Pelletizing of TiO_2 and other sticky material for XRF analysis

Abstract

Pelletizing of sticky and cohesive material for XRF analysis like, e.g., pigments or lime can cause various problems. Here we present a simple approach for manual and automatic sample preparation to overcome material-related difficulties and obtain good and stable pressed pellets with a smooth homogeneous surface.

Key words

• **XRF** • **Pressing** • **Mylar foil** • **Cohesive powders**

Introduction

Quick, precise and accurate results are one of the most important factors in industrial and scientific quality control and research. Usually, grinding and pelletizing followed by XRF are cheap and fast sample preparation methods. However, highly cohesive powders can cause serious problems while pelletizing.

One of those materials is TiO_2. Due to its special properties like the high refractive index it is a frequent ingredient in paints, plastics, cements, pharmaceuticals and many other products. When pressing fine-ground TiO_2, the pressed pellet might break apart when lowering the piston of the pressing tool. This is caused by the strong adhesion properties of the sample material (see Figure 1). Measuring in a liquid cell of the loose powder only gives poor and inaccurate results. Here we present a method how TiO2 and similar powders can be prepared successfully as a pressed pellet.

Manual presses

For pelletizing sticky materials it is recommended to use pressing into steel rings (40 mm) and avoid free-pressing or the usage of aluminum cups.

Step 1:

Dose enough Boreox or microcrystalline cellulose into the pressing tool (approximately 2 spatulas) to ensure that the TiO_2 does not get in contact with the piston.

Step 2:

Dose mixture of sample and binder on top of the bottom layer.

Step 3:

Cover the ring with a Mylar foil to avoid direct contact of the counter pressure plate and the sample material.

Step 4:

Pressing at medium forces (50 – 150 kN).

Figure 1: Miscellaneous sample preparation difficulties, when pressing strongly cohesive powders for XRF analysis:

A) Material sticking to the counter pressure plate
B) Sticking of material in the piston
C) Typical pitfalls in manual sample preparation are pressure marks by improperly applied Mylar foil (intensified contrast for visualization).

Automatic presses

The described method can also be partly applied in automatic presses like the HP-PA. Here, a roll of Mylar foil is used to avoid sticking and contaminating the counter pressing plate.

Cleaning of the pressing tool

The preparation of sticky material requires regular cleaning of the pressing tool. For cleaning, the pressing tool is disassembled. Subsequently, the piston and the matrix are cleaned individually. As cleaning agent, technical alcohols like isopropanol or similar should be used (do not apply methanol). Cleaning agents leaving any residuals are not recommended.

In order to dismantle the pressing tool, three bolts have to be removed. This can be done by pushing the pressing tool down and pulling out the bolts. After removing the bolts, the pressing tool can be disassembled and piston and matrix can be cleaned. Cleaning should be done using a lint-free towel. It is not advisable to use a scratch brush in order to remove sticking materials. After cleaning, the pressing tool can be reassembled.

Ring cleaning

To clean the analyzed steel rings HERZOG offers a combined multistep ring cleaning device (Figure 2) that can be mounted to the press (HTP 40), a wall or on a stand. Automatic presses like the HP-PA have a ring cleaning included.

Figure 2: Manual ring cleaning device.

Appendix - Cleaning Procedure for the pressing tool

Pressing tool cleaning procedure

A) Remove matrix
B) Disassembling
C) Cleaning of piston
D) Cleaning of matrix
E) Assembling

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