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Key words: Silica gel, fluidised bed drying, acoustic chemometrics, Partial Least Squares (PLS-R), Process Analytical Technology (PAT), process monitoring

# **Acoustic chemometrics for on-line monitoring and end-point determination of fluidised bed drying**

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## Abstract

An emerging process analytical technology (PAT) technique, acoustic chemometrics, was applied for monitoring of heated fluidised bed drying process. The feasibility of quantitative on-line monitoring of the drying progress and end-point determination of silica gel dried in a heated fluidised bed was investigated. Silica gel was used owing to its high water adsorption property and stability to heat. Acoustic signals were acquired using four accelerometers mounted at different locations on the wall of the fluidized bed. The accelerometer located close to the base was the best based results from this study. Prediction models validated with independent acoustic data (test set validation) were developed using Partial Least Square Regression, PLS-R. Some data pre-processing techniques were applied to improve the developed prediction model. The final prediction results were satisfactory for monitoring of the drying progress and end point determination. The prediction results based on the independent data indicated a slope = 0.97, correlation coefficient,  $R^2 = 0.99$  and the root mean square error of prediction was 1.71 water %, within the range 0 – 35.69 water % of sampled reference. It was concluded that the results from this feasibility study shows that acoustic chemometrics is a viable on-line technique for monitoring the drying progress and for determining the end-point during drying of particulate matter. This on-line monitoring technique for the drying process developed in this study can be applied in many relevant industries in order to improve the overall economics of material drying by optimising fluidised bed drying technology using this decisive end-point determination approach.

Key words: Silica gel, fluidised bed drying, acoustic chemometrics, Partial Least Squares (PLS-R), Process Analytical Technology (PAT), process monitoring

## 1 Introduction

Fluidised bed technology is widely applied for drying of particulate matter in the industry. Because fluidized bed dryers offers efficient mixing, high heat and mass transfer rates, smooth drying operation, as well as continuous drying and handling of large quantities of materials. These are important properties in order to achieve fast, gentle and uniform particle drying. Due to the high drying rate and associated economic gain, fluidised bed drying has been proposed as the method of choice as compared to other drying techniques [1–6]. However, for many drying processes involving fluidisation technology, the analytical method applied for monitoring and control purposes is a critical factor regarding the assessment of the fluidised bed and even cost of monitoring drying processes in general. Reliable and low cost methods help to achieve consistent results and reduce the overall cost of drying processes.

In recent years, several Process Analytical Technology (PAT) techniques have been applied for moisture content determination and/or material drying in heated fluidized beds in research work as well as industrial installations [2-5, 7]. However, monitoring methods based on pressure measurements still remain as the conventional and most used monitoring techniques [8]. One of the drawbacks with pressure sensors is the intrusive deployment. Intrusive sensors are highly prone to blockage and thus it is possible to acquire pressure signals that are non-representative of the hydrodynamics in the bed being monitored [8]. Furthermore, among the PAT approaches adapted for fluidised bed monitoring, on-line techniques are preferred because of the non-invasive sensor probes. Therefore, there is still need for further research and adaptation of on-line measurement concepts for improved process monitoring and control. One of the emerging on-line non-invasive PAT approaches for process characterisation is acoustic chemometrics. However, adaptation of this technique and other acoustic methods for fluidised bed characterisation is still very low. This fact can be seen in a critical review on the various experimental measurement principles applied in fluidisation technology as presented by Omen and Mudde [8]. The experimental techniques according to the review included direct visual inspection during fluidisation, tomography, optical probe, capacitance probe, pressure measurements and acoustic measurements. They explained that no measurement technique could be regarded as ultimate for elucidation of all the properties of the gas-solid fluidised bed and that acoustic measurement techniques has the lowest number of applications as compared to the others. However, in a study by De Martin, et al. [9], the characteristic dynamics of acoustic signals were applied to monitor gas–solid fluidized beds for bulk and bubble behaviour. Statistical analyses (auto correlation and standard deviation) were utilised for analysing the time domain acoustic data. The authors stated that the results from the acoustic measurements and pressure data were similar. They concluded that this fact might thus instigate the use of acoustic sensor instead of pressure sensor for fluidised bed monitoring. Similarly, good results that are comparable to pressure measurement were obtained for acoustic measurement application for bubble behaviour measurement in fluidised bed [10]. From this point of view, the motivating factor for the current investigation was in tandem with the need for especially on-line acoustic measurement applications in particle processing, food and allied industries.

The present work is an attempt to adapt acoustic chemometrics for on-line fluidised bed drying progress monitoring and end-point determination using dedicated test material and PLS-R regression models validated with independent test data [11].

## **2 Materials and methods**

### **2.1 Acoustic chemometrics**

Acoustic chemometrics is an emerging non-invasive on-line PAT technique. Its application spans various research and industrial monitoring of systems generating sound (vibrations). The acoustic measurement concept is based on recording of passive acoustic emission from systems or processes contain information of which can be useful for their characterisation. In this regard, the calculated frequency spectra are thus related to the physical/chemical properties of the investigated materials/test system. In recent years, application of acoustic chemometric for various research and industrial process studies has increased [7,12-17, 24]. The advantages of acoustic chemometrics as compared to other on-line methods are numerous and some of them can be summarised as follows:

- On-line and real-time prediction
- Relatively low cost and no maintenance needed for the measurement unit
- Easy and non-intrusive sensor deployment
- No modification of existing systems needed
- Possibility of predicting several properties from the same acoustic spectrum
- Acoustic sensors can withstand high temperatures

Furthermore, acoustic chemometrics provides information about other parameters directly from the same acoustic spectra, e.g. information about critical failure (early warning) of which can be dead zones, clogging of the bottom plate etc. [24]. Therefore, acoustic chemometrics is considered viable, and to have some advantages compared to other available invasive measurement approaches. A widely used invasive approach is to correlate the residual moisture content against the change in gas temperature and humidity inside the fluidized bed. However, invasive sensors will not work properly in the presence of a lot of fine dust which will cover the temperature sensors resulting in erroneous temperature measurements. Invasive measurements are clearly much less problematic in the inlet/exit gas than in the bed itself.

The principles, theory and application of acoustic chemometrics in science and technology can be found in open literature [16]. Acoustic signals are sampled in time domain and conditioned to a more useful format as frequency spectra to allow multivariate regression modelling. The signal conditioning includes amplification to maximise the digital resolution, analogue to digital (A/D) conversion, window transformation to avoid spectral leakage, and fast Fourier transformation (FFT). A digital acquisition (DAQ) system from National Instruments is used for the A/D-conversion. Interested readers are referred to relevant open literatures to gain more insight on the signal pre-processing involved in the acoustic chemometric method [18, 19].

## 2.2 Partial Least Squares Regression

Partial least squares Regression is a well known method for calibration of multivariate prediction models. It is especially necessary for large data sets. This data modelling approach has been employed in many multivariate modelling applications in science, technology and industrial process monitoring and control [20, 21]. In PLS-R modelling, the relationship between two data matrices known as  $\mathbf{X}$  (independent variables) and  $\mathbf{y}$  (the dependent variable) is established.

The predictive ability of A PLS-R model and determination of the optimal number of PLS-R components are based on model validation. There are several validation techniques available [20-22]. However, test-set validation has been the recommended validation method because it provides realistic prediction errors and optimal number of PLS-R components [11]. In this regard, over-fitting or under-fitting of the prediction model is avoided. In over-fitted models also PLS-components containing noise is mistakenly included in the prediction models which will lead to non-representative predictions. In the under-fitted case informative PLS-component(s) are omitted in the model which means useful information is lost. Diagnostic results including the slope, off-set, correlation coefficient ( $R^2$ ) and root mean square error of prediction (RMSEP) are used for PLS-R prediction model evaluation. Resulting plots such as the  $\mathbf{X}$  loading weights plot, the  $\mathbf{y}$ -residual validation variance plot,  $\mathbf{t}$ - $\mathbf{u}$  plots, etc., are used for model interpretation and assessment. For instance, the  $\mathbf{X}$ -loading weights provide information concerning the important variables to be included in a prediction model [20, 21]. Equation (1) below is used for the calculation of RMSEP.

$$\text{RMSEP} = \sqrt{\frac{\sum_{i=1}^n (\hat{y}_i - y_i)^2}{n}} \quad (1)$$

The calculated RMSEP value has the same unit as the reference  $\mathbf{y}$ ,  $\hat{\mathbf{y}}$  is the predicted  $\mathbf{y}$ ,  $\mathbf{n}$  is the number of observations in the independent test set.

In multivariate calibration, the multivariate data is in most cases improved by using some data pre-treatment before multivariate calibration. For acoustic data, mean centring and scaling to unit variance pre-processing is commonly applied. Another pre-treatment method applied to acoustic data is averaging to eliminate or dampen the effects of the noisy variables. However, the applied pre-processing techniques depend on the acquired data set and this is usually judged by the prediction performance of the model (e.g. RMSEP), statistical results or diagnostic plots. There are several pre-processing methods and more details on the underlying principles are available in literatures [20, 21].

## 2.3 Test material

The test material used in the current study is silica gel known as *indicator gel*. This name is due to the fact that the silica gel during absorption of moisture changes from its original orange colour and becomes transparent. This colour changing feature of the indicator gel will

allow for visual inspection of the moisture content during drying. Orange gel is odourless, non-toxic, and non-corrosive, with stable chemical and thermal characteristics. Silica gel ( $SiO_2$ ) is a hard, very porous and crystalline substance (spherical or irregularly-shaped particles). In general, silica gel is the most commonly used desiccant, and is regarded as the highest humid adsorbent in desiccant community. With active, interconnected pores forming a vast surface area, silica gel is the most capable desiccant for dehumidification. The voids are about 50 – 70% by volume and can adsorb moisture 40% or up of its weight at 100% humidity environment. The size of the gel is 250-1800  $\mu\text{m}$  and the bulk density is 670Kg/m<sup>3</sup>. The maximum drying temperature for this product was 140°C. But to avoid extreme situation, 120°C was used in the current study. The driving force for using orange gel for this investigation is due to its thermal stability and ability to retain high degree of crystalline water that can only be removed by heating. This however, will allow for monitoring of material drying due to loss of water over a reasonable time period. Most of industrial drying processes have been designed to avoid harsh and destructive drying to keep the quality of products. Also, long time drying will make obtaining of enough acoustic data and reference for calibration and validation purpose possible, which in this case, is a prerequisite for drying progress monitoring using acoustic chemometrics.

### 3 Experimental set-up

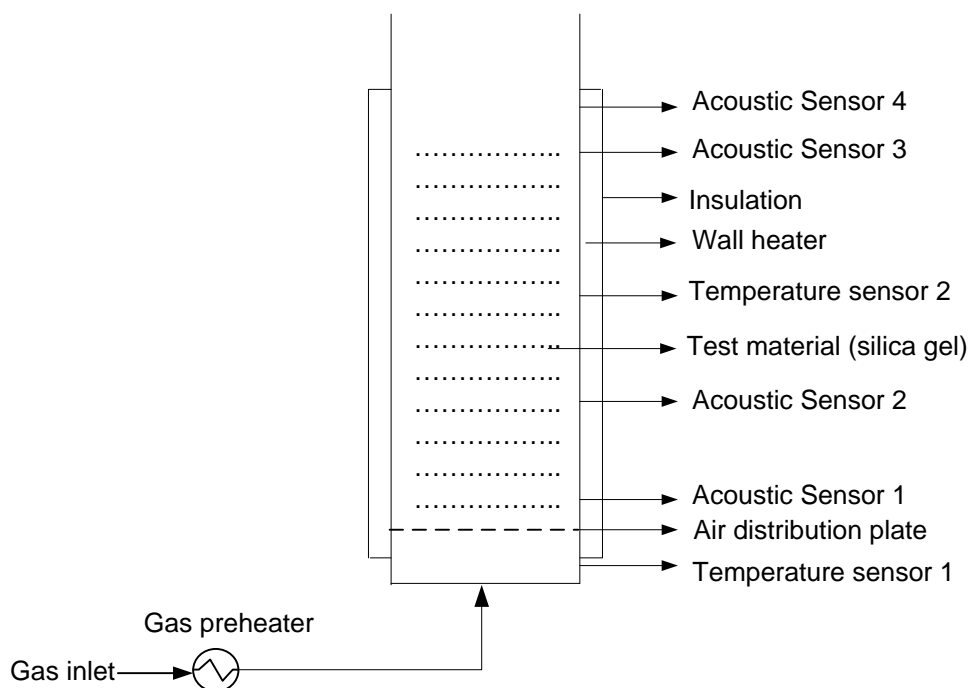


Figure 1. Schematic view of the test rig (hot fluidised bed) used for the drying of silica gel. Acoustic sensors were used for data acquisition for subsequent PLS-R modelling.

The experiments were conducted using a cylindrical pilot-scale fluidised bed in the powder research laboratory of POSTEC, Norway. A sketch of the fluidised bed including temperature transmitters and acoustic sensors are presented in figure 1. The picture in figure 2 shows the fluidised bed and the acoustic monitoring system used in the drying experiments. The



fluidised bed was constructed with stainless steel and the diameter and height of the bed were 162 mm and 1050 mm, respectively. A computer with National Instruments LabVIEW software was used to control the temperature and air flow rate in the fluidised bed during the experiment (see figure 2). Two temperature sensors were used to monitor the inlet air temperature and the temperature inside the fluidised bed. The air entering from the bottom of the drying bed was pre-heated by an electrical heater. Even hot air distribution at the column base was ensured by a uniform perforated air distribution plate made of stainless steel (see figure 1). In addition, a set of heating elements wrapped around the external wall of the bed contributed to the heating of the bed. In order to maintain uniform and stable air temperature, insulation material of about 5 cm thicknesses was used to cover the exterior wall of the bed and wrapped with aluminium foil (shown in figure 2). The temperature set point was always maintained at 120°C and controlled by means of a PID controller. The actual temperature of the test material however, varied from the initial temperature of about 19°C to approximately 92°C at the end of each test run (end point). The optimal airflow rate used for the present investigation was 14.6 Nm<sup>3</sup>/h. This flow rate was chosen to allow for initial sampling of the materials. Another factor taken into consideration when determining this optimal flow rate was avoiding spouting of materials from the bed as the drying progressed. This is because the weight of the material was always higher before drying (7.77 kg) due to high water content than when dried (5.60 kg). Using an airflow rate above the optimal would have led to loss of material from the bed over time. Loss of material during drying would have affected the acoustic measurement. For material sampling purpose, a cylindrical 500 mL container made from stainless steel and welded to 1 m long rod was constructed as a sample taker.

Four stainless steel rods (8 cm length and 1cm diameter) were welded onto the exterior wall of the bed (see figure 1). On each of the rods an accelerometer (Brüel & Kjær® 4518-002) was glued on to the end of each rod to avoid thermal damage of the sensors and cables. Four sensor locations along the bed height were used in order to capture the hydrodynamic profile of the bed. The considerations made while choosing sensors locations (see figure 1) were to place sensor 1 as low as possible to the base of the bed. Sensor 2 was located at the middle of the height covered by the material. Sensor 3 was placed exactly at the level of the material (height covered by the simulated material) whilst sensor 4 was placed 5 cm above sensor 3. The reason for locating sensor 4 above the height covered by the test material was to capture the acoustic signature of particle spouting and impacting the bed wall at the top of the bed. The sensors were connected to the acoustic signal conditioning system. These include an amplifier which was connected to a DAQ unit (USB-6361Series) and a computer with dedicated LabVIEW software (see figure 2). The time domain acoustic signals (window size of 4096) were sampled at a sampling rate of 300 kHz. The signal conditioning applied to the acoustic signals included amplification, analogue-to-digital conversion, filtering, Blackman Harris window transformation [18], Fast Fourier Transformation (FFT), and averaging. Each acoustic spectrum was calculated from a linear average of 500 FFT spectra and covered a frequency range 0 – 150 kHz.

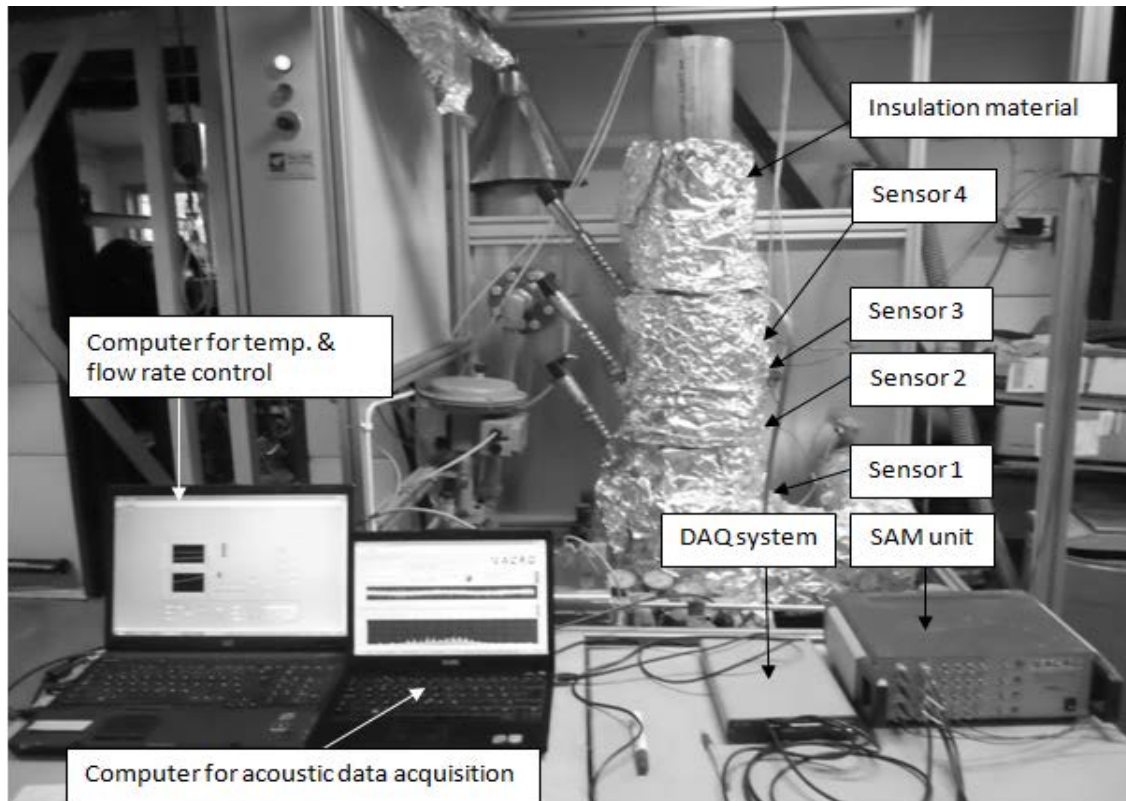


Figure 2. The experimental set-up for the hot fluidised bed drying experiment. The control and acoustic signal acquisition system can be seen in the lower part of the picture.

#### 4. Test Procedure

The test material used in this feasibility study was prepared by weighing 5.60 kg of orange gel. This quantity was required to get to the height of fluidization column, where sensor 3 was mounted. The material was soaked in water for 1 hr, drained and air-dried. On re-weighing the simulated test material, it was found that the weight was 7.77 kg. This implied that the water absorption was about 38.75% of its weight. The same sample preparation protocol was used to obtain another batch of test material used in a repeated drying experiment to obtain independent data for the test set validation of the PLS-R prediction model.

The drying experiment was started by filling in the bed with a fixed amount of silica gel to be dried. This was followed by furnishing the bed with the required conditions (temperature of 120°C and air flow rate of 14.6 Nm<sup>3</sup>/h). The bed was allowed to stabilise for about 10 min. Once optimal setting was achieved and the bed was fluidised, acoustic measurement was initiated by recording 10 replicate acoustic spectra. Then, three individual samples were taken from the fluidised bed using the cylindrical sample taker. The sampled material were weighed and poured back into the bed. The mass of the reference sample was used to calculate the moisture content in the silica gel. The moisture content was used as the reference ( $y$ ) in the modelling stage where PLS-R was used to calibrate a prediction model for the moisture content in the silica gel. The sampling procedure was carried out every 10 minutes. It was found that the variation in mass of these three reference samples collected at specific time intervals were maximum 3 gram. The average mass of each group of three samples were later

converted into water percentage (moisture content) this was used as the reference value ( $y$ -vector) in this work. The duration of each drying experiment was approximately 7 hours. By this time however all the particles of indicator silica gel turned from white to orange colour, which indicated the loss of their moisture content. After the experiment, the experimental systems were turned off and the bed was emptied using a suction vacuum system with a cyclone. The same experimental procedure were repeated another day to obtain independent test data for validation of the prediction model.

The acoustic spectra were calibrated against their respective water percentage ( $y$ -vector). The acoustic data, however, recorded during the sample extraction from the fluidised bed were deleted prior to PLS-R modelling. The acoustic signals were influenced by the introduction of the sampling container in the bed and therefore the acoustic spectra would not be representative of the drying material properties only. If the spectra from the sampling periods were included, they would have constituted gross outliers which cannot be used for modelling. Therefore, only PLS-R prediction results after removing the acoustic data recorded during material sampling) is presented.

It was necessary to apply some pre-processing to the data before the PLS-R modelling step. The initial pre-processing done before modelling were auto-scaling, reduced average of factor four on the variables; moving average using a rectangular window size which corresponds to 5.86 kHz. Subsequently, low pass filtering of the final model prediction output. The window size in the output filter was five predictions corresponding to 2 sec.

## **4 Results and discussion**

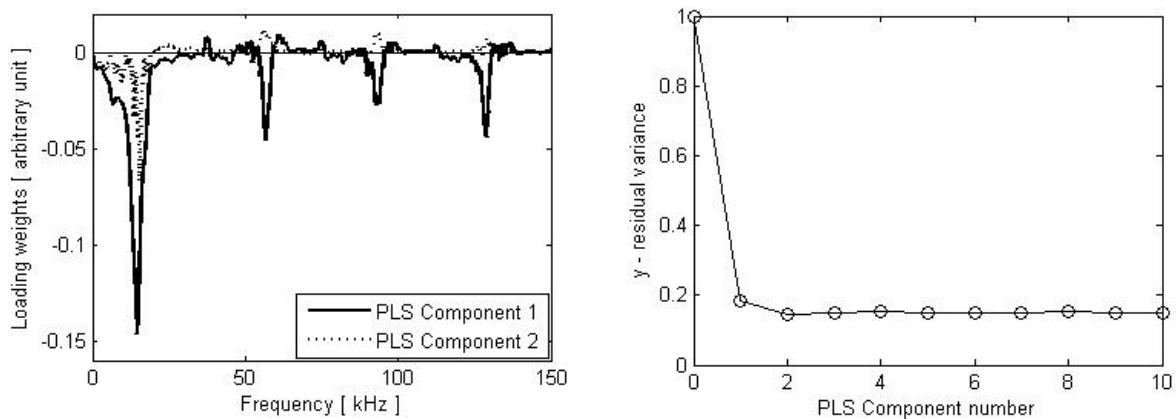
Drying of materials using hot fluidised bed technology has been of great interest and has been widely used for drying of materials in industries. This can be attributed to the accompanying high mixing rate as well as increased heat and mass transfer that promote fast and uniform loss of moisture content of the wet materials during drying process.

In the present study, drying process of water soaked silica gel was monitored over a considerably long time period by taking samples and weighing them. After some period of time, a point was reached when there was no change in mass of materials sample over time (end-point). At this time period, the colour of the silica gel turned completely back to orange colour (representing complete loss of moisture its content). The accompanying statistical results and diagnostic plots for interpreting the developed PLS-R prediction model for this drying progress monitoring of silica gel in a heated fluidised bed is presented.

### **4.1 Initial PLS-R modelling results**

In the initial PLS-R modelling, the entire acoustic signals for the four sensors were used. The prediction model was promising as can be seen in figure 2. From the residual  $y$ -validation variance plot (see figure3, lower left), two PLS-R components were required for optimal prediction of the  $y$  data. The loading weights for the 2-component PLS-R model for the 4

sensors can be seen in upper left of figure 3. It showed that the loading weight for sensor 1 was far much higher than that for the acoustic signals from sensors- 2, 3 and 4. Based on this, it was decided to use only sensor 1 for predicting the reference, as will be explained further below. The predicted verses measured scatter plot is presented in figure 3, lower left. The evaluative validation statistics comprise of a slope of 0.96, squared correlation coefficient ( $R^2$ ) of 0.94 and prediction error expressed as RMSEP was 5.08 water percentage (for the range of 0 – 35.69 water percentage of the sampled material). In addition, the predicted verses measure plot in time was also presented in figure 3, lower right. This was primarily to show the drying progress in time because; this is a drying progress monitoring study. Also, the time series plots are usually used for process elucidation in process industries. This is due to the fact that the application of this drying monitoring methodology in industries was the target for undertaking this study. As mentioned earlier, the represented predicted vs. measured plot in time does not include acoustic data during the sampling steps. Therefore, though the whole drying experiment lasted for about seven hours in each case (experiments for acquisition of calibration and test acoustic data), the total time frame judging from the plot in figure3, lower right is about five hours. This is because the acoustic data during the sampling steps were deleted because they would have constituted outliers. The important thing however, is that the acoustic data acquired represents the dynamic properties of the test system as regards the water percentage at every point in time. From the scatter plot and time plot for predicted vs. measured  $y$  it was observed that the data points were relatively scattered (for scattered plot) and noisy (for time plot), respectively. Therefore, it was concluded that applying a low pass (smoothing) filter to only the prediction was necessary. Hence, further pre-processing was applied to this initial model and the result is shown in figure 4 below.



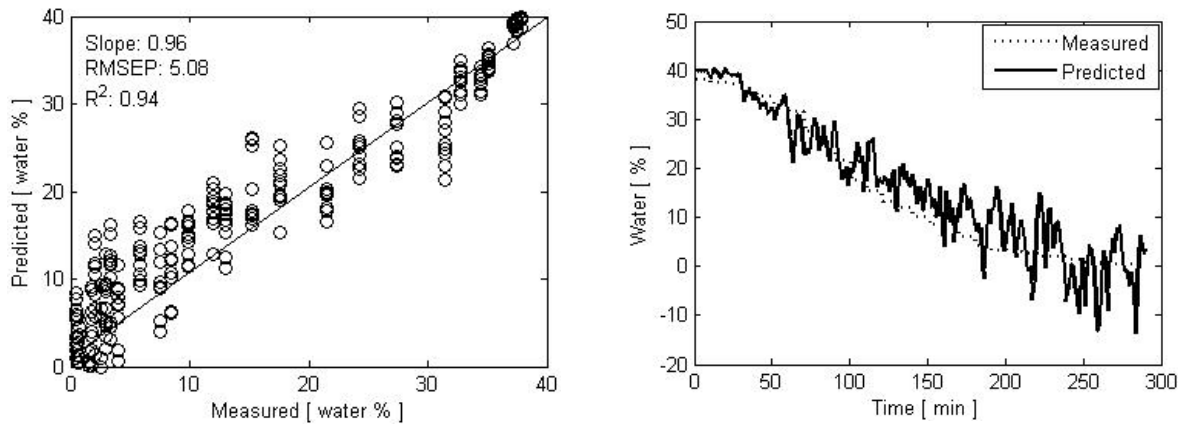


Figure 3. PLS-R prediction for all four sensors, no smoothing of the prediction output. Upper left: plot of the loading weights of two PLS-components used for modelling; upper right: residual y-validation variance plot; lower left: predicted vs. measured scatter plot; lower right: predicted vs. measured time plot.

## 4.2 Final PLS-R modelling results

As mentioned in the preceding sub-section, since sensor 1 was optimally (with highest loading weight) capable of modelling the acoustic data as compared to the other 3 sensors. Sensor 1 was thus used for further modelling of the acoustic data. Also, there was need for applying low pass filtering to the prediction output. The PLS-R modelling based on one sensor (sensor 1) is presented in figure 4. The loading weight plot is shown in figure 4, upper left and it can be seen that all the frequencies were important and included for the modelling though higher frequencies were close to zero. Using all the frequencies did not have any adverse effect on the model. The residual variance plot as shown in figure 4, upper right indicated that only two PLS-components were also needed (figure 4, upper right). The visual appearance of the plots was improved because they appeared less noisy for the time plot and less scattering for the scatter plot as compared to those in preceding figure 3. This can be seen in the depicted prediction verses measure scatter plot and prediction vs. measures time plot in figure4, lower left and figure4 and lower right, respectively. There was also improvement in the resulting prediction results slope,  $R^2$  and RMSEP were 0.97, 0.99 and 1.71, respectively, when the prediction output was pre-processed. The statistics of the final model can be seen in figure4, lower left (see also figure3, lower left for comparison). One can easily observe that there is dramatic reduction in the error of prediction (RMSP) and higher values for the slope and  $R^2$ . From the time plot of the predicted verses measured  $y$ , one could easily see that at the initial drying period, the loss of moisture from the material was low (clustered points in the scatter plot and almost stable line in the time plot). Since the water to be dried in this material was not just surface moisture but rather the moisture was entrapped in the crystal; this phenomenon can be explained on the basis that at the initial period, the material was absorbing the supplied heat. Then it was when the required temperature for the crystalline water to start evaporating was reached that water started escaping gradually from the materials by diffusion. Hence, as time went on there was noticeable increase in the loss of water represented as the loss in weight of the sampled material over time. Then the material started changing colour from transparent to faint orange until the period when it completely orange in colour (original colour) showing that its moisture content was completely lost. It

can also be seen from these scatter and time plots that the loss in weight of material were comparably low over some time period. This effect can be observed by the occurrence of more cluster points at the lower parts in the scatter plot and stable line in time plot of the predicted verses measured  $y$ . Therefore, the end-point for the drying of the material in the fluidised bed has been reached, which is a point of interest in any real life industrial scale drying operation. At this juncture further drying of the materials would not be required because virtually all its moisture content has been lost. If drying was continued, it might lead to over-heating of materials or destruction of the inherent crystalline structure of silica.

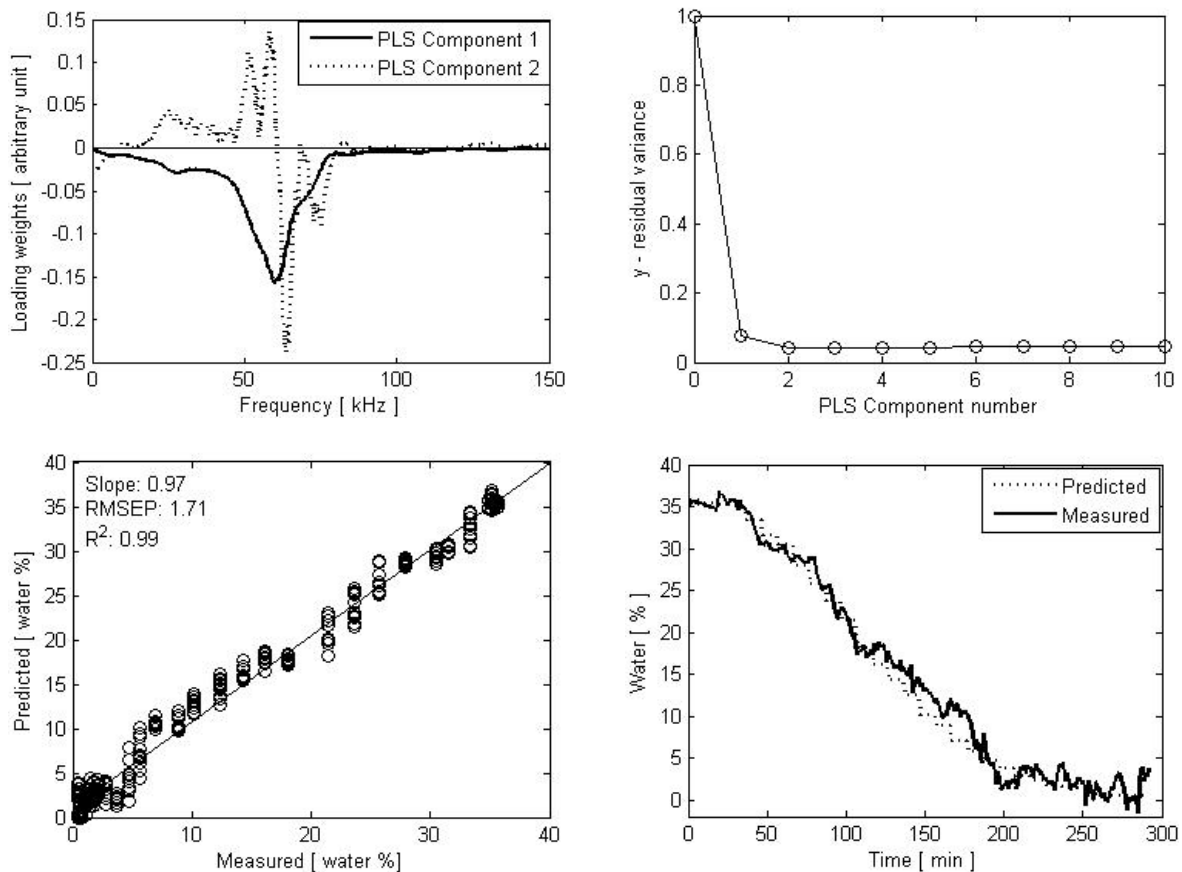


Figure 4. PLS-R prediction for sensor 1 only, with smoothing of the prediction output (rectangular window, size 4.39 kHz). Upper left: plot of the loading weights of two PLS-components used for modelling; upper right: residual  $y$ -validation variance plot; lower left: predicted vs. measured scatter plot; lower right: predicted vs. measured time plot.

From the foregoing, it can be concluded the data pre-processing applied during multivariate data analysis can be seen as a good practice herein since there were considerable improvement on the developed prediction model. The use of only one sensor for modelling was also reasonable since including the other 3 sensors introduced noise in the data. The  $y$ -residual validation variance for all components in figure 3 (upper right) is higher than that of figure 4 counterpart) result showed higher. There was reasonable change in the statistical results shown in the predicted vs. measured scatter plot, especially as regards the RMSEP value. Furthermore, application of low pass filtering to only the prediction output actually improved the visual interpretation of the prediction plots. The prediction models obtained in this investigation thus confirmed that application of acoustic chemometrics for continuous on-

line monitoring of drying progress and determining the end-point is feasible. Once a PLS-R model has been developed, it could be used for predicting the mass of materials being dried until the end-point is reached. Here, the hydrodynamic change (loss of the moisture content) of materials in hot fluidised bed was particularly captured by the acoustic sensors deployed on the wall its wall (see figure 1). Sensor 1, located close to the base of the bed, served the main objective for this present study. It was able to optimally capture the dynamics of the system based on the loss in moisture content with time. However, it was also interesting to compare the acoustic data from all the four acoustic sensors localised along the bed height.

In this study, predicting specifically the drying progress of the material and the determining the end-point was the focus. Silica gel saturated with water to create a real life situation where materials with some moisture content need to be dried in a hot fluidised bed. In some cases, a specific moisture content of the finished granules is nevertheless desired after fluidised bed drying operation in industries. This is the case in most food industries where food materials of various moisture contents are dried to certain required moisture content. Thus, utilisation of cost effective and stable method as studied in the present work will help in solving the issue of over-drying of materials since end-point assessment is possible. Once a representative acoustic data is acquire, reliable on-line and real-time prediction of drying process in industries can be achieved as well as end-point determination. These measures will allow for having the knowledge of the amount of moisture present at any point in time. In addition, consumption of large amount of power in industrial processes involving drying can be mitigated by employing this PAT measuring technique.

## **5 Conclusion**

Acoustic measurement and PLS-R analysis (acoustic chemometrics) were successfully applied, as a PAT method, in the present feasibility study for on-line monitoring of the drying progress and end-point determination of silica gel saturated with water. Out of the four accelerometers used for this investigation, only one sensor placed close to the base of the hot bed was able to capture the real change (loss of the moisture content) of materials. The satisfactory results from the test set-validated final PLS-R prediction model were an indication that this technique is reliable for on-line industrial process drying of particulate materials. The prediction error expressed as RMSEP was 1.71 water percentage (within the range of the 0 – 35.69 water percentage of sampled reference), the slope was 0.97 and the  $R^2$  was 0.99. As the drying progressed, it was possible to monitor the drying of the studied material using water content percentage as the reference.

In industries were drying of materials are employed such as in the particle processing industries, pharmaceutical and food industries, acoustic chemometrics can serve as a simple and viable on-line technique based on the present feasibility study. The overall economics of drying could be improved by using this and relatively cheap on-line PAT method. In addition, since end-point could be reliably predicted using this method, over- use of thermal energy will be circumvented. These will translate into improved drying process monitoring and reduced drying costs in industries.

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**List of figure captions:**

Figure 1. Schematic view of the test rig (hot fluidised bed) used for the drying of silica gel. Acoustic sensors were used for data acquisition for subsequent PLS-R modelling.

Figure 2. The experimental set-up for the hot fluidised bed drying experiment. The control and acoustic signal acquisition system can be seen in the lower part of the picture.

Figure 3. PLS-R prediction for all four sensors, no smoothing of the prediction output. Upper left: plot of the loading weights of two PLS-components used for modelling; upper right: residual y-validation variance plot; lower left: predicted vs. measured scatter plot; lower right: predicted vs. measured time plot.

Figure 4. PLS-R prediction for sensor 1 only, with smoothing of the prediction output (rectangular window, size 4.39 kHz). Upper left: plot of the loading weights of two PLS-components used for modelling; upper right: residual y-validation variance plot; lower left: predicted vs. measured scatter plot; lower right: predicted vs. measured time plot.