How to avoid errors in HPGR product characterization

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ABSTRACT

HPGR have been very successfully implemented in the mineral processing for grinding of various ores. Currently the worldwide interest in pellet feed preparation of iron ore concentrates (re-grinding) is rising as well. In addition to the known low specific energy consumption, high availabilities of the grinding system and less environmental load (no water consumption), the improvement of the pelletizing process is an additional bonus of the re-grinding HPGR.

It is known that the HPGR product can differ from ball mill product. One example is the possible formation of agglomerates (flakes) by the HPGR grinding process depending on the ore properties.

The above mentioned advantages of the HPGR can only be verified if the applied analytical and deagglomeration methods for the fineness analysis are comparable. With regard to the above mentioned possibility of agglomeration in the HPGR the fineness measurement methods need to pay attention to this effect. This paper details the need of careful sample preparation for iron ores in view of the results of laser particle size analysis, wet and dry screening as well as the measurement of the specific surface according to Blaine. Several test results will be used and the error impact on the industrial practice will be addressed.
INDRODUCTION

During recent years a worldwide trend towards comminution installations using High Pressure Grinding Rolls (HPGR) has been established (Klymowsky, 1998). This comminution practice was made famous in the 1980s by the research work of the German Professor Schönert. The technology had its foundations much deeper in the past (Tundermann & Singer, 1968) (Schönert, 1966). Köppern, as one supplier of HPGR, has worked for almost 120 years on roller presses, starting with the formation of coal briquettes between two rollers. Subsequently, other fields of application, e.g. for fertilizer compaction, emerged. The first HPGR grinding solutions were used in the cement industry where the advantage of energy savings was very welcome on this medium cost product (Klymowsky et al., 2002).

Nevertheless, it was a difficult step to transfer this grinding principle to minerals applications due to the much higher wear caused by the abrasive materials. One of the first applications started in iron ore concentrate grinding and were established for hard rock with improved wear lining of the roller surfaces. Examples can be found for several plants (Odenwald, Oliveira & Goulart, 2005) (Ehrentraut & Kudremukh, 2001). HPGR are available in a wide size range. Depending upon the required plant throughput, grinding tests are necessary to predict the operational behaviour of the machine for a certain material (Hees, 2005) (van der Meer & Maphosa, 2012) (Forsmo, 2007) (Kortmann & Kurghardt 1973). The most important parameters are the specific throughput and specific energy consumption which enable to size the HPGR for the required product fineness (Eisele & Kawatra, 2006) (van der Meer & Oenol, 2014) (Heinicke, Günter & Lieberwirth, 2016). The product fineness itself is defined by the transfer size to the downstream application such as flotation, magnetic separation or pelletizing and will vary with the ore characteristics (McNab et.al., 2009).

Due to the high applied pressure in the HPGR grinding process and depending on the ore characteristics it is possible to create agglomerates (flakes) in the HPGR product. One example for a gold ore is given in Figure 1.

![Figure 1 example of a flake made of gold ore](image)

Normally those flakes are easy to deagglomerate and will rupture during the transfer from HPGR to the following belt (destroyed by drop impact). Nevertheless it is questionable if small agglomerates could survive the transport regime and can find their also way to the laboratories. There they could affect the fineness analysis.
The above mentioned advantages of the HPGR can only be verified if the applied analytical and deagglomeration methods for the fineness analysis in the laboratories are comparable. The following chapters therefore detail the need of careful sample preparation for iron ores in view of the results of laser particle size analysis, wet and dry screening as well as the measurement of the specific surface according to Blaine.

**METHODOLOGY**

**Dry deagglomeration**

Both standard HPGR lab scale test or industry measurement deliver high amounts of samples. Those samples contain a certain moisture range. For iron ore concentrates the value of 0-12 % can be found on several worldwide installations with HPGR. Due to the high amount of sample of >100 kg the material has to be representably splitted. It is common to dry the material before analysis as well. During the drying process it is possible that the material creates additional packing on the drying plates. Those agglomerates have to be dissolved.

To dissolve the agglomerates of the HPGR for dry analytics a lab scaled ball mill was used (Figure 2). With a length of 80 mm and a diameter of 90 mm approximately 100 g iron ore concentrate can be deagglomerated. The deagglomeration speed is set to 80 rpm. To make the deagglomeration more efficient special rubber balls are used. Thereby no additional comminution and good deagglomeration is ensured.

![Figure 2 lab scaled deagglomeration ball mill](image)

**Laser diffractometer measurement**

The used device is a SYMPATEC Helos H2023 with selectively wet (SCELL) and dry (RODOS) dispersion unit (Figure 3). The technical information is presented in Table 1.

![Figure 3 Laser Diffractometer SYMPATEC Helos H2023](image)
Table 1 details for laser diffractometer measurement

<table>
<thead>
<tr>
<th></th>
<th>Sympatec GmbH</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Producer</strong></td>
<td></td>
</tr>
<tr>
<td><strong>Type</strong></td>
<td>HELOS H2023</td>
</tr>
<tr>
<td><strong>Dry dispersion unit</strong></td>
<td>RODOS 1 – 4 bar dispersion pressure</td>
</tr>
<tr>
<td><strong>Wet dispersion unit</strong></td>
<td>SUCELL 2 agitators; 60 watt ultrasonic bath</td>
</tr>
<tr>
<td><strong>Typical measuring fluids</strong></td>
<td>air, water, isopropanol, ethanol</td>
</tr>
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The dry dispersion is realized by the transport of particles in a pressurized airstream in two steps. In the first step, particle – particle and particle – wall contacts will break up the agglomerates. Furthermore in the second step an abruptly pressure decreasing at the transition from pressurized airstream through the nozzle into the measuring chamber will eliminate the rest of the agglomerates. This pressurized airstream can range from one through four bar.

In the wet dispersion unit, the breakage of the agglomerates is a combination of an ultrasonic bath with adjustable ultrasonic energy and agitation of the suspension with adjustable number of revolutions. The agitation makes sure that the suspension gets homogenized.

A big difference between wet and dry dispersion unit is the way to measure the particles. In dry dispersion the sample is measured one time with a higher necessary sample mass. In wet dispersion a circulation system is used which allows to lower the sample mass.

To make sure that the used laser is not blocked by too big particles a protection screening on 0.315mm is necessary.

**Air jet screen**

The air jet screening was done according to DIN 66165. The termination condition was set within one minute to 0.1 % of the mass change based on the feed mass. The screen cuts for these investigations are 20 μm, 32 μm, 45 μm, 63 μm, 100 μm and 200 μm.

The deagglomeration of flakes in the air jet screen is a combination of two effects. First, the contact between air jet and particle which causes a takeoff from the screen. Second, caused by the negative pressure, an acceleration towards the sieve and with this a contact between particle and screen surface.

For any test, the negative pressure was set constantly to 60 mbar. The used air jet screen is shown in Figure 4.

![Figure 4 air jet screen](image-url)
Wet screen

For wet screen analysis, a vibrating screen machine from Retsch type AS 200 was used (Figure 5). To ensure the comparability between wet screening and air jet screening, the same screen cuts as in air jet screening are used. The screen time was set to 20 minutes and the vibrating amplitude to 2 mm. The water flow set constant to 0.5 l/min.

![Vibrating screen machine from Retsch type AS 200](image)

Figure 5  vibrating screen machine from Retsch type AS 200

Blaine measurement

Any determination of the specific surface according to Blaine was done according to DIN 66126-2. All investigated products have been pre-screened at 0.315 mm. The undersize material was then tested in Blaine device. For some material with residues on 0.315 mm the Blaine value was corrected according to its residue mass. The time for each test was stopped with a stop watch. One Blaine value is based on three times of measuring one material with three times of measuring one sample.

As the Blaine device is not able to deagglomerate particles on its own physical measurement a separate deagglomeration with the above mentioned lab scaled ball mill is essential.

![Manually Blaine device](image)

Figure 6  Manually Blaine device

RESULTS AND DISCUSSION

Dry laser diffractometer measurement

Figure 7 shows the particle size analysis of dry laser diffractometer for various deagglomeration times with the lab scale ball mill. All curves are similar and do not vary. The same effect can be seen by varying the pressure of the dispersion unit RHODOS according to Figure 8. It is to be considered that the deagglomeration ability of the Rhodos unit is replacing any missing deagglomeration of the ball mill unit. Therefore it is not necessary to use additional deagglomeration before which saves lab time.
The reproducibility of the above mentioned laser diffractometer measurements was tested according to ISO 13320. A very small error could be found according Figure 9. This emphasizes that the dry laser diffractometer measurement is a good method for analysis of iron ore. While the time for one analysis is quite short with about five minutes only the purchase price is very high for a small industrial lab in comparison to other analysis methods. For comparing industrial results with lab scale test work it has to be remarked that different units might produce different results. This influence has to be acknowledged in industrial guarantee contracts.
Wet Laser diffractometer measurement

Figure 10 shows the particle size analysis of wet laser diffractometer for various deagglomeration times with the lab scale ball mill as well as measurements with and without ultrasonic deagglomeration supply of the SUCELL unit. Also an additional measurement with isopropanol is given to show the variance of several measurement fluids. The particle size curves for wet laser diffractometer measurements show higher variations. Also the reproducibility (ISO 13320) is out of the range (Figure 11).

![Figure 10](image1.png)

**Figure 10** differences in wet laser diffractometer measurement by use of ultrasonic and Isopropanol

![Figure 11](image2.png)

**Figure 11** accuracy of wet laser diffractometer measurement after 10 min deagglomeration

The error range of wet laser diffractometer measurement was measured with up to 10%. The client is normally aiming at a certain fines degree in the product. If a lower amount is measured the HGPR would have to be bigger in size depending on the installed flowsheet and the possible higher amount of recirculation load. A difference in 10% can easily result in an increase of 500,000 € in the CAPEX of a HPGR.

More variations have been found when measuring Magnetite samples in wet and dry ambience. The shapes of the curves are very different. It was considered that due to the magnetic valves in the SUCELL unit Magnetite particles might be astrayed. As the content of Magnetite and Hematite can vary depending on the mine conditions the wet diffractometer measurement might product non comparable results. Thereby the wet laser diffractometer measurement method was not found to be useful during this test series with iron ore concentrates.
Air jet screen measurement

Figure 13 and Figure 14 show the particle size analysis and reproducibility of an air jet screen for various deagglomeration times with the lab scale ball mill. With remarks to Figure 7 and Figure 9 the method can be classified as also producing representable results as the laser diffractometer measurements.
The challenge with this method is to be seen in the time consumption. Figure 15 shows the importance to act in accordance with the norm DIN 66165 in view to the screen time. The cumulated percent passing is varying for more than 15% from five to 40 minutes in analysis time. Thereby this method is not useful for high amounts of sample analysis. As different results of various machines are not known to the authors this method is useful for comparing industrial results with lab scale test work in relation with single guarantee values.

![Figure 15](image)

**Figure 15** influence of screening time in air jet sieving for 0.020mm screen cut

**Wet screen measurement**

Figure 16 shows the particle size analysis and reproducibility of a wet jet screen for various deagglomeration times as well as measurements with different fluids (water and isopropanol) and additional ultrasonic support. Again the reproducibility is very good (Figure 17). Even if the time consumption of up to four hours is remarkable higher than above mentioned analytical methods this option is good for an industrial application as it is commonly used on mine sides.

![Figure 16](image)

**Figure 16** differences in wet screen measurement for various deagglomeration times and different fluids and ultrasonic impact
Blaine measurement

Figure 18 shows the particle size analysis according to Blaine method for several material and various deagglomeration times. The method shows high differences in results of up to 400 cm²/g depending on the preparation method while the representability was very good for the single measurement. Thereby the exact method has to be fixed during contract negotiations if used for guarantee purposes. As the measurement device is commonly used in iron ore application it is still a good option to get an overview of industrial performance of HPGR in iron ore concentrate grinding.

CONCLUSIONS

This paper presents different results of laser diffractometer, air jet screen, wet screen as well as Blaine measurements. The particle size distribution results vary depending on the analysis device but also depending on the deagglomeration method. Differences of up to 15% or 400 cm²/g have been found with several materials. This might lead to an increase of the size of HPGR which has to be installed in industry application. Due to the nature of heavy duty machinery this will increase the OPEX of the machine.
There is no wrong measurement methodology nor is there the best method if compared to each other. So there is no instruction manual on how to avoid errors but it can be stated that the methods need to be depending on the production goals of the client. There is no systematic error which can be avoided but the method for analysis including deagglomeration procedure has to be discussed with the client. In view of evaluation of test work and industrial performance of HPGR the procedure has to be fixed before deviation between industry and test results are forming claim battles.

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