Application Note AN-002

BOISE STATE UNIVERSITY

Quantitive Phase Analysis using XRD

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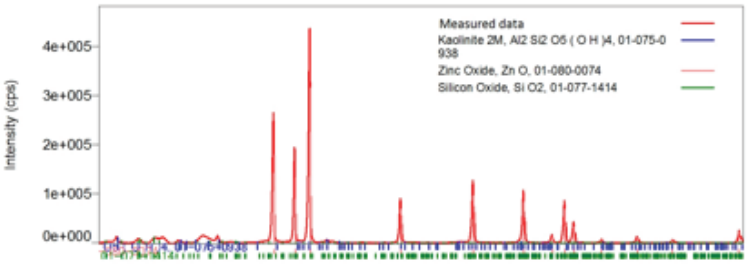
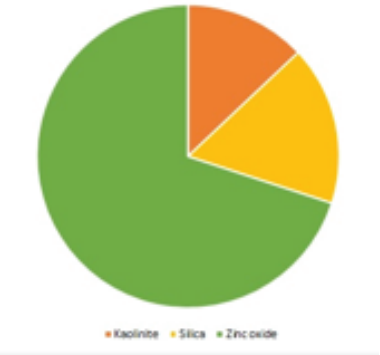
In this note, the quantitative phase analysis of a commercial medicated baby powder is presented. . X-ray diffraction (XRD) is a useful technique not only to identify the crystallographic phases present in a sample, but also to quantify the concentration of each constituent phase in case of multiphasic materials as encountered in ceramic processing, mining, medicine, etc. The XRD peak intensities of any given phase depend on many different parameters such as crystal structure, texture, concentration, etc. If the factors other than concentration are scaled to a standard reference, such as corundum, then a quantitative phase analysis of the sample can be carried out by using the ratios between the peak intensities of the constituent phases. The ratio of peak intensities from the pure sample of a phase to be quantified (I) and a known concentration of corundum (Ic) is a constant for any particular phase, and values of I/Ic can be experimentally derived or calculated from crystal structures.

Instrument: Rigaku Miniflex 600
Detector: D/teX 1D detector
Radiation : Cu Kα
2θ Scan range: 10° to 90°
Scan speed: 5° / min
Software: Rigaku PDXL



X-ray diffraction pattern of the baby powder.



Results of quantitative phase analysis using RIR method.

Calculated values can also be obtained directly from the ICDD powder diffraction files (PDF). This technique is known as the reference intensity ratio (RIR) method. The RIR method is much easier compared to full profile fitting techniques such as Rietveld refinement.

Our sample was analyzed using a Rigaku Miniflex 600 x-ray powder diffractometer using Cu Kα radiation. The resultant XRD pattern is shown in figure 1. Automatic phase identification was carried out with the help of ICDD PDF-2 powder diffraction file database.

Phase	I/Ic	Wt %
Zinc Oxide	5.44	69.9(3)
Kaolinite	1.07	13.4(15)
Silica	1.71	16.7 (15)

I/Ic values of the oxide components obtained from ICDD database and their measured concentration.

The results show that the powder consists of a mixture of three crystalline oxide phases, namely kaolinite ($Al_2Si_2O_5(OH)_4$), zinc oxide (ZnO) and silica (SiO_2); and all the peaks can be indexed to one of these three phases. Table 1 shows the I/Ic values for the component phases obtained from the PDF-2 database and their concentration present in the sample calculated using the RIR method. The quantitative phase analysis results are also presented as a pie chart shown in figure 2.

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