ABSTRACT

Batch processes are difficult to control, because the critical points that require a better control to achieve an adequate final quality are often unknown. Most of these processes are regulated automatically using the information recorded on line by means of electronic sensors, whose data are usually stored in databases. The statistical analysis of these databases can be useful to diagnose these processes, detect faults and obtain monitoring models aimed at maintaining or improving the final quality of the product.

Using the methodology proposed by Nomikos and MacGregor (1994a) for the multivariate statistical control of batch processes, based on Principal Component Analysis (PCA) and Partial Least Squares Regression (PLS), this thesis diagnoses the causes of variability of two quality parameters (the hydroxyl number and the residual water content) of an industrial process of PPOX (polypropylene oxide). The database analysed contains, for a set of 69 batches, the trajectories of 47 process variables registered on line. Since the duration of the process is not constant for all batches, it is necessary the application of several alignment techniques to obtain a three-dimensional data matrix (batches $\times$ process variables $\times$ time). Carrying out a PLS with this matrix, unfolded into a structure of batches by variables, it results a model able to predict the final quality. However, there is a strong interest to obtain a good predictive model using mainly those process variables correlated with the response variables with a cause-effect relationship. The identification of these variables implies the detection of the critical points of the process, what is not easy. For that purpose, a procedure for variable selection has been developed, that uses technical information of the process with a consistency analysis. Moreover, other advanced methods have been applied to check if the diagnosis can be improved. The Orthogonal Signal Correction technique (OSC) has not given good results. Nevertheless, this thesis has proposed a new hierarchical method based on PLS and PCR with consistency studies, that simplifies a lot the procedure of diagnosis.

The study carried out reveals that the main cause of variability of the hydroxyl number might be a fault in a flowmeter. Besides, several indications point at the final dehydration as the critical phase for the variation of the residual water content. Rectifying the faults detected and controlling the critical points, will produce as a result an improvement of the final quality of the product.

With the experience acquired in the research, a comprehensive methodology has been proposed for the diagnosis of batch chemical processes that comprises basically the following steps: definition of the problem, study of the quality parameters, analysis of the static variables, study of the technical information of the process, selection of the batches to analyse, alignment of the trajectories, exploratory analysis with PCA, identification of the latent variables correlated with the quality using PCR and PLS, application of hierarchical models, consistency analysis and finally validation of hypotheses with a design of experiments.