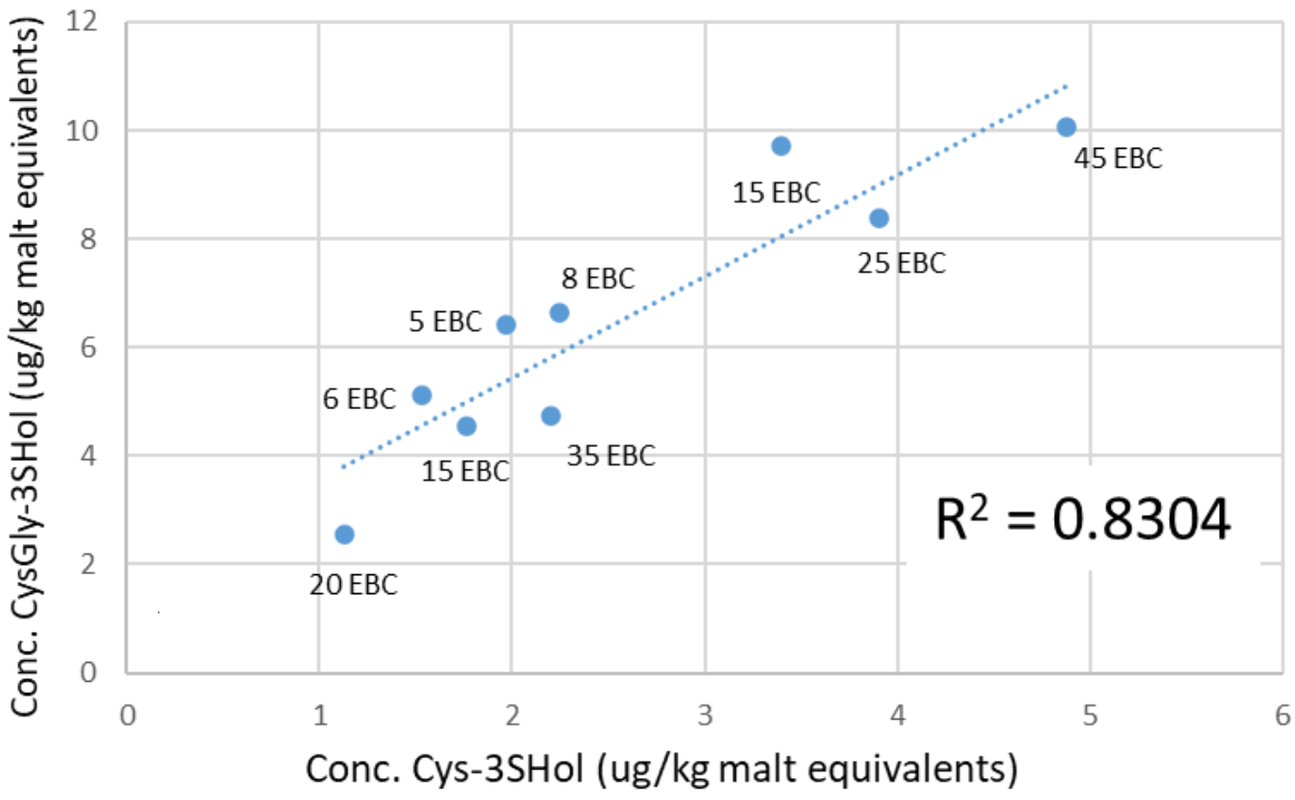
KEYWORDS

β-lyase;
Hop (*Humulus lupulus* L.);
γGT;
polyfunctional thiols;
S-conjugates

R_3 Les thiols polyfonctionnels

Relargage par touraillage dans le malt





C. Chenot, S. Collin, L. Suc & A. Roland. J. Am. Soc. Brew. Chem. (2023), in press.

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R_4 Enjeux environnementaux : malt vert, sorgho, toxines, filtration régénérable, ..

Research article

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(wileyonlinelibrary.com) DOI 10.1002/jib.690

First evidence of the glutathione *S*-conjugate of 3-sulfanylheptanol in green malt: discrepancy with the ubiquitous 5- and 6-C analogues

Cécile Chenot,[1] Raphaël Robiette[2] and Sonia Collin[1]*

100%

JOURNAL OF AGRICULTURAL AND FOOD CHEMISTRY

ARTICLE

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Potentiality of Red Sorghum for Producing Stilbenoid-Enriched Beers with High Antioxidant Activity

Meike Bröhan, Vesna Jerkovic, and Sonia Collin*

Unité de Brasserie et des Industries Alimentaires, Earth and Life Institute, ELIM, Faculté d'Ingénierie Biologique, Agronomique et Environnementale, Université catholique de Louvain, Croix du Sud, 2 bte 7, B-1348 Louvain-la-Neuve, Belgium

ABSTRACT: *trans*-Piceid and *trans*-resveratrol were authenticated for the first time by high-resoution mass spectrometry in red sorghum grains. A 0.4−1 mg/kg amount of *trans*-piceid and up to 0.2 mg/kg *trans*-resveratrol were quantified by reversed phase high-performance liquid chromatography−atmospheric pressure chemical ionization(+)-tandem mass spectrometry. The white sorghum samples contained only traces of *trans*-piceid (up to 0.1 mg/kg), and *trans*-resveratrol was absent. In much lower amounts than procyanidins, stilbenoids are not able to contribute significantly to the exceptional antioxidant activity of red sorghum (ORAC, 83−147 μmol TE/g; AAPH, 0.61−1.79 min/mg kg^{-1}). More than 10 mg/kg of total stilbenoids have been reported in some hop varieties. Yet, as hop is a minor wort ingredient as compared to cereals, red sorghum could be the main source of *trans*-piceid in beer. Hop remains, however, the single source of *cis*-piceid.

KEYWORDS: Sorghum, beer, resveratrol, piceid, stilbenoids, antioxidant activity

Food Chemistry 369 (2022) 130917

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Occurrence of (suspected) genotoxic flavoring substances in Belgian alcohol-free beers

A. Dusart[a,*], B. Mertens[a], E. Van Hoeck[a], M. Simon[b], S. Goscinny[a,1], S. Collin[b,1]

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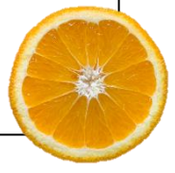

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