

Acoustic chemometrics for material composition quantification in pneumatic conveying - The critical role of representative reference sampling

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Abstract

Reliable monitoring of pneumatically conveyed particulate materials is critical for on-line detection and controlling material composition changes in the regimen of Process Analytical Technology (PAT), e.g. as the case investigated here: determination of varying concentration levels of *extraneous material* in a source stream. Results are reported from an experimental test campaign on a pilot-scale pneumatic conveying facility. Optimal sensor deployment and material flow rates are decisive parameters for signal quality and prediction performance. The test campaign resulted in an optimal sensor location/flow rate combination, based on which we present a validated prediction model (Partial Least Squares Regression model) for prediction of extraneous material concentration levels, with a $RMSEP_{(rel)}$ of 17.7% ($RMSEP$: 0.92) and r^2 of 0.95. The present approach is based on acoustic chemometrics (a.c.). The impact of nominal reference values vs. representative reference values used as response variables in prediction models is discussed. Optimal reference values were obtained through the use of representative sampling equipment (based on Theory of Sampling, TOS), specifically designed for pneumatic conveying systems, and compared with nominal concentration levels, allowed an improvement of the prediction model: ($RMSEP_{(rel)}$, of 15% ($RMSEP$: 0.85) and r^2 0.95). While the present experimental rig test resulted in relative minor quantitative improvements only, representative reference samples, required for prediction models, are essential when nominal concentration levels cannot be determined or are unknown, which is usually the case in enclosed pneumatic conveying systems, the target for this study. All prediction results are validated with independent data (test set validation).

Key words: Process Analytical Technology, Acoustic chemometrics; Pneumatic conveying; Chemometrics; Reference sampling

1. Introduction

Pneumatic conveying systems for transporting particulate materials are widely used in processing industries. The main advantage of pneumatic conveying is full encapsulation of the transported material, preventing e.g. loss and/or material modifications by external factors such as moisture for example. In many cases it is of particular importance to monitor material composition during pneumatic conveying. Variation in concentration levels of the analyte of interest, or changes in the concentration of extraneous material levels (contaminants, pollutants etc.) in the transported material are important monitoring objectives.

Acoustic chemometrics and monitoring, one recent PAT modality (Process Analytical Technology), has been applied in several earlier studies and cases for on-line monitoring of pneumatic conveying systems [1-6, 21-24], but quantification of material composition based on acoustic chemometrics has previously not been reported for particulate, aggregate systems.

By acquisition, processing and chemometric analysis of the relevant acoustic signals, acoustic chemometrics allows to predict the parameter(s) of interest – e.g. material composition quantifications (matrix, components, trace constituents). Signal quality is critically dependent on sensor location on the conveying system as well as material flow behaviour, for which two different sensor locations and two flow rates were compared and evaluated. Locations and flow characteristics were greatly helped by more than 10 years experience with acoustic chemometrics, *ibid*. This evaluation was performed for two test materials: wheat flour and pulverized alumina, for both using LDPE plastic pellets as a spiking material, standing in as extraneous material at various concentrations. These tests materials were selected within the context of an extensive testing campaign for a newly developed representative sampler (termed ‘EF-sampler’) designed for suspended particulate material streams in horizontal pneumatic conveying systems [13].

For calibrating an optimal model and in order to be able to predict the extraneous material concentrations with acceptable accuracy and precision, reliable reference samples of the various calibration composition levels are required. Only representative reference samples will allow complete optimization of a multivariate calibration the model. The successful validation of the EF-sampler allowed to extract the required representative reference samples for the present research [20].

This study focuses, besides sensor location and optimal flow conditions, on the critical role of representative reference sampling for the final prediction performance by showing the difference in prediction results between *nominal* and *representative* reference values used as input X-data in calibration and validation of the model (nominal values are explained below).

2. Methods

2.1 Acoustic chemometrics

Acoustic chemometrics is a non-invasive PAT measurement technique for on-line monitoring of industrial processes. It involves an interwoven signal acquisition/conditioning and data analysis approach that spans several fields in science, engineering and technology. Acoustic measurement technique is suitable for characterisation and monitoring of several types of industrial processes, based on “acoustic noise” (vibration energy), produced during manufacturing, processing or material transport. The acoustic chemometrics approach has been applied in various areas of research and industrial process monitoring installations [1-6, 21-24].

Some of the inherent advantages of acoustic chemometrics are summarised below:

- On-line and non-invasive – no interruption/ modification of system
- Real time prediction
- Easy sensor deployment (often “clamp on”)
- Passive acoustic energy output from the process is the source; no active signal generation is needed
- Relatively inexpensive sensor technology and easy installation
- Prediction of several parameters of interest from the same acoustic spectrum
- Fast Fourier Transformed acoustic spectra obtained at different resolutions

Acoustic signals are acquired from suitable acoustic sensors deployed on the system involved; data from the acoustic sensors are subsequently subjected to digital signal processing and multivariate calibration, such as PCA or PLS-R. Readers are referred to [1-6] for more details on the principles, theory and application of acoustic chemometrics technique. After the acoustic signal acquisition stage, the signals undergo digital signal conditioning [7,8].

The acoustic chemometric signal path can be summarised as:

- Acoustic emission from the source is measured by the sensor (accelerometer)
- Amplification to the highest obtainable digital resolution (with the aid of a Signal Amplification Module unit)
- Filtering using band-pass filters to attenuate unwanted frequencies (spectral noise)
- Analogue to digital (A/D) conversion. Window transformation in order to reduce spectral leakage
- Fast Fourier Transformation (FFT) from time domain to frequency domain
- Multivariate calibration (Multivariate Data Analysis - MVDA) and proper validation (test set validation)

The acoustic spectra (**X**-data) contain many types of information about the system, e.g. system state, flow characteristics, material composition(s). This unspecified,

potential information is subsequently data analysed, and the parameter of interest is designated as the Y-variable, whereby the correlated information is extracted in the multivariate calibration stage.

2.2 Partial Least Squares Regression (PLS-R)

Advanced measurement and monitoring techniques give rise to complex multivariate data due to the fact that many variables are measured on numerous samples and/or over a long time period. Therefore, a combination of methodologies, or a data analysis approach that has the capacity to elucidate the properties of interest in such data, is needed. PLS-R modelling is a powerful empirical approach, which has the potential to extract the relevant latent information from such complex data structures. For PLS1-R modelling the relationship between \mathbf{X} matrix and \mathbf{y} (vector), which could be linear or non-linear are in focus. The \mathbf{X} matrix contains the descriptor variables and the \mathbf{y} vector is the response variable. In process monitoring/PAT cases the \mathbf{X} data matrix may also include data from many measurement approaches e.g. acoustic signals, spectroscopic measurements, pressure data, temperature, flow data, chromatographic data whilst \mathbf{y} contains reference values of the parameter of interest. In PLS-R \mathbf{y} information is used directly in the decomposition of the \mathbf{X} data in order to derived optimal lower dimensional data spaces that are usually also more interpretable. PLS-R involves non-iterative bilinear modelling by NIPALS algorithm (**N**on-linear **I**terative **P**ARTIAL **L**east **S**quares), which need not be described further here; readers are referred to dedicated chemometric literature for more details on principle and the theory of PLS-R [8-11]. Applications of PLS-R modelling span numerous fields such as science, technology, engineering and industrial process monitoring and control [10,11].

An obvious requirement for successful application of PLS-R is that representative calibration data, and test data, is a must in the modelling stage. The test data (test set) are utilised for model validation, both to obtain the optimal number of PLS-R components and to estimate the pertinent RMSEP (Root Mean Square Error of Prediction) of the prediction model. This will prevent both over-fitting and under-fitting of the prediction model. By over-fitting, more PLS-components than necessary are used for modelling and this leads to modelling of the noise in the data. In contrast, under-fitted models are deficient in information since the whole information contained in the analysed data is not extracted during the modelling step. In this regard, it is pertinent to apply the proper validation approach. There are several validation techniques in multivariate calibration available in the chemometric literature [9-11], but criteria for selection and optimality are scarce, or many times absent. Recently however, test-set validation has been pointed to as constituting the universally best validation method [12], and this is followed here. Esbensen & Geladi outline the in-depth analysis and argumentation behind this resolution [12].

In order to determine the predictive capability or goodness of PLS-R prediction models, the associated diagnostic plots and statistical results are evaluated. These plots include both visualisation plots, such as the **X** loading weight plot (X-variable correlations and influence of the components); only **X**-variables with loadings significantly different from zero influence the prediction model. Complementary the y-validation variance plot provides information about the optimal number of PLS-R components in the model and furthers the data for calculation of RMSEP. The T-U plots show the latent, “inner” relationships between **X** and **y**; this type of plot illustrates well observations or samples that deviate so significantly from the regression line, that they may be considered outliers [11]. Diagnostic tools for multivariate calibration/prediction abound, readers interested in this topic are referred to the details in the literature [9-12].

The equation presented below gives an insight on how the RMSEP is calculated during PLS regression application.

$$RMSEP = \sqrt{\frac{\sum_{i=1}^n (y_{i,predicted} - y_{i,reference})^2}{n}}$$

where the calculated RMSEP has the same unit as **y**, n is the number of reference observations/samples contained in the independent test set data.

Data pre-processing are widely used in multivariate calibration. In general, some of the commonly applied pre-processing techniques include auto-scaling (mean-centring and variance scaling), moving average smoothing, variable selection, etc. In this work however, details of the numerous pre-processing tools used in multivariate data analysis will not be presented but the following literature is recommended for more information on this topic [9,11].

2.3 Experimental set up

All experiments were carried out on a semi-industrial pneumatic conveying rig situated at the Department of Powder Science and Technology, Tel-Tek in Porsgrunn, Norway. A schematic overview of the conveying rig is shown in figure 1. The experimental rig consists of approximately 30 meters of 3-inch steel pipeline with a wall thickness of 3mm. The material is filled from the receiving tank into the feeding tank, where a vibrating mechanism supports the flow of material into a rotary feeder. By adjusting the feed rate of the rotary feeder, the material flow into the pipeline can be controlled. In combination with introduced compressed air, the particulate material can be transported with a desired mass flow rate. The material flow direction is marked with an arrow in figure 1. The two different sensor positions – at a pipeline bend and after the feeder - are marked with “X” in figure 1 and are shown in more

detail in figure 2. The left-hand picture shows the sensor position as a top view of the pipeline bend, mounted on the outer sidewall of the 90-degree bending. The right-hand picture depicts the sensor location on the feeder, glued on to the sidewall of the feeder outlet. The dashed boxes show where the determination of the “nominal reference values” (y_{nominal}) and “representative reference values” ($y_{\text{representative}}$) originate, as used in the second part of this research. The nominal plastic pellet concentrations are calculated by adding the required concentration level to the matrix test material, based on the weight of test material, which is measured by a weighing system located under the receiving tank. These concentration levels are referred to as the “nominal concentration levels” (y_{nominal}) below. It is important to note that in particular the imprecision of the weighing system, on which the spiking concentration percentage has been based on, increases the uncertainty on the actual nominal concentration levels.

The “representative reference values”, referred to as $y_{\text{representative}}$ are based on representative samples extracted by an experimental sampling device (“EF-sampler”), which has been installed at the marked position in figure 1. The EF-sampler is a newly developed sampling device for suspended particulate materials in horizontal pneumatic conveying systems, designed in maximum possible compliance with the Theory of Sampling (TOS) [13]. The EF-sampler has recently been validated in terms of accuracy, precision and technical functionality at the same test facility the presented experiments were performed on. Based on the comprehensive approach for representative sampling – the Theory of Sampling (TOS) - the successful validation of this sampling device documents truly representative samples, which are used in the following as the “representative reference values” for plastic pellet concentrations. In order to secure a solid, representative reference value, a composite sample consisting of 75 increments was extracted for each test round (compare table 3). For an introduction to the Theory of Sampling the interested reader is referred to the following selected literature [14-19].

Figure 1: Schematic drawing of pneumatic conveying system, POSTEC (modified by the authors).

Both selected sensor positions favour particles hitting the pipe wall on which the sensor is attached, significantly increasing signal strength. For the sensor position on the bend the redirection of the material stream achieves this requirement. The acoustic signal acquired from the sensor on the feeder outlet is strengthened by the gradual constriction of the pipe diameter (see figure 2). Readers who are interested in the general background regarding physical vibration phenomena of the pipes caused by the transported material, which are picked up by the sensors, are referred to a study by Kupyna 2008 [25].

Figure 2: Acoustic sensor deployment on bend (left side) and on feeder outlet (ride side).

In the first part of the experimental test campaign wheat flour has been conveyed, focussing on the comparison of the depicted sensor locations with two different flow rates. Subsequently the test campaign was repeated but now transporting alumina, confirming earlier results. Characterising values for density, particle size distribution, as well as amount of both conveyed materials used are stated in table 1, which also includes the characteristics of the spiking material. The spiking material was added to each of the transported materials in order to span realistic levels of extraneous concentrations. It was decided to use LDPE plastic pellets because this material has a density intermediate between the two test materials, while its particle size is substantially larger w.r.t. to both test materials. The LDPE concentration levels in wheat flour were set to 0%, 2% and 5%, serving as a feasibility study for the subsequent test campaign on alumina where the concentration levels were set to 0%, 2%, 5%, 8% and 11%.

Table 1: Physical characteristics of test materials

| Material | Density | Particle size distribution | | | Amount of material |
|-----------------|-----------------------|----------------------------|--------|---------|--------------------------------------|
| | | D10 | D50 | D90 | |
| Wheat flour | 0.46g/cm ³ | 13.3µm | 66.5µm | 161.3µm | 240kg |
| Alumina | 1.25g/cm ³ | 35.7µm | 85.7µm | 134.5µm | 150kg |
| Plastic pellets | 0.58g/cm ³ | ~3mm | | | Depending on simulated concentration |

The conveying mass/air ratios are listed in table 2 with the corresponding mass- and airflow rates of wheat flour and alumina - both transported in a dilute flow regime. The airflow was varied between 750Nm³/h (Flow rate 1) and 950Nm³/h (Flow rate 2) with a constant feeding rate for both scenarios and test materials.

Table 2: Tested mass/air ratios of wheat flour and alumina

| Material | Naming of flow rate | Mass flow (kg/s) | Airflow (Nm ³ /h) | Mass-air-ratio |
|-------------|---------------------|------------------|------------------------------|----------------------|
| Wheat flour | FR 1 | 0.30 | 750 | $\frac{1.2}{1}$ |
| | FR 2 | 0.20 | 950 | $\sim \frac{0.6}{1}$ |
| Alumina | FR 1 | 0.50 | 750 | $\frac{2}{1}$ |
| | FR 2 | 0.350 | 950 | $\sim \frac{1}{1}$ |

Each test scenario [factors: test materials (2), spiking concentrations (3 or 5) and flow rate (2)] was repeated in order to produce an independent calibration and test set [11], resulting in 12 test rounds for wheat flour (3 concentration levels * 2 flow rates * 2 transportation rounds) and 20 for alumina (5 concentration levels * 2 flow rates * 2 transportation rounds).

The acoustic signatures from both sensor locations involved individual recording of 150 replicate signal spectra from each sensor per test round for wheat flour and 60 replicate spectra (for each sensor location) per test round for alumina. By applying reduced averages, the 150 wheat flour replicate spectra were reduced by a factor of 15, giving 10 spectra per concentration level for calibration purposes, while the alumina spectra were reduced by a factor of 6, also resulting in 10 spectra per concentration level. The smaller number of spectra for alumina was due to a smaller total amount conveyed therefore a faster transportation time. Table 3 gives a full listing of all test scenarios for alumina. The test scenarios for wheat flour are equivalent, however using only three spiking concentrations.

Table 3: List of test scenarios including all test parameters

| Test scenario* | Test material | Spiking concentration (%) 'nominal concentration levels' | Flow rate | Repetition |
|----------------|---------------|---|-----------|------------|
| A_0_FR1_1 | Alumina | 0 | 1 | 1 |
| A_0_FR1_2 | | | | 2 |
| A_0_FR2_1 | | | 2 | 1 |
| A_0_FR2_2 | | | | 2 |
| A_2_FR1_1 | | 2 | 1 | 1 |
| A_2_FR1_2 | | | | 2 |
| A_2_FR2_1 | | | 2 | 1 |
| A_2_FR2_2 | | | | 2 |
| A_5_FR1_1 | | 5 | 1 | 1 |
| A_5_FR1_2 | | | | 2 |
| A_5_FR2_1 | | | 2 | 1 |
| A_5_FR2_2 | | | | 2 |
| A_8_FR1_1 | | 8 | 1 | 1 |
| A_8_FR1_2 | | | | 2 |
| A_8_FR2_1 | | | 2 | 1 |
| A_8_FR2_2 | | | | 2 |
| A_11_FR1_1 | | 11 | 1 | 1 |
| A_11_FR1_2 | | | | 2 |
| A_11_FR2_1 | | | 2 | 1 |
| A_11_FR2_2 | | | | 2 |

* W=wheat flour, A= Alumina; FR1, FR2 = see table 2; _1, _2 = repetition round used as calibration (_1) and test set (_2)

The acoustic signal acquisition system included accelerometers/sensors (Brüel & Kjær® 4518-002), which are glued on the marked positions as show in figure 1 and 2, being in direct contact with the piping system. The specific data acquisition conditions include a sampling rate of 300kHz and an averaging of 100 acoustic

spectra into one spectrum_{avg}. A Blackman- Harris window (weighting) transformation is applied to minimise spectral leakage during the subsequent FFT step [7], for which each Fast Fourier transformed acoustic spectrum is a result of 100 averaged individual acoustic spectra. The time averaged over for each spectrum is approx. 3 seconds. The frequency range covered by each spectrum is 0–150kHz.

3. Analysis and results

In the following section selected results will be shown from part 1: comparison of sensor locations and airflow rates, and part 2: the effect of representative reference values ($y_{representative}$) on prediction results in contrast to nominal reference values ($y_{nominal}$).

3.1 Part 1: Comparison of sensor locations with different airflow rates

The wheat flour feasibility study shows a clear tendency favouring the higher airflow rate (FR2) for both sensor locations, using the prediction model as basis for comparison (see table 4). All prediction results are validated using an independent test set, acquired by the second experimental round, repeating the entire experiment.

Comparing the bend vs. feeder sensor locations, prediction results are slightly better if based on acoustic signals acquired at the feeder location. The following tabulation shows RMSEP for all four scenarios (two flow rates, two sensor locations):

Table 4: RMSEPs for wheat flour, FR1 vs. FR2, bend vs. feeder.

| RMSEP | FR1 | FR2 |
|-----------------|------|------|
| Bend location | 1.17 | 0.67 |
| Feeder location | 0.81 | 0.45 |

The feasibility study allows to conclude that plastic pellet concentration in wheat flour can best be predicted if based on a higher airflow rate (FR2) with the sensor located on the feeder outlet.

Analysis of two typical (averaged) spectra, selected from the recordings during transportation of alumina with flow rate 1 and flow rate 2 (nominal concentration of 11%, sensor location also at the feeder), confirms these wheat result. The upper spectrum in figure 3 represents the acoustic signal corresponding to material transportation with the higher flow rate FR2, showing that in most parts of the frequency range the signal is stronger compared to that for transportation with the lower flow rate FR1.

Figure 3: Spectra comparison of FR1 vs. FR2 for alumina with 11% spiking concentration, sensor location on feeder.

In figure 4 the two sensor locations (feeder vs. bend) are compared again pertaining to alumina, also here confirming the feasibility test results for optimal sensor location. The sensor located on the feeder (upper spectrum) displays in general higher amplitudes for the majority of the frequency range. Hence FR2 was chosen as fixed transportation speed, using the sensor located on the feeder outlet for the prediction models of plastic pellet concentration in alumina below.

Figure 4: Spectra comparison of feeder vs. bend location for alumina with 11% spiking concentration, flow rate 2.

3.2 Part 2: Nominal reference values vs. representative reference values

Figure 5 shows the validated chemometric prediction results based on the nominal plastic pellet concentration (y_{nominal}) in alumina under optimal flow rate and sensor deployment conditions (FR2, sensor location on feeder).

The X-loading weights from the model of the raw data, shown in the upper left part of figure 5, reveal that the higher frequency range carries a systematic, structured periodic noise in the range 90-150kHz. A comparison of spectra from initial transportation pre-tests revealed that this is caused by the vibrating mechanism located on the feeding tank for supporting the material flow into the feeder and piping systems respectively. These vibrations are transferred to the sensor located on the feeder outlet below the feeding tank by the support system for the rig, which acts as a waveguide. This part of the frequency spectrum is therefore corrupted and not needed for calibration. Removal of this frequency range, as shown in the upper right part of figure 5, improved the prediction model. Furthermore, the loading weight spectrum w_1 (upper right figure) reveals equal model contributions from frequencies distributed over the entire frequency range now in use, with w_2 (PLS component #2) and w_3 (PLS component #3) adding important information mainly in the lower frequency range (0-10kHz) and also in the high frequency range for w_2 (70-80kHz) respectively. Three outliers in the validation set were removed from the relevant TU-score plots, leading to a final model based on 3 PLS components, the characteristics of which are depicted in the validation Y-variance plot in figure 5. This test set validation shows that three PLS-components are significant. A model based on two or even one component(s) would lead to an under-fitted model, since important calibration information would be

left out. A thorough background of the need for universal application of test set validation in complex, noisy real-world systems (a.c. is a prime example) has been given by Esbensen & Geladi [12].

The prediction evaluation, successfully validated by the independent test set, shows a prediction error (RMSEP) of 0.92, with a regression line slope very close to 1.00 (0.98). Further pre-processing of the data did not further improve the model. For comparison purposes, we prefer to express this RMSEP in a relative % format, $RMSEP_{(rel)}$, w.r.t the average y-value = 17.7%.

Figure 5: Upper left: X-loading Weights of raw data; upper right: X-loading Weights without systematic noise; lower left: Residual Validation Variance; lower right: Predicted pellet concentration vs. nominal reference.

The depicted model predicts the concentration levels of the extraneous material with a quite satisfactory average accuracy (slope), but as the prediction error, $RMSEP_{(rel)}$ cannot be said to reach a similar status, a further study was performed analysing the effect of nominal reference values in contrast to representative reference values used as the response variable in the prediction models. The nominal concentration levels are only *assumed* to represent the actual concentration levels of the plastic pellets in the transported alumina, but with some reservation as outlined above. Inaccuracy of the weighing system is the likely main reason for possible deviation between the actual pellet concentration levels in the alumina and the nominal concentrations. The objective was therefore, to find to what degree the prediction performance of the prediction model could be improved by introducing more representative y-values.

For this evaluation, the newly developed representative sampling device ('EF-sampler') designed for horizontal pneumatic conveying systems has been installed at the position shown in figure 1 [13].

Table 5 depicts the alternative nominal and representative basis for calibration and validation, revealing minor, but significantly different concentration levels.

Table 5: Comparison of nominal vs. representative LDPE concentration levels.

| Nominal concentration level (y_nominal) | Representative concentration level – calibration set – (y_representative_cal) | Representative concentration level – test set – (y_representative_val) |
|--|---|--|
| 0% | 0% | 0% |
| 2% | 1.78% | 1.89% |
| 5% | 4.89% | 4.76% |
| 8% | 7.68% | 7.34% |
| 11% | 10.77% | 10.39% |

Again after removal of the noisy, higher frequency range interval as argued above, seven individual measurements in the validation set were excluded and the remaining spectra averaged as described. This prediction model, “representative y-values model”, is also based on 3 PLS components as shown in the residual variance plot in figure 6. Comparing the validation Y-variance plot based on $y_{\text{representative}}$ with the validation Y-variance plot based on y_{nominal} (figure 5) it transpires that the additional two PLS components are only correcting for deviations between the nominal and the real physical-present y-values ($y_{\text{representative}}$). This observation supports the reasoning for only using representative reference values (see also further below).

The predicted vs. measured plot in figure 6 shows the improvement of the RMSEP, from 0.92 (y_{nominal}) to 0.85 ($y_{\text{representative}}$) as well as a lower bias, decreasing from 0.23 to 0.10. The corresponding $\text{RMSEP}_{(\text{rel})}$ is lowered to 15.5%.

Figure 6: Upper left: X-loading Weights of raw data; upper right: X-loading Weights without systematic noise; lower left: Residual Validation Variance; lower right: Predicted pellet concentration vs. representative reference concentrations.

It can be concluded that the representative reference concentration indeed improves the prediction precision, albeit only marginally in the present case. Even though the difference between these two prediction models only has a minor quantitative impact on the prediction results in this example, it is generally so that nominal reference values may well lead to both a higher RMSEP and/or to a higher inaccuracy, for example due to uncontrolled process variations and even higher uncertainties in the determination of the reference value. These results also confirm the representativity of the EF-sampler, being able to secure representative material characteristics in pneumatic conveying systems. Furthermore, in real-life applications of such prediction models for which extraneous concentration levels are unknown and are therefore focus of interest, prediction models can only be based on representative reference sample, since no nominal concentration levels can be determined.

It is a general conclusion regarding acoustic chemometrics that while the *average* prediction accuracy over the entire calibration range is often good (slope close to 1.0), the associated prediction error (RMSEP) is not yet in a completely satisfactory state, as witnessed by a $\text{RMSEP}_{(\text{rel})}$ close to 15%. There are evidently a not yet fully realised a.c. regimen regarding even better sensor localisation and general flow conditions.

4. Conclusions

Comparison of sensor locations and different airflow rates revealed a locally optimised setup for on-line acoustic chemometrics prediction of extraneous

concentration levels in wheat flour and alumina transported in pneumatic conveying systems. In particular an increase of the airflow rate and herewith a lower mass-air ratio improved the prediction results of plastic pellet concentration levels in alumina.

The discussion of the difference between nominal reference values and representative reference values pointed out the risk of unknown uncertainties inherent in non-representative reference values, risking an inferior calibration model. Comparison of concentration levels based on analysis via a representative sampling device, with the more uncertain nominal concentration levels, showed that the LDPE concentration in the pneumatically transported material was universally lower than set for the corresponding nominal concentration levels. In this particular case this difference is most likely due to the inaccuracy of the weighing system located on the conveying rig, which served as the basis for calculating the required amount of plastic pellets to reach the nominal concentration levels.

Calibrating the model with $y_{\text{representative}}$ instead of y_{nominal} further improved the prediction model, reducing RMSEP and the offset (bias). While the present experimental rig test resulted in relative minor quantitative improvements only, representative reference samples, required for prediction models, are essential when nominal concentration levels cannot be determined or are unknown, which is usually the case in enclosed pneumatic conveying systems, the target for this study. One is always obliged to use only representative y reference levels in modelling and validation, especially in complex multivariate calibration settings.

Further research is planned which will shed light on whether the present a.c. approach also allows on-line prediction of the actual particle size fraction distributions of the conveyed material. This is a much tougher ambition level; the present feasibility study was initiated to indicate the likely prospects for such an endeavour. It can be concluded that a major critical success factor will be improvements in the a.c. prediction precision. There are hopeful indications from a wide swath of a.c. applications that this would appear to be within reach [1-6]. It is likely that sensor localisation will play a very important role. It is necessary to search extensively, and exhaustively, for optimal sensor localisations. The present feasibility study serves to point the way.

Full evaluation of the EF-sampler, a minor player in the present study, is given in a parallel study, but presented elsewhere [20].

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FIGURE 1

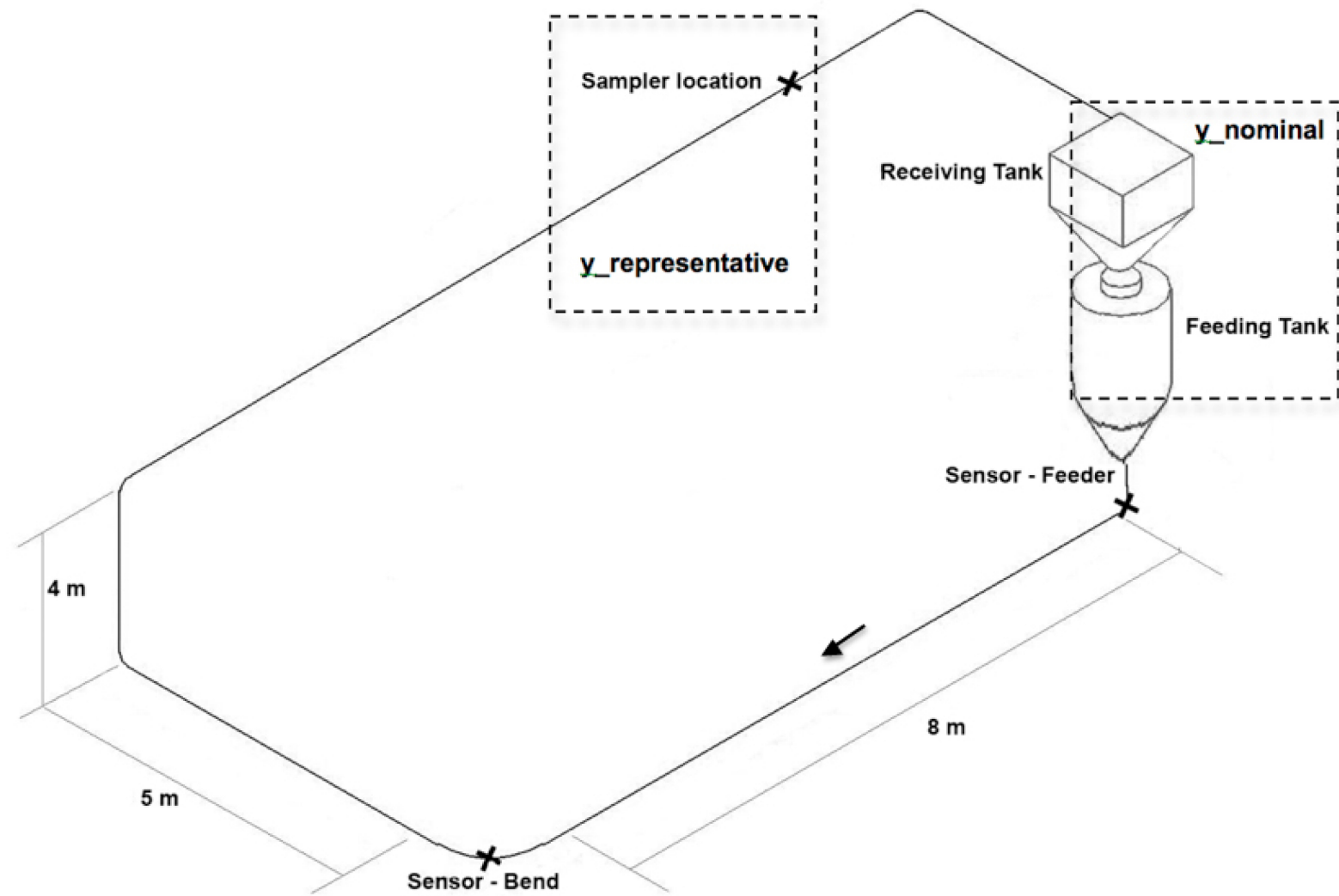


FIGURE 2

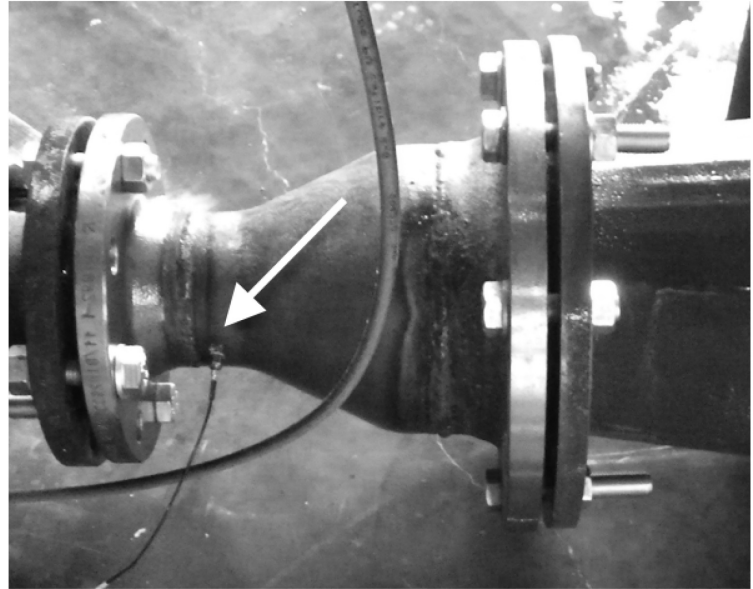
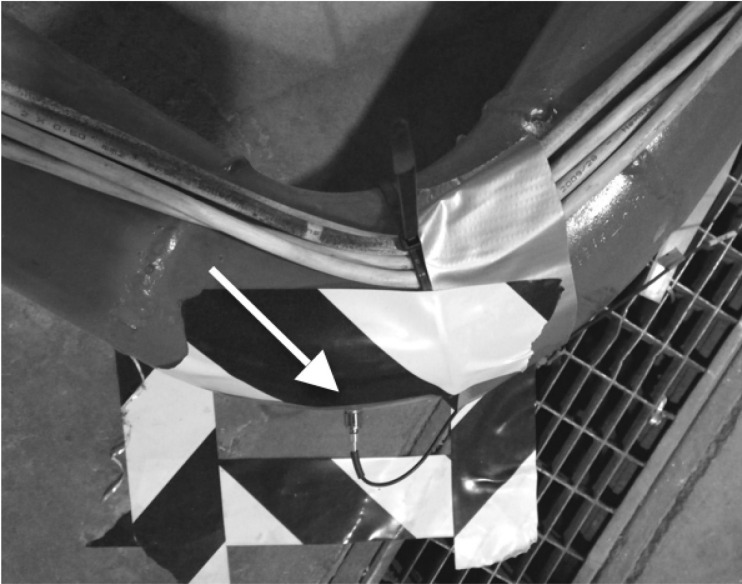


FIGURE 3

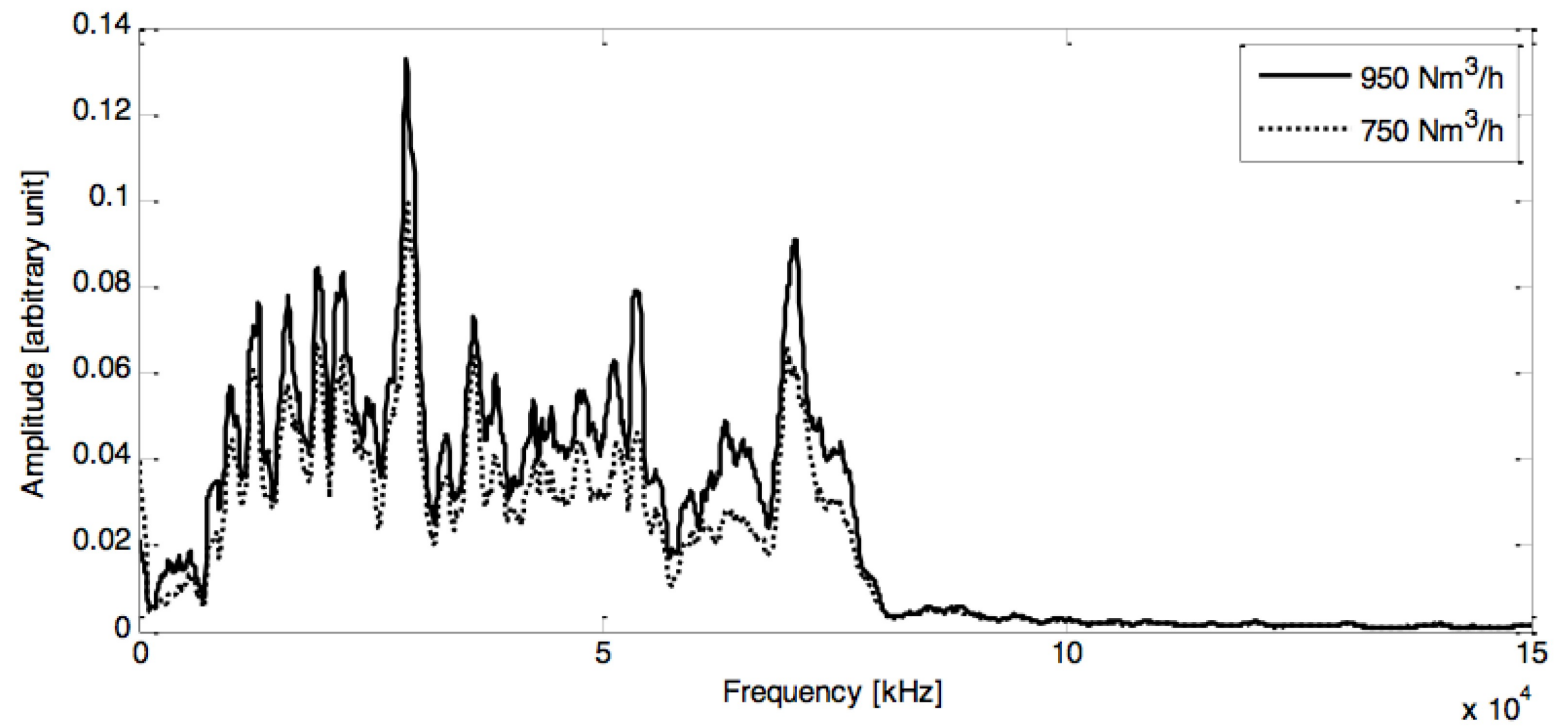


FIGURE 4

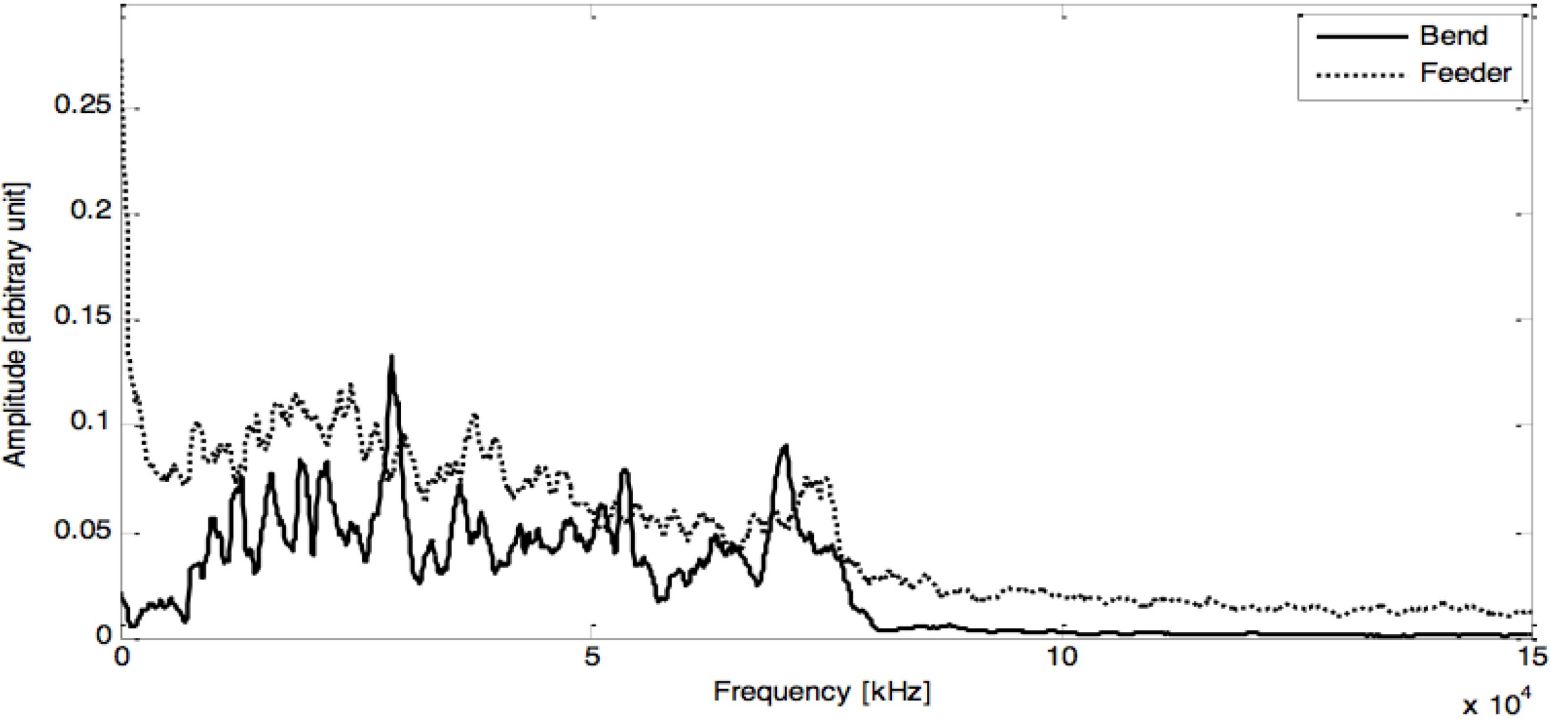


FIGURE 5

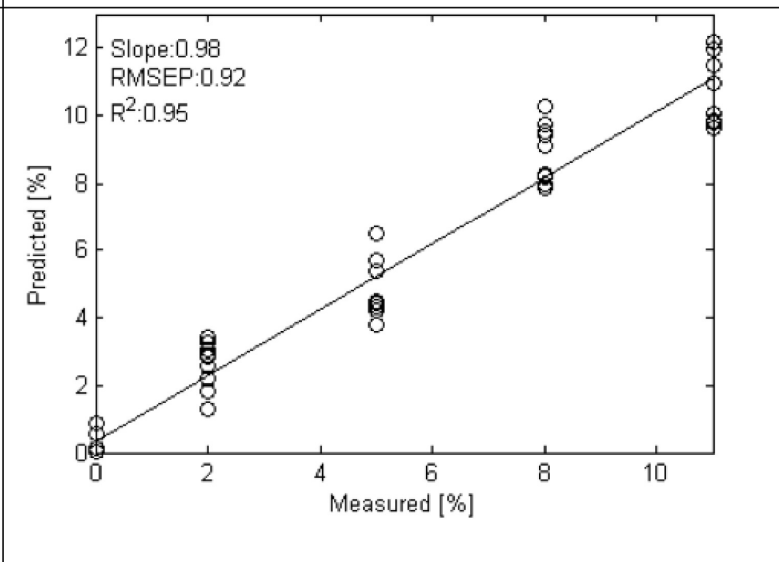
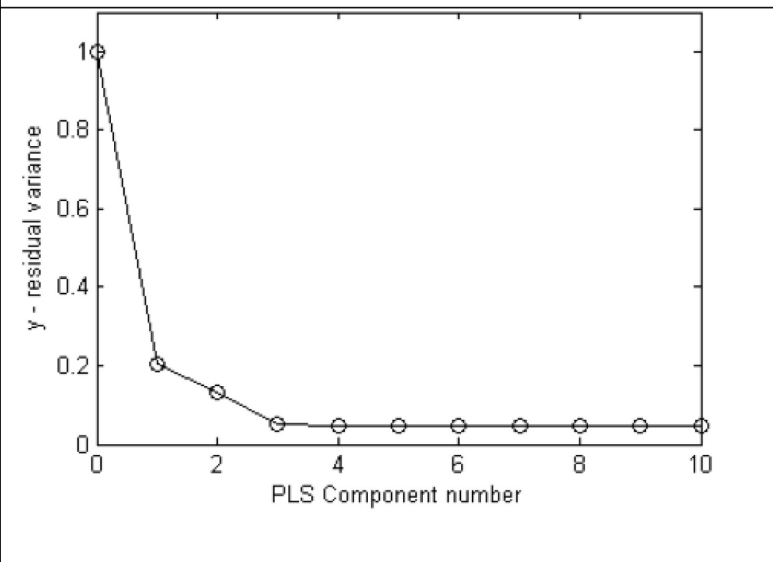
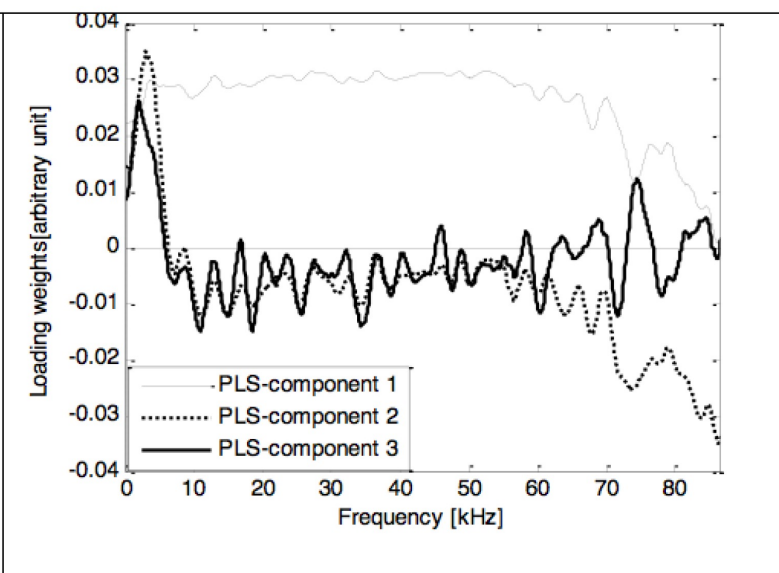
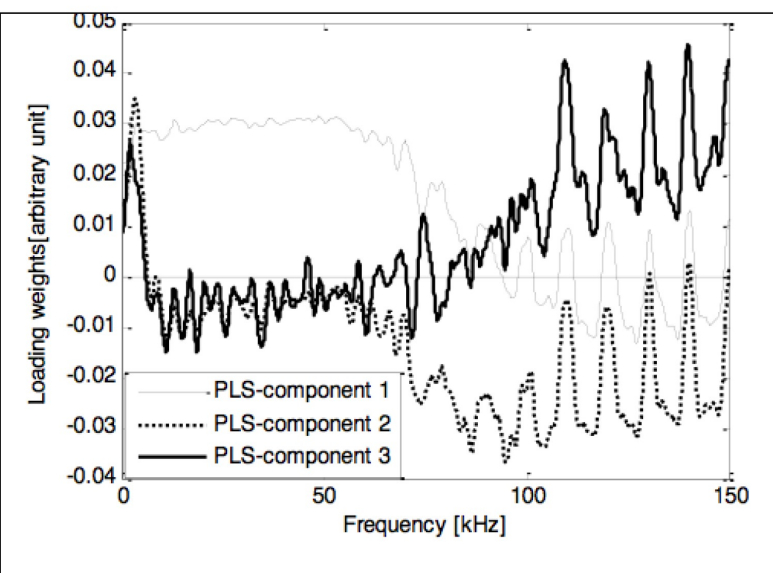


FIGURE 6

