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Innovative software devices to monitor the primary drying phase of freeze-drying processes

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Monitoring of the primary drying phase can be very important for quality control and optimisation, and is extremely valuable in the development step.

Although the insertion of thin thermocouples in some of the vials is a widely used method to measure the product temperature during the operation, it is well known that this invasive procedure can not be considered representative of the whole batch and is not reliable and very useful.

Because of this, in recent years some innovative non-invasive software methods to monitor the lyophilization of pharmaceuticals have been developed; in particular two of these, developed by our group and recently patented, are presented here: the “smart vial” a low cost device based on a Kalman type observer which allows to follow the behaviour of several selected individual vials, and a new Dynamic Parameters Estimation method. These innovative tools have been tested in a pilot-scale freeze dryer (Lyobeta–Telstar), specially instrumented (Pirani and Baratron pressure sensors, humidity sensor and mass spectrometer for the measurement of the composition of the chamber atmosphere).

An observer, or soft-sensor, combines a priori knowledge about the physical system – the mathematical model – with some experimental data – the on-line measurements – to provide real-time estimation of states and/or process parameters. The variables and parameters considered for setting up the observer are the external temperature of the vial (measured), the position of the moving interface and the heat and mass transfer coefficients, respectively at the bottom of the vial and in the porous matrix (estimated by the observer).

The second technique investigated is the Dynamic Parameters Estimations (DPE), which consists of a non intrusive method that allows to estimate the average values of the state of the system (product temperature, frozen layer thickness, mass and heat transfer coefficients) measuring and interpreting the pressure rise which occurs when the valve between the drying chamber and the condenser is closed, throughout all the primary-drying. This approach is not new at all but the DPE method is based on a unsteady state model of the process. Moreover, this method takes into account also the dynamics of the product temperature during a DPE test and estimates the temperature profile in the whole vial. However the pressure rise methodology does not allow a continuous measure during the drying phase, and should be used with care when approaching the end of primary drying.

The possibility of using the informations coming from the observer and/or from the DPE tests, eventually taking into account their synergy, in a model-based control algorithm, having the goal of optimising the process, beside satisfying the operative constraints, is the current focus of our research work.