# Effect of seasoning time on volatile compounds of Toscano dry-cured ham: first results

*Francesco Sirtori*, Luca Calamai**, Carolina Pugliese**

*Dipartimento di Scienze Zootecniche - Università di Firenze
**Dipartimento di Scienza del Suolo e Nutrizione della Pianta – Università di Firenze

**The objective of this study is to evaluate the effect of seasoning time on aromatic profiles of Toscano dry-cured ham.**

**The Tuscan dry-cured ham is a traditional pork meat product from Central Italy with a Protected Denomination of Origin (PDO) registration**

*Agilent 5975C MSD spectrometer with Gerstel MPS2 XL equipped with SPME automatic fiber exchange option*

## Method

- ✓ *Lean or fat tissue sampled from 10 intact hams at different seasoning times*
- ✓ *The volatile compounds were sampled by SPME*
- ✓ *CARBOXEN/PDMS/DVB fiber.sorption times 15 min at 60 °C*
- ✓ *0.5 g sample blended with 2 ml H2O + 1 g NaCl in 10 ml vials*
- ✓ *Addition of labeled internal standard mix to normalize responses.*
- ✓ *Column J&W Innowax 30 m, 0.25 mm, ID 0.5 µm DF.*

| Compound | RT | Compound | RT | Compound | RT | Compound | RT |
|---|---|---|---|---|---|---|---|
| Etill acetato (istd 1) | 3,22 | 2,3-Octanedione | 20,51 | 2-Octen-1-ol, (Z) | 32,78 | Tetradecanal + 2dodecen-1-olo | 43,50 |
| Butanal, 2-methyl- | 3,86 | 1-Hexanol | 21,84 | Butanoic acid | 33,53 | Heptanoic acid | 44,08 |
| Butanal, 3-methyl- | 3,87 | Nonanal | 23,73 | 2-Decenal, (E) | 33,99 | 2(3H)-Furanone, dihydro-5-pentyl | 45,57 |
| Pentanal | 5,36 | 3-Octen-2-one | 24,16 | Decanoic acid, ethyl ester | 33,79 | Octanoic Acid | 45,84 |
| Butanoic acid, ethyl ester | 7,17 | 2-Octenal, (E) | 25,09 | 2,4-Nonadienal, (E,E) | 36,24 | Hexadecanal | 46,84 |
| Toluene | 7,27 | 1-Octen-3-ol | 26,09 | Benzaldehyde, 3-ethyl | 36,47 | Nonanoic acid | 47,07 |
| Butanolo (istd 2) | 12,37 | 1-Heptanol | 26,31 | 2-Undecenal | 38,21 | Phenol, 3,4-dimethyl- | 47,57 |
| Hexanal | 8,87 | Pentadecane | 27,51 | Oxime-, methoxy-phenyl | 38,66 | Hexadecanoic acid, ethyl es | 47,80 |
| Heptanal | 13,35 | 2,4-Heptadienal, (E,E) | 27,93 | Ethanol, 1-(2-butoxyethoxy) | 38,67 | n-Decanoic acid | 47,89 |
| Etil esanoato (istd 3) | 15,25 | 3,5-Octadien-2-one | 29,07 | 2,4-Decadienal, (E,E) | 40,27 | Octadecanal | 48,41 |
| Furan, 2-pentyl | 15,72 | Benzaldehyde | 29,13 | Hexanoic acid | 41,51 | 9-Octadecenal, (Z) | 48,60 |
| Hexanoic acid, ethyl ester | 15,72 | 2-Nonenal, (E) | 29,63 | Trans-geranilacetone | 42,09 | Octadecanoic acid, ethyl est | 49,02 |
| 3-Octanone | 15,72 | Acetic acid (istd 4) | 26,15 | Benzyl Alcohol | 42,07 | Phenol, 3,4-dimethyl- (istd 6) | 47,57 |
| 1-Pentanol | 17,26 | Decanal | 28,07 | Phenylethyl Alcohol | 43,20 | Dodecanoic acid | 49,12 |
| Octanal | 18,66 | 1-Octanol | 30,57 | Esanoic acid (istd 5) | 41,06 | Tetradecanoic acid | 50,52 |
| 2-Heptenal, (Z) | 20,30 | 3,5-Octadien-2-one, (E,z) | 31,12 | 2(3H)-Furanone, 5-butyldihyd | 43,37 | n-Hexadecanoic acid | 52,49 |

**About 80 compounds were identified by comparison with NIST 05 spectral library belonging to: esters, aldehydes organic acids, ketones, alcohols and furans.**

On lean tissue the trend is not clear. This is probably due to the high data variability.

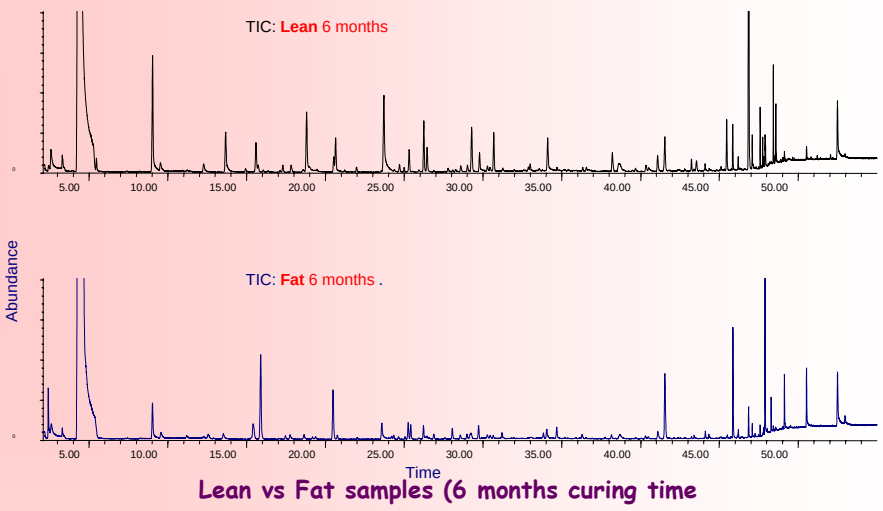
On adipose tissue a significant increase, during curing time, of organic acids, alcohols and esters was registered. For aldhydes, ketones and furans a increase for the first three months was observed. In the following three months there was a decrease.

### Lean - ANOVA on normalized areas of volatile compounds by compound class

| Compounds | Time of seasoning (months) | | | | Sign. |
|---|---|---|---|---|---|
| | 0 | 1 | 3 | 6 | |
| Aldehydes | 20.397 | 15.496 | 20.186 | 28.710 | ns |
| Organic Acids | 3.744 | 16.663 | 21.240 | 14.634 | ** |
| Alcohols | 26.830 | 5.541 | 7.486 | 7.442 | ns |
| Ketones | 4.420 | 10.988 | 11.264 | 7.158 | ** |
| Esters | 1.849 | 1.956 | 3.187 | 1.713 | ns |
| Furans | 1.990 | 4.636 | 5.417 | 5.943 | ** |

### Fat - ANOVA on normalized areas of volatile compounds by compound class

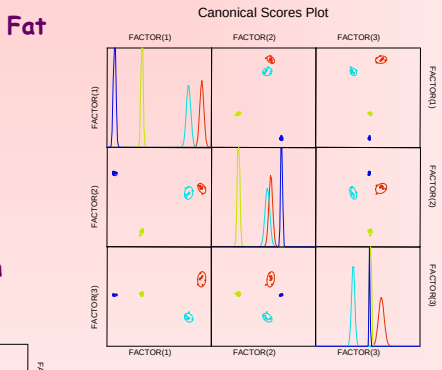
| Compounds | Time of sesoning (months) | | | | Sign. |
|---|---|---|---|---|---|
| | 0 | 1 | 3 | 6 | |
| Aldehydes | 7.946 | 13.343 | 43.075 | 26.536 | ** |
| Organic Acids | 3.966 | 8.143 | 15.524 | 24.894 | ** |
| Alcohols | 1.010 | 1.755 | 2.699 | 3.175 | ** |
| Ketones | 2.084 | 2.049 | 8.072 | 4.982 | ** |
| Esters | 1.694 | 1.588 | 7.277 | 13.618 | ** |
| Furans | 2.180 | 6.538 | 6.075 | 3.898 | ** |



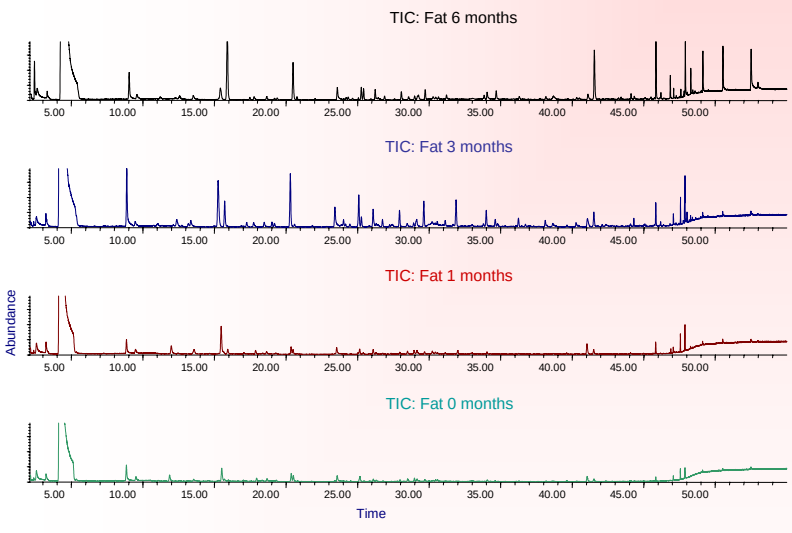

Lean vs Fat samples (6 months curing time

| Jackknifed Classification Matrix | | | |
|---|---|---|---|
| | Lean | FAT | %correct |
| Fat | 37 | 1 | 97 |
| | 5 | 34 | 87 |
| Total | 42 | 35 | 92 |

Discriminant analysis separate fat from lean samples

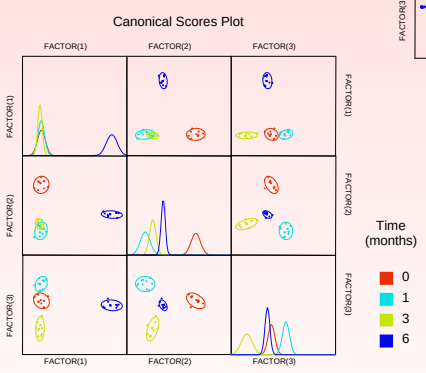



Fat




Evolution of volatile Compounds in ham fat during seasoning

Lean




Jackknifed Classification Matrix - TIME

| | 0 | 1 | 3 | 6 | %correct |
|---|---|---|---|---|---|
| 0 | 8 | 0 | 0 | 1 | 89 |
| 1 | 1 | 9 | 0 | 0 | 90 |
| 3 | 0 | 2 | 8 | 0 | 80 |
| 6 | 0 | 0 | 1 | 9 | 90 |
| Total | 9 | 11 | 9 | 10 | 87 |

Discriminant analysis on normalized peak areas of identified compounds separate specimens at different curing time for fat and lean

Canonical Discriminant Functions : Standardized by Within Variances

| | 1 | 2 | 3 |
|---|---|---|---|
| BUTANAL__2_METHYL | 1.31979917 | 1.95534261 | -0.94279847 |
| BUTANAL__3_METHYL | 0.4495598 | -2.05211862 | -0.49086817 |
| PENTANAL | -0.61225527 | 0.28066822 | 0.47326013 |
| BUTANOIC_ACID__ETHYL_ESTER | 0.00819795 | -1.36288676 | 0.76069968 |
| TOLUENE | 0.27059211 | 0.80143086 | -1.30847803 |
| HEXANAL | -0.97039356 | -0.35630394 | 1.30548265 |
| HEPTANAL | 1.57230246 | -0.57629512 | -0.72709224 |
| FURAN__2_PENTYL | | | |
| HEXANOIC_ACID__ETHYL_ESTER | -1.73162126 | 1.55921798 | -3.53790892 |
| 1_PENTANOL | -1.17371754 | -1.97324031 | -1.651288 |
| OCTANAL | -0.15141898 | -0.16394711 | -1.13752496 |
| 2_HEPTENAL__Z | -2.60991114 | 3.30574814 | 0.04273035 |
| 2_3_OCTANEDIONE | 0.91646833 | 0.80862664 | 2.91601173 |
| 1_HEXANOL | -21.4422418 | -4.55982333 | 8.41163883 |
| NONANAL | 0.53435957 | -0.93760125 | 0.65649596 |
| 3_OCTEN_2_ONE | | | |
| 2_OCTENAL__E | | | |
| 1_OCTEN_3_OL | | | |
| 1_HEPTANOL | | | |
| PENTADECANE | 3.64874024 | -2.36295928 | 0.45397957 |
| 2_4_HEPTADIENAL_E_E | 21.4057518 | 2.26733991 | -6.62290295 |
| | 8 | | |
| 3_5_OCTADIEN_2_ONE | | | |
| BENZALDEHYDE | | | |
| 2_NONENAL_E | | | |
| DECANAL | | | |
| 1_OCTANOL | 0.71384689 | 1.28434203 | 1.77375444 |
| 3_5_OCTADIEN_2_ONE_E_Z | -0.49934899 | 0.06733377 | 1.28719069 |
| 2_OCTEN_1_OL_Z | -0.94789866 | -2.01531958 | -3.94607924 |
| BUTANOIC_ACID | 0.30299068 | -0.47496596 | 0.52302486 |
| 2_DECENAL E | | | |
| DECANOIC_ACID_ETHYL_ESTER | | | |
| 2_4_NONADIENAL__E_E | | | |
| BENZALDEHYDE_3_ETHYL | | | |
| 2_UNDECENAL | | | |
| OXIME_METHOXY_PHENYL | | | |
| ETHANOL_1_2_BUTOXYETHOXY | | | |
| 2_4_DECADIENAL__E_E | | | |
| HEXANOIC_ACID | | | |
| BENZYL_ALCOHOL | -0.25137385 | 0.42612885 | -0.32705109 |
| PHENYLETHYL_ALCOHOL | | | |
| 2_3H_FURANONE_5_BUTYLDIHYD | | | |
| TETRADECANAL_2DODECEN_1_OLO | | | |
| HEPTANOIC_ACID | | | |
| 2_3H__FURANONE__DIHYDRO_5_PENTY | 0.23401878 | -0.30191866 | -0.44258321 |
| L | | | |
| OCTANOIC_ACID | | | |
| HEXADECANAL | | | |
| NONANOIC_ACID | | | |
| PHENOL_3_4_DIMETHYL | | | |
| HEXADECANOIC_ACID_ETHYL_ES | | | |
| N_DECANOIC_ACID | | | |
| UNK_Z | | | |
| 9_OCTADECENAL_Z | | | |
| OCTADECANOIC_ACID_ETHYL_EST | | | |
| DODECANOIC_ACID | | | |
| TETRADECANOIC_ACID | | | |
| N_HEXADECANOIC_ACID | | | |

Canonical Discriminant Functions
Standardized by Within Variances

## CONCLUSIONS

- About 80 compounds were identified belonging to: esters, aldehydes organic acids, ketones, alcohols and furans.
- There were significant differences by compound class during curing time in only in fat specimens
- Discriminant analysis an normalized peak areas separate specimens at different curing time