MICRO SPRAY DRYING AS A STRATEGY FOR OPTIMISING CLAY MINERALS CHARACTERISATION

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One of the most widely used technologies for characterising the mineral components in ceramic materials of a clayey nature is the X-ray diffraction (**XRD**) technique (Fig 1). At present, on an industrial level, in the ceramic sector there are no specific protocols for full speciation of the minerals in a ceramic product [1]. Optimum raw materials characterisation would enable a product's end properties to be more effectively predicted, on being able to quantify the minerals contained in the ceramic composition much more accurately. This requires optimising the sample test and preparation strategies used in quantifying clay minerals by XRD.

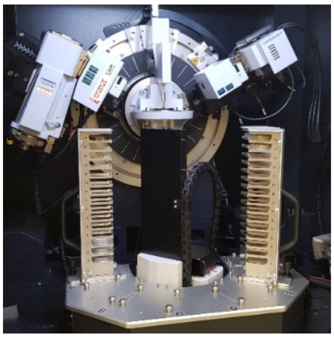

Fig. 1 *X-ray diffractometer*

One of the important constraints in quantifying crystal structures by XRD is the presence of **preferred orientations** of minerals [2], quite a recurrent problem in the ceramic industry as ceramic products basically exhibit a clayey nature and/or asymmetric structures. This phenomenon can give rise to significant errors in quantification when specialised quantification tools and software programs are applied, as stronger diffraction peaks generally appear than would correspond to them for the percentage in which they appear.

In light of the need to improve the quantification of crystalline species, a **micro spray-drying unit** of clay suspensions has been developed at ITC at laboratory level, with a view to eliminating undesired effects such as the presence of preferred orientations in the determination of crystal structures by X-ray diffraction.

Unlike traditional spray drying [3], the products dried by micro spray drying are able to generate spheres of 50–60 µm in diameter, which can be readily loaded in the XRD powder sample holder, providing a smooth surface with a relatively low and constant bulk density for the X-ray beam.

The unit developed at ITC improves other solutions [4]. It consists of an automatic sprayer, involving a comprehensive configuration of all the components integrated into a single work unit, a PID temperature controller for a working range between 25 ºC and 180 ºC, which allows use of different solvents and, in addition, it features a compressed air pressure regulator for controlling the final size of the generated agglomerates.

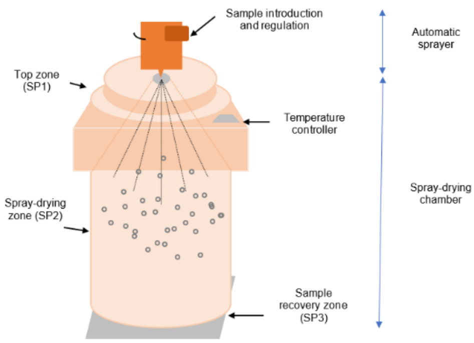

Figure 2 *Micro spray-drying unit developed at ITC*

Sample preparation with this unit effectively avoids generation of preferred orientations especially in micas, zircon, and feldspars such as albite. In addition, process efficiency ranges from 80–85% of the original product, which makes it extremely interesting for analysis of small quantities of sample.

Figure 3 shows the result on the effect of the preferred orientations in the diffractograms obtained in a clay sample after analysis as powder without spray drying and after analysis subsequent to a micro spray-drying process.

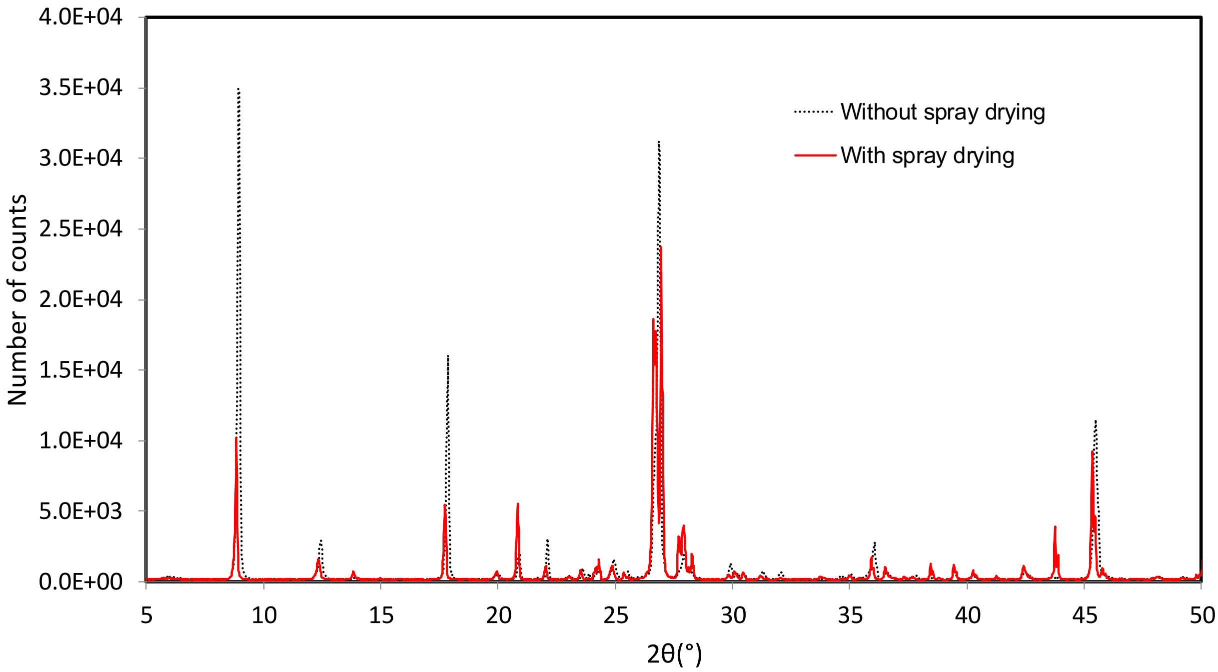

Figure 3 *Diffractogram of a clay sample without and with the spray-drying process in the unit developed at ITC*

As may be observed in this figure, treating the sample to a micro spray-drying process largely eliminated the preferred orientations, particularly of micas. Therefore, the uncertainty in quantifying crystal structures, owing to correction of the anomalous diffraction signal that is not related to a greater percentage of the crystalline phase but to a preferred orientation, decreases enormously, without having to resort to mathematical corrections in the quantification processes.

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