THE EVOLUTION OF AROMATIC PROFILE OF SANGIOVESE WINE DURING FERMENTATION AS REVEALED BY A QUANTITATIVE SPME-GCMS APPROACH: METHOD DEVELOPEMENT

Luca Calamai[1,2], Irene Lozzi [1], FedericoValori[1], Francesca Boscaro[2]

1 Centro Interdipartimentale di Spettrometria di Massa, Università di Firenze
2 Dipartimento di Scienza del Suolo e Nutrizione della Pianta, Università di Firenze

During winemaking process, the profile of aromatic compounds evolves and the primary varietal scent from the grapes are complemented and/or transformed by the yeasts' activitey. Also, condensation, hydrolysis and trans-esterification reactions take places recurrently as the concentration of alcools and organic acids continuously change during wine making catalyzed by must and wine acidic pH .

Is it possible to gather quantitative information on the volatile compound during wine making by SPME- GCMS as a function of different oenological tretments to be used as feedback process indicators?

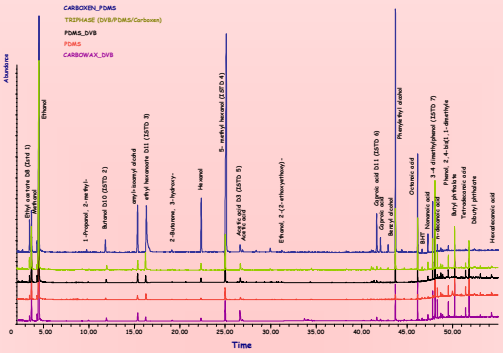
Experimental

-Samples collected 1 day after crushing and destemming
- Different wine making treatments:
Cold prefermentation maceration at 5 °C
Crio macetation with liquid nitrogen
Traditional winemaking

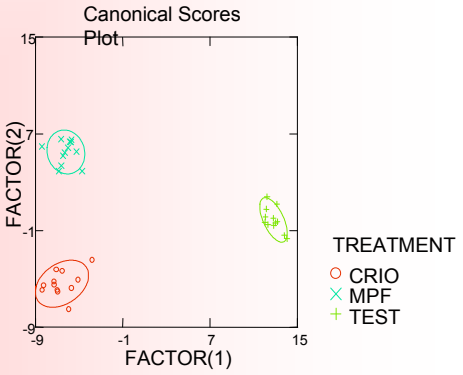
Method developement

-5 ml of must or wine supplemented with 2 g of NaCl in 20 ml vials
-5 types ofSPME fiber: DVB/CARBOXEN/ PDMS fiber,
- sorption times, f rom 5 , 10, 30 min min
-Addition of labeled internal standard mix to normalize responses.
-column J&W Innowax 30 m, 0.25 mm, ID 0.5 µm DF; injection temperature 250°C, splitless mode, oven program: 40 degrees for 1 min then 2°C/min to 60 °C, then 3°C/min to 150°C, then 10°C/min to 200°C, then 25°C/min to 260°C for 6.6 min

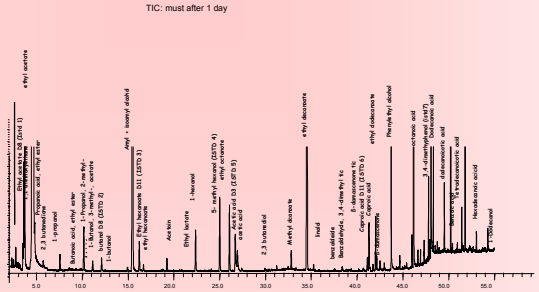
Agilent 5975C MSD spectrometer with Gerstel MPS2 XL equipped with SPME automatic fiber exchange option

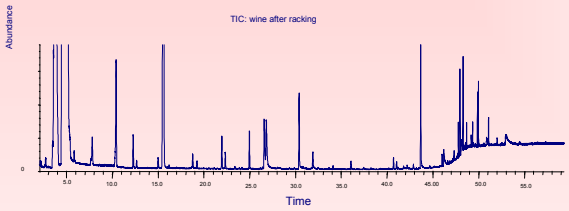


The best overall results were obtained with a 65 um carboxen/PDMS/ DVB SPME fiber exposed in the head space of a 20 ml vial under continuous agitation for 20 min at 80°C

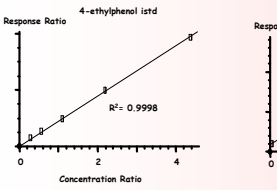


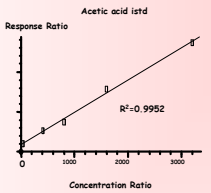
Discriminant analysis on compound concentration indicate statistically significant grouping with treatments

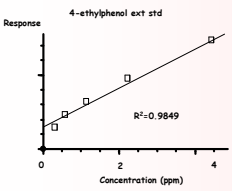


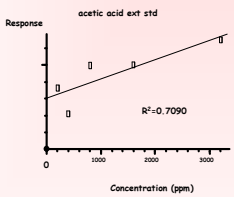


Both primary and secondary aromatic compounds are identified in the samples.
SPME-GC ms analysis during winemaking may provide insights on the fermentation dynamics









Reliable calibration lines were obtained when the appropriate internal standards (ISTD) was used for calibration. An ISTD mix composed of two compounds (fast and late eluting) for each class was added to all samples.

Canonical Discriminant Functions : Standardized by Within Variances

	1	2
1_1_DIETHOXYETHANE	0.36309	0.047257
ETHYL_ACETATE	4.780526	-1.35864
PROPANOIC_ACID_ETHYL_ESTER	-4.77559	-0.71234
BUTANOIC_ACID_ETHYL_ESTER	-4.00784	1.030564
3_BUTANOLO	0.29489	0.368357
1_PROPANOL_METHYL	2.531798	-1.33972
AMYL + ISOAMYL ALCOHOL	-3.66267	13.293
1_BUTANOL_2_METHYL_ACETATE	1.294225	10.1613
ETHYL HEXANOATE	-3.69002	-1.28373
ETHYL LACTATE	3.436858	2.949362
ETHYL DECANOATE	1.805871	4.620106
ETHYL DODECANOATE	-2.03198	-2.56303
ETHYL TETRADECANOATE	-6.30733	-2.14502
3_HEXANOL	-3.18469	-2.56937
2_HEXENAL-E	-0.36237	-1.84538
3_HEXEN_1_OL_Z	-1.23956	-0.66318
1_OCTANOL	2.98669	0.772886
1_DODECANOL	-2.2893	-2.27307
OCTANOIC ACID	0.397371	14.59236
N_DODECANOIC ACID	-3.68283	1.045351
N_DECANOIC ACID	5.40138	10.0614
BENZOIC ACID	-5.3553	-4.00649
TETRADECANOIC TIC	4.790019	0.504301
HEXADECANOIC ACID		
GERANIOL	-2.33336	-1.16235
LINALOL		
BENZALDEHYDE	-0.39774	-1.26008
BENZALDEHYDE_4_ETHYL	2.443666	2.239546
BENZYL_ALCOHOL	1.017217	1.036393
PHENYLETHYL_ALCOHOL		
PHENOL_4_BIS_1_1_DIMETILETHYL	-2.12442	-4.53027

almost all identified compounds are included in the composition of discriminant factors

CONCLUSIONS

-Both primary and secondary compounds are revealed by SPME-GCMS analysis
-Triphasic *DVB/CARBOXEN/ PDMS fiber afforded the best performances*
-Reliable quantation curves were obtained by the use of a multiple ISTD mix composed of labelled compounds