XRD Testing

JKTech offers a mineral identification and abundance quantification service based on the X-ray Diffraction (XRD) technology to the mining industry.

X-ray diffraction (XRD) is a mature x-ray technology that is widely used in the minerals industry for mineral identification and quantification. XRD uses the unique crystal structure and derived x-ray fingerprint of any crystalline material, e.g. mineral, for identification. XRD, unlike other mineral or bulk techniques such as SEM-EDS (MLA) or bulk assay (respectively), is unique in being able to unambiguously identify a mineral in a bulk or pure mineral sample, irrespective of the mineral type - sulphide or gangue, anhydrous or hydrated. While it can be used to provide gross solid solution mineral chemical changes (e.g. in carbonates, feldspars) the method is insensitive to minor or trace element changes in a mineral lattice.

Every crystalline substance is made up of an intrinsic regular arrangement of atoms in the form of its crystal lattice.

When the lattice is illuminated by a collimated beam of x-rays the lattice reflects the x-rays at angles (θ) specific to the distance spacing of the individual lattice planes (d-spacings, or "d") and the wavelength of the illuminating x-radiation (λ).

The Bragg Equation relates these parameters:

\[ n\lambda = 2d\sin\theta \]

A diffractogram showing peak intensities against position (2θ) for a simple three-component mineral mixture of quartz, calcite and pyrite is shown below (Figure 1). Each peak at a 2θ position corresponds to a reflection from an individual lattice plane for a given mineral. Computer-assisted searching of commercial and/or user-defined reference databases matches peaks to the minerals present.
Figure 1. Diffractogram showing reflection peaks for a quartz - calcite - pyrite system. The individual peaks for each mineral are colour coded (quartz - red, calcite - blue, pyrite - green).

**XRD data may be reported in three different modes:**

1) Qualitative/Semi-Quantitative identification, i.e. minerals present and estimation of major/minor/trace components;

2) Standardless quantification of minerals present to ±1 wt% (absolute) using the Rietveld 'full profile' method of analysis;

3) "Full" (single peak) Quantification, which is analogous to conventional XRF analysis and standards-based quantification.

Of these modes, the first two are most commonly employed particularly for mineral applications.

A typical Reitveld-refined quantification is presented below (Figure 2) showing matched peaks and estimated mineral abundances (in wt%).

Figure 2. Diffractogram and Rietveld quantification of minerals in a typical rock sample.

Mineral detectability, identification accuracy, and abundance quantification precision are dependent on a number of factors including the mineral complexity of the bulk sample, individual mineral crystallinities, and instrument analysis parameters. Detection of minor
Cluster analysis of raw XRD spectral scans allows the rapid visualisation (using PCA cubes and dendograms) and assessment of large suites of mine or process plant samples for potential mineralogical variation. Two examples are given. The first allows the overall mineralogical variation of the sample suite (>150 samples) to be assessed. The second example from a process plant shows time sequence samples and highlights the period when the plant failed due to a change in feed mineralogy. Potential future problems can be identified by XRD analysis of feed samples and rapid comparison of the raw data scan with historical data of 'good' and problematic mineralogy. No detailed interpretation is necessary.

Example 1 - Porphyry copper mine samples

The first example shows the dendogram (Figure 3) and Principal Component Analysis 'cube' (Figure 4, with the P1, P2 and P3 principal components as the X, Z and Y axes, respectively) of clustered XRD spectral data in a set of over 150 samples from a single porphyry copper system.
Figure 4. Principal Component Analysis (PCA) cube showing variations in P1, P2 and P3 principal components using cluster groupings shown in Figure 3.

After mineral identification and Rietveld quantification the PCA cube delineates the gross changes in sample mineralogy from relatively unaltered biotite-plagioclase feldspar-dominated rocks (grey-brown groups), through biotite-alkali feldspar-minor quartz rocks (green-light blue-purple groups) to highly altered muscovite-illite-quartz rocks (dark blue group). The red and yellow dots delineate the clay-dominated sample in the suite.

Example 2. Mineral processing plant operation samples

The sample suite consists of over fifty feed samples collected over an eight month period in a minerals processing plant. At some stage the plant experienced problems with the feed material and an assessment of the mineralogy was sought to a) define the problem and b) provide information to flag in advance potentially problematic samples.

Figure 5. Dendogram showing simple clustering of over 50 plant feed samples. The figure highlights the relative XRD spectral, and by inference mineralogical, differences of
the ‘problematic’ sample group (shown in red) compared to the remainder of the sample suite associated with normal plant operation.

Figure 6. PCA cube showing P1, P2 and P3 principal components in raw XRD data shown in Figure 5.

Cluster analysis of the raw XRD scans, that is, without any mineral identification or quantification, shows the samples received in a relatively short time window (red samples) that caused problems in plant operation. Subsequent identification and quantification defined the problem mineralogy, and provided the plant with information to assess future feed samples before they reached the plant.

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