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EXPERIMENTAL INVESTIGATION OF THE EFFECT OF THE POWDER AND THE SUSPENSION RHEOLOGY ON GRINDABILITY

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Abstract: The grindability of raw materials is a key parameter of mineral processing. It is also very important for some kinds of secondary raw materials, such as demolition and construction wastes. The rheological behavior of the ground slurry or suspension evidently affects grinding because of the well-known viscous dampening effect. However, the grindability of materials as a function of the moisture content and rheology has not been studied on-line. Therefore, the grindability of a soft adhesive (limestone) and a hard non-adhesive (quartz) material was examined in the Universal Hardgrove Mill during room temperature dry and wet (in tap water) grinding. The powder flow behavior of the dry ground samples was measured in an FT4 powder rheometer, while the rheological properties of the wet ground suspensions were measured in a rotational rheometer.

Keywords: *grindability, Universal Hardgrove Mill, moisture content, grinding, quartz*

1. Introduction

The issue of the high energy demand of comminution is widely known in the literature and continuous research is ongoing in this field. On the other hand, the process engineering design of crushing and grinding units is typically carried out by using different kinds of grindability indices characterizing the given raw material. This is the reason why the fundamental research of grindability test methodology development and the development of the auxiliary complementary test methodology is so important. At the Institute of Raw Materials Preparation and Environmental Technology of the University of Miskolc, it has a long tradition, the Universal Hardgove Mill and the Universal Bond Mill had been developed by with grindability can be measured at so called universal circumstances, namely at high temperature and in any kind of (neutral, acidic, alkali) media (Csőke et al., 2003; Mucsi et al., 2006).

Deniz (Deniz, 2022a and 2022b) determined the effects of kinetic breakage parameters on calcites in a Hardgrove mill. Yang et al. (Yang et al., 2020) studied experimentally the effect of moisture on Shengli lignite breakage behavior and energy efficiency. They concluded that the energy-size reduction process for grinding lignite is markedly influenced by moisture occurrence and content. Removing surface moisture from 37.90% to 16.61% (the air-dried condition) resulted in a slight increase of input energy by 0.04 kWh.t−1 per 10 s. However, with further drying to inherent moisture to 0%, the consumed energy significantly increased by 0.16 kWh.t−1 per 10 s. Meanwhile, the mass fraction of the top size decreased from 42.29% to 24.73% and then to 13.00%, while the pulverized coal production increased sharply from 6.28% to 10.68% and to 23.64%, both at a grinding time of 6 min. The energy efficiency was also significantly improved as the moisture content of lignite was reduced to below the air-dried level. The air-dried moisture content was the inflecting point for lignite grinding in the Hardgrove mill. A two-stage pre-drying system was proposed accordingly. The experimental results of Vuthaluru et al. (Vuthaluru et al., 2003) suggest that no relationship exists between the coarse fraction moisture and Hardgrove index in the case of coal. Beyond these, lack of knowledge about the effect of the moisture content on grindability can be found in the literature in the case of quartz or limestone. The effect of the moisture content of coal (Vuthaluru et al., 2003; Yang et al., 2020) was measured.

Another current issue is the question of viscous dampening in comminution machines. Therefore, two model materials, - a soft and adhesive material: the limestone and a hard and non-adhesive material: the quartz - were selected. After the preparation of the taken samples the so-called dry (< 20 m/m% moisture content) and wet (> 50 m/m%) grindability tests were carried out in the Universal Hardgrove Mill. At this time auxiliary tests had also been carried out. Powder flow features were measured in a powder rheometer and suspension rheology was measured in a rotational rheometer.

2. Materials and methods

The limestone samples - with a particle density of 2680 kg/m^3 (measured by liquid pycnometer in water) were taken in Tornanádaska, Hungary. The quartz samples - with a particle density of 2631 kg/m^3 (measured by a liquid pycnometer in water) were taken in Alsózsolca, Hungary. The Hardgrove grindability index was measured by the earlier developed - but a newly built and improved - Universal

Hardgrove Mill (Figure 1 and 2). Parts of the Universal Hardgrove Mill are: 1. Grinding balls, 2. Shaped closing assembly for pushing down the balls, 3. Grinding crucible, 4. Crucible furnace: heat-insulating enclosure with three 230 V/500 W heating wires and three PT100 platinum thermal resistors, 5. Electric connection field for connecting the heating wires and thermal resistors, 6. Electric connection field for connecting the heating wire and thermal resistor, 7. Cover of crucible furnace: heat-insulating enclosure with one 230 V/500 W heating wire and one PT100 platinum thermal resistor, 8. Axially moving shaft with bearing, 9. Bearing, 10. Worm-gear drive, 11. Weight, 12. Asynchronous motor, 13. Lever, 14. Measurement electronics, 15. Measurement data acquisition and A/D card, measuring and controlling computer, measuring and controlling software, 16, Axial bearing, 17. Force measuring transducer.

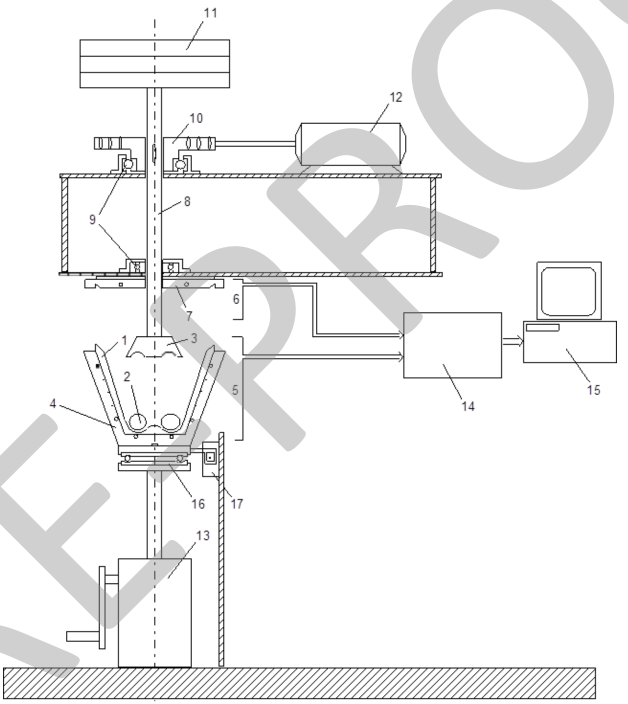

Figure 1
Schematics of the Universal Hardgrove mill

According to the standard Hardgrove protocol 50 g prepared sample, namely only the x = 590… 1190 μm particle size fraction is fed into the grinding chamber. There are eight 25.4 mm diameter balls in there. The vertical force on the grinding balls is set constant to be 290 N. The constant revolution number of the rotor is 20 1/min and a grinding lasts until 60 full revolutions. After grinding, the ground solids are removed and sieved in a Retsch sieving machine for 20 minutes using a 0.075 mm aperture size screen. The mass of the fine fraction (m_{75}) is measured in gram unit and the Hardgrove index is calculated by Equation 1. However, the

Hardgrove index (H) is widely used in the industry, this index characterises the specific energy need only indirectly. Csőke et.al. (2013) suggested a simple conversion equation (Equation 2) by with the Bond index ($W_{B,H}$) can be calculated from the Hardgrove index.

$$H = 16 + 6.93 \cdot m_{75} \quad (1)$$

$$W_{B,H} = \frac{468}{H^{0.82}} \quad (2)$$

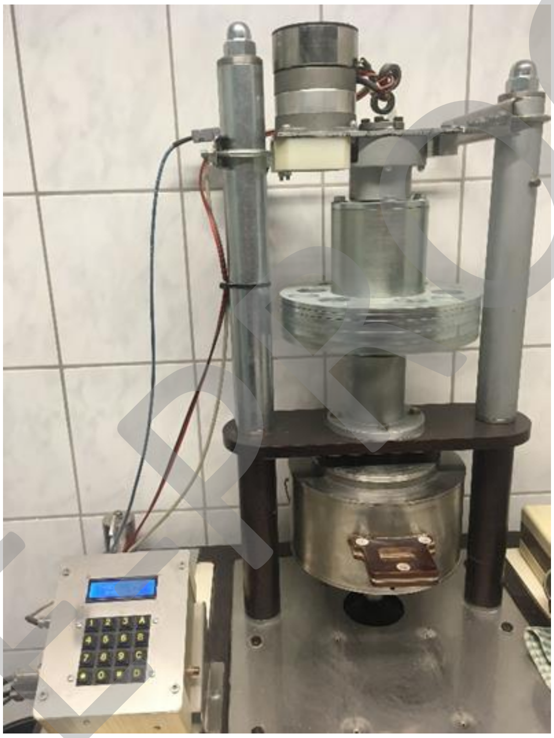

Figure 2
Photo of the upgraded newly built Universal Hardgrove mill

Powder flow properties were measured in a FreemanTech FT4 powder rheometer according to the so-called Specific Energy (SE) protocol. SE is a measure of how powder flows in an unconfined or low-stress environment. It is calculated from the energy required to establish a particular flow pattern in a conditioned, precise volume of powder. This flow pattern is an upward clockwise motion of the blade, generating gentle lifting and low-stress flow of the powder. The powder samples were placed into the 25 mm diameter and 60 mm height sample holder. The shape of the blade is shown in Figure 3.

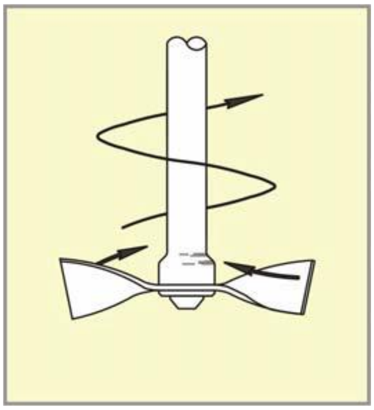

Figure 3
Shape of blade of SE tests

During each SE tests 8 test cycles were done, and the tip speed of the blade was always constant 100 mm/s. One test cycle contained first a total down and an upward moving of the blade into the powder bed for conditioning purposes without measurements. Afterward, the blade moved down again, and measurement started when the blade started to move up again. Since the revolution number and torque were measured, the total energy that the blade needed for the total upward movement was calculated by the data acquisition software by numerical integration. The measured SE values as a function of the 8 consecutive test cycles can be considered as the function of powder flow and cohesion and the friction angle can be determined as the energy axis intersect and slope of the fitted straight line.

The rheological properties of the suspensions were measured in an Anton-Paar Physica MCR51 rotational rheometer using a cylinder – cylinder measuring system with a 40 mm diameter bob and 0.5 mm Couette gap when the necessary sample quantity for one test was 50 cm^3. The revolution number of the bob was set when the shear rate was gradually decreased from 1000 1/s down to 100 1/s. The data acquisition system of the rotational rheometer records the measured torque and set revolution values and calculates the pseudo shear rate and pseudo shear stress values, namely the points of the pseudo shear curve. Afterwards, the rheological model and the parameters can be determined by curve fitting.

3. RESULTS AND DISCUSSION

During the systematic tests two different materials, limestone (LS) and quartz (QA) and five initial moisture contents were tested. Moisture contents of 0, 10 and 20 m/m% can be considered as dry grindability tests and the ones of 50 and 70 m/m% can be considered as wet tests. The exact moisture contents were set by drying and wetting before the Hardgrove tests. After the Hardgrove tests the ground samples were dried at 105 °C until mass equilibrium, because without this, sieving with the 0.075 mm opening size screen was not feasible. This simple operation just shows why the moisture content is not considered during regular Hardgrove testing. The results of the Hardgrove tests are shown in Table 1.

After the Hardgrove testing the entire fine (<0.075 mm) and coarse fractions were mixed again and the Specific Energy (SE) tests were performed. Figure 4 shows the sample holder of the FT4 powder rheometer with a quartz sample when the blade was rotated in it.

Figure 4

Sample holder of SE testing

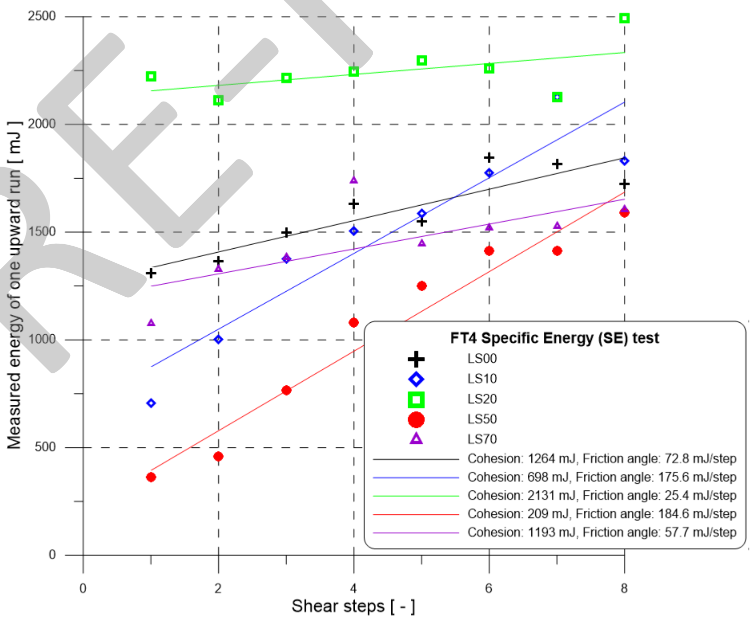

Figure 5

Specific energy as function of test cycles of limestone SE tests

Figure 4 illustrates what the problem was with the SE tests of quartz. The particulate system was a little too coarse for the device and the powder was gradually dug up by the blade. The carrying out of quartz SE tests was not feasible, but with limestone it was without problems. Figure 5 shows the measured powder flow points and fitted flow curves of the limestone SE tests. The results of linear curve fitting and the measured Hardgrove indices, cohesions and friction angle values - based on Figure 5 - are shown in Table 1.

According to Table 1, grindability is strongly influenced by the initial moisture content. It is well known in the literature that sieving at 15 m/m% moisture content is critical because the capillary force between the adjacent particles is the highest in such cases, practically materials with such moisture content cannot be sieved. Above 20 m/m% moisture content, wet sieving has occurred. At the so-called dry grinding range (0 and 10 m/m%) of quartz the measured Bond grindability indices are considerably higher because the particles with themselves and with the balls are bonded by the capillary forces. At wet quartz grinding (20, 50 and 70 m/m%) the required energy is lower, because the inter-particle bond decreases but the particles still can be bonded to the surface of the balls.

Table 1

Results of the Hardgrove and SE tests

Mark of sample	**H index -**	**$W_{B,H}$ Wh/t**	**Spec. work Ws/50g**	**Coh. mJ**	**Frict. angle mJ/step**
LS00	58.48	16.7	26.8	1264	72.8
LS10	57.93	16.8	26.8	698	175.6
LS20	70.26	14.3	26.8	2131	25.4
LS50	71.51	14.1	26.7	209	184.6
LS70	70.75	14.2	26.8	1193	57.7
QA00	47.05	19.9	26.7	-	-
QA10	42.47	21.6	26.7	-	-
QA20	51.07	18.6	26.7	-	-
QA50	54.05	17.7	26.8	-	-
QA70	53.70	17.9	26.8	-	-

Probably, grinding also deteriorates at extremely high moisture contents because particles cannot be bonded to the balls anymore. From these observations the conclusion must be drawn that the Hardgrove grindability testing should be carried out at the actual moisture content of industrial grinding, especially when the measured values are used for on-line grinding regulation. Regarding the limestone grindability tests similar conclusions can be drawn except that the adhesive limestone can be efficiently ground at 70 m/m% moisture content too, because limestone can still be bonded to the balls.

After the powder flow tests the same material was used for the rotational rheometer tests. Because of the Couette gap width (0.5 mm) between the cylinders and according to our earlier experiences, particles typically coarser than 0.16 mm should not be measured in the MCR51 rotational rheometer with this measurement system. This is a general rule of sampling and test methodology, that the smallest size of the equipment should be at least three times the typical particle size ($L_{min} \geq 3 \cdot X$).

Table 2

Results of rheological tests of limestone samples

Original sample	**Volumetric concentration %**	**Absolute viscosity mPas**	**Absolute viscosity (repeated) mPas**
finer than 0.16 mm			
LS10	2.2	4.0	3.9
	4.4	4.0	4.1
LS20	2.2	4.0	4.0
	4.4	4.2	4.1
	6.7	4.4	4.4
LS70	2.2	3.9	4.0
	4.4	4.0	4.0
	6.7	4.3	4.3
finer than 0.5 mm			
LS70	16.3	4.6	4.4

Therefore, the solids were sieved again, but now with a 0.16 mm aperture size screen. Unfortunately, this method decreased the quantity of fine solids; therefore only low solids concentrations could be measured. There is one exception, in the case of the LS70 solids a higher concentration and the <0.5 mm fraction were measured successfully, but this result has to be handled with care (the simple sampling rule of $L_{min} \geq 3 \cdot X$ was not satisfied in this single case). Table 2 shows the results of rheological tests of limestone samples; these low concentration suspensions were Newtonian fluids with low absolute viscosity values. Table 3 shows the results of rheological tests of quartz samples.

It is well seen that the many sample operations with the ground solids (drying, sieving, SE testing, sieving, mixing) probably destroyed the original effect of Hardgrove testing or there is no such effect in this case. The measured absolute viscosity values follow the well-known Einstein concentration-viscosity relation for dilute fine suspensions.

Table 3

Results of rheological tests of quartz samples

Original sample	**Volumetric concentration %**	**Absolute viscosity mPas**	**Absolute viscosity (repeated) mPas**
finer than 0.16 mm			
QA00	2.2	4.0	4.0
	4.4	4.0	4.0
QA10	2.2	4.1	4.1
	3.0	4.1	4.1
QA20	2.2	4.1	4.1
	4.4	4.1	4.1
QA50	2.2	4.0	4.0
QA70	2.2	4.0	4.0

4. Conclusion

The carried out wet mode grindability testing has proved that the moisture content is also a very important technological parameter. At the so-called dry grinding range (0 and 10 m/m%) the measured necessary grinding energy was typically higher, at wet grinding (20, 50 and 70 m/m%) the required energy was lower. 50 m/m% moisture content resulted in the lowest grinding energy need. The same trends were observed for both model materials, namely for the limestone (soft adhesive) and the quartz (hard non-adhesive).

Two important phenomena were revealed, namely that the places of capillary force acting among the particles and among the particles and the balls are also important and opposite effects can be observed in the two places. The particle-particle bonds are typically not advantageous because they deteriorate grinding; however, particle-ball bonds are advantageous because in ball and ring mills it helps for the formation of more stressing events.

It was also concluded that on-line grindability testing for technological regulation should be done at a moisture value equal to the actual moisture content of the material in the industrial equipment.

This study also has revealed that further measurement methodology development is also necessary. Just think about the problem when Hardgrove grindability is measured in the 10-20 m/m% moisture content range, but sieving at this range is impossible, because of the high capillary forces. Measurement technique difficulties were also found at the powder flow and the rotational rheometer testing, but it is thought that this initial work can pave the way for later developments.

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