



Improving the efficiency of fluidized bed comminution of limestone samples

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ABSTRACT

Purpose: The aim of the research was to describe the properties and methods of using limestone for desulfurization of flue gases and to analyse the process of fluidized bed comminution and the influence of selected parameters and stand modification on quality of product of fluidized bed comminution.

Design/methodology/approach: Tests of the grinding process on the fluidized bed were carried out using a modified fluid bed mill for operational variable parameters and their results were compared with the results received before modification. The modification of the test stand consisted of increasing the height of the grinding chamber, which ensured an increase in the volume of the fluidized layer where the grinding process takes place.

Findings: Main parameters that determined the effects of comminution in the analyzed case were: overpressure of working air and the rotor speed of the classifier. The introduction of the modifications of the test stand ensured an increase in the volume of the fluidized layer in which the grinding process takes place. As well as a greater gravitational classification, which caused larger grains to be stopped in the grinding chamber and shift of characteristics of grain compositions towards finer grains.

Research limitations/implications: It is assumed that the diameter of sorbent grains used in the fluidized bed can not exceed 6 mm. The granularity of the offered sorbents ranges from 0.1 mm to 1.2 mm. The quality of the desulfurization process depends on the overall granulation of used sorbent grains.

Practical implications: Appropriately selected sorbent grains used in wet and dry flue gas desulphurisation plants ensure improved efficiency of the desulphurisation process and lower operating costs of the installation.

Originality/value: Thanks to the comminution method used, a sorbent is obtained without impurities and with an increased specific surface, which can be used in fluidized bed boilers.

Keywords: Comminution, Grinding, Limestone, Desulfurization

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PROPERTIES

1. Introduction

The process of grinding is a technological process of mechanical degradation of materials (both organic and artificial) into smaller pieces. The grinding of the working material happens because of external factors, that cause tension stronger than the maximum material's strength. When the grinding takes place, the shape of grains changes and the surface area of the material expands. The aim of the grinding process is to:

- achieve an optimal size grade that meets the specifications of the technological process (used when further processing requires feed of specific grain size),
- create a product of a set maximum grain size,
- separate the product from unwanted materials.

The process of grinding requires large intakes of energy but is widely used everywhere, therefore there is a great need to find a more effective and cheaper solution. There are many different ways of grinding, each with its own set of specifications. This article's focus is on fluidized bed comminution, complexity of which depends on many different factors [1].

2. Material and methods

2.1. Properties and application of limestone

Limestone was used during the research of the comminution process because of its characteristics: low hardness, brittleness, abrasibility, low absorption, high cold resistance, and numerous uses. Various branches of economy use limestone in its many forms: limestone powder, burnt lime and hydrated lime.

Nowadays, because of the norms that restrict the emission of fumes, it is important to use limestone sorbents to desulfurize flue gases. The negative impact of industry on the environment brings about the implementation of increasingly more demanding policies restricting the pollution of fumes. One of these is aforementioned desulfurization of combustion gases. Limestone grains are fed into the combustion chamber and after reaching a certain temperature undergo the calcining process. It is assumed that the diameter of sorbent grains used in the fluidized bed can not exceed 6 mm. The granularity of the offered sorbents, called in the industry limestone sands, ranges from 0.1 mm to 1.2 mm. The quality of the desulfurization process depends on the overall granulation of used sorbent grains [2-4].

During the calcining process limestone combines to create calcium oxide CaO and carbon dioxide CO₂. Calcination of calcium carbonate only happens when the pressure of carbon dioxide in the gas is lower than the

balancing pressure of the hearth furnace. CO₂ contents depend on the excess air coefficient. As it rises, the temperature of the calcination process drops. During the calcination, as CO₂ releases, a pore structure made of micro and macro pores appears on the surface of grains. CO₂ escapes outside through the pores. Parallel to the calcination process, the sintering process takes place, which causes larger pores to appear. It is unlikely for the grains to sinter inside fluidized bed boilers, since the temperature inside is lower than the softening point of CaO. The size of pores depends on the size of grains, qualities of limestone used, calcination duration and contents of the gas in which calcination process occurs. Produced CaO reacts with sulfur dioxide SO₂, created during the sintering process to make calcium sulfate CaSO₄. Because of these reactions a layer of CaSO₄ appears on the surface of the grains and on the pores, which obstructs them. The obstruction of pores slows the desulfurization process. If the sorbent stays inside the combustion chamber for a too short amount of time, only a portion of CaO undergoes the reaction. The extent of sulfur reduction can be defined by a coefficient Ca/S. Said coefficient describes the number of moles in the sorbent necessary to compound with the sulfur contained within the fuel. As the amount of limestone increases, the process of energy creation worsens. It also means that large amounts of waste, consisting mostly of unreacted limestone sorbent, are created. This causes problems with the storage and industrial utilization. On the other hand, if the amount of calcium based sorbent is not adequate during combustion, the desulfurization process is then relatively weak and significant amounts of sulfur compounds remain unreacted and are released into the environment. Reduction in the emission of sulfur oxide using limestone sorbents is possible, inter alia, by the application of following actions:

- direct binding of sulfur oxides in the combustion chamber,
- removal of sulfur oxides from flue gases.

Direct binding of sulfur oxides happens when certain substances are brought into the hearth furnace, which due to thermal decomposition create calcium oxide CaO. Substances that are used most commonly to that effect are: calcium carbonate CaCO₃, dolomite CaCO₃•MgCO₃ or calcium hydroxide Ca(OH)₂. Limestone reacts directly with SO₂ creating CaSO₄ in the process. The efficiency of the sulfur binding process inside the furnace depends on the temperature of the reaction area. The binding can only take place in temperatures ranging from 700°C to 1100°C. This fact explains the difficulty with realizing those reactions inside pulverized coal furnaces, which flame temperature reaches 1600°C.

In order to remove sulfur oxides from flue gases the following processes can be used: wet, dry and semi-dry. Wet

process achieves high efficiency of desulfurization (up to 95%). It depends on calcium or calcium carbonate's stoichiometric ratio in relation to that of removed sulfur. From the economical standpoint it is about 75% cheaper to use limestone instead of calcium. Dry process, on the other hand, produces an easy to manage, dry side product. The process, however, achieves lower efficiency of desulfurization (around 50%) than the wet one. Dry process of desulfurization can be conducted by:

- dosing a sorbent into coal inside a mill,
- insufflating a sorbent into a combustion chamber above the flame.

Semi-dry process combines high efficiency of the wet process and low cost of the dry process. Reduction of costs results from the facts that the dry side product is easy to manage, the process requires less energy, less water, and is an overall cheaper investment. What is more, the desulfurization process is also simpler. In the semi-dry process, the sorbent is dosed with such an amount of water so that the side product is dry and the fumes are above the water dew point. There are numerous different varieties of wet, dry and semi-dry processes that all differ in the apparatuses, devices used as well as technical and economical indicators [5-9].

The limestone used for the research on the grinding process was taken from the "Czatkowice" Limestone Mine in Krzeszowice near Kraków. The company is involved in extracting crushed limestone and is the leading manufacturer in Poland of high quality limestone sorbents used in wet flue gas desulphurization installations as well as in fluidized bed boilers. The average chemical composition and physical properties of the tested raw material examined are given in Tables 1 and 2. The material delivered from the mine was characterized by grain size ranging from 0-2000 μm . The material used has been divided into narrow grain classes prior to testing.

Table 1.
Chemical composition of the limestone used [10]

Ingredient	Content
CaCO ₃	96.00%
SiO ₂ +NR	1.50%
MgCO ₃	1.50%
Fe ₂ O ₃	max 0.2 %
Al ₂ O ₃	max 0.2 %
Na ₂ O	0.02%
K ₂ O	0.37%
Heavy metals	trace
CaCO ₃	96.00%
SiO ₂ +NR	1.50%

Table 2.
Physical properties of limestone used [10,11]

Features	Values
bulk density	1.40-1.70 t/m ³
apparent density	2.68 g/cm ³
water absorption	medium 0.35%
porosity	0.96%
Mohs hardness	3-4
compressive strength when dry	90-110 MPa
abrasion in the Deval drum	4.10%
abrasion in the Los Angeles drum	max 25%
frost resistance	0.20-0.50%
thermal conductivity	200 W/mK
color	cream-gray

2.2. Grinding methods

Industrial equipment for mechanical processing mineral raw material uses the following grinding technologies: hammer, vibrating, ring, disc, ball, jet, mix, colloid, fluidized. Each of these grinding methods has specific characteristics. However, regardless of the grinding method used, the working conditions of industrial shredders are sought which on the one hand guarantee the purity of the grinding product by the selecting the appropriate parameters of the working factor and, on the other hand, reduce the energy consumption of the grinding process and ensure the improvement of environmental protection requirements.

There are many methods for classifying grinding equipment in the available literature. The general breakdown of grinding equipment can be found in the standard PN/M-47270. After analysing various methods of classifying mills, it was proposed to divide equipment into five main groups, in which sub-groups were identified:

- 1) Crushers:
 - jaw crushers,
 - conical crushers,
 - roller crushers,
- 2) Grinding mills:
 - drum mills,
 - tube mills,
 - screen mills,
 - vibration mills,
 - pearl mills,
- 3) Ring mills:
 - ball-bowl mills,
 - roll-bowl mills,
 - disc roller mills,

- 4) Jet mills:
- rotor mills,
 - counter rotating mills,
 - fluidized bed mills,
- 5) Other:
- hammer mills,
 - beater wheel mill,
 - disc mills,
 - colloid mills.

A feature that distinguishes among the above technologies the use of jet mills is the purity of the grinding products. Obtaining a pure product, without the intrusion of undesirable substances, is due to the absence of intermediary bodies during the energy transfer process. In addition, these mills are characterized by high crushing of the product. Both of these features are fundamental arguments for the development of jet grinding technologies. Due to the grinding mechanism, jet mills can be divided into two groups: the first group consists of mills whose size degradation occurs due to friction, the second group consists of mills in which grinding occurs during mutual collisions of the material under consideration.

Of the known designs of jet mills, three main groups can be distinguished

- vortex (spiral) jet mills,
- counter-jet (counter rotating) mills,
- fluidized bed mills.

The principal of operation of spiral jet mills is based on mutual collision and abrasion of grains suspended in the gas stream which are reduced. The grinding chambers of spiral mills have the following shapes: annular oval or conical. The gas nozzles are mounted tangentially around the circumference of the grinding chamber which allows for strong turbulence thanks to which grinding takes place practically without contact with the inner lining of the chamber. Spiral mills are characterized by a preliminary classification - coarse grains are held at the periphery of the grinding chamber, while fine grains can be more easily lifted with the gas (swirl). In counter-rotating jet mills, grains gaining high velocity from the gas stream in the acceleration tube are crushed by collision of two or more two-phase gas-solid streams, or by impact of the two-phase stream against a stationary plate. Mills equipped with a stationary plate are called jet-disc mills. The grinding chamber of these mills is usually cylindrical, while two or four two-phase jets are placed opposite each other on the circumference of the cylinder. The grains located in the central zone of the grinding chamber can reach a supersonic speed, the higher the grain speed during the collision, the better the crushing effect.

Grinding in a fluidized bed mill occurs as a result of collision and abrasion of the grains forming the fluidized bed. Fluidization is the process of forming a dynamic suspension of grains in a flowing stream of gas or liquid. A conventional fluidized bed mill is constructed with a vertical grinding chamber, in the lower part of the chamber counter-rotating air nozzles are mounted, and the material is poured above the nozzle. The air coming out of the nozzle expands, causing the material to fluidize in the grinding chamber. Soft materials are broken by impact, while hard materials by grain abrasion. It follows that the structures of jet mills are determined by the grinding method, another criterion of is the type of working factor: gas mills (most often compressed air), steam mills (most often superheated water vapour) [12-19].

2.3. The process of fluidization of granular materials

Intensive mixing of raw granular materials during fluidization promotes diffusion phenomena, intensifies the course of chemical and physical processes, such as drying, combustion, heat transfer. The creation of a large interfacial surface enabling rapid heat exchange during fluidized contact of two factors makes the fluidized bed technique used in a large number of technological processes, such as: catalytic cracking of crude oil, heat treatment process and thermo-chemical processes of metals, drying and mechanical processing of substances, calcination of calcium carbonate, combustion of sulfur ores in the production of sulfuric acid, combustion of low-calorific solid fuels and sludges, and direct flue gas desulphurization in furnaces of fluidized bed boilers in the power industry. In industrial practice, technological processes in a two-phase flow system are very often used for grinding granular materials. Two-phase flow is defined as the joint flow of two phases. There are a continuous phase and a dispersed phase. The continuous phase is a fluid, i.e. a liquid or a gas, while the dispersed phase is a substance of any state of aggregation, i.e. a solid, liquid or gas. There are three basic forms of two-phase flows, Figure 1:

- 1) gas-liquid or vapor-liquid,
- 2) gas-solid,
- 3) liquid-solid.

The process of fluidization of granular materials is a form of two-phase flow gas-solid, while the term fluidization is defined as a two-phase process, in which the layer of fine powdery material becomes fluid as a result of a direct gas or liquid interaction. Under fluidization conditions, the force of gravity acting on solid material grains is balanced by the force of fluid resistance acting on

them. As a result, the grain layers are suspended in space due to which the fluidized bed acquires the characteristics that make it liquid:

1. the surface of the bed remains horizontal regardless of the inclination and shape of the vessel,
2. the bed material can flow out like a liquid through the opening in the side wall and the bottom of the vessel,
3. objects with a density higher than the density of the bed will move downwards, while objects with a density lower than the density of the bed will remain on the surface,
4. proportional pressure drops,
5. the grains mix well and the bed has a homogeneous temperature during heating.

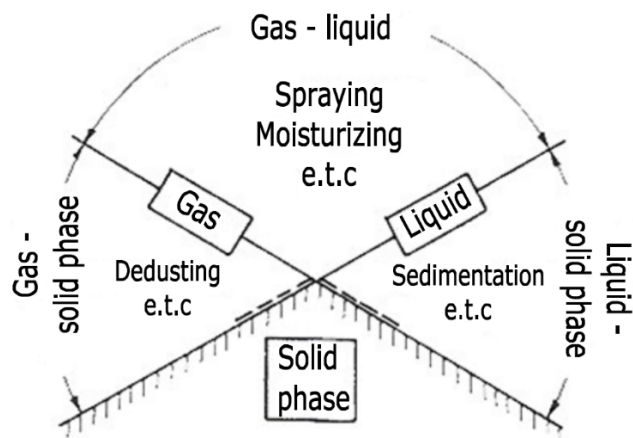


Fig. 1. Three basic forms of two-phase flows

As the velocity of the gas flowing through the material of the layer increases, there are some changes in its behaviour due to the contact of the gas and the solid material.

Initially, the bed remains in a static state (it is stationary), but with increasing gas velocity it successively changes into: bubbling state, turbulent, fast and finally pneumatic transport [18, 20-23].

Figure 2 shows the successive stages of the formation of the characteristic states of the fluidized layer. The stationary state of the bed is defined as state 1 (Fig. 2b). The particles are then stationary and supported by contact with other particles. The pressure drop increases with the increase of the apparent velocity $v = v_0$. The bed has a minimum porosity ϵ_{min} and a height H_{min} corresponding to this porosity. This is the typical flow of fluid through a solid, loose or porous layer. The moving bed corresponds to state 2, it is formed during the loosening of the stationary bed. In this state, the particles are in contact with each other, moving relative to the column wall without changing their position. They perform oscillatory movements with small amplitudes, as if they were suspended in a stream of fluid. Loosening of the bed occurs when the overpressure of the fluid equals the pressure exerted by the bed. This state determines the start of fluidization (minimum fluidization – mf). The dotted line r (Fig. 2a) defines the pressure change with a slow decrease in velocity u , which is accompanied by the formation of a loose solid bed with freely deposited particles with porosity ϵ_{mf} . The R point is the theoretical point of transition of a fluidized bed into a stationary bed with a slow decrease in the fluidization rate. The fluid bed corresponds to states 3 and 4, the gaseous fluid bed is accepted for consideration. The height of the H-bed is form tens to thousands of times greater than the diameter of the particles. When the minimum fluidization velocity u_{mf} is exceeded, a fluidized bed with a dense phase I is formed, which has a clearly defined upper boundary. An increase in gas velocity increases the volume of the bed, which becomes a turbulent fluidized bed with intensive mixing of the particles

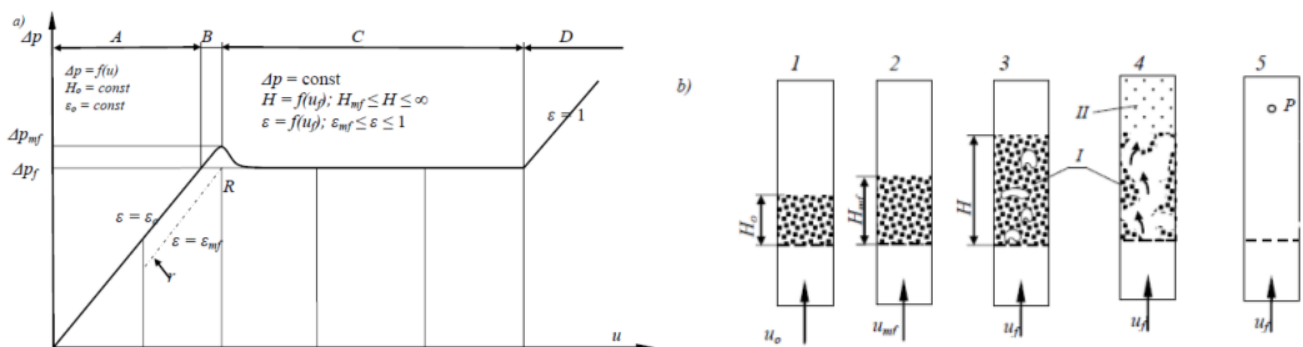


Fig. 2. Single sized fluidized bed: a) plot $\Delta p = f(u)$; b) different states of the bed: A – stationary bed, B – moving bed, C – fluidized bed, D – empty column; u_f – fluidization velocity, u_{mf} – minimum fluidization velocity, ϵ – porosity, H – height of the bed, R – theoretical transition point of the fluidized bed to the stationary bed, P – motionless free particle [23]

throughout its area. The boundary condition defined as state 5 (Fig. 2b.) occurs when the flow velocity equals the free-fall velocity of particles $\varepsilon=1$, then the fluidization ends. This state is symbolized by the motionless free particle P. The velocity of the free fall of the particle in the motionless gas v_0 is equal to the fluidization velocity, which is referred to as the suspension velocity $u_f = u_t$, i.e. $v_0 = u_t$.

3. Result and discussion

The tests of the grinding process on limestone samples was carried out in a laboratory fluid mill that was adapted for fine grinding of granular substances with the grain size of the product below $100 \mu\text{m}$. The research was carried out before and after the modification of the grinding chamber, which was extended by 17 cm. The schematic diagram of the test stand is shown in the Figure 3.

Grinding of limestone samples was carried out in the laboratory fluid-bed mill. The process of each trial was similar. A sample with a mass of 1500 g and a grain size of $800\text{-}1200 \mu\text{m}$ was fed using gravity from the hopper into a cylindrical grinding chamber, where it underwent intensive fluidization. Working air with a regulated mass flow was supplied from a system of three converging nozzles (diameter 2 mm) arranged concentrically on the perimeter of

the chamber, and a fourth nozzle of the same diameter, mounted on the bottom of the grinding chamber. The volume of the air mass stream was regulated by setting the appropriate value of the working medium overpressure, which also depended on the speed of the flowing air. The air flowing out of the nozzles expanded, creating a rise of high-energy fluidized layer, enabling comminution of the tested material. In the upper part of the grinding chamber, an impeller classifier is located, which, using the inverter, allows for setting a specific rotor speed. It ensures the division of the product into fine class, that's fed to the cyclone, and thick class, that's returned to the grinding chamber. In the cyclone, the two-phase mixture was separated into two streams: a stream of dusty working air directed to the fabric filter, and a stream of grinding product 1, fed to the grinding product container. In the fabric filter, very fine grains that were not retained in the cyclone, were separated from the air. These grains, marked as product 2, together with the grinding product 1 determine the total fluid bed grinding product. The effects of the comminution process can be checked using various parameters. One of the most important parameters is the grain composition of the product. Therefore, the weighed, packaged and properly described samples were brought to a grain composition analysis, which was carried out using the KAMIKA Instruments electronic Infrared Particle Sizer (IPS) analyzer shown in the Figure 4.

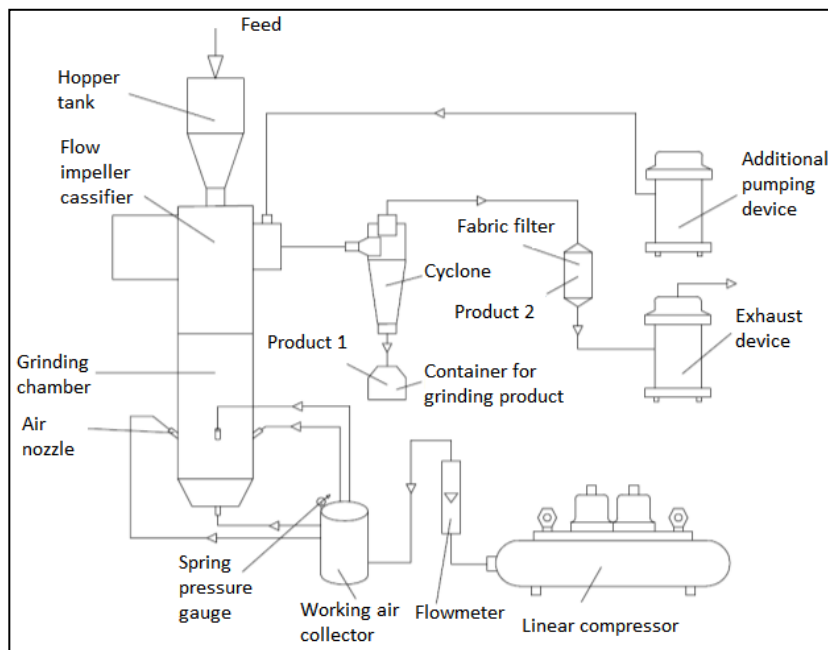


Fig. 3. The schematic diagram of the test stand



Fig. 4. Electronic IPS particle size analyzer

The electronic particle analyser IPS (Infrared Particle Sizer) by KAMIKA Instruments, Warsaw, Poland, is an indirect measurement device used to determine the particle size distribution under laboratory conditions, irrespective of its chemical and physical properties, within a measuring

range of 0.5 to 2000 μm . It also allows reading of the second mean particle size and determining the aspect ratio. The basic system of the analyser is an IPS measurement system whose task is to emit and record a beam of infrared radiation that radiates over the measuring area of the probe.

The measurement with the IPS analyser consists of measuring the infrared radiation flux scattered by grains moving in the measuring plane of the probe. The electronic system receives an analogue electrical signal from the measuring system, representing the response of the measuring system to the attenuation of the emitted radiation beam. The resulting signal is properly processed, amplified and transmitted to the A/D analog-to-digital card. Then the digital signal is processed on the computer using the appropriate program. An electronic grain dosing system is integrated with the analyser's measuring system, ensuring measurement continuity and controlling concentration of grains in the measuring area (all controlled by a computer). The actuator in the automatic dosing system is a miniature compressor with specific characteristics adapted to smooth regulation of the air flow.

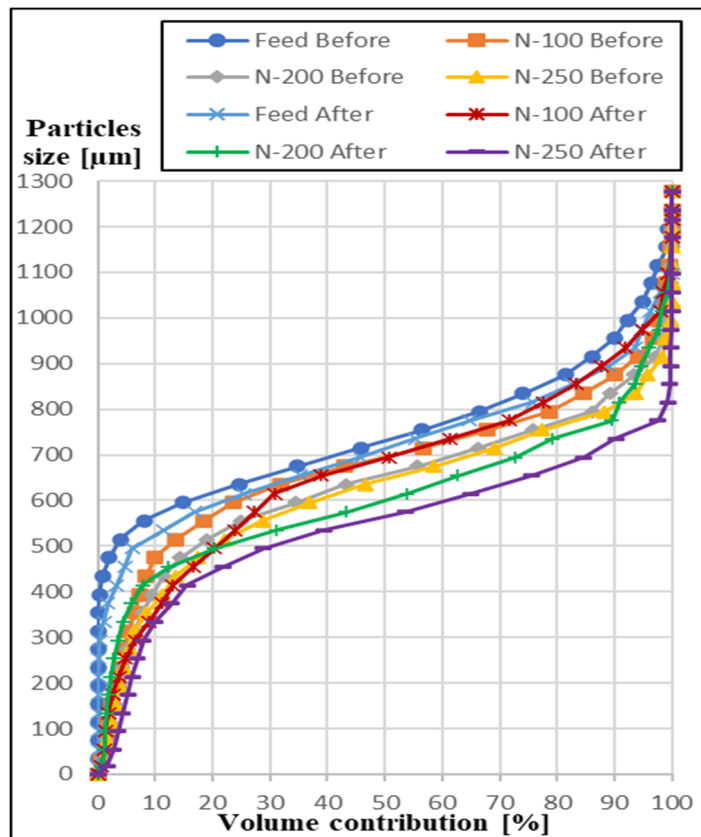


Fig. 5. The influence of rotor speed on the comminution process

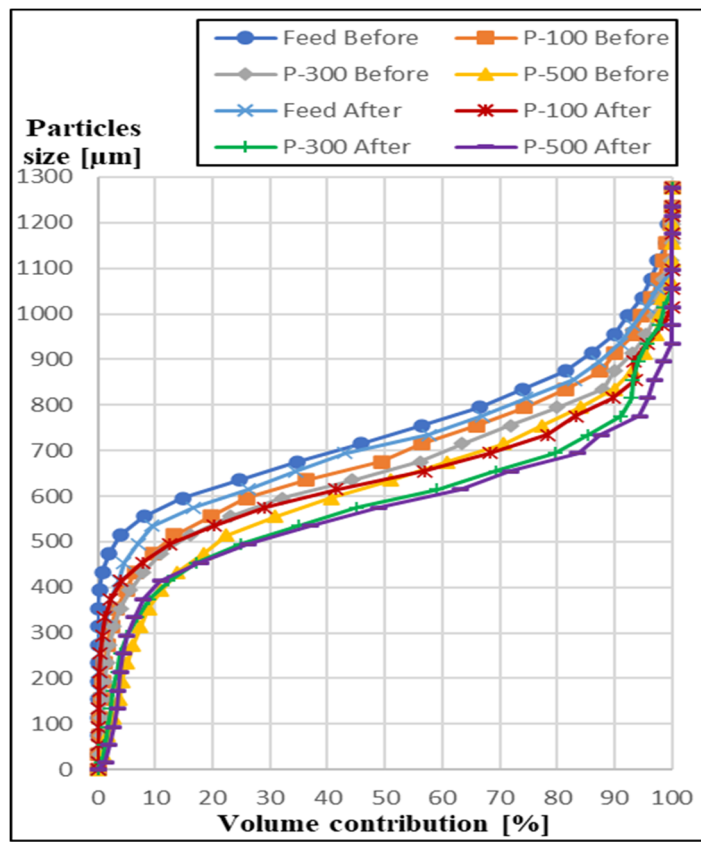


Fig. 6. The impact of working air pressure on the comminution process

To ensure efficient dosing, it is also necessary to control the air jet, lifting the previously separated grains and transporting them to the measuring zone. The flow control is possible at 300 levels, such a precise control of the dispenser allows quick measurement (up to several thousand grains per second) of individual grains and avoids overlapping of grains in the measurement space. The collection of grains is measured into 4096 size classes and calibrated into 256 user-accessible classes. Measurement results are available in the form of graphs and tables [19,24,25].

Impact of selected parameters on the comminution effect

Because of its complexity, the comminution process depends on many different parameters. That is why in order to define which of the parameters fundamentally determines the effects of the process, the following parameters were altered:

- rotation speed of the classifier rotor was set on three different levels 100 (N-100), 200 (N-200), 250 (N-250) [1/s],
- similarly, working air pressure was set on three levels 100 (P-100), 300 (P-300), 500 (P-500) [kPa],

- Before indicates the results before the modification of the grinding chamber,
- After indicates the results after the modification of the grinding chamber.

Afterwards, based on the results of particle size measurements done with the help of the IPS analyzer, two charts were drawn: one depicting the influence of rotor speed on the comminution process (Fig. 5) and one depicting the impact of working air pressure on the comminution process (Fig. 6).

4. Conclusions

Main parameters that determined the effects of comminution in the analyzed case were: overpressure of working air, tied to the change of the working air flow, and the rotor speed of the classifier. Increasing the overpressure value forces the larger working air stream, which is supplied to the nozzles from the compressor. Thus, it increases the energy that is transferred to the grains by the air particles in the grinding chamber, which causes an increase in the

intensity of the fluidization process. This in turn leads to a shift in the cumulative grain composition curve toward finer grains. Higher rotor speed ensures a smaller average grain size characterizing the grinding product. This is caused by the centrifugal force impacting the grains falling into the classifier, which increases with the increasing rotor speed. Under the influence of this force, larger grains are returned to the milling chamber enabling secondary grain material degradation, which is the final product. Similarly, increasing the working air pressure causes a shift in the cumulative grain composition curve toward finer grains. This is due to the fact that, as the working air pressure increases the air velocity in the grinding chamber also increases, which intensifies the fluidized grinding process. It results in the final product of smaller grain size. The introduction of the modifications of the test stand ensured an increase in the volume of the fluidized layer in which the grinding process takes place. As well as a greater gravitational classification, which caused larger grains to be stopped in the grinding chamber and shift of characteristics of grain compositions towards finer grains.

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