Impact of manual sample preparation on efficiency of grinding

Abstract

Sample preparation for XRF analysis is often done manually by crushing, splitting, pulverizing, and pelletizing. Here we show that the way in which the grinding vessel is loaded with sample material has a significant impact on the pulverizing efficiency. In our case study, the difference in the fraction < 45 µm was in the range of 15 %. This highlights that even minor and possibly involuntary variations in manual sample preparation have a major influence on the reproducibility of laboratory results.

Key words

• **Grinding** • **Pressing** • **Automation** • **XRF** • **Avoid errors**

Introduction

The chemical composition of granular materials like, e.g., ores, slags, clinker is often determined by means of x-ray fluorescence (XRF) spectroscopy. In preparation for analysis, the sample is usually crushed, split, pulverized and pelletized. Deviation from the standard sample preparation procedure may introduce a bias into the whole process and eventually increase the total analytical error.

In this application note, we are assessing the impact of sample loading into the grinding vessel on the outcome of the pulverizing process. We demonstrate that variation of material distribution within the grinding vessel significantly influences grinding efficiency.

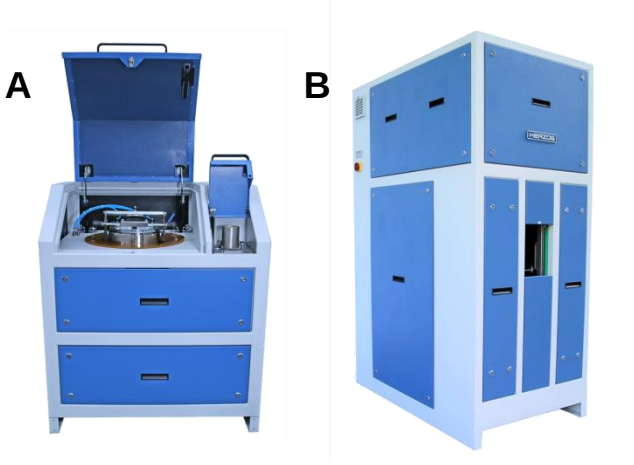

Figure 1: *(A) Manual pulverizer HP-M 500. The grinding vessel needs to filled, cleared and cleaned by the operator.*

(B) Pulverizing mill HP-M1500 with automatic material infeed, discharge and cleaning.

Method

As test samples, we used 250 g of quartz sand. Each sample originated from the same lot of raw material and was identical in terms of grain size distribution and moisture. Each trial was performed in the same manual pulverizing disc mill (model HP-M 500, Figure 1, A) using the same chrome steel grinding vessel (500 ccm) and preparation parameters (rotation speed 1000 rpm, grinding time 60 sec.).

We used three different ways to load the grinding vessel with the sample material (Figure 2):

In **case A**, the sample was filled in the gap between ring and the grinding vessel wall.

In **case B**, the material was poured in the space between stone and ring at the center of the grinding vessel.

In **case C**, the material was equally distributed among both gaps.

After completion of the grinding step, we determined the grain size distribution of each sample using a vibratory sieve shaker.

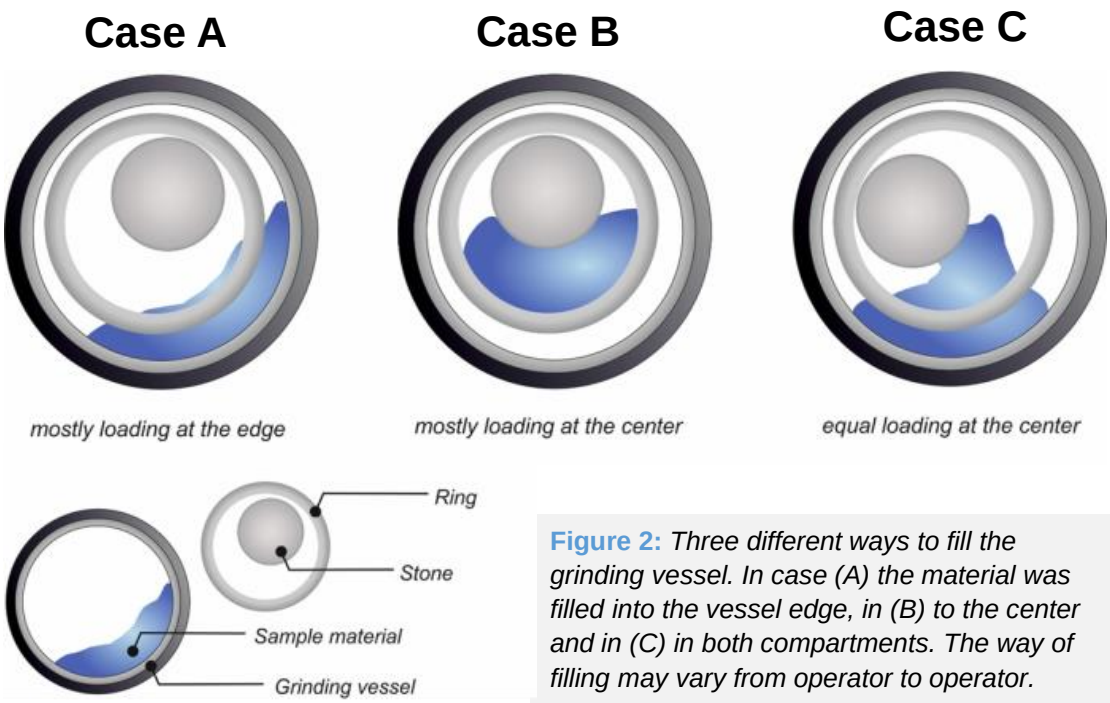

Figure 2: *Three different ways to fill the grinding vessel. In case (A) the material was filled into the vessel edge, in (B) to the center and in (C) in both compartments. The way of filling may vary from operator to operator.*

Results

In **case A** (sample loading in the edge of the vessel), the fraction < 45 µm was 69 %, whereas fractions <500 µm and <150 µm accounted for 14 % (Figure 3).

In **case B** (sample loading the center), the fraction <45 µm was larger with 79 % while fractions < 500 µm and <150 µm were reduced to 4 %.

Case C (equal distribution) led to less efficient grinding with 64 % <45 µm and 19 % < 500 µm and <150 µm, respectively.

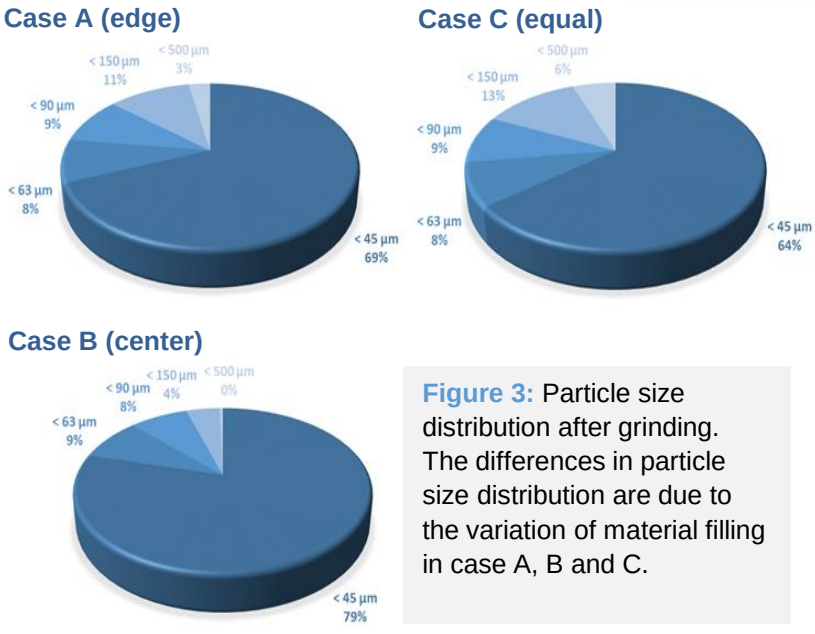

Figure 3: Particle size distribution after grinding. The differences in particle size distribution are due to the variation of material filling in case A, B and C.

Discussion

The differences in particle size distribution show clearly the impact of sample loading on grinding efficiency. Regarding the size fraction < 45 µm, we observed a difference of up to 15 % between all three cases. During a manual sample preparation process, different sample loading by different operators can cause varying outcome in grain size distribution. Discrepancies in grinding results can directly influence accuracy and precision of the XRF analysis particularly because instrument calibration is dependent from a specific particle size distribution.

Therefore, it is important to eliminate the operator's bias on sample preparation as far as possible. If fully manual methods are applied, laboratory management should provide a detailed instruction how each preparation step has to be performed by the staff. Random inspections are recommended to ensure the compliance of instruction.

Alternatively, automatic disc mills like the HP-M1500 (Figure 1, B) can be used. This machine has a mechanical loading mechanism using reproducible input settings and leads to significantly less fluctuations in particle size distribution. Finally, this guarantees a more even and constant sample preparation with higher precision and accuracy of analytical results.

Germany

HERZOG Maschinenfabrik
GmbH & Co.KG
Auf dem Gehren 1
49086 Osnabrück
Germany
Phone +49 541 93320
info@herzog-maschinenfabrik.de
www.herzog-maschinenfabrik.de

USA

HERZOG Automation Corp.
16600 Sprague Road, Suite 400
Cleveland, Ohio 44130
USA
Phone +1 440 891 9777
info@herzogautomation.com
www.herzogautomation.com

Japan

HERZOG Japan Co., Ltd.
3-7, Komagome 2-chome
Toshima-ku
Tokio 170-0003
Japan
Phone +81 3 5907 1771
info@herzog.co.jp
www.herzog.co.jp

China

HERZOG (Shanghai) Automation Equipment Co., Ltd.
Section A2, 2/F, Building 6
No. 473, West Fute 1st Road,
Waigaoqiao F.T.Z., Shagnhai,
200131
P.R.China
Phone +86 21 50375915
info@herzog-automation.com.cn
www.herzog-automation.com.cn