On-line Process Monitoring of Water Content in Powder Samples

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1. Introduction

Analysis of water content is very important since water affects the physical and chemical properties of raw, intermediate, and products in powder processes. There are many analytical method for measurement of water content such as infrared, microwave, neutron technique[1-4]. But it is not easy to control water content in powder process due to the variety of powder materials in industry. Therefore there are many kinds of analytical methods to analyze water content in powder.

In this study, X-ray method was used for online monitoring of water content in power samples. X-ray method was a useful online monitoring method for realtime measurement of water content because X-ray methed has a very short measuring time and good penetrating property [5].

2. Material and Methods

2.1 X-ray measurement system

X-ray generator consists of X-ray tube and its control box. In X-ray tube, anode target material is a tungsten. X-ray power is controlled in the range of 30W to 1kW. Irradiation area is controlled on the aperture of X-ray generator.

X-ray is detected by using Cadmium Telluride (CdTe) detector (Amptek, Model XR-100T-CdTe), which is a high performance X-ray and gamma ray detector, preamplifier, and cooler system using a $3 \times 3 \times 1$ mm Cadmium Telluride (CdTe) diode detector mounted on a two-stage thermoelectric cooler.

X-ray measurement system is a dynamic system for online monitoring of water content in sample. It has a rotating sample plate with width of 20cm, depth of 10cm and a diameter of 400cm. The rotation speed can be controlled in the range of 1.5 to 60 rpm.

2.2 Measurement of scattered X-ray

Activated carbon powder (ACP) was used for the measurement of water content. The preparation of the samples for the X-ray measurements was performed by adding a certain amount of water to the cylindrical plastic bottle containing 10 g of ACP. After sealing the

bottle tightly, the bottle was heated for 60 min at 100 °C and then was shacked for complete mixing. Mettler LP16 infrared moisture analyzer was used as a reference method to measure the water content before and after the X-ray measurement.

The samples with various water content were placed on the rotating plate of X-ray measurement system. Irradiation time of X-ray was one minute.

The counting time of X-ray were 0.1s. During the X-ray measurement, sample plate was rotated at 3rpm.

3. Results

There were two kinds of scattered X-ray signal detected in X-ray measurement system. One was a Rayleigh-scattered X-ray from the sample plate and the other was a Compton-scattered X-ray from the powder sample containing water.

Fig.1 shows the a representative X-ray spectrum for Rayleigh and Compton-scattered X-ray. Rayleigh-scattered X-ray signal was observed at the 100-200 channel number. Compton scattered X-ray signal was observed at the 300-1000 channel number.

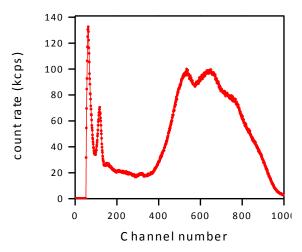


Fig.1 X-ray spectrum of activated carbon powder containing water.

On-line monitoring of water content in powder samples was performed under the dynamic condition. To observe the change of water content in the samples, the samples with various water content were placed on the rotating sample plate.

Fig.2 shows the result of on-line measurement of the water content in powder samples. X-ray measurement system monitored the change of water content in samples. The intensities of X-ray were increased as the water content increased.

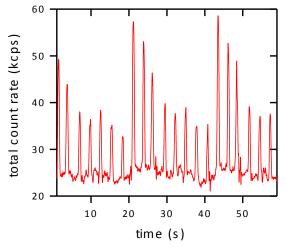


Fig.2 The result of on-line monitoring of water content in powder samples

4. Conclusions

The X-ray method introduced in this work are a useful online monitoring of water content in powder samples. Because X-ray measurements are possible within a hundred milliseconds, it is a promising technique for online process monitoring in the powder processes.

REFERENCES

[1] S.A. Margolis, P.H. Huang, Water Determination, in: A. Townshend, C.F. Poole, P.J. Worsfold (Eds.), Encyclopedia of Analytical Science, Elsevier Ltd., p. 357, 2005.

[2] R.L. Green, G. There, N.C. Pixley, A. Mateos, R.A. Reed, J.P. Higgins, In-line monitoring of moisture content in fluid bed dryers using near-IR spectroscopy with consideration of sampling effects on method accuracy, Anal. Chem. Vol.77, p. 4515, 2005.

[3] R. Wellock, A. D. Walmsley, Applications of microwave spectroscopy in process analysis, Spectroscopy Europe, Vol. 16, p. 23, 2004.

[4] R.R. Benke, K.J. Kearfott, Soil sample moisture content as a function of time during oven drying for gamma-ray spectroscopic measurements, Nucl. Instr. and Meth. A, Vol. 422, p.817, 1999.

[5] Y.S. Choi, J.-Y. Kim, S.-B. Yoon, K. Song and Y. J. Kim, Determination of water content in silica nanopowder using wavelength-dispesive X-ray fluorescence spectrometer, Microchemical Journal, Vol. 99, p.332, 2011.