ON-LINE X-RAY DIFFRACTION FOR QUANTITATIVE PHASE ANALYSIS: AN INDUSTRIAL APPLICATION OF THE RIETVELD METHOD

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Material properties and plant operations are governed by mineralogy not chemistry. However, they are most often controlled in production by simple measurement of bulk chemistry. If used at all, mineralogy is derived from chemistry through normative calculation. Methods of this nature are fraught with inaccuracy due to the necessary assumptions regarding the exact chemical formulae of each of the component phases. One example is the Bouge method which is the industry standard for Portland cement production. Using X-ray diffraction (XRD), Portland cement remains a notoriously difficult material to quantify due to (i) the number of phases present, and hence degree of peak overlap, (ii) the variable composition of individual phases, and (iii) the presence of polymorphism. Typical cement plant control relies upon intermittent (say, four-hourly) X-ray fluorescence (XRF) analyses of bulk chemistry from which mineralogy is derived. These limitations in material characterization within the cement industry led to the design and construction of an on-line XRD instrument for direct minute-by-minute measurement of mineralogy. The complex nature of cement XRD patterns required that the instrument use the whole-pattern, Rietveld method for phase quantification rather than traditional single peak methods. The necessity for rapidity and stability of the analytical method required that the classical Rietveld approach be modified to a more restricted refinement strategy. Instruments incorporating these design criteria have now been installed in working cement plants. Preliminary results show that the rapid and direct measurement of mineralogy can be used in feed-back loops for plant control in real time and has the potential to be used in feed-forward loops for prediction of material properties.

Keywords: (ON LINE X-RAY DIFFRACTION) RIETVELD CEMENT

IMPROVING THE ACCURACY OF SIZE/MICRO STRAIN ESTIMATION BY FIRST PRINCIPLES-MONTE CARLO RAYTRACED FUNDAMENTAL PARAMETERS PROFILES

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In the Monte Carlo raytraced FPA (Fundamental Parameter Approach), the true slit dimensions, their distances and other geometric features are used as input parameters to follow the beam paths inside the diffractometer. In a first trial, this method was used for describing the profile (Rietveld reference material) as measured using different devices and X-ray tubes. The reason could be addressed to the emission profile of the commercial X-ray tubes: 5%...30% of the X-rays do not originate from a proper rectangular X-ray focus. The intensity coming from both sides of the focus was called 'tube-tails'. In a second step, the raytraced FPA has been corrected by measuring the focus intensity distribution and including this measurement in the Monte Carlo raytracing algorithm. Now, SRM 660 gave a small micro grain diameter (size) of 725(8) nm and a micro stress of 0.000075(4). For the new SRM 660a a zero micro strain and 1164(13) nm size have been determined. However, the Rietveld plot of SRM 660a deviated significantly from a clear random difference curve. Additionally, a dynamic extinction/absorption correction to the diffraction profile was introduced. Now, a micro strain of 0.000075(4) and an insignificant large size of 1(1+4+10) microns were calculated for SRM 660a. SRM 660 did not show a dynamic effect. The micro strains of SRM 660 and 660a are identical and may result from surface tension. SRM 660 has a clear micro size structure. SRM 660a seems to have a size value near to the macroscopic grain size.

Keywords: XRPD RIETVELD METHOD PROFILE ANALYSIS

AN ACCURATE MOLECULAR STRUCTURE DETERMINATION USING CONVENTIONAL X-RAY POWDER DIFFRACTION DATA OF SMALL ORGANIC MOLECULE AND POLYMER MIXTURE N. Jaiboon1 1 Department of Chemistry, Faculty of Science, Chulalongkorn University, Bangkok, Thailand, 10330 2 Department of Physics, Faculty of Science, Thammasart University, Pathumthani, Thailand, 1212 3 Metallurgy and Materials Science Research Institute, chulalongkorn University, Thailand, 10330

An accurate determination of crystal structure and atomic positions of a small organic drug molecule in a mixture of the drug and polymer using conventional X-ray powder diffraction data is demonstrated. Polymer is used to control or release pharmaceutical substances from controlled release drugs or sustained release drugs. Crystal structure determination of the pharmaceutical substances in a drug-polymer mixture is necessary, although difficult, since the presence of polymer or the preparation method might cause changes in physical properties of the pharmaceutical substances, e.g. morphology, polymorphism, or intermolecular interactions to the polymer. Diclofenac in the matrix of chitosan-diclofenac was studied. The X-ray powder diffraction data were extracted using the Le’ Bail method. The crystal structure was solved by direct methods from the extracted data and were refined using the Rietveld method. The results were compared with the crystal structural data obtained from single-crystal X-ray diffraction data. The differences in bond lengths are in the range of 0.30 Å and the differences in bond angles are in the range of 5.5°.