Application

Classification of Coffee Powder and Instant Coffee
with PEN2 and EDU2 instruments

Introduction

The capability of a Sensor Array Instrument (Electronic Nose) is to classify samples by investigating the gas composition accompanying the sample and by comparing to a given classification that has been trained before. The PEN2 Sensor Array instrument comprises an array of 10 Metal Oxide Sensors (MOS) and a sample flow system. During the application setup, a number of series of analyses were carried out while changing the parameters for the measurement and data evaluation.

The principal goal of the application described is the classification of the quality of coffee powder and instant coffee powder (final product).

Experimental

The instruments involved in the application setup are :

- PEN2 – Sensor array instrument (standard sensor set) with software WinMuster V1.5.2., optional additions: autosampler connection
- EDU2 – Trap&Thermal Desorption Unit – involved in the analysis for one measurement series
- Headspace Sampler HSS32 (Analytik Jena) expanded for dynamic headspace sampling. This instrument is involved using only parameters that can be reproduced with a manual sampling procedure.

The instruments are coupled via 3mm Teflon tubes having ambient temperature or heated transfer lines.

Fig 1 : HS-Sampler coupled to Sensor array instrumentation

After incubation, the sample is driven by means of the internal sampling pump of the PEN2 instrument or the EDU2 system, respectively.

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The following samples were given for carrying out the investigation :

Good Powder	Good Instant Coffee	Middle Powder	Middle Instant Coffee	Bad Instant Coffee
844	848	846	842	854
845	849	847	851	855
	850		852	857
			853	858
			856	

The names of the columns indicate two characterizations of the samples :

a) the given classification of the samples – obtained as the result of a panel test
b) the physical characteristic. ("powder" indicates that the samples consist of grinded coffee, the other samples consist of instant coffee)

This distinction is necessary, because the grinded coffee releases a completely different headspace compared to the other samples (see sect. "Results").

The given samples were filled into 10ml vials using 1,0g of one sample. 40ml vials filled with 3g have been used for manual sampling respectively. The sampling vials were capped using a Teflon coated silicon rubber septum.
After filling, the samples were equilibrated at room temperature for different periods, between 3h up to 24h, assuming that the storage time is not influencing the analysis as long as equilibrium in the headspace can be considered.

Zero gas is provided by a zero air generator (active charcoal filtering).

Procedures

For a measurement series each sample was filled in three vials, so the total number of samples for a series is 48. One empty sample was measured in advance of each series.
Some samples consist of very fine powder particles.
All procedures are based on dynamic headspace sampling. The gas from the headspace of a sample vial is sucked by the measuring instrument for a given sampling period.

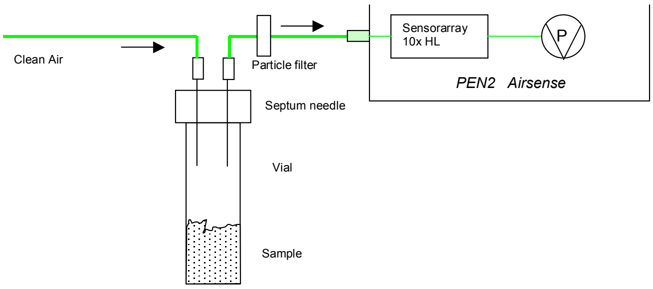

Fig 2 : connection of the sample

In order to prevent particles from entering the sensor array, a particle filter was included in the sampling line. A brief investigation on the memory behaviour of the Teflon coated filter

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showed that no influence on the measurement occurs, as long as the backflush time during the zero gas period is long enough.

The procedures carried out are :

	Name	Comment	Method
1	Headspace ambient temperatue PEN2 - direct sampling	Simple preparation of samples and measurement, short cycle time	PEN2 parameters Injection flow 400ml/min, no autom. dilution, flushing time 420sec, Measurement time 92secs. Acquisition Mode “with Autosampler”1) Pattern extraction at 80 to 82 secs. HSS parameters Ambient 25°C; incubation 4min; dynamic headspace procedure, no pressurization, cycle time 11min, transfer lines ambient
2	Headspace 50°C	Increased sample temp result in higher sample concentration, compounds with higher boiling points	PEN2 parameters Injection flow 400ml/min, no autom. dilution, flushing time 1200sec, Measurement time 92secs. Acquisition Mode “with Autosampler”1) Pattern extraction at 80 to 82 secs HSS parameters 50°C; incubation 15min; dynamic headspace procedure, no pressurization, cycle time 11min, transfer lines ambient
3	Headspace plus Trap/thermal desorption	Enrichment of relevant compounds, eventual selection of analytes during sampling	PEN2 parameters Injection flow 400ml/min, no autom. dilution, flushing time 435sec, Measurement time 85secs. Acquisition Mode “with Autosampler”1) Pattern extraction at 80 to 82 secs. EDU2 parameters Sampling 30sec HSS parameters Ambient 25°C; incubation 4min; dynamic headspace procedure, no pressurization, cycle time 11min, transfer lines ambient

1) Meaning that an automatic alternating sequence of flushing and measuring is provided

Procedure 1 is mainly investigated because it is the most simple and quickest way of analysis. The second procedure has been investigated to determine the influence of an elevated temperature of the headspace. A possible benefit of including the trap&thermal desorption unit into the analysis will be estimated from the 3rd procedure.

Results

Measurement signals

Figures 3 and 4 show the response of the sensors when exposed to the different coffee samples using procedure 1 described above. One example for each class is shown. The signals derived from the coffee samples are high enough (room temperature) to derive reproducible pattern and to enable statistical evaluation.

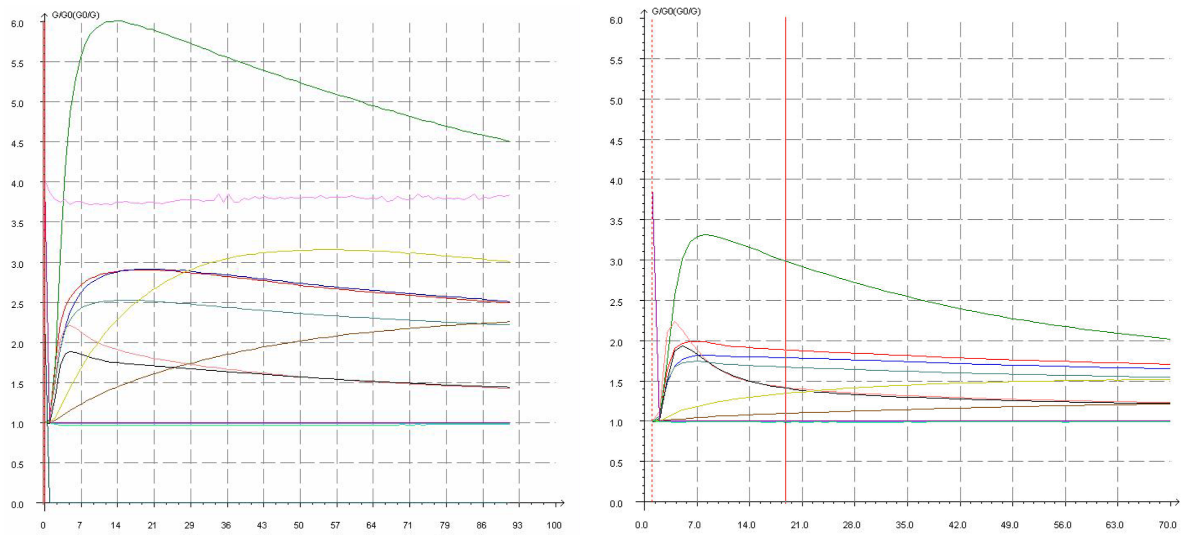

Fig 3 : Sensor signals of samples „10 1000 849“ and „10 1000 852“

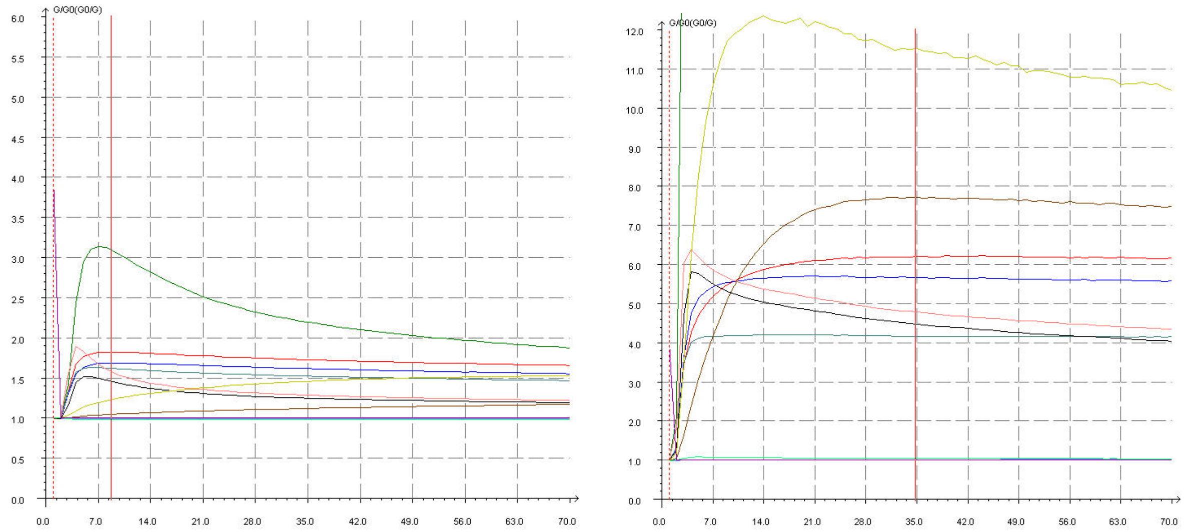

Fig 4 : Sensor signals of samples „10 1000 857” and „10 1000 845“ (double scaling)

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From the measurement signals it can be seen, that samples containing coffee powder exhibit a much stronger signal and a very different pattern compared to the samples of instant coffee.
Two samples of instant coffee (namely „10 1000 854” and „10 1000 855“) consist of very fine powder. Having a sample flow of 400ml/min through the sample causes particles to be dispersed in the vial and sucked into the sampling system. Particles must not enter the sensor chamber, therefore it is strongly recommended to include a particle filter (e.g. Roth Teflon Filter Rotilabo PTFE, 0,45µ) into the sampling line. Using the backflush mode the particle filter is cleaned after each measurement. As stated above, the particle filter shows no distortion of the measurement after 3 measurement series (48 measurements) but it is recommended to replace the filter latest after one week.

Discrimination of Coffee Powder – Instant Coffee

The Principal Component Analysis (PCA) is used to reduce the data set and to display the maximal variation of the single measurement points (pattern) in a two dimensional plot.
In order to explain the huge differences between the coffee powder and the instant coffee a PCA-plot from measurements with procedure 1 is displayed.

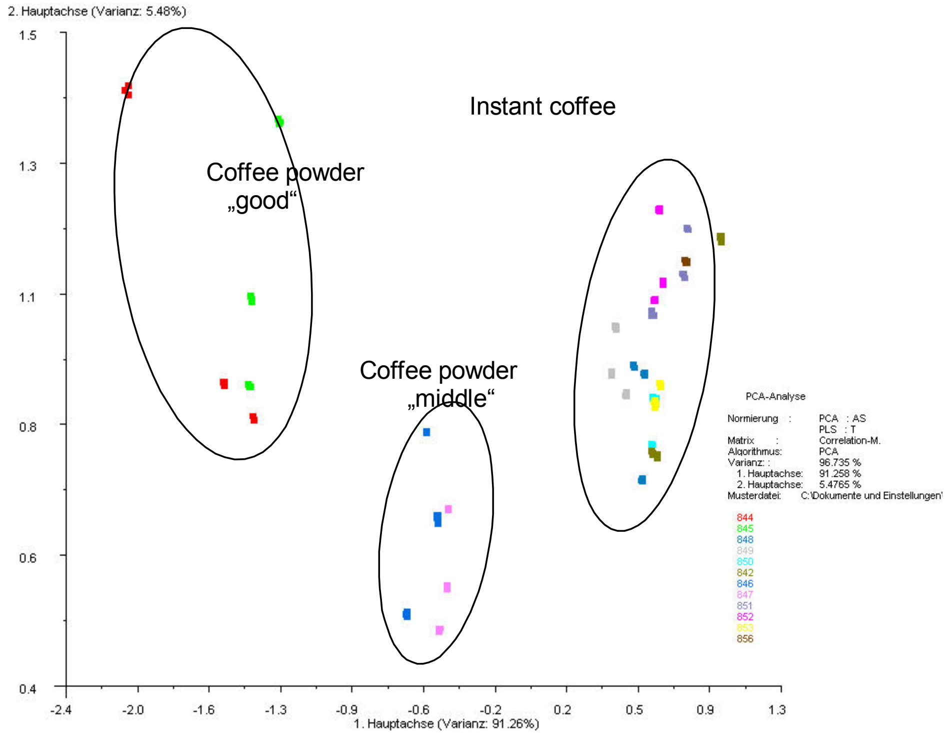

Fig 5 : PCA of coffee powder and instant coffee samples

From this plot can be seen, that the differences between the coffee powder (good and middle) and the instant coffee samples is bigger than the differences in classification of the instant coffee (good and middle). As a result, it is not recommended; to merge the pattern of the powder and the instant coffee product, say to combine the pattern of “good” powder samples and “good” instant coffee samples within one class.

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Therefore, it will be necessary, to separate the data evaluation of both types of samples. The user has to select the corresponding pattern database for the later analysis of unknown. In the ongoing description, the results of both types of samples are investigated separately.

Grinded coffee powder

The coffee powder exhibits strong measuring signals and can be discriminated with all of the three procedures. A PCA-Plot of two measurement series which were acquired at two different days and using procedure 1 is shown.

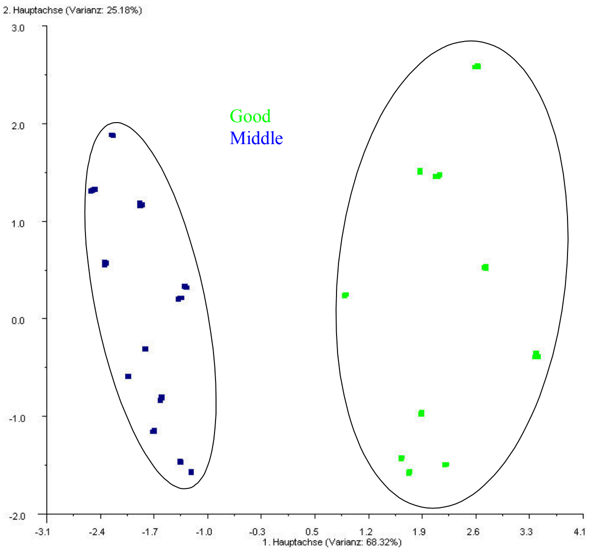

Fig 6 : PCA of coffee powder, procedure 1

The plot shows a first axis discrimination of the two given classes, meaning that a good reproducible discrimination is achieved.

Instant Coffee - Comparison of sampling procedures

The following graphics show pattern data derived from measurements with the instant coffee samples and using the three different procedures.

In terms of discrimination, the procedures 1 and 3 look quite similar. So there is no important positive influence on the classification using the slightly elevated headspace temperature. Despite that, the measuring signals are definitely higher with procedure 2.
In both cases (P1 and P2) there is a good discrimination between the samples named “good” and the samples of class “middle” and bad. The two latter ones are not separated.

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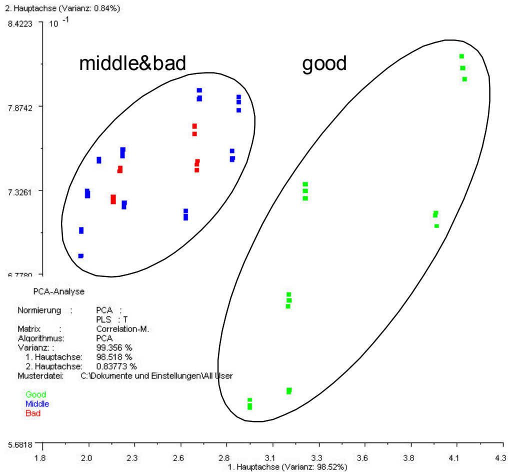

Fig 7a) : instant coffee, procedure 1 (25°C)

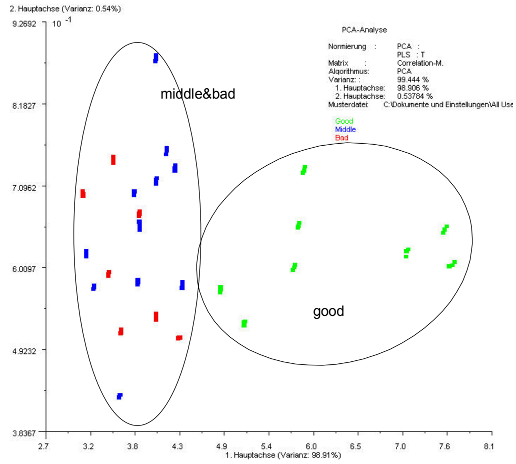

Fig 7b) : instant coffee, procedure 2 (50°C)

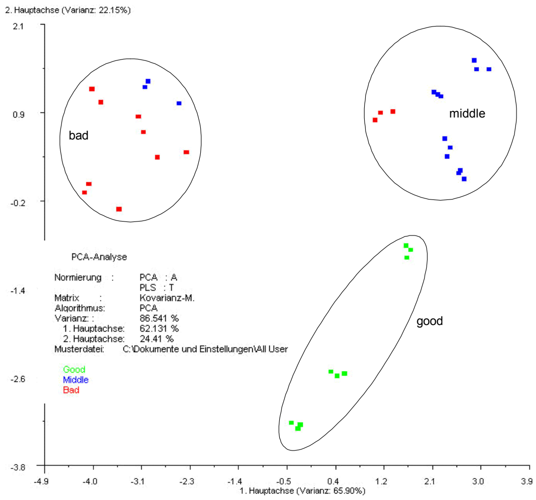

Fig 7c) : instant coffee, procedure 3 (EDU)

In the case of the procedure including the EDU trap&thermal desorption system the groups of “middle” and “bad” seem to have a reasonable discrimination. So the procedure 3 could be used to perform this classification. In Figure 7, also the two samples (namely 856 and 854 show the clear opposite behaviour. The reason for this is unknown, eventually, the samples have been swapped. Furthermore, the number of samples is too small within this pattern file to derive a final statement. This is one point that needs further investigation.

As a result, it will be feasible to discriminate the samples of the class “good” from the others. The procedure 1 (with some modifications in time) is taken as the standard procedure, because of its simple use and that the other procedures do not lead to much better results.
A discrimination of the coffee powder samples is also possible using procedure 1.

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Data evaluation

Using the measurement procedure 1, two series have been carried out at two different days (11 days in between). Mixing the data from the two series (all samples of instant coffee) leads to the pattern shown below:

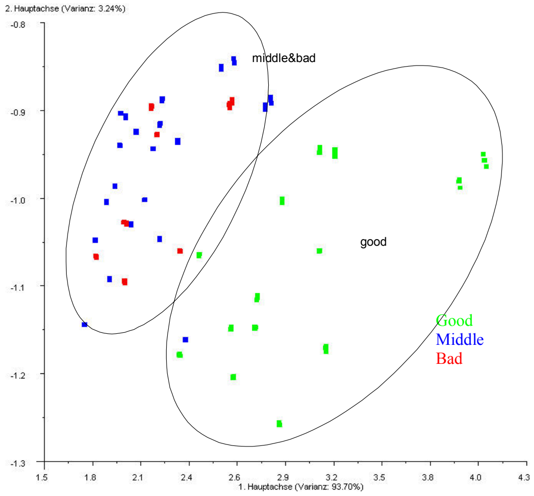

Fig 8 : coffee samples, procedure 1

This pattern has the same characteristics as mentioned for one series. There is a clear distinction of the samples named “good” and the other two classes “bad” and “middle”.
A number that is representing the amount discrimination between classes can be calculated and is called “discrimination power”. The discrimination power calculates the relation between the distances between class centers and the variances of the classes. For the given classes, this number has the following values:

	Good	**Middle**	**Bad**
Good	0,0000	0,4880	0,5428
Middle	0,4880	0,0000	0,1538
Bad	0,5428	0,1538	0,0000

A discrimination power above 0.5 is a reasonable discrimination for an application. This is achieved between the classes “good” and “bad”. Also between “good and “middle” the classification is good enough for the application. Between “bad” and “middle” only a poor discrimination is found.

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