17TH EUROPEAN SYMPOSIUM ON COMMINUTION & CLASSIFICATION

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ABSTRACT BOOK

Sunday 26th June 2022 - Welcome reception and Registration

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Monday 27th June 2022 - Morning Session

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Poster List

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Autonomous comminution circuits – Opportunities and challenges

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Keywords: Autonomous systems, Mineral processing, Comminution.

Mining covers a significant footprint in the excess of 57,000 km2 of the earth's surface [1] and consumes almost 3% of the world's water [2]. Mining activities are responsible for 4-7% of Greenhouse gas emissions on the global scale [3]. Because of the scale of the impact, the mining process has on Environmental, Social, and Governance (ESG), the regulatory limits and industry standards are reshaping the mining industry.

Process autonomy is the key tactic for the minerals industry to achieve its ambitious ESG goals set for 2030 and 2050. Comminution and classification processes significantly impact energy consumption, carbon and environmental footprint, and process performance. Therefore, developing and implementing process autonomy for the comminution circuits presents a considerable opportunity.

There is a general misconception that autonomous systems are primarily a means for reducing the cost of the workforce. However, process autonomy could deliver value to operations through:

- stable operation, which leads to higher overall throughput, consistent product quality and improved productivity;
- reliable and predictable process, which enables targeted and accurate economic optimisation of the operation;
- optimum operating condition, which increases the equipment life and consequently reduces operating costs and downtime;
- reliable data on water, energy, emissions and consumables usage, which can be used for managing and forecasting the costs and optimising utilisation;
- autonomy of more mundane tasks, which enable operators to focus on more rewarding and higher-skilled tasks; and
- use of autonomous units in hazardous environments, which improves safety.

An overview of advances in applying Autonomous systems in Minerals Extraction and its key features are presented in Figure 1. Each stage in the process benefits from introducing technologies that automate aspects of the stage, and different stages current vary in their automation maturity. However, optimising locally does not guarantee an overall benefit. We assert it is the linking between the phases that is critical and requires address

Comminution circuits, in general, are falling behind the development and uptake of autonomous systems relative to mining and extraction. The main barrier preventing the transition from automation to full autonomy is the scarcity of reliable and trustable technologies to replicate the numerous human decisions necessary for comminution circuits to operate effectively in an unpredictable and complex environment.

Addressing the challenges of the development and implementation of autonomous systems for comminution and classification processes, in particular, and mineral processing plants, in general, will enable the industry to harness the benefits listed above.

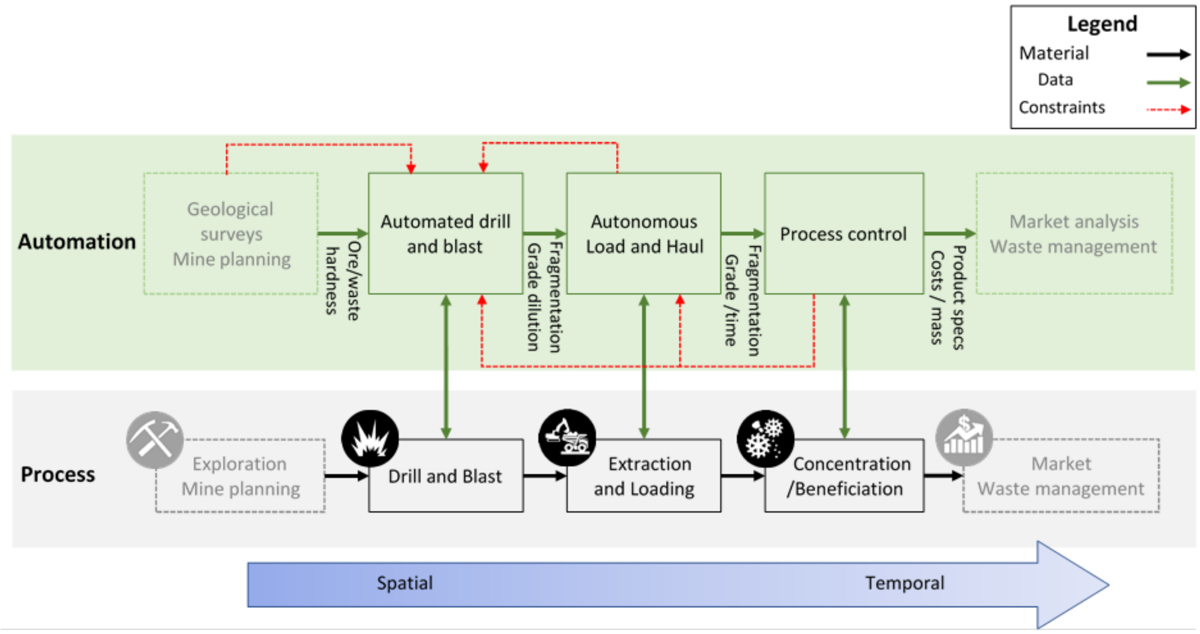

Figure 1. Overview of automation in the mineral extraction process.

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[3] McKinsey: Climate risk and decarbonization - January 28, 2020

The 4M business for drug nanoparticles and nanocomposites: making, manipulating, measuring, and modeling

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Keywords: drug nanoparticles, nanocomposites, particulate processes, modeling

Due to their low water-solubility, a great majority of recently discovered drug molecules dissolve slowly and exhibit low bioavailability as well as wide variability in fed vs. fasted state. Among various approaches in formulating such drugs, preparation of drug nanoparticle suspensions via wet stirred media milling (WSMM) and their conversion into nanocomposites via drying (**Figure 1**) have been shown to be effective [1,2]. WSMM is preferred as it can handle most poorly soluble drugs, while being scalable and environmentally benign. The nanosuspensions prepared by WSMM appeared in more than a dozen pharmaceutical products [2]. This talk aims to expose the 4 major facets of WSMM and nanoparticles–nanocomposites, i.e., making, manipulating, measuring, and modeling, in a holistic and integrated fashion. As often the case, *modeling* and *measuring* go hand in hand and allow us to develop a deeper understanding of the practice of *making* and *manipulating* such fine particles.

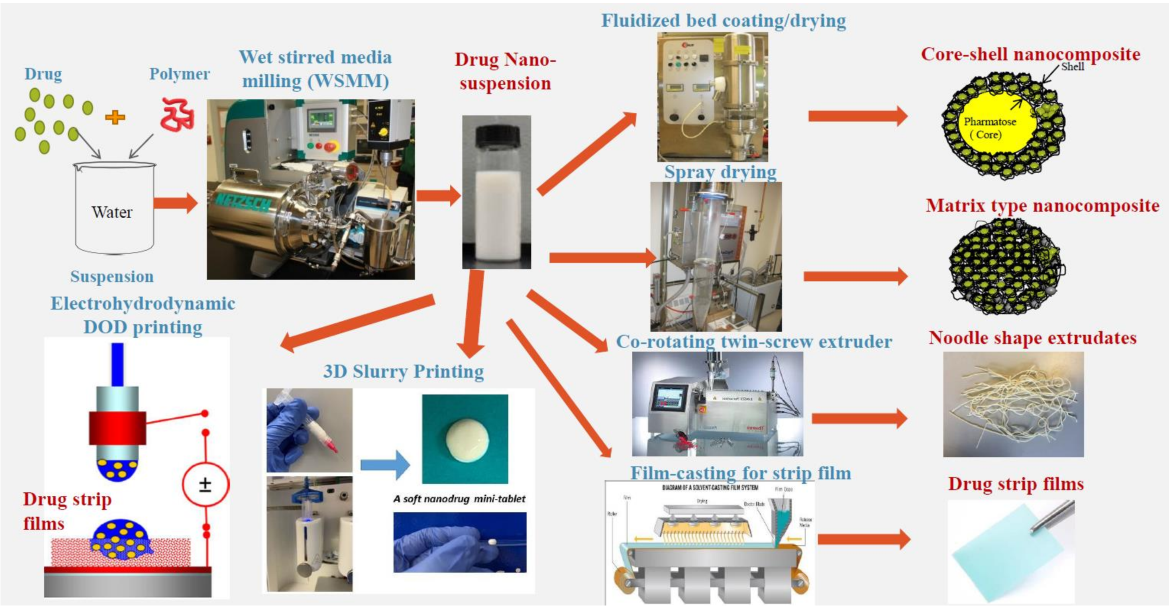

Figure 1. A schematic illustrating the *making* of drug nanosuspensions via WSMM and their drying into various forms of nanocomposites, i.e., powders, extrudates, films, and tablets. The powders/extrudates are further processed into final solid dosages such as tablets, capsules, and sachets, via milling/blending, lubrication, and tableting/encapsulation.

In the first part of this talk, I will present the recent advances in the *making* of drug nanosuspensions via WSMM, a top-down method, along with the use of microhydrodynamic modeling and various characterization tools for better process understanding. An intensified WSMM, upon rational selection of bead size and processing conditions, achieves fast preparation of sub-100 nm particles with minimal media contamination [3]. Moreover, mixtures of two different types of beads, crosslinked polystyrene and zirconia, enable superior process performance as compared with the individual beads at the same volumetric bead loading [4]. This part of my talk will conclude with the prediction of the breakage rate constant under a wide variety of processing conditions–bead properties using a microhydrodynamics-based multilinear regression model [5].

In the second part of this talk, I focus on the *manipulation* of drug particle sizes and surfaces in the WSMM process upon judicious selection of various stabilizers such as polymers, surfactants, and their combination. Growth of particle size through aggregation and Ostwald ripening during the processing and storage is a major challenge, which can be mitigated by pharmaceutically acceptable, adsorbing polymers/surfactants [1]. Considering that bare drug nanoparticles seldom possess an absolute zeta potential above 30 mV, the physical stability is provided by the adsorption of the polymers/surfactants onto the particle surfaces, which modulate steric, electrostatic, or electrosteric mechanisms. In formulating drug nanosuspensions, one must exert utmost caution considering potential toxicity of the stabilizers and their acceptability to various regulatory agencies. Also, the impact of these stabilizers on the WSMM process, e.g., through viscous dampening, and the downstream processing of the drug nanosuspensions, and their ultimate end-use by the patients must be considered [1,2]. Our efforts on the synergistic stabilization via polymer–anionic surfactant combination [6–8] and surfactant-free stabilization via colloidal superdisintegrants will be highlighted [9].

The third part of my talk will focus on the impact of nanoparticles on the dissolution enhancement and the manipulation strategies on the drug nanoparticle recovery, redispersion, and *in vitro* drug release from various types of nanocomposites. Significant dissolution enhancement upon use of drug nanoparticles with large surface area and novel surfactant-free formulations [10,11] that make use of colloidal superdisintegrants are highlighted. The talk will conclude with future research directions and opportunities in this field: (i) the need for combining CFD or microhydrodynamics model to a population balance model for prediction of the whole particle size distribution under a variety of process conditions [12], (ii) development of material-sparing approaches for assessing short-term physical stability [13], (iii) prediction of physical stability using molecular dynamic simulations [14], (iv) design of intensified and optimized processes for drug nanoparticle–nanocomposite production [1–3], and (v) design of drug nanosuspensions as inks for 2D and 3D slurry printing [15].

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Recent developments and future prospects in comminution and classification in chemical industry

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Keywords: wet comminution, dry comminution, classification, developments

Comminution and classification are old technologies dating back to ancient times. They were especially used for food and ore processing. Still nowadays research results give rise to further developments of these technologies for use in industrial scale. This paper presents the trends during the last 30 years driven by cost reduction and rising product quality standards. An estimation covering future trends concerning comminution and classification is included.

In **wet fine grinding** the demands on the mean particle size did not change much during the last 30 years. Mean particle sizes of 0.2 µm to around 1 µm are required for e.g. colour pigments and crop protection formulations. The research in the generation of nano formulations around 15 years ago was successful but resulted in a rather low number of products. The demands on the steepness of the particle size distributions showed a trend to narrower particle size distributions, e.g. lower d_{80}- or Hegmann-values. In 1990 stirred ball mills were predominantly horizontal stirred ball disk mills with a bead separation by a rotating gap or vertical disk mills. Both types limit the bead size to bigger than 1 mm, allow rather low flow rates only and for the vertical mill low tip speeds only as well. In the early 90s it was shown that smaller milling beads shorten the necessary grinding time significantly [1]. A higher flow rate through the mill results in the usually employed circuit grinding mode in a considerably shorter grinding time as well [2].

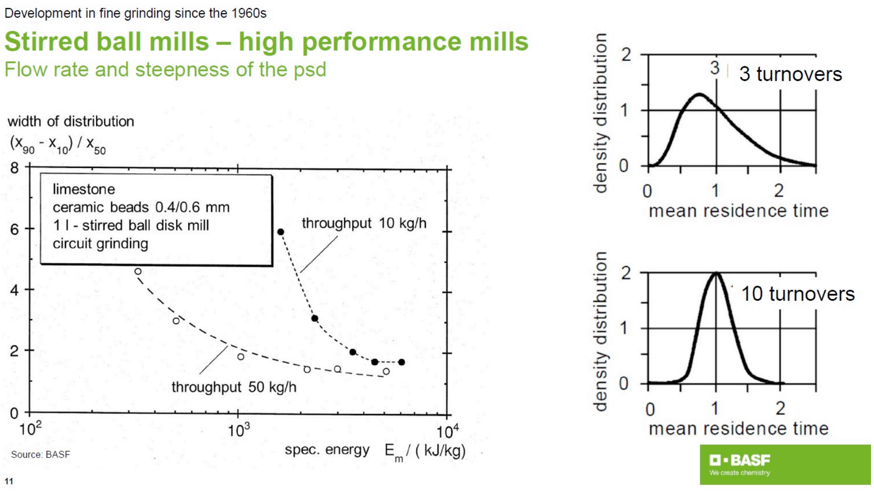

Figure 1. Flow rate and steepness of residence and particle size distribution in circuit grinding mode

Figure 1 shows on the right hand side an example of the residence time distribution of a stirred ball milling in circuit grinding mode after the same milling time. The high flow rate (10 turnovers) shows a significantly steeper residence time distribution which is coupled with a steeper particle size distribution, left hand side. Plotted is the dimensionless width of the particle size distributions $(d_{90} - d_{10})/d_{50}$ against the specific grinding energy for a lab scale grinding. The plot shows that product specifications like a d_{80} will be reached much faster at high flow rates avoiding the energy-intensive generation of unwanted fines. Consequently, high performance stirred ball mills were developed in the late 80s and early 90s that can employ smaller milling beads and allow high flow rates around 4 times higher than with stirred ball disk mills. This cuts milling costs and enhances the capacity of a milling line significantly. The trend is to even steeper particle size distributions.

In the last years a need for a more efficient **wet pre-grinding** revealed. The pre-grinding with wet rotor (colloid) mills existing since decades usually does not grind fine enough to permit the use of more efficient small milling beads < 0.5 mm for true comminution because the pre-ground suspensions contain bigger particles that cannot be destroyed by smaller beads resulting in screen clogging and mill failure. Two manufacturers have developed pre-grinders based on stirred ball milling recently. These pre-grinders are designed for a high hydraulic load resulting in a high number of turnovers and the use of milling beads around 3 mm. They have been tested successfully at production plants of BASF. They are more expensive than wet rotor mills but free capacity of the fine grinding mills being used for pre-grinding tasks until now.

A new technology for comminution are **laser ablation** and **laser fragmentation.** A high intensity pulsed laser beam generates a plasma on the surface that evaporates material that de-sublimates to nano particles (laser ablation) or shoots nano particles out of the surface (laser fragmentation). Tests showed a wear free generation of pure inorganic nano particles. The costs are high in the range of €/g.

In **dry grinding** at this time new mill types like the classifier mill and the opposed jet mill integrating deflector wheel classifiers into the mill were developed in the 1970s and 1980s allowing the generation of steeper particle size distributions and predominantly grit-free products contrary to e.g. spiral jet mills or rotor impact mills with static classifiers. During the last decades mainly progress in details which may have high impact on the capacity or the product quality like new nozzle designs for jet mills or grinding tool geometries for rotor impact mills have been developed. There is no general trend concerning the fineness of the products because the fineness depends on the targeted application. A trend toward still narrower particle size distributions seems to continue.

In **air classification** a trend to finer and to sharper cuts could be observed. Deflector wheel classifiers with higher tip speeds and dispersing zones have been developed for finer cuts. Cut sizes smaller than 1 µm are usually avoided due to the high adhesive forces in between the particles. In 1992 it was discovered [3] that eddy currents are present in between the lamellas of the deflector wheel resulting in a fuzzy cut that disappears when the tangential velocity of the gas and the particles equals the tip speed. Counter measures like pre-acceleration of the two-phase flow entering the sifting chamber and special lamella design resulted in classifier types allowing a much sharper cut as before.

In **screening** the main developments were the ultrasonics supported screening and the multifrequency screener that allow fine cut sizes < 100 µm making the use of the more cost efficient screening possible for cut sizes formerly reserved for air classifiers.

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KEYNOTES

About the significance to run media milling processes under optimal conditions

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Keywords: stress model, stirred media mill, optimization,

Media milling is a widespread unit operation in dry and wet grinding applications for many types of products. The product specifications range from nm- to mm-scale and from soft organic compounds like pharmaceutics, foods or agricultural products to very hard and abrasive materials like carbides or oxides. Especially wet operated stirred media mills are used often as they deliver a very broad range of power densities from very low values for soft grinding or dispersing applications to very high stress intensities for ceramic grinding. They are available from volumes of several ml up to m^3-scale, with working principles in batch, semi-batch or continuous working modes and a high variability in geometrical variants.

Questions regarding scale-up or transferring results from one mill type to another are hard to answer. The origin stress model introduced by Kwade [1] delivers a tool to optimize the energy consumption of a milling process with a fixed mill and a fixed product based on experimental results. Scale-up rules for self-similar mills are available. [2-3]

The enhancement of the origin stress model follows the idea to introduce geometric and material dependent parameters to open its usage to a wider range of products and mill types without increasing the experimental effort. The result is an enhanced version of the stress model, which gives in a first instance the possibility to predict optimum stress conditions for materials with known material parameters [4-5]. The geometry of the mill is recognized by using the stress energy distributions first introduced by Stender based on fluid flow assumptions [3].

Upon this first enhancement step for finding optimum conditions a second step follows which has the target to predict the need grinding times for a chosen set of parameters to get the desired product particle size. Therefore, the breakage behavior was proved for values beside the optimum stress conditions and stress or collision number of the grinding media was introduced. Herewith the residence time of the material inside the mill can be estimated.

The third step shows up an easy possibility to connect this residence time the with real grinding time in dependency of the operation mode and with the power consumption of the mill under certain circumstances.

The sum of the enhancements delivers a tool to improve processes with regard to specific energy or throughput (see Fig. 1), to set up new processes, to scale or transfer them or even to decrease the grinding media wear very easily.

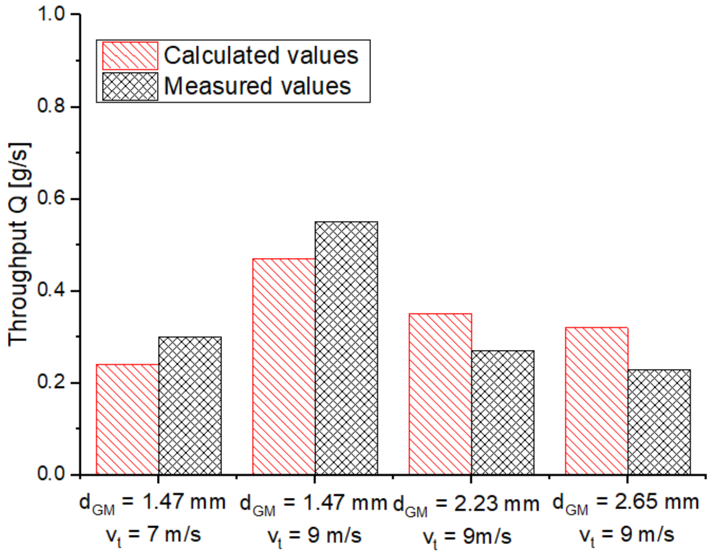

Figure 1. Comparison of measured and with the enhanced stress model calculated throughput values for grinding silica to a particle size $x_{50,3}$=2 µm

Acknowledgement

I gratefully thank my former colleagues at the Institute for Particles Technology of TU Braunschweig, Germany, namely:

Arno Kwade for the deep discussions and the feedback at the different stages of the model, Frederik Flach, David Sterling, Didier Schons and Arne Lüdecke to give me the chance to prove the model in their experimental data and especially Helmut Seeland who performed many of the shown experiments and realized the idea of the changed rotor geometry.

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Analysis of Dynamics of Spiral Jet Mills by Coupled CFD-DEM Simulations

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Keywords: spiral jet mill, DEM, CFD, modelling, dynamics

The spiral jet mill has a simple design and operation procedure, but has a very complex gas and particle flow fields, making it challenging to predict the milled particle size distribution without an empirical knowledge of the system. The particles are in a fast shearing dense phase near the wall, and as their size is reduced, the gas drag entrains them eventually out of the mill. The gas flow pattern changes from forced vortex near the wall to free vortex mid-way in the mill approaching the classifier region. Therefore, there exists a dynamic relationship between particle hold-up and the gas flow, as well as an interdependence between breakage and classification, all occurring in the same chamber. To provide a predictive capability, the dynamics of spiral jet mills has been analysed by coupled CFD-DEM. The role of particle hold-up and how it affects both particle dynamics and gas behaviour are addressed. The kinetic energy transfer from the gas to particles, grinding jet penetration, coarse graining methodology and size reduction simulations are presented.

The dynamics of the jets has been analysed as a function of the grinding gas pressure and particle hold-up. As the grinding gas pressure is reduced, the jets can no longer penetrate through the densely packed bed. This inhibits particles reaching the highest velocities needed to cause fragmentation and chipping.

The greatest challenge posed to CFD-DEM method is the number of particles in the system. The coarse graining method is applied to explore its capability to circumvent the challenge. This method replaces groups of smaller particles by a single larger one. Both the kinetic energy and dissipated energy are modelled successfully for the lowest scaling values of four or fewer particles per group. The predicted behaviour of the particles in the bed agrees with the base case. However, particle velocity in the lean region differs from the original simulation due to the scarcity of particles present in this region.

Finally, the breakage model of Ghadiri and Zhang has been implemented to allow for size reduction by chipping. By recording the mass of fines produced as a result of chipping as a function of time at various gas pressures, the total fluid expended work leading to breakage has been estimated. The methodology developed here provides a capability of predictive milling for various applications based on the mechanical and physical properties of single particles.

Selective fragmentation of primary and secondary resources

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Keywords: provide four keywords separated by commas.

Due to growing population, urbanization, digitalization and transition to clean energy, the demand for raw materials (metal ores and industrial minerals) is facing a steep increase over the past twenty years and will continue to grow globally. Satisfying this increasing demand will require addressing several challenges. In particular, access to the resources is critical. Since high-quality ores are being exhausted, it is crucial to develop new technologies for an efficient processing of low-quality ores and of secondary resources especially related to their comminution. Indeed, comminution, which is the first step in mineral processing, providing qualified material for the separation operations, uses about 1.8% of the generated electrical energy in the world [1]. An increase of the energy consumption of the comminution operations is expected in the coming decades due to the processing of ores of lower quality which have lower head grades, meaning processing a higher tonnage to produce the same amount of product, and are sometimes more resistant to breakage.

Currently, comminution is mainly performed through the use of mechanical techniques which are not energy efficient. For example, the energy efficiency (defined as the ratio of the energy of the new surface created during size reduction to the mechanical energy supplied to the machine performing the size reduction) of a tumbling and a ball mill is estimated to be about 1% [2]. Moreover, these techniques are not selective which means that the whole material is ground uniformly. In addition to a high energy consumption, this leads to several issues since it usually requires fines grinding for the liberation of the particles of economic values which leads to losses in the tailings and to questions around tailings management. Several innovative selective fragmentation methods are been studied for improving the processing of primary and secondary resources. These methods are usually based on anisotropic properties of the ore, usually related to varying properties of constituent minerals (e.g. mechanical, electrical or acoustic properties, thermal expansion). Two examples are Electric Pulse Fragmentation (EPF) and microwave embrittlement. EPF exploits differences in electrical and acoustic properties of component phases in a rock to selectively fragment them while microwave embrittlement is based on the selective effects of internal heating on different mineral phases when submitted to microwaves which create mechanical stresses due to differential thermal expansion. The potential of these methods was demonstrated for the fragmentation of primary and secondary resources. In this keynote, these two innovative fragmentation methods will be presented and examples of case studies investigated at BRGM will be given.

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ORAL COMMUNICATIONS

Formulation Screening, Parameter Optimization and Scale-up of Grinding Processes with Agitator Bead Mills

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Keywords: Wet grinding, Screening, Scale-up, Agitator bead mill

The challenges in the development of new products are the screening of the formulation, the optimization of operating parameters and the scale-up to the production scale. Regardless of whether it is a paint, a coating, a technical ceramic or a pharmaceutical active ingredient, the goal is to be able to realize the development with a minimum amount of the partly expensive material and in a short time.

For example decreasing particle sizes and, at the same time, increasing specific surface area of APIs (active pharmaceutical ingredients) lead to an increase in dissolution rate and bioavailability of the drugs and, therefore, a better performance in the human body. Oftentimes, the dose of poorly soluble drug substances can be reduced which leads to less side effects and lower costs to the patient.

The main challenges for process development from initial studies to the final market launch are the prize as well as the availability of the APIs in the earliest stages of the development.

In this contribution the influence of essential operating parameters for wet agitator bead milling and their optimization will discussed. Furthermore, the transfer of process for formulations developed on the smallest laboratory scale to the production scale will described.

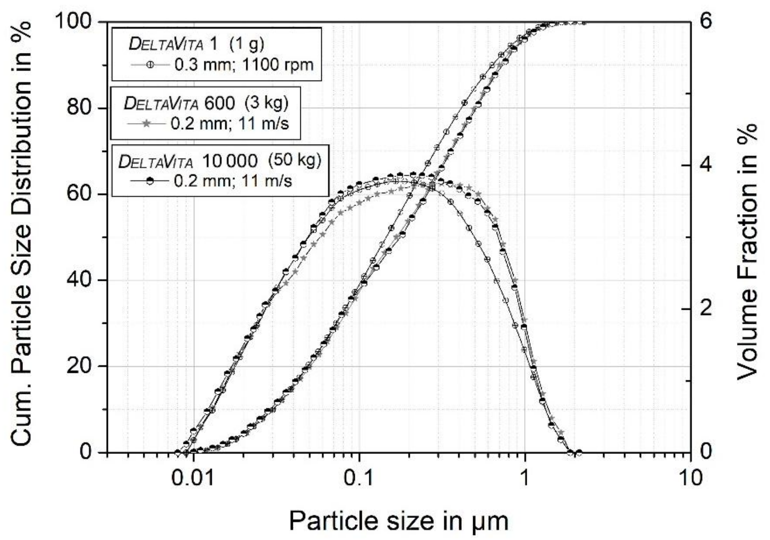

Figure 1. Comparison of grinding results with batch sizes of 1 g, 3000 g and 50000 g of the same model API formulation

Tailoring hydroxyapatite suspensions by stirred bead milling as a pre-processing step for additive manufacturing technologies

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Keywords: hydroxyapatite, ceramic suspension, wet milling, powder bed fusion

Hydroxyapatite (HA) bio-ceramic is a well-known material in bone tissue engineering and bone defect repair due to its good biocompatibility and bioactivity. The use of additive manufacturing (AM) technologies would allow the production of patient-matched scaffolds with defined and reproducible internal structures [1]. For the powder bed fusion (PBF) category of AM technologies, such as the selective laser sintering (SLS/M) process, a powder feedstock is used to produce a homogeneous powder bed prior to laser scanning. It is preferably achieved by flowable spherical spray-dried aggregated particles of a few tens of microns (30–70 µm) with a certain polydisperse distribution [2]. In a spray-drying process, higher heat transfer efficiency (resulting in the lowest moisture content of the granules) and more solid, spherical, and dense granules are obtained when using highly concentrated suspensions containing ceramic primary particles having an x_{50} commonly in the range of 0.2 to 1.5 µm [3–6].

Since as-synthesized powders usually do not meet these required sizes, the efficiency of using a stirred bead milling process to tune the hydroxyapatite particles size was investigated. The control of the suspension formulation was also of special relevance since higher solid concentration can lead to an increase in the slurry viscosity. If the viscosity is too high, it can cause the grinding media to be stuck on the surface of the grinding chamber affecting the comminution of the particles and the fineness of the obtained particles [7]. It could also bring handling difficulties during the spray-drying process itself, especially during the pumping and mixing of the slurry [8]. The successful spray-drying of ceramic slurries having viscosities up to 0.1 Pa.s has been reported previously [3].

In this communication, the production of spherical powders with a correct flowability through a multi-step process (Figure 1) involving synthesis, milling, spray-drying, and mixing with absorption additives steps will be briefly presented. However, the present work focuses on the wet grinding conditioning of ceramic suspensions prior to spray-drying process. The characterization of slurries stability (through rheological behavior, zeta potential, and morphology measurements), as well as powder chemical and structural properties (SEM, UV-VIS spectrophotometry, FTIR, and XRD), was decisive to produce and study the different process feedstocks.

A Darvan C dispersant agent dosage of 2.1 $mg.m^{-2}$ (active component) to the specific surface of the ceramic was found to be the most outstanding dosage for the stabilization of the dispersion. Milling chamber, bead wear, and energy consumption were evaluated and minimized, finding the most efficient operating parameter setting for the process (Figure 2). For the studied material, the use of a fast stirrer speed, smaller grinding media size, and higher solids concentration increased the energy efficiency. This work confirmed the presented laboratory-scale milling process as a promising method to study the processability of hydroxyapatite-filled slurries, as a first-step product, on the manufacture of hydroxyapatite microspheres for the additive manufacturing industry, more concretely powder bed fusion technologies.

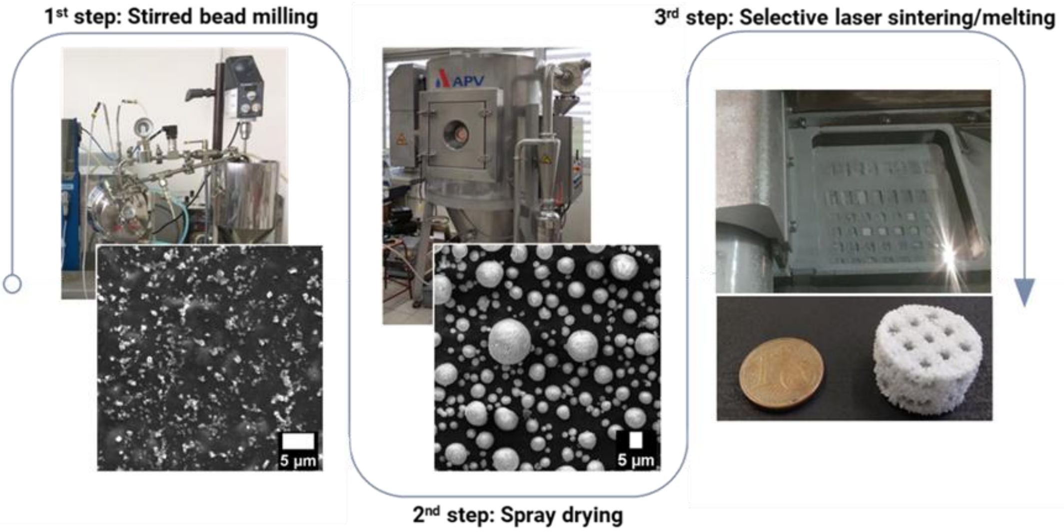

Figure 1. Multi-step process for the production of HA powder feedstock for PBF

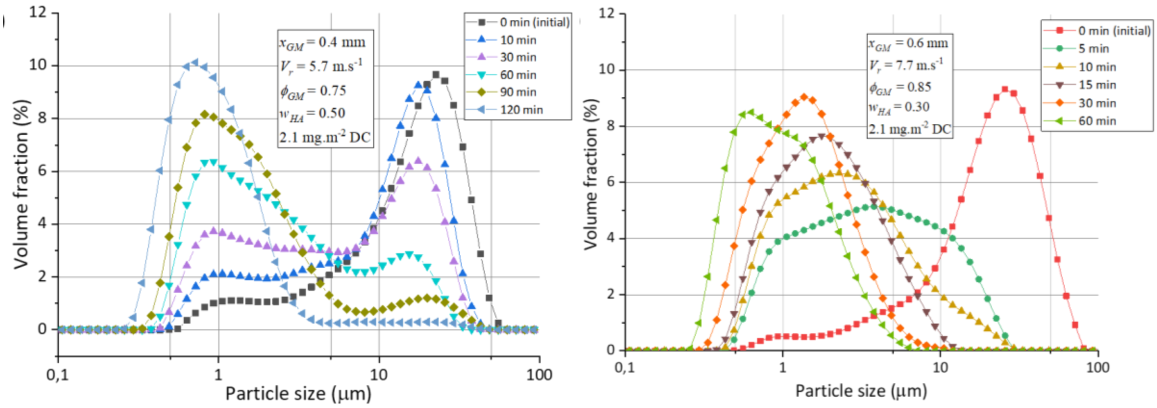

Figure 2. Evolution of PSD of a HA slurry with the grinding time for two different sets of parameters. x_{GM} = Grinding media size, V_r = Stirrer speed, ϕ_{GM} = Chamber filling ratio, w_{HA}= Solid mass fraction, DC = Darvan C dispersing agent.

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Energy-efficient solution in ultra-fine grinding of ceramic raw materials

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Keywords: fine grinding, energy efficiency, ceramic raw materials, stirred media mill.

In many industries an increasing demand for finer and steeper particle size distributions of raw materials can be observed. This is specifically true for the raw materials for technical ceramics, such as Al_2O_3, glass, ceramic pigments and other hard materials. This is due to the fact, that the granulometric characteristics of the powders affect the properties of the final product. For many applications, e.g in electronics and sensors, very fine particle sizes in the range of a few microns only, are required.

Since most of these materials are abrasive and the applications require very low levels of metal contamination, wear protection plays an important role for the utilized mills, classifiers and other equipment. Moreover, these applications are very cost sensitive and therefore, investment and operation costs, and in particular the specific energy consumption are important factors for the selection of suitable comminution and classification techniques.

In the past, these requirements could be met by the utilization of wet grinding processes, which demands a number of process steps, such as slurry preparation and storage, grinding in stirred media mills, drying, deagglomeration etc. As an alternative, dry processing in fluidized bed jet mills could be utilized, which results in simpler process technologies but requires a very high specific energy consumption.

To overcome these issues, the PULVIS stirred media mill system with integrated classifier was developed. The system is suitable for the dry production of very fine powders in the fineness range of $x_{97} < 10$ µm with steep particle size distributions and has various equipment options to optimally adapt the machine to the requirements of the product. It is very compact and consists – besides the mill – of a dosing unit, a filter with product discharge and two fans for the process gas but has a clearly lower specific energy consumption compared to a fluidized bed jet mill. Although the system has proved its ability in many applications, it was now further improved. The combination with the ATP-PRO classifiers, which are wear protected, ensures very fine cut sizes at very low pressure drops [1]. Thus, the energy consumption of the fan used to convey the process gas and material through the system is reduced considerably.

In comparative trials with a PULVIS and a fluidized bed opposed jet mill, it was shown that the specific energy consumption of the PULVIS was up to 80% lower. As a result, the operating costs of the PULVIS are correspondingly lower than those of the jet mill. Depending on the material and the used set of process parameters, a maximal product fineness x_{97} of approx. 1 µm can be achieved with the PULVIS. This was proven for instance for aluminium oxide, as can be seen in Figure 1 that shows the particle size distributions of the feed material and the ground Al_2O_3.

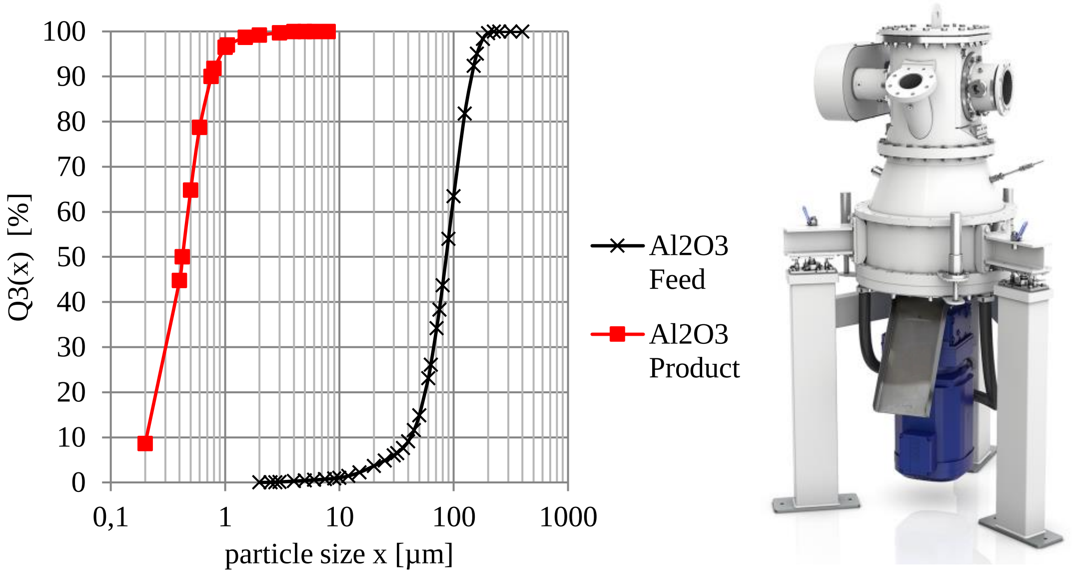

Figure 1. Particle size distribution (left) of a feed material and an aluminum oxide ground on the PULVIS 600 (right)

The other advantage of the PULVIS is the wide variety of different classifier variants available. Depending on material properties and process requirements such as fineness, wear, and throughput, an ATP, an ATP-PRO, or a TTD classifier can be used. Whereas the ATP ceramic classifier can be used for very abrasive products and the TTD classifier can be used for very fine products.

To further improve the performance of the PULVIS plant, the mill can also be equipped with multi-wheel ATP ceramic classifiers or with multi-wheel wear-protected ATP-PRO classifiers. Multi-wheel classifiers, compared to single-wheel ATP or ATP-PRO classifiers, allow the production of finer low-contamination products at similar throughputs or the production of significantly larger throughputs at similar fineness.

Reference tests with aluminium oxide using a PULVIS 600 with an ATP-PRO multi-wheel classifier and an ATP-PRO single-wheel classifier showed that throughput could be increased by 61.4%. At the same time, the required specific grinding energy was significantly reduced by 46 %. In both trials, a product fineness of $x_{97} = 2$ µm was achieved. Additionally, it was found that the particle size distribution of the product processed with the ATP-PRO multi-wheel classifier was steeper.

It can be concluded that the PULVIS is a machine with many unique selling points compared to the current state-of-the-art grinding processes for ultra-fine grinding of abrasive hard materials.

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Wet comminution of engineering thermoplasts in stirred media mills

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Keywords: wet comminution of polymers, thermoplast microparticles, grinding limit, grinding media wear

Comminution of plastic or visco-elastic materials like polymers is known to be energy-intensive and costly. The mass-specific energy required for size reduction of materials with visco-elastic or plastic deformation behavior typically is one to two orders of magnitude higher than for brittle matter [1]. Often, impact mills, cutting mills and jet mills are applied for comminution and the feed material frequently is pre-cooled using liquid nitrogen or solid carbon dioxide. Typically, minimal product particle sizes in the range of several 10 microns typically can be obtained with the aforementioned devices. Production of very fine polymer particles of less than 10 microns size by dry grinding methods is challenging for amorphous thermoplasts like styrenes or acrylates and almost impossible for engineering and high performance thermoplasts like, for example, polybutylene terephthalate (PBT), polyoxomethylene (POM) or polyaryl ether ketones (PAEK). Although, polymer microparticles are conveniently accessible by a wet comminution approach [2, 3]: in case of amorphous polystyrene particle sizes $x_{50,3}$ as small as 2 microns may be obtained. Fine microparticles of engineering or high performance thermoplastics are of high interest for the modern manufacturing methods of industry 4.0, i.e. additive manufacturing (AM) processes, like laser powder bed fusion of polymers (PBF-LB/P) and may be obtained by wet grinding as a feed material for a process chain [3, 4] allowing for novel feedstock powders of good flowability for PBF-LB/P. Within this contribution, wet comminution of engineering thermoplasts in stirred media mills will be discussed. In PBF-LB/P, besides flowability the thermal properties of the thermoplast, i.e. its melting and solidification temperature as well as the melt crystallization kinetics are of immanent importance, thus, damage of the polymer during comminution and contamination of the product with grinding media wear should be minimized. The grinding products are thoroughly characterized by vibrational spectroscopy, X-ray diffraction and dynamic scanning calorimetry. The dependency of the grinding result on fundamental process parameters, i.e. stressing conditions such as e.g. stirrer tip speed or grinding media size as well as on the process time, process temperature and the system composition will be discussed with a focus on energy efficiency and minimal grinding media wear [5]. Moreover, experimentally determined grinding limits are discussed in the context of the mechanical properties of the thermoplast and the process chain approach [3, 4] is briefly sketched.

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Sequential multicomponent comminution of anode slurries for lithium-ion batteries

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Keywords: stirred media mill, multicomponent comminution, anode slurry, lithium-ion battery

In the context of the energy transition, research into lithium-ion batteries is becoming increasingly important, as these are seen as a key technology for electromobility, consumer electronics and stationary energy storage systems. One focus of current research activities is the investigation of silicon-containing anodes, as these enable higher volumetric energy densities compared to purely graphite-based anodes. The disadvantage of using silicon is a strong volumetric expansion during the charging process, which can only be eliminated by using nanocrystalline silicon. A major challenge at present is the efficient and cost-effective production of the silicon-containing anode slurries. The conventional production process consists of an upstream nano-grinding of the silicon and a downstream dispersion of nano-sized silicon, graphite, carbon black and binders in parallel working batch mixers, which is associated with high personnel and infrastructure costs. Furthermore, the scalability of the dispersion process step is severely limited due to the machines used.

To counteract these limitations, a new process route has been developed as part of a current research project, which enables the integration of the nano-silicon grinding and the dispersion of the other slurry components in the form of a sequential multi-component comminution. The aim of the project was to design a process route that has a high scalability for industrial applications and allows higher throughputs than standardised processes with the same product quality. The designed process route is shown in Figure 1 and comprises the following process steps:

First, silicon is wet comminuted in ethanol to an average particle size of 120 nm using an established batch grinding process in a stirred media mill [1]. The other anode components such as graphite, conductive carbon black and binder are subsequently added to the nano-silicon slurry and dispersed. The main challenge is to efficiently fracture the highly agglomerated carbon black, whereby the stress energy must not be set too high in order to avoid further comminution of produced nano-silicon and graphite, as an overgrinding has a negative effect on the performance of the anode. For the selection of suitable process parameters, findings from multi-component grinding in stirred media mills [2] as well as experimental data on particulate fracture strength were used. The particle size distributions of the discontinuous reference process were successfully approximated by means of a detailed process study in which the number of passages through the stirred media mill and the volume flow rate of a single-passage process were varied (cf. Figure 2). Compared to the reference process, the sequential multi-component comminution enables an increase of the throughput on a laboratory scale from 0.44 kg/h to 2.18 kg/h with comparable product quality, which is confirmed by the electrochemical characterisation of the anode slurries. Furthermore, it offers the potential of an overall more energy-efficient anode slurry production, as essential steps of the reference process, such as the dry mixing of reactants, are omitted. Corresponding investigations are currently being carried out.

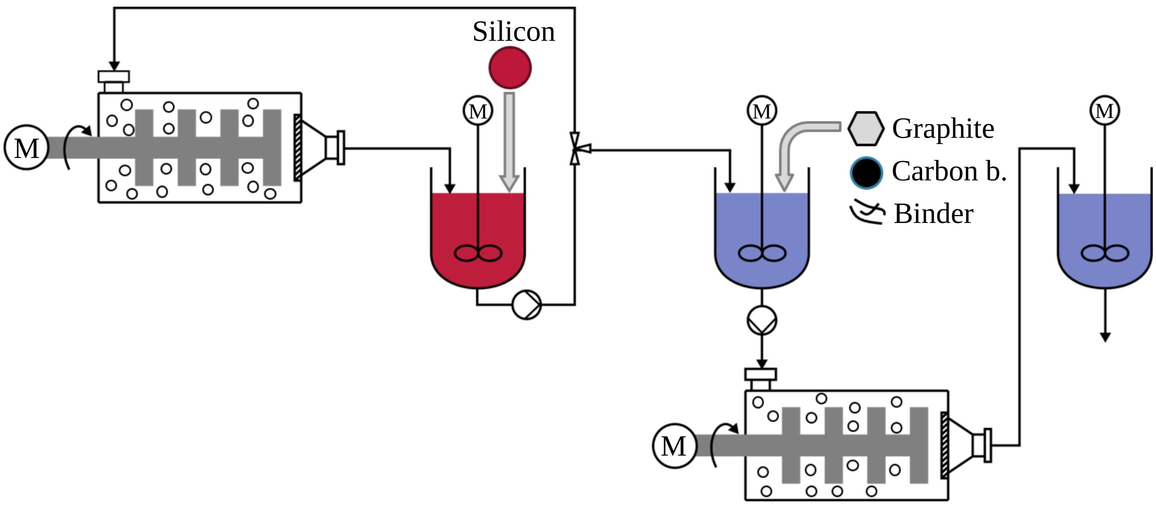

Figure 1. Process diagram for sequential multi-component grinding of anode slurries in stirred media mills.

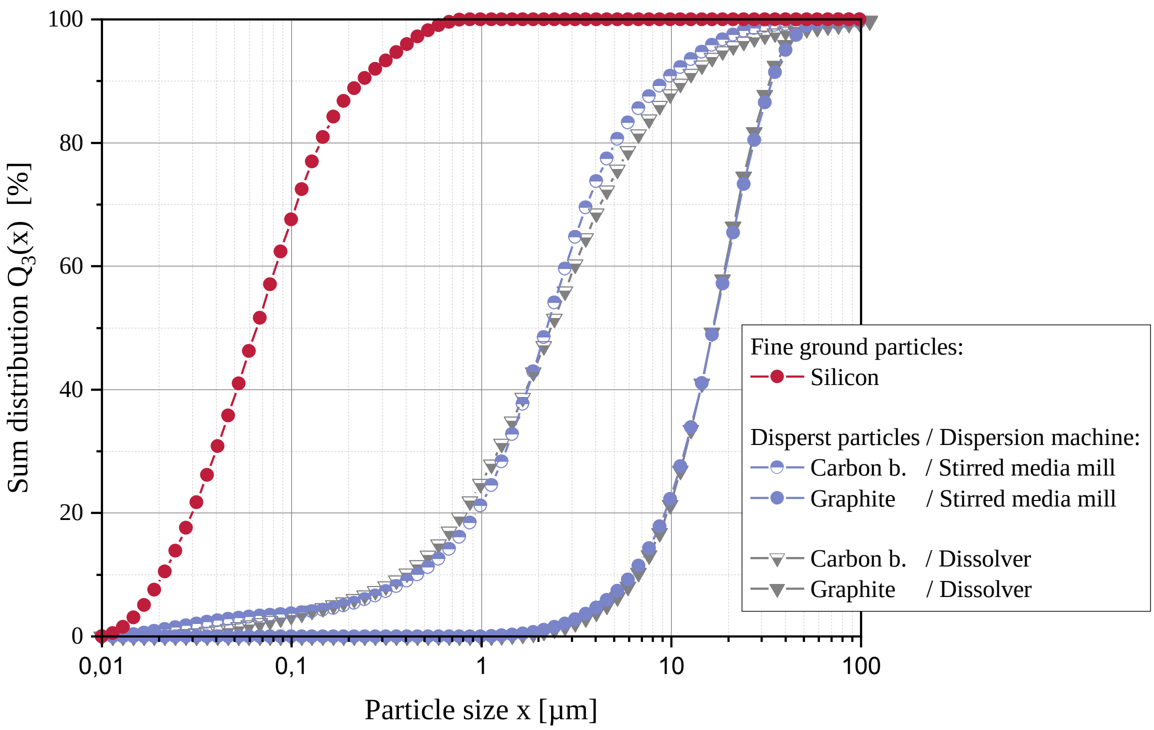

Figure 2. Particle size distributions of silicon, carbon black and graphite of anode slurries depending on the dispersion machine used.

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Wet nanomilling of drug materials using a Zr-complex as stabilization mechanism

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Keywords: wet milling, Naproxen, grinding limit, electrostatic stabilization

A high number of drug candidates developed during the last decades exhibit poor bioavailability due to their sparingly solubility in water. Size reduction of drug particles to sub-µm or even nanometer range by wet milling is an effective and scalable method to achieve enhanced oral bioavailability due to higher dissolution rate [1,2,3,4]. Traditionally, surfactants, polymers or combinations of both are used as milling additives in drug formulations [5,6]. These stabilizing agents, however, do not only prevent agglomeration of the drug particles, they also increase the solubility of the drugs. This leads to ripening and recrystallization of the milled drug particles.

Recently, we produced stable drug nanoparticles by antisolvent precipitation using complex formation between ZrO^{2+}-ions and carboxylic acid groups of the drugs as an alternative stabilizing mechanism [7]. In this contribution, we transfer this unconventional stabilizing agent to stirred media milling of carboxylic acid containing drugs like Naproxen or Ibuprofen. Using this approach, Naproxen particles with a primary particle size of 29 nm are produced under optimized process conditions. X-ray diffraction studies show that the product consists of monocrystalline nanoparticles. In contrast to that, if polyvinylpyrrolidone (PVP), a conventional stabilizer is used, the minimum particle size that can be obtained via comminution is 50 nm. Increased suspension viscosity in PVP/Naproxen mixtures and therefore dampening of the milling bead motion was excluded, indicating that the increased efficiency of size reduction can be explained by the strong electrostatic interactions between the carboxylic acid groups of the particle surfaces and the highly charged metal ions. The ZrO^{2+} to drug concentration ratio must be in an optimum range to provide excellent electrostatic stabilization and to avoid too high drug solubility and de-stabilization. Moreover, as long as quasi-perfect colloidal stability is provided, the grinding limit is not a function of stabilizer concentration but solely depends on intrinsic material properties. Varying the size of the milling beads and therefore the stress energy (SE_{mb}) and the number of stress events (SN), we found that small milling beads are favorable for product fineness and nanoparticle yield, indicating that a high number of stress events is critical for nanomilling in comparison to a high stress intensity in combina-

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tion with low stress number. It should be noted, that the threshold SE_{mb} must be above a critical value to achieve significant fracture. X-ray diffraction, Raman scattering and differential scanning calorimetry (DSC) indicate some loss of crystallinity and substantial lattice strain. Nanomilling was previously investigated for inorganic materials [8,9] and becomes now also available for the production of Naproxen nanoparticles. Nanomilling of organics may thus become a promising method for the manufacture of pharmaceutical solids of high quality and with increased bioavailability. Future investigations include the transfer of the developed method to other drug systems.

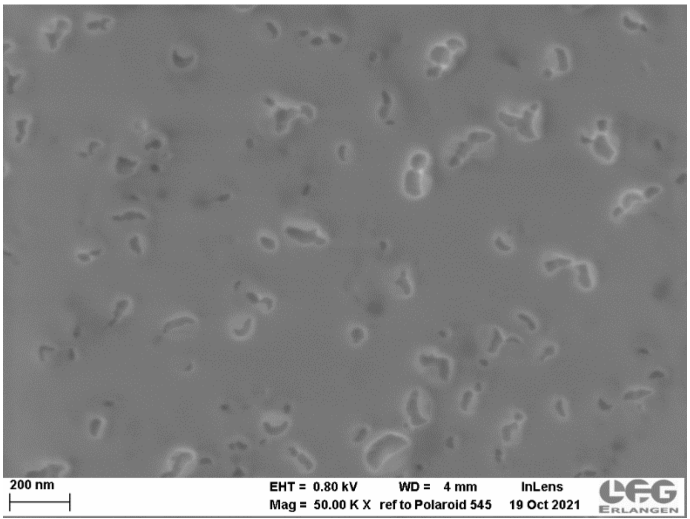

Figure 1. SEM image of the wet milled Naproxen nanoparticles with irregular geometries.

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Influence of grinders loading modes on the dissociation and hydration properties of wheat brans

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Keywords: Wheat bran, grinding, electrostatic separation, hydration

Wheat bran is a co-product of the conventional roller milling process. Bran has a multi-layered composite structure (testa, pericarp, aleurone and residual starchy endosperm), whose different tissues present contrasting physicochemical and mechanical properties. The different tissues are characterized by distinct compositions. Some of them contain molecules with interesting nutritional and functional properties [1, 2, 3]. Currently, bran is mainly intended for animal feed purposes, principally due to the difficulty to separate the high nutritional value tissue (aleurone layer) from the low nutritional ones (pericarp). In particular, the aleurone layer contains many bioactive compounds like vitamins, as well as about half of the minerals and a high protein content. There is strong interest in separating wheat bran into different purified fractions that could be used as ingredients and incorporated directly into food products for human consumption or from which bioactive compounds can be extracted.
Incorporation of bran in cereal-based products receives more attention these last years. Most of the studies investigated the incorporation of whole common wheat bran under the form of fine powders into wheat-based food [4, 5, 6]. It has been reported that the milling process influences the rheological properties of the dough, the quality of the final food products and the consumer acceptance [7]. This could be explained by differences in water retention capacity related to the size, and dissociation of the different layers constituting the bran. In particular, the dissociation state among the bran layers in ground common wheat bran can be evidenced by electrostatic separation after milling [8]. In the bran, different binding strengths between bran components and water molecules can be distinguished. Recently, a distinction was made between weakly bound water in bran, *i.e.* water between stacked bran particles and in micropores of empty pericarp cells and strongly bound water in bran, *i.e.* water bound in cell wall nanopores and through hydrogen bonding [9]. However, the relation between the milling process used, the state of dissociations of the different bran layers and the molecular impact on hydration water in bran was not yet elucidated.

The aim of this study is to investigate the influence of the loading modes (high shear grinder and impact grinder) generated by two different grinders on the hydration properties and dissociation of the common and durum wheat brans. An original study at the molecular scale to target the distribution and the intensity of the bonds of hydration water was carried out by gravimetric (sorption isotherms) and spectroscopic (Fourier transform infrared spectroscopy, low field NMR) methods. Results were analyzed in regards to the particle size and shape, the dissociation state of the sample evaluated by electrostatic separation, biochemical composition and multispectral imaging (see figure 1).

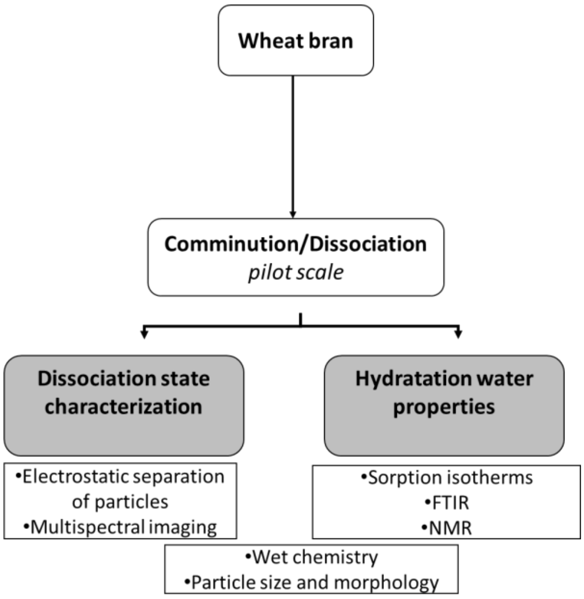

Figure 1. General strategic study plan of the impact of grinding destructuration processes on tissue dissociation and hydration water properties of common and coarse durum wheat brans.

Our study highlights that durum wheat bran has lower water sorption capacity than common wheat bran in relation with neutral sugars content. Based on sorption isotherms and FTIR measurements, we demonstrate that impact grinder has a stronger effect on sorption capacity and FTIR multimer water at 3600 cm^{-1} of durum wheat bran and induces a high decrease in residual starch crystallinity degree. On contrary high shear grinder tends to increase the proportion of water strongly bound. Electrostatic separation has been shown to be more effective for common wheat bran with a significant enrichment in aleurone layer in the positive fraction. This has been related to a better dissociation rate. As for common wheat bran a correlation is found between lower dissociation efficiency and less interactions between hydration water and the bran's backbone as found by NMR low relaxation times T_2 of most mobile protons. This study allowed to have a better mechanistic understanding of the importance of intrinsic hydration water interaction in ground wheat bran. This brings new elements to optimize the processability and the quality of cereals products enriched in bran and converges towards a better valorization of common and durum wheat bran with fractions enriched in compounds of interest (fibers, vitamins and minerals).

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Control of cell destruction and proteins extraction of *Tetraselmis suecica* by stirred-bead milling: correlation of process parameters to cell lysis

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Keywords: Microalgae, Mechanical cell destruction, Stirred bead milling, Proteins

Microalgae are single-celled organisms of interest for a wide range of applications: biofuels, human food or animal feed. These microorganisms are currently considered as one of the solutions to meet the high demand for proteins caused by the expected growth of the human population in the coming decades. *Tetraselmis suecica* is a green marine microalgae of 10µm covered by a cell wall relatively resistant containing 35-38% DW (w/w) protein. To produce these proteins on an industrial scale and in order to become competitive, a biorefinery process must be designed. This usually includes the steps of cell destruction and extraction of the target molecules. Cellular destruction can be defined as the loss of membrane or cell wall integrity, resulting in the release of intracellular contents [1]. The main objective of this study is to evaluate the effect of various stirred-bead milling parameters and how it affects the cell destruction of a fresh *Tetraselmis suecica* cells suspension cultivated as a biofilm.

A Labstar stirred-bead milling from Netzsch, Germany was used for the comminution experiments. It consists of a 0.6 L grinding chamber with a double jacket in which the cooling fluid circulates to prevent overheating during the experiment. Stirred-bead milling were operated under two modes: recycling mode or pendular mode [2] with 1 to 3 passes. For each running mode, the effect of some operating parameters was analysed: bead size, chamber filling volume and stirring speed, as well as the grinding time for the recycling mode and the number of passes for the pendular mode. Each treated sample lysate was clarified by centrifugation. The cell disruption level was studied by combining four evaluation methods on the pellet and the supernatant: recovery of proteins by elemental analysis for extraction efficiency, laser diffraction granulometry for cell wall fragment sizing, optic microscopy for viable cell count and scanning electron microscopy for morphological analysis of the cells, before and after each mechanical treatment. Protein extraction efficiency and energy consumption were also compared to other mechanical cell destruction methods: high-pressure homogenization and ultrasound.

Microscopic observations for the three mechanical processes show several kinds of cell destruction. Indeed, SEM observations coupled with laser granulometry show that cells do not fragment in the same way depending on the method used. Fig. 1 shows, as an example, the size distribution of *T. suecica* suspension using ZrO2(Y) beads with a diameter of 1 mm beads, a bead filling volume of 85% and a stirrer speed of 2000 rpm for various milling times (recycling mode). Three size-populations are observed for each milling time: aggregates with a peak centered at 100 µm, individual cells at around 10 µm and cell wall debris with a size of around 1 µm. Increasing the grinding time causes cell disaggregation and increases cell destruction leading to the formation of a majority of cellular debris (1 µm). SEM images show that 30 minutes of milling results in the disappearance of intact cells in favor of shredded cells.

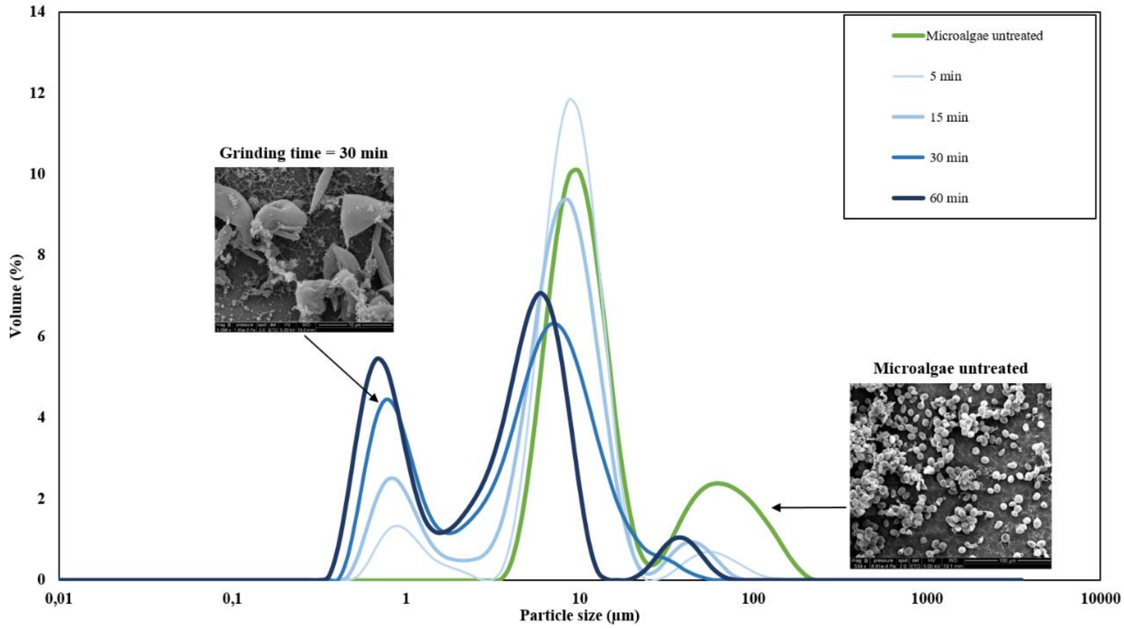

Figure 1: Example of Particle size distribution of Tetraselmis suecica cells before and after 60 minutes of stirred-bead milling under standard conditions. SEM images show the morphologic aspect of cells after the mechanical treatment.

Overall, the high-pressure homogenizer and the stirred bead mill appeared to generate cell debris around 1 µm in size, whereas a milder ultrasound treatment permeabilised the cells without necessarily causing complete cell damage. It was also observed that the protein yields were not the same depending on the process: for the high pressure homogenizer a single pass at moderate pressures (20 to 40 MPa) makes it possible to recover up to 80% of proteins, by damaging the cell membrane, without achieving full rupture of all *T. suecica* cells. Using the stirred bead milling process under standard operating conditions such as those used for the run shown in Figure 1, an equivalent yield of 80% was achieved after 30 minutes when milling 2 liters of cell suspension containing 10% of dry matter. Slightly lower maximum yields, around 70%, were obtained with ultrasonic treatment. This study revealed the possibility to associate operating conditions with several levels of cellular destruction, allowing a better control for the next steps of the downstream processing.

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How does milling impact the aspect ratio of miscanthus stem particles ?

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Keywords: particle, elongation, grinding, lignocellulose

The increasing demand for recyclable and biodegradable materials has led to a growing interest in the use of natural fibers in technical composite applications. Natural fibers have been known to decrease the environmental impact of composite materials and to lightweight them strengthening their interest for automotive, building and construction sectors. However, the word "fiber" can take many different meaning and many plant organs have been studied: from pure bast fibers extracted from fiber plants (e.g. flax, hemp, or ramie) to particles from whole plant (e.g. miscanthus, cereal stems) or leaves (e.g. sisal, curauá, coir fibers), leading to a large range of plant-based composites performances. Among lignocellulosic crops, miscanthus seems to be a good candidate considering the high agronomic yield of the main cultivated species (Miscanthus giganteus), its good water use efficiency and low intrant requirements [1]. However, miscanthus reinforced biocomposite materials exhibits lower mechanical properties than other plant fiber-composites (as flax or hemp fiber composite). The decrease in mechanical properties could be attributed to the fact that particles are more lignified, exhibits a lower aspect ratio, and has an important proportion of dust [2-4]. The production of tailor-made fibers (length and size) from miscanthus is thus a priority to improve the mechanical performances of such composite materials.

In this study, miscanthus fibers were obtained by grinding the whole plant then sieving the powder in order to select the most suitable fraction for further compounding process. The objective of this work was to establish a relationship between the comminution process and the aspect ratio of the miscanthus particles. In particular, the impact of the dominant mechanical stress and the mechanical work intensity in the grinder were studied**.**

Material and Methods.

Materials. Whole miscanthus giganteus stems were harvested after senescence by Novabiom. Leaves were discarded and internode were manually isolated to be further milled.

Milling technologies. Different milling technologies have been used: knife mill SM100 (Retsch), impact mill (Hozokawa alpin™ UPZ100) and centrifugal mill ZM200 (Retsch) using different screen size selectors (6, 4, 2 and 1mm) to generate different mechanical work intensity.

Particle characterization. The particle size distributions were obtained through mechanical sieving using adapted mesh sizes. The particle morphology was obtained by image analysis using a QicPic (Sympatec) with Gradis dispersion unit. The fibre length (Lefi) and diameter (Difi), and elongation factor (defined as the ratio of Difi to Lefi value) and their distributions were determined using Paqxos software.

Results.

Miscanthus was ground with knife mill, impact mill and centrifugal mill generating different main mechanical forces : shear and cut (knife mill) , impact (impact mill), and a combination of both impact+shear (centrifugal mill) [5]. Different experiments were conducted with screen size selectors set at 1mm, 2mm and 4mm (Figure 1) to generate different mechanical work intensity.

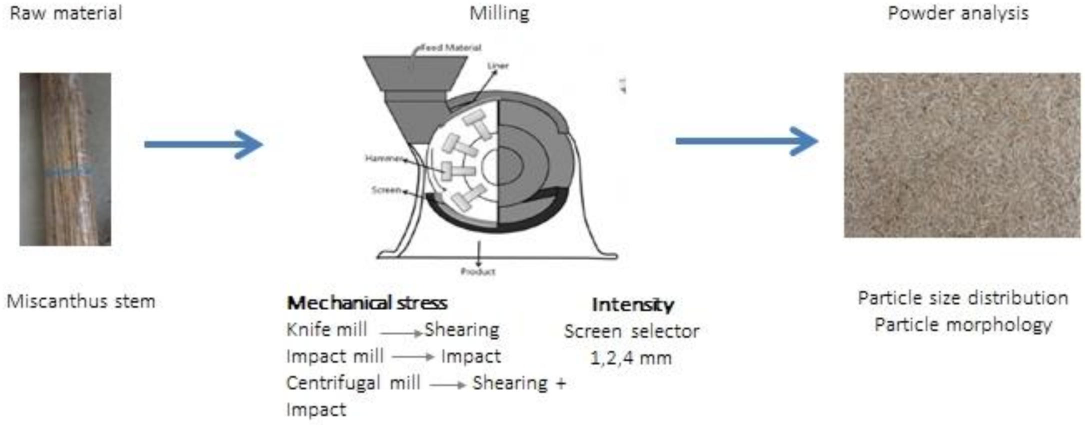

Figure 1. Experimental design

According to screen size selectors used, significant differences in particle size distribution were observed between the milling technologies. Knife mill tended to produce slightly smaller particle sizes and a larger amount of dust (<200µm) than impact mill.
Subsequently, more attention was paid to a sub-fraction, obtained between the 200µm and 500µm sieves, which appeared to be the most suitable for composite polymer applications. As expected the yield of this fine fraction increased as the screen size selector decreased for each type of grinder and reach values above ~40% for screen selector size equal to 2mm or below. The particle morphology of this fraction was studied using image analysis. Whatever the technologies considered, the coarser particles were more elongated. The particle length (Lefi) was more related on the selector screen size (mechanical work intensity) than on the mechanical stress generated by the milling technology. Indeed, even though collected on the same sieve ([200µm; 500µm[) the median particles length and elongation differ according to the screen size selector used. Smaller screen size selectors lead to shorter fiber length and less elongated particles. Thus, milling mainly affect the particle length rather than their diameter. The impact of the mechanical stress was most pronounced with the finest screen size selector (1mm). A medium elongation factor of 0.05 (=aspect ratio of 20) could be obtained for some fractions. The more elongated particles were obtained in the centrifugal mill (combination of shear and impact stress) compared to impact mill (mainly impact) and then knife mill (mainly shear and cut). This could be explained by the anisotropy of the miscanthus stem and the direction of the application of the constraint. In a knife mill, shearing is applied both parallel and orthogonal to the direction of the fiber, resulting in shearing but also in cutting of the fiber length. In the centrifugal mill, the centrifuge force drives the particle tangentially to the selector favoriting shearing in the lengthwise of the fiber.

To conclude, our study shows that the aspect ratio of miscanthus particles can be improved using tailored fractionation diagram while keeping acceptable yield of production. Multi scale milling has been shown to be potentially interesting to preserve the initial particle shape and maximize the particle aspect ratio.

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Production of complex glass flakes by mechanical compression in a mill: From single particles to process design

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Keywords: Glass flakes, stirred media mill, Particle processing, micro compression testing,

A major trend in particle technology is the ever increasing complexity of particles in terms of shape and composition. SiO_2-based glass flakes of complex chemical compositions are an ascending material group in research and industry, e.g. biomedical applications include composite membranes of bioactive glass flakes in chitosan [1], protective coatings [2] and flake-like substrates for bone replacement. Complex glass compositions are also discussed for next generation applications as electrodes in batteries. The well-controlled and scalable production of such glass flakes is possible via compression in the liquid phase in stirred media mills [3]. Particle stressing and size reduction takes place between colliding grinding beads, i.e. by two-sided compression [4-6]. The *process function* defines the stressing conditions in the mill in terms of stress energy and stress number while the *material function* describes the reaction of a material to the external stress input [7]. Both functions are highly distributed and in many cases unknown. Remarkably, stress energy distributions in mills (e.g. stirred media mills but also in jet mills) can be determined by recent progress based on stressing of mechanically well-characterized model particles in a custom-made scanning electron microscope (SEM) supported single particle compression device [8-10].
In this contribution, we correlate the deformation of silica-based glasses of different chemical compositions with the stressing behavior in a stirred media mill [3]. Our unique nanocompression technique provides a large dataset of over thousand single particle compression experiments. Shape formation consists of two distinct steps: i.) starting from relatively particle feed particles brittle breakage occurs until the material dependent brittle-to-ductile transition particle size is reached; ii.) then plastic deformation leads to flake formation which can be correlated with the respective single particle experiments. In addition, the formation of the glass flakes in the mill and in the single particle experiments is compared with the stress energy distribution of the respective mill, derived by Strobel et al. [9].
In order to obtain the needed large data set with high precision and high statistical significance within an appropriate timeframe, the compression experiments were conducted in an advanced version of our previously reported SEM-supported single particle compression device [10] which allows to measure mechanical particle properties including Young's modulus, yield strength, elastic-plastic loading index, elastically and inelastically absorbed energy uptake as well as particle strength. Interestingly, the mechanical response can be correlated with intrinsic material composition and properties. This way, our device opens new ways towards the systematic determination of the widely missing mechanical particle properties.

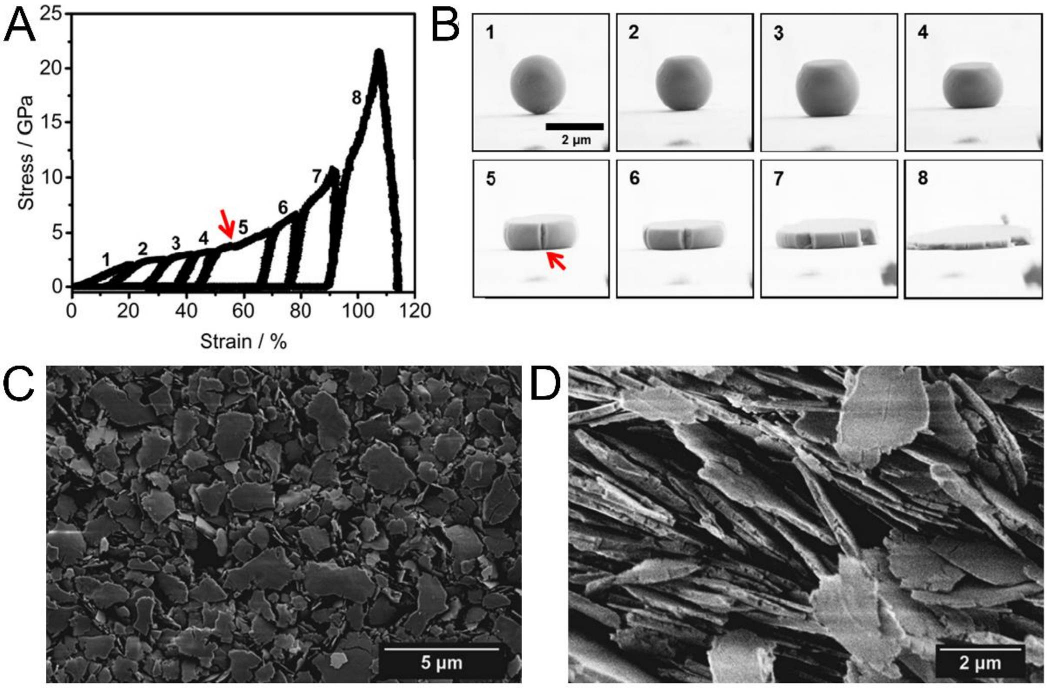

Figure 1. (A) Consecutive Stress-strain curves of a 2 mm soda lime glass particle. SEM images of (B) the corresponding steps of the consecutive stressed particle, (C) top and (D) side view of glass flakes produced by a stirred media mill [3].

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Understanding the Relationship Between the Initial Particle Bed Packing and Compression Comminution

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Keywords: Fuller curve, compression comminution, piston press, packing density, size reduction.

Nowadays, there is an increased interest in the High-Pressure Grinding Rolls (HPGR) technology in the mining industry due to its lower energy consumption than a conventional grinding circuit [1]. Despite the benefits, the high quantity of material required to size the HPGR makes it difficult for new mines to consider this technology. The piston press test has been used in The University of British Columbia to predict the behaviour of the HPGR. It is possible to predict the energy requirements and size reduction utilizing a small amount of material to size the HPGR. However, a throughput prediction model is still missing.

The HPGR throughput depends on several operational parameters, such as the rolls speed and the operating gap. However, the operating gap results from the equilibrium in between the force applied by the HPGR floating roll and the resistance to compression from the material. The resistance from the compression of the material depends on several material properties, such as the moisture content, the fines content, the particle size distribution (PSD), and particle breakage resistance.

The PSD of the bulk material plays an important role in how the particle bed will pack. Fuller and Thompson [2] determined a specific PSD shape that can achieve the highest packed density called Fuller Curve. When a compressive force is applied to a particle bed, the contact points between particles will affect how the energy is propagated and dispersed. As particles break, the PSD also changes, affecting the packing of the particle bed, and thus, the applied energy distribution.

This study investigates the differences of compressing three different sample PSDs obtained from the same copper ore, which was crushed to passing 12.5 mm (Figure 1). The first PSD corresponds to the natural distribution resulting from the crusher. The second is built artificially to recreate the Fuller curve. The third corresponds to a truncated feed, built from removing the fines (-300 µm) from the natural feed. The test is performed utilizing a piston-and-die press test (PPT) apparatus with an 86 mm diameter die. Three pressures of 140, 190, and 240 MPa are applied to 240 ml of sample from each PSDs.

An increase in the final compressed thickness can be seen when using the Fuller Curve feed compared to the natural feed, while the truncated feed compressed less. This result is contrasted in the PSD of each of the products (see Figure 1). The Fuller curve feed resulted in a finer PSD, while the truncated feed resulted in a coarser PSD. The compression of the Fuller curve feed shows to be more energy-efficient in the generation of new fines under 300 µm. The natural and truncated feed are more energy-efficient in generating new particles in the intermediate range (+300 µm -6.7 mm).

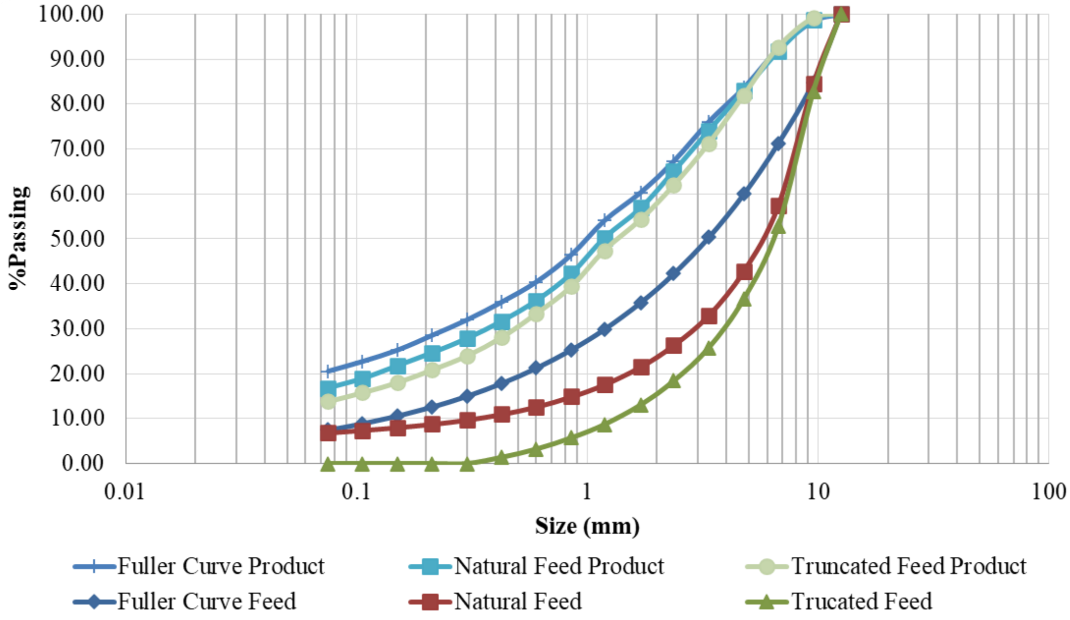

Figure 1. Particle size distributions for each feed used in the piston press testing and their respective products after the 240 MPa compression.

The specific energy consumption of each compression is greatly affected by the feed PSD as well. The finer the PSD of the feed, the less specific energy consumption the compression will have. A 37% lower specific energy consumption was obtained when comparing the Fuller curve feed compression with the truncated feed independent of the pressure applied. Due to the wider and finer PSD of the Fuller curve, the material can pack easier while the compression is being performed. However, a good material packing has an adverse effect on the grindability of the sample. The more supported the particles are in the packed bed, the more energy is dispersed through the particles and not used for breakage. A more efficient packing allows the pressure target to be reached faster, and thus, reducing the energy applied during the compression.

Entirely different behaviours can be expected while compressing different PSDs of the same material. Differences in the specific energy consumption, grinding efficiency and final compacted densities are some of the differences that can be observed. The change of these responses significantly affects the throughput of the HPGR. Future work is required to quantify these responses and correlate them with the behaviour during the HPGR compression.

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Analysis of Particle Impact Damage by Material Point Method

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Keywords: Material Point Method, impact, deformation, mechanics.

Metal and ceramic coatings are commonly developed through methods that require high-temperature processing of materials. These high processing temperatures often lead to complications such as oxidation and adverse structural changes. Therefore, interest in solid-state coating methods like aerosol deposition and cold spraying has increased rapidly over the past two decades. In these techniques, powder particles are accelerated to high velocities by a supersonic gas jet and impinge on a substrate. When the impact velocity exceeds a material and process-dependent critical velocity, the particles deform and adhere to the substrate, building up a thin functional film. Deformation behaviour of the impacting particles plays an important role in their effective adhesion to the substrate and consequently, the quality of the deposited film. In this work, the deformation mode of the particles as a function of their mechanical properties is analysed. Material Point Method (MPM) is used to simulate the impact of particles with different ratios of Young's modulus to yield strength. Contours of strain in the vertical direction and plots of energy versus time show that as the impact velocity increases, the particles undergo more noticeable plastic deformation. Also, qualitative observation of contours of strain suggests that as the ratio of Young's modulus to yield strength increases, the vibrating motion of the particle due to elastic waves disappears and plastic deformation becomes the dominant deformation mechanism.

Influence of rheological behavior of lactose powders on the weight consistency of tablets

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Keywords: powder, characterization, pharmaceutical, rheology, tableting

The processability of pharmaceutical powder plays a key role in the design and improvement of production processes for oral delivery systems (e.g. tableting, capsule filling). To control and to optimize processing methods, material properties and behaviour of bulk powder should be characterized. Moreover, good flowing properties of a powder are also required for flow through the tableting system, resulting in uniform flow into the die cavities. Insufficient flow can lead to uneven filling of the dies, resulting in large weight and dosage variations of the final tablets. At high tableting speeds, the time to fill dies is reduced, making the flow properties of the blend even more important. The particle properties, mainly size and shape, strongly influence the flowability of the powder and thus the critical filling velocity achievable in die filling. However, having access to the micro properties (size distribution, shape, roughness) does not directly allow to predict the macroscopic behaviour due to the lack of a complete theoretical micro to macro relation. Furthermore, the bulk characteristics always arise from complex interplays between different physical mechanisms. A good example is the electrostatic charges buildup that is observed in powders due to the triboelectric effect, which depends on numerous parameters like the particle size and shapes, the material chemistry, the presence of capillary bridges…. Therefore, using properly chosen macro characterization methods is required to get relevant information allowing us to predict the powder behavior in industrial processes.

Lactose is one of the most widely used excipients in the pharmaceutical industry. There are many reasons for its popularity, such as the fact that lactose is largely inert, relatively inexpensive, safe, many different grades are available, and it has a long history of usage in successful formulations world-wide. For direct compression processes like tableting, lactose excipients can be used as a filler-binder to provide bulk density, compaction and flow to the formulation. Good flow of a pharmaceutical formulation is critical to produce uniform dosage forms.

In this study, the rheological properties of lactose powders have been investigated with the rotating drum method [1] (GranuDrum) and correlated with the actual consistency of flow achieved in a tableting system. The investigation of powder flowability at different drum rotating speed gives useful information on the evolution of powder processability at a higher shear rate. Indeed, with the classical repose angle characterization method the stress state at which the powder is submitted is far from what the powder experiments in the tableting machine. We observed that some grades exhibit shear-thinning behaviour, i.e. an increase of flowability with increasing applied stress, while others show the opposite. The classification of powder cohesiveness is therefore inverted at high shear. These observations have been correlated with the mass variation of tablets produced with a RoTab tableting press at different feeder speeds. We present the correlation between the flowability assessments obtained with the rotating drum and the weight consistency of the produced tablets. We thus highlight the importance of powder characterization at a stress state equivalent to those at which the powder is processed is thus emphasized.

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Attrition/fragmentation of limestone particles during fluidised bed sorption-enhanced gasification

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Keywords: attrition, fragmentation, CO_2 capture, fluidised beds.

Sorption-Enhanced Gasification (SEG) is a promising technology that can be based, Figure 1, on the use of Ca-based sorbents (like limestone) to selectively remove CO_2 from the gasification environment for production of a hydrogen rich syngas [1–5]. The SEG process benefits from the extensive understanding of the "calcium looping" process, a post-combustion technique aimed at removing CO_2 from combustion flue gas [6–9]. Calcium looping is most typically carried out in a Dual Interconnected Fluidised Bed (DIFB) reactor system, where the Ca-based sorbent is cycled between a carbonator, where it captures CO_2 from flue gas, and a calciner where the sorbent releases CO_2 and is regenerated for another cycle. Design of sorbent looping processes in DIFB reactors must consider: 1) sorbent deactivation (i.e., decay of CO_2 capture performance) over repeated cycling; 2) loss of sorbent material due to elutriation, that may be enhanced by attrition and fragmentation. The aim of this study was to investigate the performance of six different commercial limestones in terms of sorbent performance and attrition/fragmentation tendency under simulated SEG conditions.

The experimental campaign was carried out in a batch-operated lab-scale DIFB reactor, electrically heated. The six sorbents were different limestones coming from different parts of Europe. A synthetic gas including air, CO_2 and N_2, was used to simulate SEG conditions. A "test" consisted of ten complete cycles of calcination/carbonation. Calcination was performed at 850°C fluidising the bed with a stream of 10% CO_2 (balance air) so as to simulate oxidising conditions typical of the combustor-calciner. In the carbonation stage, the temperature was kept at 650°C and the CO_2 concentration was set at 10% (balance nitrogen) to account for reducing conditions typical of the gasifier-carbonator.

During each carbonation stage, the CO_2 concentration at the exhaust was continuously monitored to calculate the CO_2 specific capture performance. The sorbent attrition rate was determined by working out the mass of fines elutriated at the exhaust and collected in the filters, for each calcination and carbonation stage. After a test, each exhaust sorbent sample was sieve-analysed to obtain the particle size distribution and the fraction of generated fragments (Figure 2).

The preliminary characterisation of the six sorbents presented in this abstract shows that three of them (termed EBW, CZA and SAR) have good CO_2 capture performance, coupled with a limited production of both in-bed fragments and elutriable fines, while the sorbent named TAR is the worst in terms of the three features listed above. While results here presented can be useful for the determination of the make-up of fresh sorbent required for steady operation, and for optimal design and operation of sorption-enhanced gasification, further work is currently being carried out and concerns: i) the morphological (via SEM analyses) and porosimetric characterisation of the samples, in order to explain the features observed here; ii) the study of the impact fragmentation tendency of these sorbents, under a variety of impact velocity conditions.

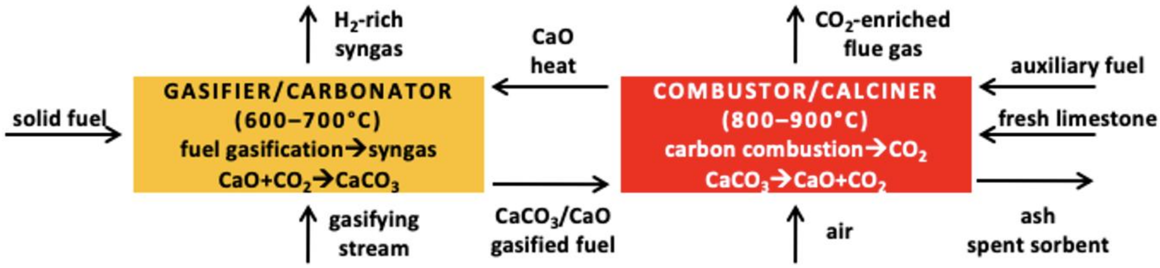

Figure 1. Sorption-enhanced gasification in dual interconnected fluidised beds reactor.

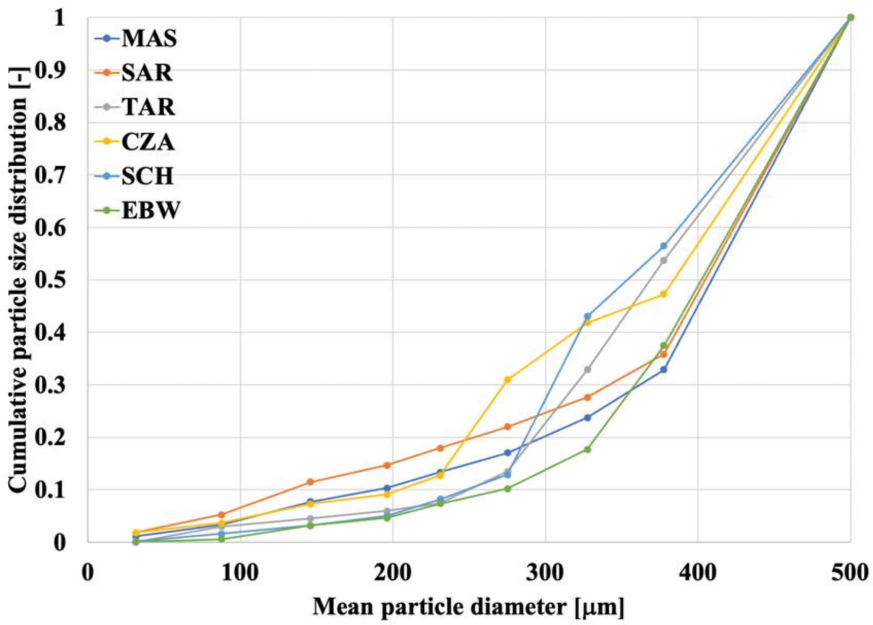

Figure 2. Cumulative particle size distribution for in-bed sorbent particles retrieved at the end of simulated DIFB-SEG tests (six different limestones).

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Exploring Particle Breakage Regimes via DEM

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Keywords: DEM, JKR theory, breakage regimes

This research involves meso-scale, discrete element method (DEM) modeling of micron-sized particles impacting a substrate. Using DEM, physical insight into the details of particle response upon impact are obtained. Particles interact via contact forces and Newton's second law of motion. Impacting particles were modeled as spherical agglomerates consisting of a large number of small, cohesive constituent spherical particles of equivalent properties. Via DEM, details of the force transmission within a particle, material failure, crack locations and particle breakup are explored. Cohesive forces resulting from van der Waals attractions in normal contact are determined based on the Johnson-Kendall-Roberts (JKR) theory and are characterized by a particle surface energy [2]. Calculation of cohesive effects in tangential contact forces is based on the work of Mindlin & Deresiewicz [3] and Savkoor & Briggs [4].

The spherical agglomerates are made to impact orthogonally with a flat surface of the same material under a wide range of impact velocities and particle surface energies. Mannitol powder material properties were used in order to compare the simulation predictions with previously published work [5] which investigated a limited set of impact velocities and surface energies only. Both the impact velocity and the particle surface energy varied over several orders of magnitude. With this wide range of conditions, five distinctive particle behaviors post impact (denoted by the different colors in Figure 1) were observed. In general, for a fixed impact velocity, as the particle strength decreases, the post impact behavior is rebound, fragmentation, and shattering, respectively.

In the rebound regime, the impacting particle remains intact and bounces off the impacting surface with a given coefficient of restitution. The predicted value for the coefficient of restitution decreases with increasing impact velocity as in the experimental work of Hassani-Gangaraj et al. [1]. In the vibrating regime, the particle also remains intact but does not possess sufficient initial kinetic energy to strongly rebound from the impacting surface and, instead, merely vibrates near the impacting surface.

In the fragmentation regime, the impacting particle breaks into different, smaller fragments. We show how force is transmitted within the impacting particle and the formation of cracks leading to particle breakage. Using DEM, compressive and tensile forces occurring within the particle are also determined. It was found that as the impact velocity increases for the same particle surface energy, the agglomerate breaks into a larger number of primary particles and smaller fragments. Higher impact velocities provide the energy necessary to break

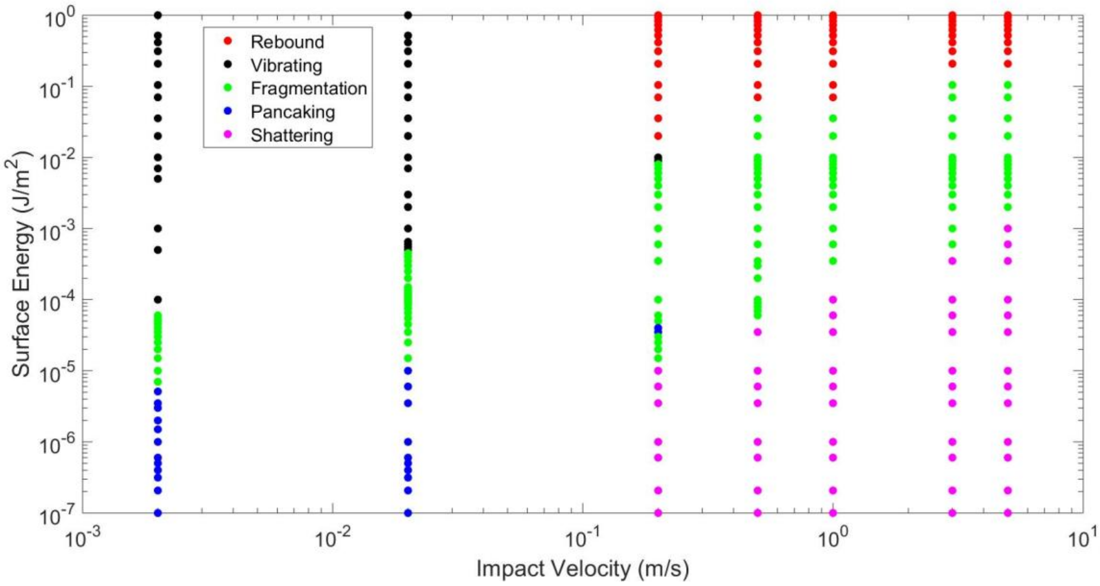

Figure 1. Regimes of Post-Impact Particle Behavior

the normal adhesive forces between constituent particles. Smaller fragments tend to have a larger rebound velocity than larger fragments, consistent with the experimental work of Hassani-Gangaraj et al. [1]. Some of the smallest fragments have rebound velocity magnitudes which exceed the impact velocity of the agglomerate. This suggests that there are collisions between small fragments and primary particles after the initial impact which accelerates their motion. In the pancaking regime, the impact particle breaks up while the largest fragment remains on the plate. Finally, in the shattering regime, the impact velocity is sufficiently large such that only constituent particles remain after particle impact with the surface.

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Investigation of effect of roller wear in HPGR operation using a hybrid DEM-PBM approach

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Keywords: Discrete element method, Population balance model, High-pressure grinding rolls, simulation

In spite of the important developments in mathematical modeling of high-pressure grinding rolls (HPGRs) in the last three decades, predicting their performance for a wide range of geometries, wear conditions of the rolls and types of lateral confinement remains a challenge. The UFRJ research group, using the discrete element method (DEM) coupled with multibody dynamic (MBD) and a particle replacement strategy (PRM) [1], has been able to predict key variables of pilot HPGRs processing iron ore pellet feed [2] and study the effect of worn rolls, aspect ratio and confinement system [3] on HPGR performance. Due to the computational effort for the large number of particles in HPGRs simulation using DEM, predictions of product size distribution were performed only qualitatively [2]. Subsequently, the outputs of DEM simulation (i.e. throughput and power, both total and distributed along the rolls) were used to feed a population balance model (PBM) and make predictions of product size distribution quantitatively, with a relative error around 7% [4]. The application of the PBM was based on the Torres and Casali model, and modification carried out by the authors in an earlier study [5,6].

In the present work, the coupled DEM-PBM was used to study the effect of worn rolls in product size distribution along the rolls for both pilot-scale and industrial-scale HPGRs. The methodology proposed by Rodriguez et al., (2021) was used, where DEM-MBD-PRM simulations were performed in a pilot-scale HPGR with 1 m and 0.82 m in diameter and length, respectively. The worn rolls were designed in detail in a CAD design with two symmetric patterns: parabolic and trapezoidal. These patterns were based on the literature and experimental measurements, and can be seen elsewhere [3]. The parabolic pattern was designed with a greater wear in the center of the rolls (2.5 mm) and circular conical surface until the edge, while the trapezoidal or “bathtub” shape was designed with equally intense wear in the center of the rolls (2.5 mm) and modest wear in the edge region. An additional simulation was performed with an intense trapezoidal wear with 5 mm in depth in the center region. From these simulations, power, throughput, pressure and throughput profile were extracted and coupled with PBM. Parameters from the non-normalizable breakage function [7] and specific selection function proposed by Herbst and Fuerstenau (1980) [8] were based on previously calibration using the modified Torres and Casali model [5].

Figure 1 presents the percentage passing the 45 µm sieve of the product predicted using the DEM-PBM approach. Simulations showed worn rolls surfaces producing greater heterogeneity in the predicted percent passing in 45 µm along the rolls, being the trapezoidal shape responsible for the most heterogeneous profile. New rolls and parabolic worn pattern achieved more homogenous product size distribution. In all the cases with worn rolls, coarser product size distribution is in the roll center region, and mainly for trapezoidal pattern; towards to the edge, finest particle size distribution could be seen. These quantitative results make available the understanding of roll wear effect and provide valuable information to support decisions in HPGR operations.

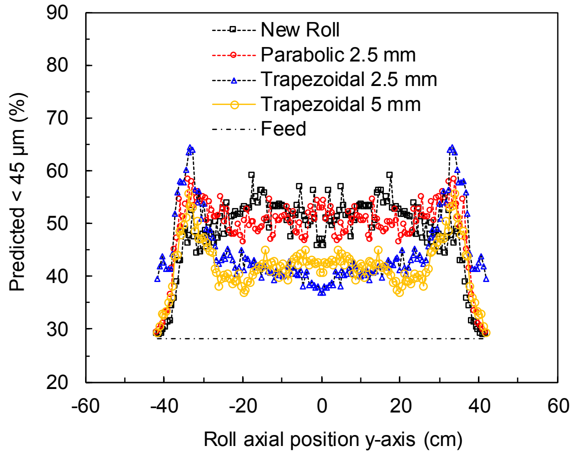

Figure 1. Predicted percentage passing the 45-µm sieve in the product for the pilot-scale HPGR for different surface wear conditions operating at specific force of 3.5 N/mm^2 and roll velocity of 0.5 m/s.

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Mechanistic simulation of dry media mills – from millimeter down to the sub-micrometer scale

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Keywords: Media mills, dry grinding, powder flowability, modelling, simulation.

The mechanistic approach to media mill models is a very flexible and powerful tool once it accounts for the contributions of the different stress and process mechanism, allowing full separation of material and process aspects. However, the mechanistic models proposed so far that can be applied to dry grinding operations, have generally focused on coarser particles (>100 µm) milling, in special for tumbling ball mills [1]. That type of mill and particle size scale can, by itself, lead the author to adopt certain assumptions in order to simplified the mathematical model, such as, absence cohesive forces among the particles, a monolayer of stressed particles between the grinding media and free flowing powder as mill product. Nevertheless, in the case of fine dry grinding, especially when median particles sizes below 30 µm are achieved, those assumptions are no longer reasonable [2].

This work proposes a formulation for a media mill mechanistic model capable of predicting grinding of particles sizes from the lower millimeter size range down to the sub-micrometer scale. The model should also be independent of mill design or operation mode, therefore, no distinction is made here between the traditional tumbling ball mill, a stirred media mill or a vibration ball mill, for instance. The effects of powder cohesiveness and reduction in flowability are also accounted for.

Based on experimental evidences, the following hypothesis were assumed as valid for this application: (a) The population of particles have a size dependent distribution of fracture energies – in other words each particle with a given size requires a certain value of applied energy to suffer fracture [1]; (b) Particles are not stressed individually, but as part of a bed of particles captured between either two grinding media or a grinding medium and a mill surface [3]; (c) All kinetic energy dissipated during a grinding media collision is transferred to the captured particle bed as applied stress [3].

Considering that the three assumptions cited above relate to particles stressing and breakage, but not to fragmentation once breakage occurs, mechanism such as powder cohesion, impact energy dissipation, stressing energy and fracture detection, were incorporated into a material breakage rate. The general breakage rate (S_i) over time was adapted from the formulation proposed by King [3] and represents the integration of the fraction of material in size class i broken by applied stress energy E, over all energies values.

$$S_i(t) = \int_0^{\infty} \frac{m_{capt}}{m_{holdup}} \cdot N_{col}(E,t) \cdot \left[\int_0^1 F_i(eE) \cdot p(e) \cdot de\right] \cdot dE \qquad (1)$$

where N_{col} is the frequency of collisions between grinding media with dissipated energy E and can be estimated from discrete element methods (DEM) simulations [1]. $F_i(eE)$ represents the fraction of material in size class i that breaks due to applied stress energy eE transferred to it and $p(e)$ is the stress energy partition amongst particles inside the captured bed. $F_i(eE)$ is solely a material aspect and reliable models for this fraction, as a function of size are present in the literature [1].

m_{holdup} is total mass of powder inside the grinding chamber, while term m_{capt}/m_{holdup} represents fraction of powder captured between two grinding media during a collision. Both the mass of stressed particle (m_{capt}) and the kinetic energy dissipation into the bed from colliding grinding media are highly affected by powder flowability and particle size distribution [3]. For instance, higher powder flowability and smaller adhesion to grinding media surface allows a bigger amount of powder to flow out of the capturing zone during a collision, resulting in a smaller captured bed mass. Prziwara et al. proposed that m_{capt} can be calculated as a function of grinding media diameter (d_{GM}) and impact velocity (v_{imp}). For the case of a collision between two spheres, m_{capt} is given by

$$m_{capt} = {}^{\pi}/_{4} \cdot \left(d_{GM} \cdot \sin\alpha_{capt}\right)^2 \cdot \rho \cdot x_{max} \quad \text{and} \quad \sin\alpha_{capt} = a \cdot {v_{imp}}^{b}. \qquad (2)$$

The parameters a and b, together with the powder bulk density (ρ) and the maximum particle size in the bed (x_{max}) are a function the bed particle size distribution and powder cohesion, therefore, powder stressing and dissipation evolves alongside product size. In order to main energy consistence and allow implementation of eq. 2 in the breakage rate, the impact velocity (v_{imp}) can be rewritten using the definition of dissipated energy (E) between two colliding grinding media of mass m_{GM} as $v_{imp} = \sqrt{4 \cdot E/m_{GM} \cdot (1 - COR^2)}$, where COR is the coefficient of restitution.

The presented breakage rate was implemented, alongside an energy dependent breakage function [4], in a population balance model and validated against experimental data from a batch and a continuous ball milling. The results are presented in Figure 1. It must be pointed out that the model presented very good agreement with experimental data without parameter fitting. In fact, the material characterization was conducted in bench scale test procedures without the use any mill.

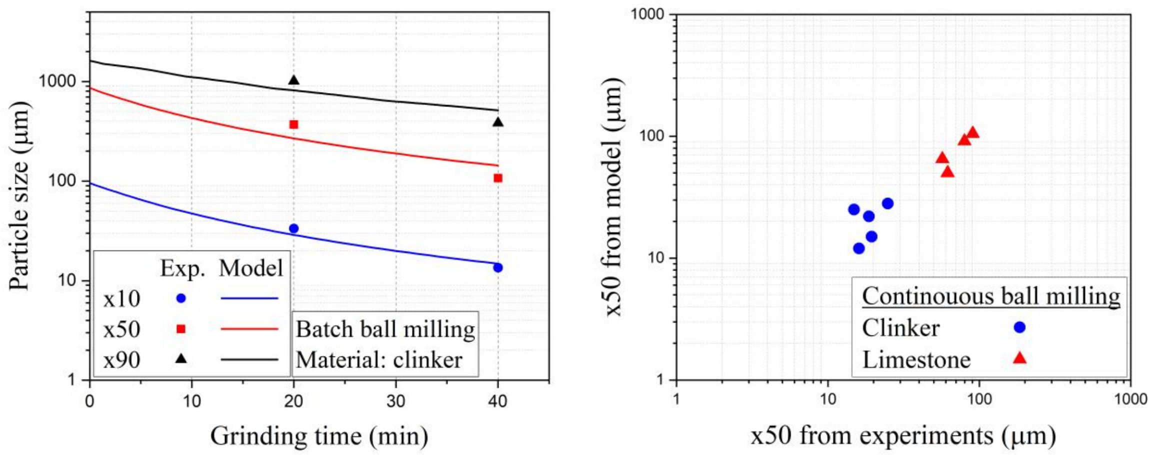

Figure 1. Comparison between experimental and model results for (left) batch tumbling ball milling of cement clinker and (right) a continuous ball milling of limestone and clinker in different conditions.

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Mechanistic modeling and simulation of multi-component batch ball milling of iron ore

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Keywords: ball milling, multi-component, iron ore.

In order to supply de demand of metals in a scenario of depletion of the richest ore deposits around the world, the most part of mining companies are facing a key challenge when dealing with reductions in head ore grade and increase in competence. In the context of ball milling, greater complexity in ore mineralogy, representing a multi-component environment, demonstrates the growing importance in understanding the breakage behavior of the different components contained in the ore and their interaction.

Indeed, several studies have been carried out with the aim of assessing multi-component ball milling using experimental results in industrial-scale [1] and mathematical models in laboratory mills [2;3]. Although detailed investigations and relevant results have been achieved by the use of these phenomenological models [2;3], it is worth mentioning that there is still a lack of proper understanding of how components behave and how they interact in a comminution environment. A potential way to overcome this issue is the application of a mechanistic ball mill modeling approach. Advanced modeling approaches using DEM simulations that provide information on the mechanical environment in ball mills and size-mass balance models coupled to particle breakage models has been proposed [4-6].

The present work analyses the application of the UFRJ mechanistic mill model [6] to predict multi-component batch ball milling results. The system studied corresponded to an itabirite iron ore from Brazil, which may be treated essentially as a binary mixture of Iron Minerals (mostly composed by hematite) and Gangue (mostly Quartz).

Experiments consisted of batch grinding tests in a 30 cm-diameter, 30 cm-length laboratory-scale mill. The mill is installed on a steel frame and is equipped with a torque sensor connected to a computer, allowing measurements of torque. The top ball size used in the tests was 36.5 mm, whereas the solids concentration in the slurry was maintained constant at 57% v/v, with voids fillings of 100%. Two different narrow size ranges (2.36 – 1.18 mm and 1.18 – 0.6 mm) and also the natural feed size distribution were tested in batch tests. Components were previously prepared using high-intensity magnetic separation and batch grinding tests were carried out considering each single component and also in a mixture 1:1 by volume. The experiments ran for a set time, the product was discharged and filtered, and the torque was recorded. Size analyzes were then conducted by combining dry and wet sieving.

The work started by estimating selected parameters describing the ore characteristics by back-calculation from batch grinding tests. Validation of the model in batch grinding for different mixtures and component distributions were then carried out highlighting very good agrement between experimental and simulared particle size distributions.

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A novel online modeling and simulation approach for pressing iron ore concentrates in industrial-scale HPGR

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Keywords: HPGR, iron ore, soft sensor, modeling.

Application of high-pressure grinding rolls (HPGRs) integrated with ball milling in size reduction of iron ore concentrates reached great popularity in industry in the last 30 years. Being one of the pioneers applying the technology on this circuit configuration [1], Vale S.A. adopted the HPGR technology in all of its pelletizing plants in the Complexo de Tubarão (Vitória, Brazil), where it often operates in the regrinding stage prior to pellet formation in the so-called pellet feed preparation step. Although it is a consolidated operation, several challenges can be stated when it comes to full process integration with HPGR pellet feed pressing, as this operation occupies the boundary between the end of the pellet feed preparation stage and the begining of the pellet formation process.

Indeed, the current digital transformation in the mining industry is shifting traditional process operation towards new approaches able to correlate the multi-scale dynamic modelling and simulation with the main industrial demands, thus trying to make real-time decisions in order to improve the production capabilities. This novel modelling approach, which requires an integration into the plant network and real-time information between physical operation and digital models, allows predicting variations within the process besides being used as a robust model-predictive control. For pressing iron ore concentrates, recent works by the authors [2;3] succesfully applied offline phenomenological modeling to describe HPGR performance under several variations in operating conditions and feed characteristics. These studies relied on the so called Modified Torres and Casali model which has only been used to describe pressing under in steady-state conditions and was not previosly demonstrated to be capable of being used in more robust process integration using online information.

As such, the present work aims to propose a new HPGR modelling approach coupled with real-time information and applied as an online tool in an industrial-scale iron ore pelletizing plant. The model is first validated on the basis of data from 12 months of operation. The model is then used to map the industrial operation in order to reduce the process disturbances and to propose optimal operational strategies to increase the HPGR throughput, thus reducing the energy consumption and improving the product Blaine Specific Surface Area (BSA). The work takes advantage on recent advances in HPGR mathematical modelling [2;3] using a phenomenological approach capable to give rapid responses from several variations in operating conditions, design variables and feed characteristics. A data-driven soft sensor model is proposed to predict the HPGR feed based on an Artificial Neural Network (ANN) and developed from plant database. Application of the modelling approach demonstrated capabilities to map the physical operation and give a realistic representation of the HPGR performance. The model was also capable to give support for the pellet feed production by given extended real-time information of the process, enabling the improvement of the operational strategies and process stability.

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Experimental comparison of wet and dry batch stirred media milling and the application of the continuity theory

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Keywords: dry grinding, wet grinding, stirred media mill

Wet stirred media milling is a widely used grinding process where the particle size of the product often reaches a size range of <500 nm. Dry application of the stirred media mill, - on the other hand - is a less common method of grinding mainly due to the phenomenon of aggregation and agglomeration during grinding. Dry grinding in a horizontal stirred media mill has undergone significant development in the last decade thanks to the work of several research groups [1 - 3], however further investigations are necessary also on machine, material and process level. A possible way to develop dry stirred media milling is to compare it with wet milling, to gain a deeper understanding of the similarities and differences. In the present study, dry and wet grinding experiments were performed on quartz, zeolite, kaolin and limestone in a batch horizontal stirred media mill at the same settings, but with different dispersion medium, water and air. The experiments were carried out at different stress energy (SE) levels, where the grinding media size, density and rotor velocity were also changed. The effect of the grinding time and specific grinding energy on the product particle size distribution and specific surface was investigated. In case of zeolite, kaolin, and limestone the wet grinding resulted in significantly lower median particle size at the same SE and grinding time. However, in the case of quartz, which is a hard to grind, but non cohesive material, the dry grinding resulted in the same or finer product at the same grinding energy investment. These experimental results show well the efficiency of dry grinding if the adhesion of the material can be avoided. To explain the results and to explore differences between dry and wet milling, the continuity model was applied for the research. Faitli [4] introduced the continuity theory for solid – liquid mixtures based on hydraulic transport studies in pipelines. According to the theory, particles in a fluid can behave continuum or discrete element like depending on the characterising size of the system (machine) and the particles. The characterising size of a system might be the thickness of the laminar sublayer around a grinding bead or the wall of the equipment. If a small particle fits into the laminar sublayer, the hydrodynamic buoyant force on it is negligible, therefore the small particle can stay in that region and therefore the small particle typically remains part of the fluid and this is continuum like behaviour. At higher velocities, coarse particles cannot stay in the laminar sublayer because of the significant hydrodynamic buoyant force resulting discrete element like behaviour. In wet stirred media milling the ground particles and water formed solid-liquid mixture is generally called "slurry" in the literature. It means it is treated as continuum (equivalent fluid) automatically and the grinding beads are treated and simulated as discrete elements. There might be cases when this widely applied approach is not true. The carried-out experiments with different ground materials and media and with wide ranges of the technological parameters can be used to validate the continuum model.

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Experimental determination and optimization of the energy transfer coefficient of a stirred media mill

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Keywords: Stirred media mill, grinding chamber and stirrer materials, advanced stress model, energy transfer coefficient

Wet-operated stirred media mills are commonly used in the field of fine grinding. Depending on the application, there are different mill geometries and materials of which the grinding chamber lining and the stirrer are made. Identical materials are often used for both the stirrer and the grinding chamber lining in horizontal stirred media mills. Polyurethane (PU) and ceramics (C) are frequently implemented materials, characterized by wear-resistance and cooling properties, respectively. Besides the process-relevant effects such as cooling and wear prevention, an energy-efficient mill operation is intended, which can be achieved, for instance, by using the stress model by Kwade [1,2]. A more in-depth energy optimization or even the scale-up of a comminution process of a stirred media mill can be realized by the extended stress model of Breitung-Faes [3]. Here, besides the mill geometry, the process parameters, and the properties of the feed material, various energy transfer coefficients are also determined. In this context, the energy transfer coefficients (ν) describe which portion of the specific energy (E) are dissipated and, thus, not transferred to the product (E) in a way that is relevant for comminution.

Within this study, the mill-related energy transfer coefficient (ν_{mill}) describes how efficiently the power provided by the motor via the shaft and, thus, the stirrer is transferred to the grinding media. Furthermore, ν_{mill} is defined as the product of the median grinding media stress energy (SE_{GM}) and the grinding media stress frequency (SF_{GM}) related to the net power input, thereby leading to a linear regression slope.

In order to investigate the influence of different grinding chamber linings and stirrer materials on the power consumption, experiments were carried out with a stirred media mill on a laboratory scale within the scope of this study. For this purpose, the power consumption of the mill was investigated under variation of the grinding chamber lining and the stirrer material as well as the grinding media size (d_{GM}), the filling degree of the grinding media and the stirrer tangential speed (v_t). The experiments were carried out without feed material and in passage mode to avoid the influence of wear particles from the mill and the grinding media as well as a viscosity change due to the product. Thus, the water was conveyed from the supply line into the grinding chamber and subsequently directly into the drain. In order to ensure a homogeneous grinding media distribution as far as possible and to avoid an increased power consumption by the classifier, a screen cartridge was used as grinding media separation device as well as a perforated disk geometry of the stirrer.

The resulting net power consumptions show that higher grinding media filling ratios and stirrer tangential speeds lead to different results for varied grinding chambers and stirrer materials. While the power consumptions for a combination of PU-PU also increase with increasing grinding media filling ratio and tangential speed, a combination of C-C shows only a slight influence on the slope. Figure 1 indicates that the mill-related energy transfer coefficient is not only influenced by the mill geometry, as assumed, but also by the mill material. It is assumed that the physical material properties such as hardness and Young's modulus as well as the surface properties are responsible for the different regression slopes. Apparently, increased surface roughness decelerates the grinding media closer to the grinding chamber wall, causing these grinding media in turn to slow down nearby grinding media. The motor is consequently forced to provide an increased amount of power to rotate the stirrer at a defined speed. In addition, a lower Young's modulus of the mill equipment could cause the mill material to absorb impact energy from the grinding media, subsequently reducing the grinding media speed.

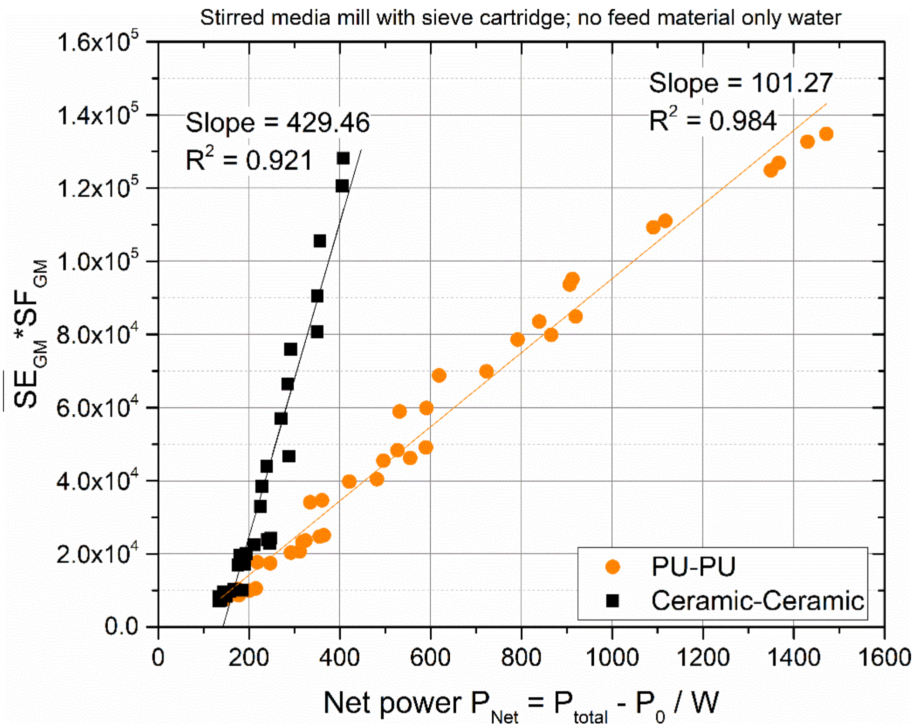

Figure 1. Mill and material related energy transfer coefficient

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Scalable production of two-dimensional materials by stirred media delamination

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Keywords: Liquid-phase exfoliation of layered materials, Stirred media mill, Process parameters, Defect generation.

Two-dimensional (2D) particles like graphene, MoS_2, WS_2, SnP_3 or GeS exhibit outstanding electric properties and are therefore promising for applications in Li-ion batteries, electronics and sensing [1-6]. Industrial application of 2D-materials requires inexpensive and scalable production methods. Mechanical exfoliation of layered materials in liquid phase is an easy to handle production route for 2D-materials consisting of few layers, as it does not require dangerous chemicals or harsh process conditions.

In this contribution we demonstrate that stirred media delamination [7-10] is a highly promising, cost effective and scalable method for production of few-layer graphene and other 2D-materials. In a stirred media mill, layered materials are exposed to shear and compression forces due to collisions with milling beads (e.g. of ZrO_2), see Figure 1. Under suitable process conditions thin layers are peeled-off from the particles, whereas lateral fracture is suppressed. If the delaminated sheets are sufficiently stabilized against agglomeration and re-stacking, few-layered 2D-particles are obtained.

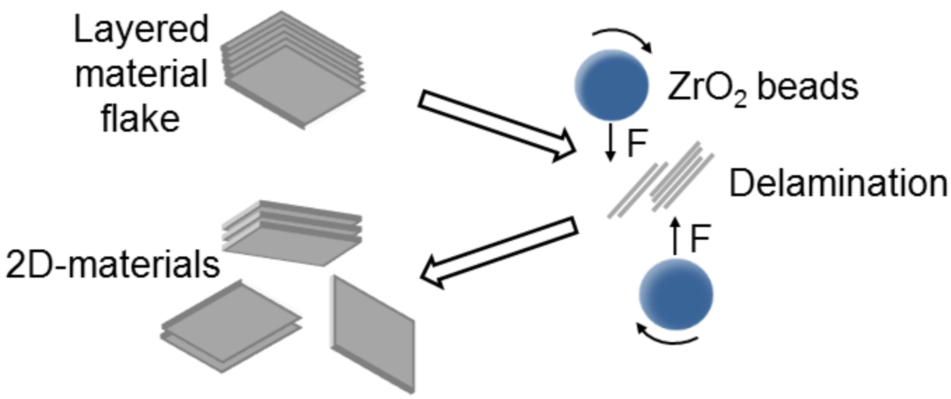

Figure 1. Principle of stirred media delamination of layered materials [7-10].

We report the influence of different process parameters (size of ZrO_2 beads, stirrer rotation speed, feed concentration, solvent viscosity) on the yield, degree of delamination and quality of the formed product exemplarily for exfoliation of graphite and MoS_2. After the stirred media delamination, the exfoliated sheets have to be separated from the not yet delaminated feed particles by centrifugation. The applied centrifugal force has major influence on the distribution of thickness and lateral size of the product particles. Characterization of the processed and separated materials by statistical Raman-spectroscopy reveals that few-layer graphene with low defect density is obtained if the kinetic energy transferred from the ZrO_2 beads to the graphite particles is below a critical threshold. Structure–property and structure–process relationships for graphene production are derived and presented in form of a roadmap towards high quality nano-sheets. When exfoliation dominates over lateral particle fracture few-layer graphene of typically 2-4 layers is obtained. The scalability of stirred media delamination is demonstrated by increasing the batch-size by the factor of 10.

In case of graphene oxide, we studied also the size reduction of monolayers via stirred media milling and ultrasound [11, 12]. Widely applicable scaling laws can be derived based on fast und highly accurate sedimentation analysis of the nano-sheets.

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Selective agglomeration of submicron particles from wet fine grinding with potential for wear separation

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Keywords: stirred media milling, selective agglomeration, wear, interfacial separation

In the field of pharmaceutical and life science products as well as for optical applications, suspensions with particle sizes in the submicron/nanometer range with the high quality standards are required. Within the top-down synthesis in stirred media mills, wear (abrasion) of the mill components, primarily the grinding media, is a major challenge. Due to an increasing contamination of suspensions with wear particles during the grinding process, the products suffer a loss of quality and can often be used only to a limited extent. A direct separation of the wear particles during/after the grinding process cannot be accomplished easily, as these usually have particle sizes similar to the product components [1, 2]. In this regard, especially for very small particles (< 1 μm), interfacial particle properties and the resulting interparticle interactions become strongly relevant, causing common mechanical separation principles to face their limits and to lose efficiency as well as selectivity.

Within this framework, a novel concept is demonstrated to separate grinding media wear particles from product suspensions achived by wet milling with stirred media mills. The basis of the approach relies on the integration of the selective agglomeration into the milling step, allowing only the wear component to be transferred to a coarser particle size range, which enables a material-selective separation through known mechanical principles. For this, the success of a selective agglomeration strongly depends on the targeted control of the material-related, electrostatic particle-particle interaction and the corresponding agglomeration kinetics between the respective particle species [3].

The investigation was carried out using the organic compound anthraquinone (AQ) as an exemplary product material. In a first step, the fundamental mechanism of the selective agglomeration is presented on the basis of binary, defined mixtures of anthraquinone and zirconium dioxide (ZrO_2). ZrO_2 was chosen as a model wear component, since it is one of the most frequently used grinding media material. The study compares the state of agglomeration as well as the resulting separation efficiencies and agglomerate sizes for different charge conditions at the particle interface, volume ratios and particle sizes of the respective components. Results reveal that, in addition to the particle-particle interaction according to DLVO theory, also the particle number ratio between the two materials has a significant impact on the agglomeration step. In fact, ZrO_2 agglomerates with a size of up to 30 μm can be obtained applying suitable conditions, proving to be easily separable and thus enabling almost complete separation of the mixture. A small insight of the results is shown in Figure 1, which depicts the material separation of AQ and ZrO_2 from the mixtures as a function of pH for a constant particle size and composition. Beginning with a pH of 11, which was identified as the starting point of the selective agglomeration in accordance with the literature [3, 4] (high surface charge of same sign; not shown), an almost entire material separation could be achieved by reducing the pH to 8.5. For lower pH values, however, it can be seen that a certain amount of AQ is also separated, subsequently decreasing the separation efficiency.

This can be attributed to the mutual coagulation between the particle species due to opposing surface charges (not shown).

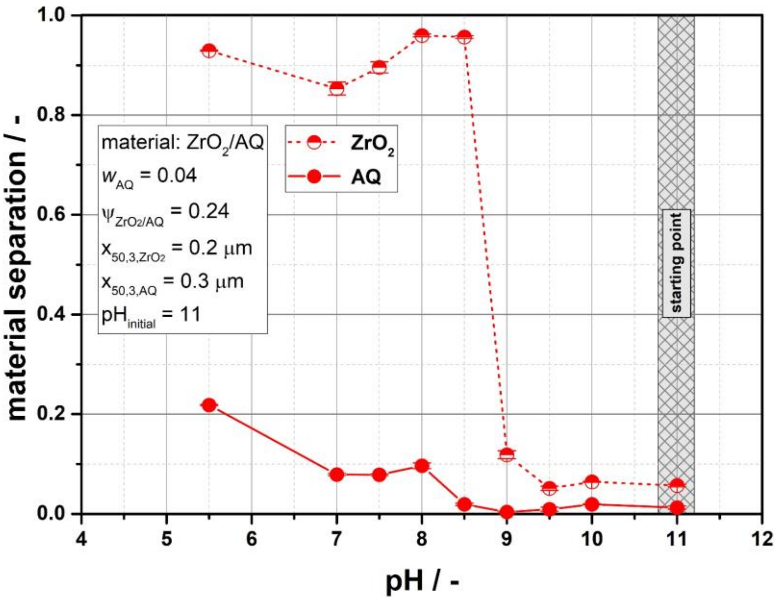

Figure 1. Material separation of ZrO2/AQ mixutre in dependence of pH.

Within the further course of the study, the integration of the selective agglomeration into the milling step is considered. Based on the preliminary results, the agglomeration of the wear particles can be induced directly in the mill after reaching a certain target particle size. The focus of the analyses is set on the identification of suitable stress conditions for the agglomeration step. The grinding media size cannot be changed at this stage, hence different agglomeration times and stirrer tip speeds are primarily examined. In this context, the mill can be operated between two limiting cases: as a mixing element or as a grinding device. The different applied stress conditions are presented and discussed in dependence of the separation efficiency of the wear. Results indicate a promising approach towards obtaining low-wear products from wet fine grinding with stirred media mills.

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Ultra-fine ceramic media for efficient nano-milling

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Keywords: wet-milling, beads, stress intensity, tungsten carbide.

Wet-milling in agitated bead mill is a standard process for the size reduction of a great variety of powders, from active pharmaceutical products to mineral ores. A theoretical framework emphasized the importance of the bead size and density with regards to the process efficiency, through the stress intensity concept [1]. An historical perspective of wet-milling media tends to confirm these theoretical consideration, and the most advanced media are today very fine yttria-stabilized zirconia beads: a ceramic with excellent mechanical properties, but also of relatively high density (6g/cc) compared to other standard technical ceramics like alumina (4g/cc).

As the pursuit of more efficient process is driven by growing application with requirements of very fine final particle size [2, 3], improving the media is still of interest. We report here some new media evaluation for milling down to 100nm: ultra-fine ceramic beads of size 30µm to 100µm. We evaluate the efficiency of yttria-stabilized beads, as well as tungsten carbide beads, on a few model systems. We show that using such ultra-fine beads can dramatically improve the milling efficiency, and using tungsten carbide beads can enhance even more this efficiency thanks to the higher density (15g/cc).

Materials and Methods

Two types of media were evaluated, both manufactured by Saint-Gobain:

- Yttria-stabilized zirconia beads (*Zirmil Y*) ;
- Tungsten carbide beads (*Ultimil*).

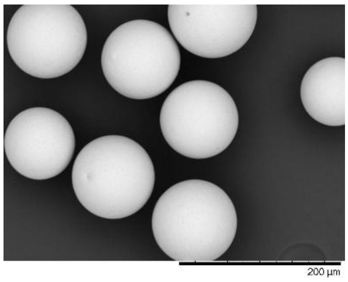

Figure 1. SEM image of ultra-fine yttria-stabilized zirconia beads.

Different model systems were considered to evaluate the milling efficiency: titanium dioxide, zirconia, alumina, and silicon carbide. Wet milling was done in recirculation in a lab mill, with tip speed of 10m/s or 13m/s. The particle size was measured by laser diffraction, from samples taken at various time during milling. Bead wear was evaluated by measuring the weight loss of the beads after the test, or by measuring the contamination in the product. The power input was measuring continuously during milling, and the Specific Energy Input was assessed by subtracting the power consumption by the empty mill.

Results

Depending on the bead type and size, the productivity of the wet-milling process could be changed very significantly. As expected, decreasing the bead size allowed to increase the milling efficiency, until the bead was too small to provide enough impact energy. In that case, switching to tungsten carbide media allowed to continue to improve the productivity. A representative example if the case of TiO_2 milling: switching to tungsten carbide allowed to increase by about three time the milling productivity, when monitoring the D50 as a function of time (see figure 2).

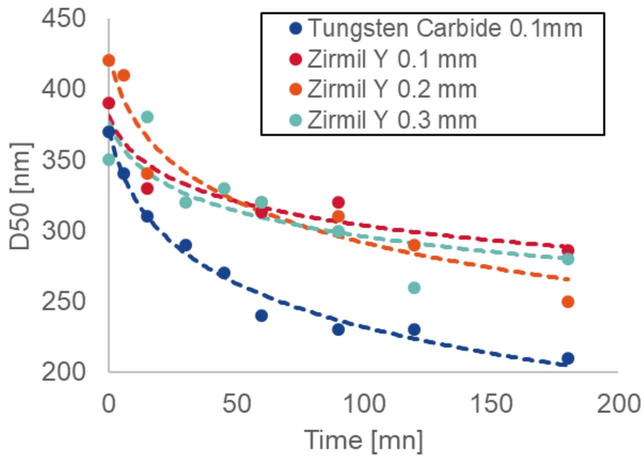

Figure 2. Particle size evolution when milling TiO_2 with different media – all other parameters being constant.

The use of tungsten carbide media proved also to be very favorable when milling particles with high hardness. For instance, when milling silicon carbide, the wear was more than times lower when using tungsten carbide beads compared to zirconia beads, while providing a more productive milling process with the same bead size.

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Autogenous grinding in stirred media mills for low-contamination production of silicon nanoparticles

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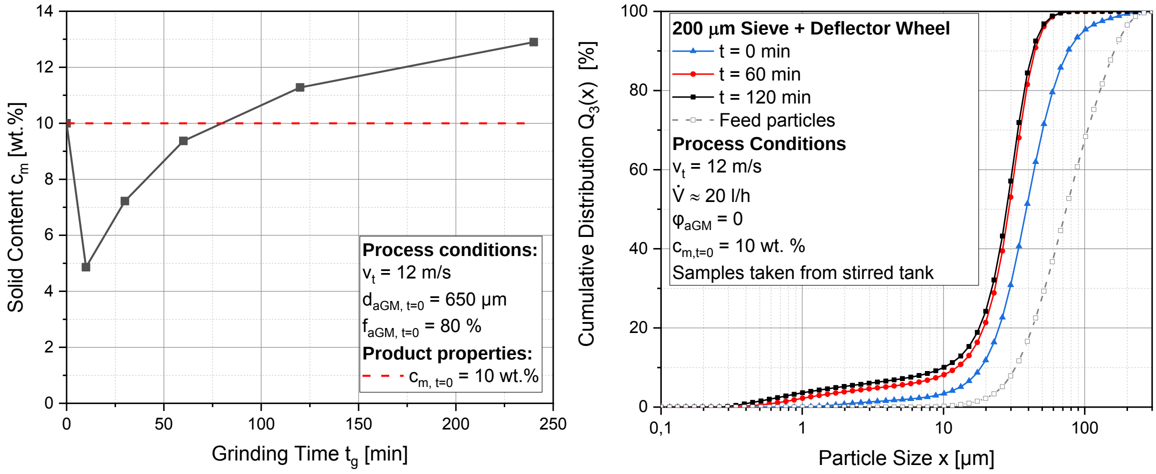
Keywords: stirred media mill, autogenous comminution, classification, silicon, lithium-ion battery.

With the energy transition in transportation, the demands on the range of future electric vehicles are also increasing. Current research is therefore focusing on lithium-ion battery cells with a high specific energy content and high energy density. One promising approach for increased energy density is the use of silicon as an anode material. However, the current challenge is that silicon undergoes significant volume expansion during battery charging, irreversibly damaging the electrode structure. Recent studies show that a combination of graphite and silicon nanoparticles can be used to significantly increase the mechanical integrity of the electrode and consequently the performance of a silicon-based battery. In this context, intensive research is currently being conducted on the production of high-quality silicon nanoparticles.

Wet comminution in stirred media mills provides a common method for the production of silicon nanoparticles. For example, Nöske et al. developed a fine-grinding method based on ethanol and electrostatic stabilization of the nanoparticles by adding sodium hydroxide [1]. However, comminution with stirred media mills is always accompanied by wear of grinding media and mill components. In order to decrease contamination and increase the purity of the nano-sized silicon, an autogenous grinding is addressed in this study. In autogenous grinding, grinding media and product are made from the same materials. As a result, grinding media wear becomes the product and contamination can be decreased significantly. While this grinding technology is well described for the processing of ores in ball mills, the use in stirred media mills for the production of nanoparticles is poorly investigated. Nevertheless, Kwade investigated the autogenous grinding of limestone in stirred media mills [2]. It was demonstrated that product particles below 10 µm can be produced.

However, modern mills for the production of nanoparticles are somewhat different to older generation mills such as Kwade [2] used. More particular, the withhold of grinding media in the grinding chamber in modern mills is supported by an integrated deflector wheel. The deflector wheel acts as a classifier and holds back particles up to a certain cut size. Since the grinding progress with autogenous grinding media takes significantly longer compared to grinding with conventional grinding media, it is expected that the role of the deflector wheel will be more important in autogenous grinding.

In this study, a first approach towards an autogenous grinding route based on silicon in ethanol is demonstrated. One of the main objectives is to obtain information about the comminution mechanisms of the autogenous grinding media (aGM). Therefore, a full autogenous grinding was applied, without adding fine feed material to the mill. Moreover, an autogenous grinding, where aGM as well as feed particles (smaller silicon particles) are used, was applied to increase the product yield. Particle size analysis showed that particles smaller than 150 nm can be obtained by autogenous grinding. Assuming that the abrasion of the aGM becomes the product, it should be possible to determine the abrasion and breakage rate of the aGM by measuring the increase in the slurries solid concentration. Figure 1a shows the solid content of the slurry as a function of the grinding time.



a) b)

Figure 1. *a) Progress of the slurries solid mass concentration as a function of grinding time. b) Impact of the deflector wheel on the feed particle size distribution at constant process conditions.*

An initial solid concentration of 10 wt. % (feed particles) was set in the beginning of the experiment. But, it can be seen that just after starting the autogenous grinding experiment, the total solid content drops to approximately 4.5 wt. %. After 60 -70 min, the measured solid content passes the initial 10 wt. %. In the end of the test, almost 13 wt. % solid content can be measured, which can be attributed to the abrasion of the aGM. The initial drop of the solid content of the slurry is caused by the deflector wheel. It withholds particles coarser than a process specific cut size. In fact, this behavior is also common in conventional grinding with stirred media mills containing a deflector wheel. But, as grinding with aGM progresses rather slow, this effect is distinct and present for a longer grinding time. Therefore, the importance of the deflector wheel was investigated. Figure 1b shows the particle size distribution of the feed particles. In order to see the impact of the deflector wheel, no aGM, but only feed particles were used in this test. The feed particles were fed into the stirred vessel while the mill and pump were running. The test was operated in circuit mode and samples were taken from the stirred vessel. It can be seen that the particle size distribution of the feed material decreases as the running time increases. This shows that the feed material accumulates inside the mill as the deflector wheel holds back particles up to a certain cut size. After 120 min running time, particles coarser than 70 – 80 µm are no longer present, meaning that only particles smaller than this size can pass through the deflector wheel. In order to analyze the impact of the deflector wheel on the grinding performance, tests were conducted at different stirrer tip speeds and throughputs. Additionally, aGM were added with different filling degrees and also tested at these process conditions. Herewith, a product yield can be defined and predicted at certain process conditions. Finally, this allows a prediction of the overall productivity of the presented autogenous comminution process.

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DEM Simulation of Particle Attrition in Mechanofusion Device

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Keywords: Attrition, Impact, Mechanofusion, DEM

Mechanofusion is a very high shear mixing device that is commonly used for dry powder coating. High speed rotation of the vessel outer wall generates a centrifugal field, causing compression of the powder against the wall. A stationary semi-cylindrical push arm is placed close to the external wall, producing a narrow gap, through which a part of the powder bed passes, causing intense shearing under very high compression. If coating powder is added, such as a lubricant or glidant, then shearing of the powder bed 'smears' the fine particles over the surfaces of the host particles, coating/fusing them together. While the high shear mixing promotes homogeneous coating, it could also lead to unwanted attrition of weak particles.

In this work, particle attrition in the Mechanofusion device is modelled by the DEM approach. The mechanistic model of Ghadiri and Zhang [1] is implemented in the DEM software package to govern the particle breakage events. Both spherical and polyhedral particles are used to represent the model crystal used in this work. The results of the simulation are compared against experimental results for validation. This presentation will report our findings and highlight the difference between using spherical and polyhedral particles in attrition modelling.

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DEM Modeling of particle breakage inside rotating drums

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Keywords: Discrete Element Method, granular media, particle breakage, rotating drum, upscaling.

In this work, we used the Contact Dynamics - Discrete Element Method (CD-DEM) to investigate the effect of material and system parameters on the grinding process in a 2D rotating drum. To model breakable particles we implemented the Bonded Cell Method (BCM) [1], in which the particles are discretized into bonded polygonal cells (figure 1a). A debonding criterion consistent with the classical framework of fracture mechanics (both in terms of yield stress and fracture energy) was employed [2]. We used a smooth drum without grinding media in which particle breakage is a consequence of granular flow. In this self-grinding or autogenous process (figure 1b), each particle breaks into fragments composed of unbreakable primary cells with different shapes and sizes depending on grinding time, surface energy, rotation speed and other mechanical properties.

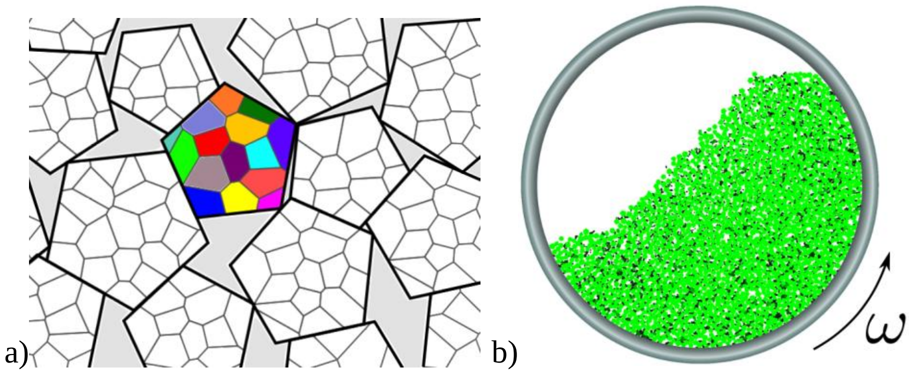

Figure 1. a) Voronoï tessellation applied to polygonal breakable particles. Each cell is presented in a different color. b) Snapshots of a rotating drum simulation. The color is proportional to damage, defined as the number of cells detached from a particle, represented on color scale from bright green for intact particles to black for highly-damaged particles.

For an extensive parametric study [3] we performed long lasting simulations with large number of particles and cells in order to get meaningful statistics of fracture events. We varied system parameters such as drum size, rotation speed, filling degree and initial particle shape. The effect of each parameter on the granular flow and evolution of grinding in terms of the mean particle size and specific surface of the material was quantified. We show that the specific surface (defined as the sum of the surface areas of all particles divided by their total weight) increases almost linearly with time up to a transition point to a nonlinear regime where many unbreakable fragments are generated, and thus the probability of breakage declines. For all values of system parameters, this point corresponds to the same amount of specific surface equal to slightly more than half the maximum specific surface that can be generated in the simulations. This transition was used to define a characteristic time associated to the grinding efficiency. For all system parameters, when the times are scaled by this characteristic time, the rate of particle breakage collapses on a master curve. Finally, we show that the rate of particle breakage can be expressed as a linear function of a general scaling parameter that incorporates all system parameters. This scaling behavior provides a framework for the upscaling of drum grinding process from laboratory to industrial scale.

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Single particle tracking in DEM for use in validation of PEPT experiments in a horizontal stirred mill

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Keywords: particle tracking, simulation, fine grinding, dynamics.

Studies of the dynamics of grinding media within a shearing environment such as an IsaMill require indirect methods of observation due to the opaque nature of the system and high shear forces any internal probes are subjected to. For this reason, Positron Emission Particle Tracking (PEPT), a non-invasive particle tracking technique, has been extensively utilised to study the grinding media dynamics in tumbling mills and IsaMills [1,2]. The assumption that the time averaged behaviour of a tracer particle represents the ensemble average of the bulk material makes a PEPT tool for studying motion of particles in opaque systems such as IsaMills [3]. This experimental technique has had a lot of success in elucidating the internal workings of different types of grinding mills, however, it requires other observation techniques to be cross validated. Thus, Discrete Element Method (DEM) computational simulations are often performed to determine limits of the application of results of PEPT experiments [4]. However, DEM simulates the bulk behaviour of griding media over a much shorter time scale and a clearer comparison between DEM simulations and PEPT experiments should be performed.

This paper compares the dynamics of grinding media in a 3 disc horizontally stirred mill as it is derived from PEPT experiments, as well as DEM simulations. Both the bulk average of all simulated particles and the time-averaged behaviour of a single particle are examined. The fraction of the total volume occupied by media as derived by each technique is shown in Figure 1.

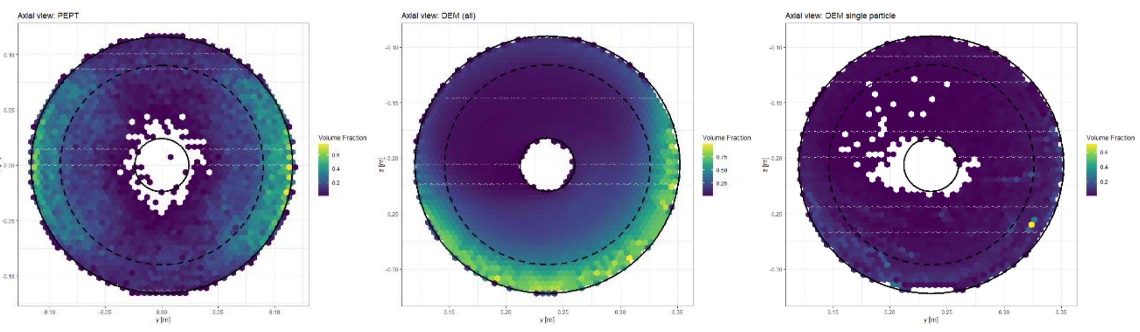

Figure 1. The fraction of volume occupied by grinding media in the horizontal mill as derived from PEPT (left), DEM (middle) and single particle DEM (right).

Figure 1 indicates that there is a clear difference between the results of the three techniques, with the full DEM simulation (centre panel) showing the best coverage of the entire system. The disconnect in the observed behaviour of the grinding media in the horizontal mill in each technique is explored, along with possible adaptations of the DEM simulation techniques to generate data in order to better improve the agreement between techniques.

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Simulation of breakage of iron ore pellets using the discrete breakage model

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Keywords: Iron ore pellets, discrete breakage model, discrete element method, particle breakage.

The design and study of comminution and classification processes relies on the simulation of the behavior of particles inside different types of equipment. For granular materials in the mining industry, the dominant simulation methodology is the Discrete Element Method (DEM), which has been used extensively in recent years for the simulation of all types of processes where a granular phase is involved. The DEM method solves the individual differential equations of motion for a large amount of particles to obtain the dynamical response of each of the particles and the overall behavior of the bulk material. In addition, the DEM method could be coupled with the Computational Fluid Mechanics (CFD) method to give more accurate results in systems where the fluid-particle interaction is relevant.

Despite the detailed resolution of the DEM method for particle trajectories, the most important feature required for the simulation of a comminution system is the mechanical response of each of the particles and the computation of particle breakage [1]. In this regard, the DEM method requires an additional Breakage Model (BM) for the computation of the particle breakage energy and, if required, the substitution of particles by its progeny fragments in the on-going simulation. Some examples of this ensemble of methods are the simulation of particle breakage by the particle replacement models or the bonded particle models [2]. Nonetheless, the more traditional approach is to use a mechanistic modeling where the particle behavior is simulated by using the DEM method and the obtained data is post-processed to account for the energy spectrum of the particle-particle and particle-geometry interactions. The pot-processed data is then used to utilize an auxiliary BM such as the Tavares model and a population balance method to simulate particle breakage and the final grading of the granular material. Inasmuch this method is not embedded in every DEM software and requires in-house coding, it has been used for the simulation of numerous comminution systems with great adaptability, accuracy and robustness [3].

A third type of DEM-BM is the Discrete Breakage Model (DBM) originally proposed by Potapov and Campbell [4,5]. The DBM is based on the discretization of the particles under study into a number of elements represented by simpler geometrical shapes such as Voronoi triangles or Dealunay tetrahedra. Each particle element is modeled as a discrete element, therefore, the DEM method is used for the simulation of its dynamical behavior. In addition, every element interacts with other elements and bounding geometries as any particle would interact in a DEM environment. As a result of the interactions, normal and tangential overlaps among the elements of the same particle determine an internal stress state. Elements within each particle are bonded to its neighboring elements by a joint. Every joint can be set to break under different circumstances such as a tension threshold, a tangential threshold or a combination of both. After the joint is broken, it remains in that state and the neighbor elements only interact by overlapping. As joints break, the particle loose its integrity modeling the breakage of the particle.

The DBM is one of the newest introductions of the commercial software Rocky from ESSS, being available for its usage by the final user. However, the validation of the model has only been presented in ideal circumstances using perfectly brittle materials and a preliminary version of the code. This work uses the DBM, as embedded in the commercial software Rocky DEM, for the simulation of breakage of individual iron ore pellets. The results of the model are compared against experimental data in order to validate it and observe its main features, advantages and disadvantages. The results of the simulations show that the DBM is a valuable tool for the simulation of particle breakage. The model could be calibrated to be used with different numbers of elements and provide accurate predictions of stresses, deformations and breakage energies.

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The Influence of Feed Component Density on the Performance of Semi-inverted Hydrocyclones

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Keywords: semi-inverted, hydrocyclone, classification, efficiency

Hydrocyclones are the most commonly used classifiers in mineral processing mainly due to their simplicity, versatility, and low capital and operating costs. However, despite their wide use, hydrocyclones are inefficient, especially when treating multi-component feeds. Density differences between the valuable mineral(s) and the barren particles as well as the fines by-passing to the coarse stream are the main causes of poor hydrocyclone performance. These phenomena can lead to over-grinding of liberated high-density fine minerals as well as misclassification of low-density composite particles to the flotation feed. The former may result in reduced milling capacity while the latter, metal loss to the flotation tailings. The overall circuit performance can be significantly improved by running hydrocyclones efficiently.

Although many studies have been done to improve hydrocyclone efficiency, limited focus has been put on how they are installed. Previous research has shown that the hydrocyclone inclination influences classification performance. Significant reduction in the water recovery to the coarse stream can be achieved at hydrocyclone inclinations above the horizontal – also known as semi-inverted positions. Less water to underflow results in less misreporting of fines to the coarse stream. Understanding how hydrocyclones perform in semi-inverted positions will thus play a critical role in improving mineral classification and consequently the overall circuit efficiency.

In total, 66 tests were conducted on a 250 mm diameter hydrocyclone to study the effect of inclination and feed component density on the classification efficiency. Synthetic feeds containing a mixture of low and high-density material in different ratios were used as a proxy for a multi-component ore. The data obtained from the test work was used to understand how the densities of different components in the feed influence the efficiency of a semi-inverted hydrocyclone. These findings can be used not only to optimise mineral classification in existing circuits but also to potentially develop new applications for semi-inverted hydrocyclones.

Pre-concentration of sulphide ores using a novel continuous high voltage pulse system design

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Keywords: Ore pre-concentration, High voltage pulse, Coarse waste rejection, Grade heterogeneity.

Ore pre-concentration has the potential to reduce milling throughput and improve comminution and recovery efficiency, whilst still achieving required metal production targets. This is achieved by separating valuable minerals from gangue minerals prior to the energy intensive grinding stages of a processing circuit [1]. From the many ore pre-concentration methods studied over recent years, the use of high voltage pulse (HVP) technology is emerging as a likely option to optimise mining operations and make the exploitation of low-grade ore bodies economically viable. Ore pre-concentration using HVP technology involves applying electrical pulses to rock particles to induce selective breakage of those containing metalliferous grains [2]. The adoption of a high voltage pulse equipment before the energy-intensive grinding stage would allow for the separation of the fresh feed into a high- and low-grade stream using size separation. The low-grade stream, containing coarse unbroken material, could be rejected as waste or diverted to a different recovery process, whilst the high-grade stream would be further processed in the downstream grinding circuit.

Research on high voltage pulse has showcased the benefits of adopting the technology in the mining industry [3-5]. However, most of the work that has been undertaken in this area adopted a batch testing setup using reduced amounts of sample. To be implemented in industrial applications, a HVP system must be scalable, operate continuously and process high tonnage rates. This work will present the concept for a new continuous HVP system proposed by researchers at the Julius Kruttschnitt Mineral Research Centre (JKMRC). The continuous system developed at laboratory-scale consists of a feeder system coupled with a high voltage processing zone, comprising a patented novel electrode design, which is able to treat and separate the feed into two product streams of different grades in a single step (Figure 1).

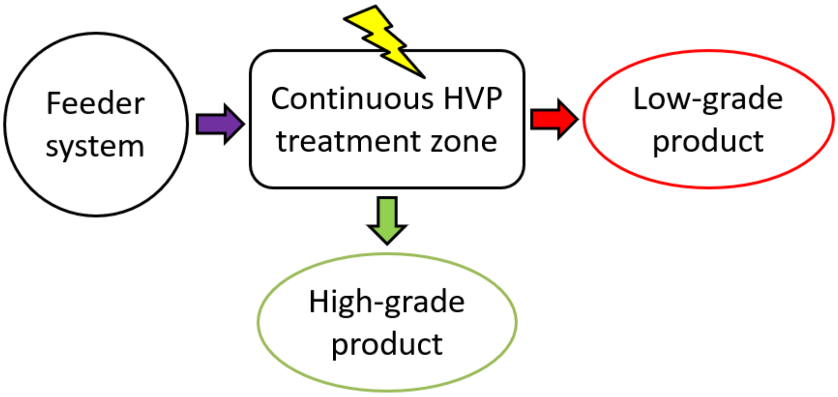

Figure 1. Schematic diagram illustrating the new continuous HVP system developed at the JKMRC consisting of a feeder system integrated with a continuous HVP treatment zone that splits the material in two streams of different grades.

The new device was used to process different Cu/Au sulphide ores and its ability for pre-concentration was assessed. The results indicate the HVP system, operating continuously at a feed flow rate between 0.5 t/h to 1.0 t/h, attained rejection rates of up to 50% of the total feed mass whilst still achieving metal recoveries of up to 75%. The pre-concentration step has consequently led to a head grade enrichment of more than 50% for different HVP operating conditions.

The performance of the device will be presented and discussed on the basis of grade heterogeneity and mineralisation texture of the ores tested. The grade heterogeneity of different ores were assessed based on X-ray fluorescence (XRF) assays on individual particles and the maximum theoretical metal recovery was determined for comparison with the HVP pre-concentration data obtained.

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Vertical Roller Mill Operation with Alternative Classification Devices in a Minerals Processing Application

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Keywords: Comminution; Classification; Vertical Roller Mill; Platinum Group Minerals

The comminution process is energy intensive and accounts for up to 70% of energy consumption in a typical minerals processing plant. This makes it a key area of interest when looking to improve energy efficiency, particularly in an industry faced with continually declining ore grades and deposits which are becoming more disseminated and will require greater energy inputs in order to maintain valuable mineral productions [1]. The vertical roller mill (VRM) is a high compression grinding device which has the potential to improve comminution energy efficiency in the minerals processing industry [2]. The device has widespread use in cement and coal dry grinding applications, where it is known to be more energy efficient than conventional tumbling mills. In these applications the vertical roller mills are typically operated as a comminution device coupled with an internal dynamic air classifier. Air classifiers were chosen for this application because they are preferred for dry grinding applications [3]. These classifiers are however more energy intensive than other classifiers used in the mineral processing industry and operating the vertical roller mill with less energy intensive classification devices could have the potential to further reduce the total energy consumption.

This paper investigates the influence of the method of classification on the mill performance in terms of energy consumption, throughput and product characteristics. Three classification devices were used namely: the internal dynamic air classifier, an external hybrid air classifier and external vibrating screen. The study was carried out using the platinum group mineral bearing ore, Platreef, which is a very competent ore and is classified as a rhelogically complex ore. It pauses several problems in wet milling circuits ranging from high energy demand to problems during classifications using either hydrocyclones or screens. The study was carried using a pilot scale vertical roller mill which allowed flexibility of changing the classification set-up.

The comparisons of particle size distributions for the vertical roller mill products generated with the different classification devices are shown in Figure 1. The information indicates that the VRM can provide products that can match the primary, secondary and tertiary grinding levels encountered in the platinum comminution circuit. The VRM testwork indicated that the device can be operated with the internal dynamic air classifier, an external air classifier and vibrating screen. For the air classification circuits, specific grind energy increased, and throughput decreased as the target grind size became finer. The throughputs achieved for the external screening and external air classification circuits were around 50% lower than those attained with the standard internal dynamic air classifier. The products generated by the internal air classification circuit were of steeper particle size distribution compared to those generated using external classification systems. The screening circuit yielded a greater sharpness of separation than the air classifiers, when targeting the coarse product grind.

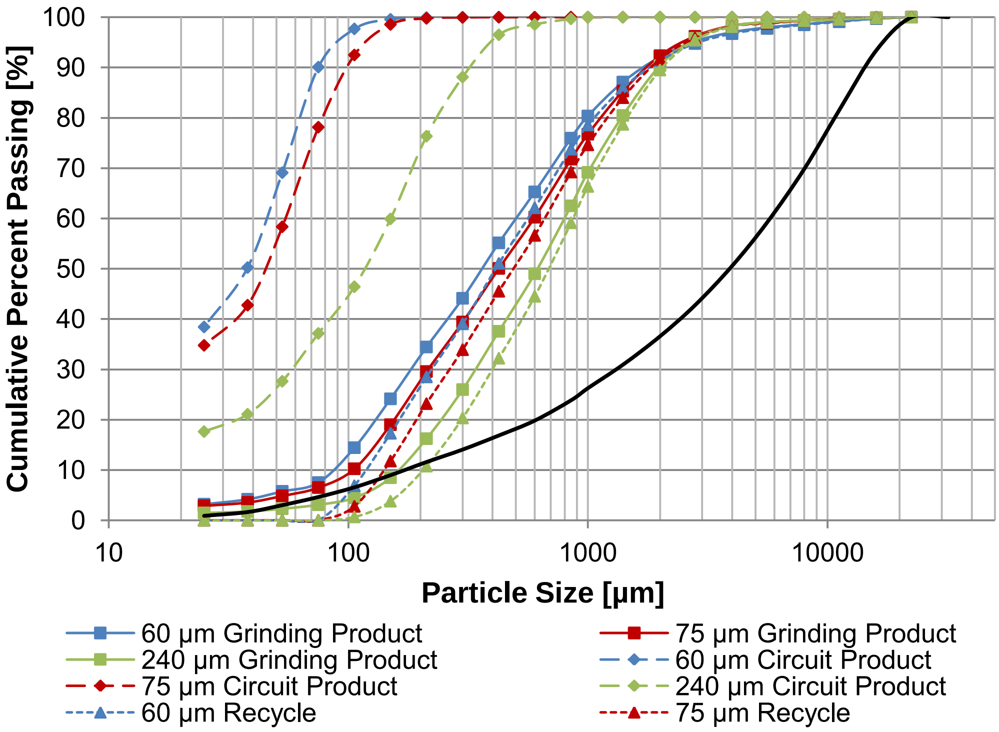

Figure 1. Measured and estimated mass balanced particle size distributions for the internal air classifier vertical roller mill circuit for the different target products sizes (P_{80}: 60, 75 and 240 µm).

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Towards Green Economy in Mining Industry: Eco-Efficient Dry Comminution by Vertical Roller Mill (VRM)

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Keywords: Energy. Dry Comminution, Energy Efficiency, Water Consumption, Wear, Vertical Roller Mill (VRM)

Introduction: Mining industry, as the cornerstone of human civilization, is directly and indirectly responsible for up to 45% of the global economy. The industry is an energy and water consumer. It is projected that about 6-7% of total world's energy is consumed by mining industry and therefore responsible for 4-7% of total carbon footprint [1-2]. Furthermore, the industry consumes between 7 and 9 Bm^3 of water per annum which is associated with water pollution and contaminations [3]. Accordingly, resource efficiency, access to energy and water, reducing environmental footprint, etc., are major challenges within the industry.
Comminution, however, is the most energy-intensive process within the mining chain, accounting for almost half of the energy consumption in mining. Accordingly, inventing, developing, and implementing eco-efficient machines, processes, and flowsheets to minimize energy and water consumptions in comminution, therefore, reducing related environmental footprints would be of great interest. Dry comminution, using Vertical Roller Mill (VRM) could be an ideal energy efficient comminution option for hard rocks in mining that offers a great opportunity to reduce energy and water consumption and related environmental footprint. This paper presents and discusses the results from comminuting two different raw materials, using VRM through a comprehensive research and development program supported by European EIT-RawMaterials.

Materials and Methods: Two different raw materials/samples were received for testing, a sulfide ore from Boliden Minerals AB and a ferromanganese slag from EraMet. The samples were characterized for their comminution behaviors. Accordingly, crushing, rod mill and ball mill Bond work indexes were determined for the materials. Furthermore, the abrasion index of the samples was defined, using standard Bond abrasion tests. The results from characteristic of the samples are shown in Table 1.

Table 1 – The comminution characteristics of the samples

	Crushing Work index (Wic)	Rod Mill Work index (Wir)	Ball Mill Work index (Wib)	Abrasion index (Ai)
Sulfide Ore	10 (kWh/t)	13 (kWh/t)	12,5 (kWh/t)	0.15 - 0.18
Fe-Mn Slag	14 (kWh/t)	17.7 (kWh/t)	17 (kWh/t)	0.38 – 0.51

After materials' characterization, the comminution tests were conducted to explore the potential of implementing of VRM technology [4], however, parallel tests were conducted by conventional ball milling. The criteria for comminution were defined based on detailed mineralogical analysis of the samples and the related processes for downstream. Accordingly, the target product sizes for comminuting were considered as d_{80}=60-65µm for sulfide ore and d_{80}≈300µm for the ferromanganese. Moreover, the media consumption for comminuting the samples were estimated based on specific energy consumption and laboratory tests. Thus, the energy needed to size down the samples was estimated for different comminution modes, while the wear in comminution practices and its related indirect energy consumption were estimated.

Results: Series of comminution tests were completed, using VRM and the energy needed for comminuting the samples were defined. Fig.1 shows the results to achieve the target sizes for the samples. Accordingly, the energy needed to gain the target sizes were at about 7.2 and 4.4 kWh/t for sulfide and slag samples respectively. Furthermore, tests were conducted by conventional milling for comparison which revealed that the energy needed for comminuting sulfide ore to the target size was between 19.73 and 21.2 kWh/t in dry mode, while for wet grinding it was 17.9 kWh/t. However, the energies to grind the slag sample were 8.75 and 10.5 kWh/t for wet and dry mode respectively.

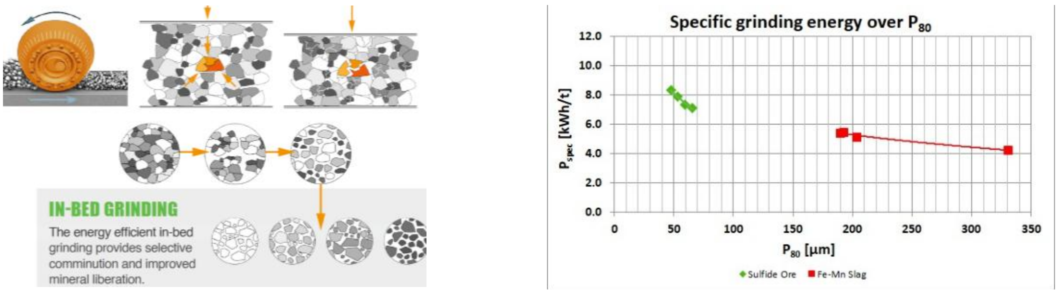

Fig.1- In-bed comminution mechanism of VRM and Energy needed to comminute materials

Adding to the energy for comminution, there are indirect energy and cost related to the wear in comminution. The results from the grinding tests revealed that wear is considerably higher in grinding by conventional wet and dry milling in comparison with VRM dry grinding, especially when grinding rolls are made from Metal Matrix Compound (MMC). In this case the media consumption reduces considerably to ~8 and ~12 g/t which contributes greatly to reduce the cost and carbon footprint in comminution. However, if mild steel is considered, the values for wear are at range of 129 and 186 g/t for sulfide ore and slag respectively. In comparison, the wear rates using conventional ball milling in both wet and dry modes are much above the values stated above (about 240 and 630 g/t respectively in dry ball milling and much higher in wet grinding mode). To produce a ton of steel/steel-alloy, the energy needed would be about 6 MW/t with related carbon emission of 2t [5-6], while the production consumes at least 3 to 4.2 m^3 water as a strategic resource [7]. Accordingly, reduction in comminution wear rate greatly contributes to resource eco-efficiency and green economy.

Conclusion: Moving towards “Green Mining Economy” requires responsible attempts to invent, develop, and implement eco-efficient technologies and related flowsheets. Dry comminution by Vertical Roller Mill (VRM) and developing smart related flowsheets to deal with comminuting hard rocks. This study explored the potential of comminuting rocks by VRM eco-efficiently. The results revealed potential reduction in energy consumption in grinding in comparison with conventional comminution practices. The technology offers great opportunity in reducing wear/media consumption in comminution that conserve energy in manufacturing media and related costs. Furthermore, as a dry comminution process, there are no needs for water and water purification system

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Measurement of size-dependent transport in a pilot AG mill

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Keywords: Mill mass transport, particle transport, tumbling mills, grinding mills.

It is understood that rocks in a mill charge do not instantaneously transport along a grinding mill and are consequently not perfectly mixed within that charge. However, this is overlooked in mill modelling due to a lack of understanding of the transport phenomenon and the difficulty of modelling realistic transport in a complex tumbling system. The authors identified that, particularly for Autogenous milling, this deficiency limits the validity of scaling up from pilot work and in predicting the build-up and discharge of competent pebbles. The need to better understand these phenomena led to a set of test work designed to measure rock transport along and out of a pilot mill.

A 1.67 m diameter by 1.4 m long pilot mill at the SGS Lakefield laboratory was operated autogenously over four test conditions, which are the combination of two fillings, 25% and 35%, and two discharge open areas, 3.5% and 7.1%, both with 75 mm ports. From T2 to T4 the only change is to double the open area, with the same mill filling. T2 feedrate is 1.03 tph, with 270% recycle, T4 is 1.14 tph with 430% recycle - far higher when operating with double the open area.

Marked rocks and ceramic balls from 19 to 102 mm were used as sized tracers. One set of tests measured transport time through and out of the mill during operation. Marked rocks were dumped in a batch and timed as seen exiting the discharge to provide a residence time distribution and thus in-mill velocity, Figure 1a. The second set was used to calculate transport velocity in the mill by dumping a batch in around 2 min before crash-stopping the mill – the idea being to stop the mill before any were discharged. This also allowed transport velocities of rocks larger than the discharge port to be measured. The smaller rocks were added as one batch then the large in 3 batches at 10s intervals as a practical matter of coping with the task. The rocks were manually picked out and the distance from the end of the mill measured. The mill was barred to reveal most of the tracer rocks after each trial. This provided a distribution of transport distance in the mill for a given time, Figure 1b, and in-mill velocities, dashed distributions in Figure 1a.

There is a wide distribution in velocities, with the coarser particles being higher. The tracer locations show that the large rocks (+102) were piling up at the discharge end prior to crash-stopping within the 2 minute transport period, truncating the natural tail of the distribution, so some rocks have even higher velocities than calculated from this data.

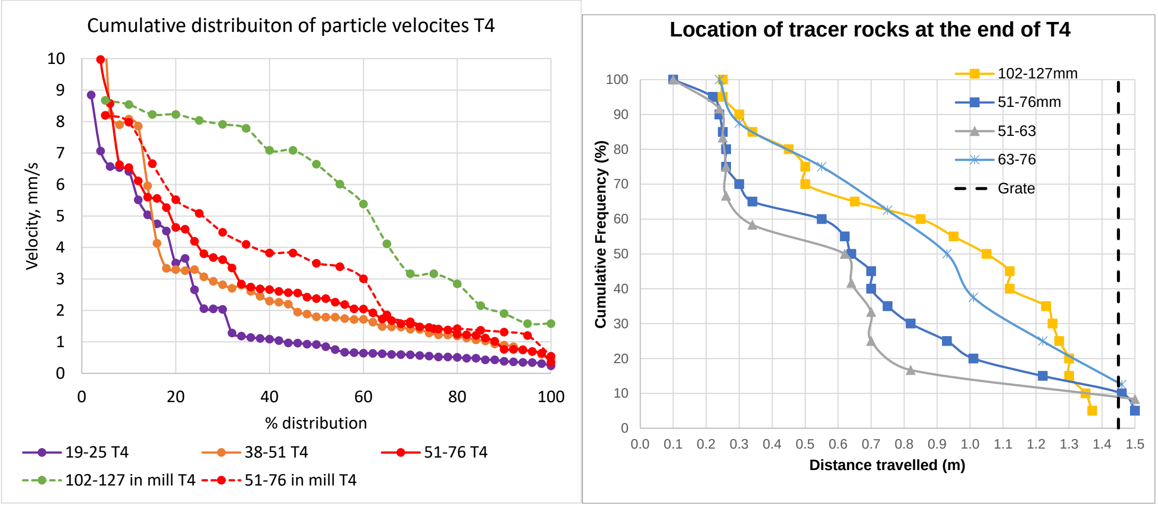

Figure 1 Measured distributions of (a) tracer particle velocity and (b) in-mill location

Figure 2 presents plots of calculated axial velocities for the tracers based on the median values of the distributions. Superficial velocity is calculated off the superficial residence time of hold-up/feedrate. Water velocity is similarly based off water holdup and addition.

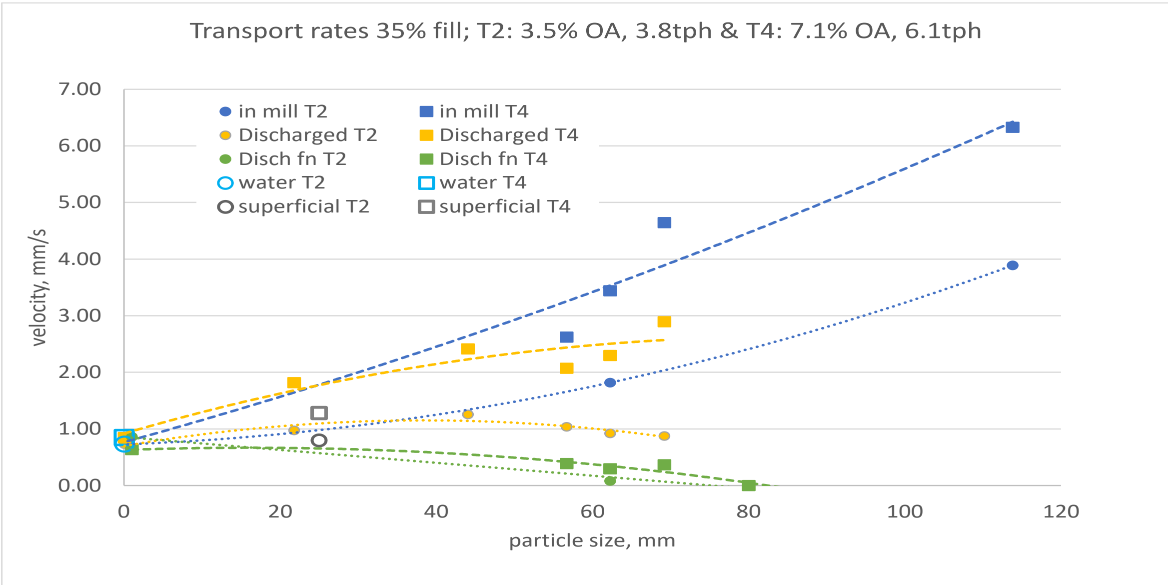

Figure 2. Measured transport speeds of different size tracers for two open areas

The large rocks have a far higher transport rate, of the order 7 times the fine particles. The higher open area influences the in-mill velocity as the total feedrate is 50% higher, with a corresponding increase in superficial velocity. The discharge velocity is the net for transport along the mill and then out the discharge. This is close to the in-mill velocity below 40 mm, but then as expected it drops off due to selectivity at the discharge grate. The Discharge function is the calculated 'velocity' through the discharge - the median rate at which the particles pass through the final slice and the discharge ports. This decays from the same as the in-mill velocity down to zero at the port size.

The data demonstrates how distributed the transport and residence time is in a tumbling mill, justifying the need to further understand and model this somewhat complex phenomenon. It should be noted the high circulation of pebbles was used to specifically highlight transport phenomena, so the differences in normal operating conditions may be less substantial. To the authors' knowledge this data is unique. The distribution of calculated transport rates is somewhat surprising and certainly enlightening.

Solubility and Dissolution Kinetics Enhancement of a Hydrophobic Drug Using Dry Milling Mechanochemical Approach

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Keywords: Dry milling; Indomethacin; Lysin; Dissolution rate.

A large proportion of currently available drugs or new molecules of potential pharmaceutical interest have limited bioavailability due to poor dissolution properties (solubility and dissolution rate), which limits the choice of technologies that can be used to deliver them to patients [1]. Several methods such as milling, micronization, solid dispersion, complexation, mechanochemical combination with suitable excipients, spray drying, or the use of supercritical fluid technologies can be used to overcome this problem. The advantage of dry milling lies in the absence of an organic solvent. The strategy adopted here consists of using two dry milling tools the Pulverisette 0® (sample named INDP2) and the CryoMill® (sample named INDCM3) to reduce the size of such an active ingredient, indomethacin (IND), and to co-mill this active ingredient in presence of a judiciously chosen excipient (Lysin – LYS) to improve the passage of the active molecule in biological aqueous liquids through a physico-chemical form allowing a significant improvement in the solubility and dissolution rate of this active substance.

The characteristics particle size of the milled and co-milled products, as presented in Table 1, show slightly decrease on D [4;3]. Besides, around 50% of decrease was observed on D [3;2]. This behaviour could be related to agglomeration during milling.

Sample	*Span*	*D[4;3] (µm)*	*D[3;2] (µm)*
Raw IND	1.7	34.2	15.3
INDP2	0.7	11.0	4.3
INDCM3	2.4	31.8	9.7
INDP2/LYS (1:0.25)	3.9	5.2	2.1
INDP2/LYS (1:1)	2.3	6.6	3.0
INDCM3/LYS (1:0.25)	2.1	4.9	2.6
INDCM3/LYS(1:1)	Data not acquired		

Table 1. Particle size data.

The solid state of samples was obtained by XRPD analysis. Figure 1 shows X-ray diffraction peaks from the crystalline form of the starting γ-indomethacin and lysin. However, it was found that an amorphous halo begins to form as the amount of lysin in the co-grind increases. This halo, more pronounced for the INDCM3/LYS 1:1 (w/w), highlights a transition to an amorphous state that is possibly related to the co-grinding process [2].

According to FTIR analysis (Fig. 2) a disappearance of the vibrational band of the free carboxylic group (C=O) at $\nu = 1714\ cm^{-1}$ and the insertion of the -NH3 strain at $1504\ cm^{-1}$ into the broad peak present around $1582\ cm^{-1}$ corresponding to the band of the ionized carboxyl group (COO-). This phenomenon may suggest the formation of a γ-indomethacin lysinate due to its ionization by mechanochemical effect [3].

Figure 3 A and B show the solubility improvement of co-grinded IND compared raw IND. The relative solubility indicates that the solubility has been improved by between 150% and 700% at 24h. Beside solubility enhancement, IND grinded stability in solution is also noticed up to 48h. The dissolution testing of the IND/LYS co-milled powders showed very fast dissolution kinetics, compared to raw IND, resulting in a high dissolution rate of the active substance, between 74 and 100% in the first 15 minutes (See Figure 3 C and D).

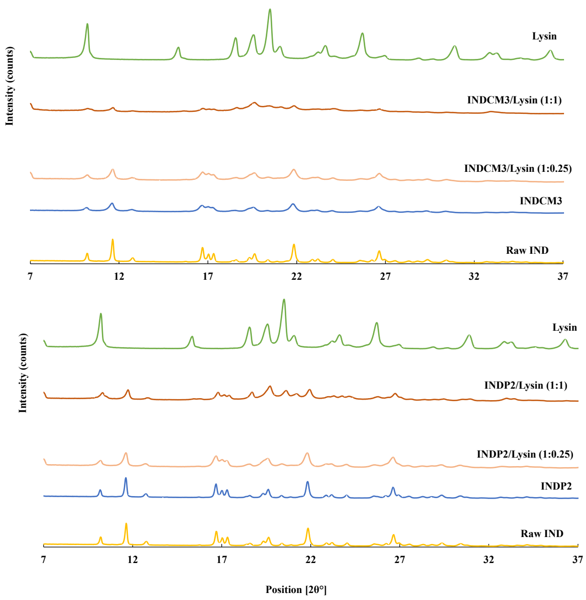

Figure 1. Diffractometer of IND raw and co-milled.

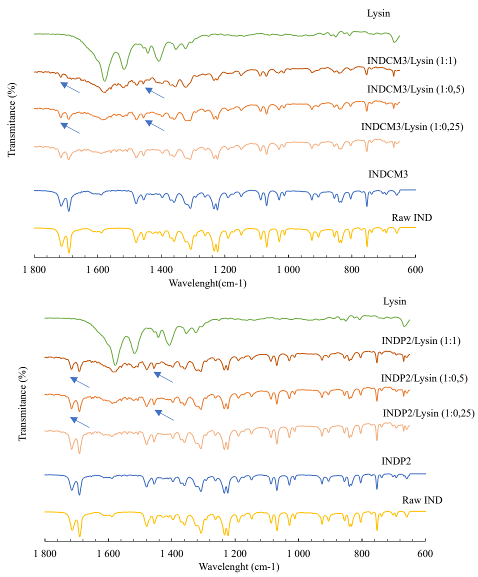

Figure 2. FTIR spectra of LYS, IND raw and co-milled

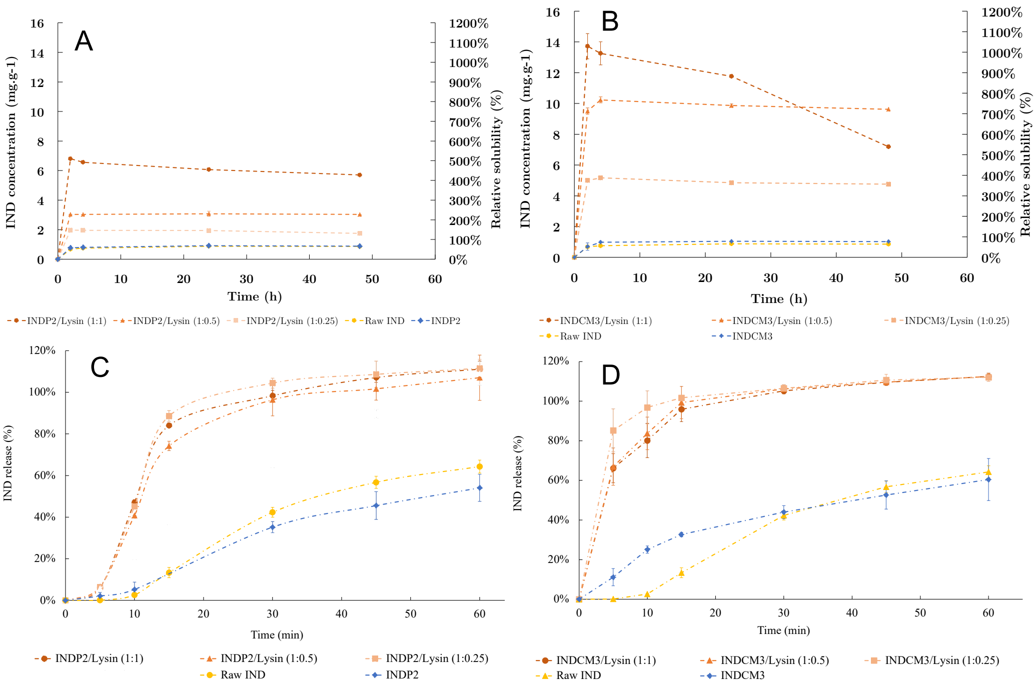

Figure 2. Solubility (A and B) and Dissolution kinetics (C and D) results of IND raw and co-milled.

CONCLUSION

The solubility and dissolution rate are improved for milled and co-milled IND. According to this study, dry grinding process, as a solvent free technique, can be a promising tool to improve solubility and dissolution kinetics of IND formulations, in particular, for the case of LYS used as an excipient. Otherwise, a better comprehension of the mechanism of indomethacin lysinate formation could be confirmed using Raman spectroscopy.

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Effect of stress conditions on mechanochemical synthesis of solid electrolytes in different high energy media mills

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Keywords: Mechanochemical reaction, high energy media mill, planetary ball mill, stirred media mill, solid electrolyte, stress energy, stress frequency, stress conditions

All-solid-state-batteries (ASSB) will most probably exceed the performance of lithium-ion batteries based on liquid electrolyte [1,2]. Against that in ASSB different solid electrolytes based either on polymers, oxides, sulfides or halides are employed. The different ASSB have pros and cons, but based on actual literature sulfides show the highest ionic conductivities. Especially thiophosphates have attracted broad interest because of their high Li+ conductivity and compliant mechanical properties [3]. However, still challenges have to be solved: beside higher energy densities and, especially, electrochemical cycling stability, the scaling of material production is an important issue. Currently, there are still a lot of hurdles for upscaling the synthesis and processing routes of solid electrolytes (SE).

The focus of our work is to evaluate and establish new scalable process strategies to produce thiophosphates by mechanochemical synthesis applying high energy ball milling. Their synthesis can be performed by high energy ball milling. A systematic parameter study was carried out in order to get a better understanding of process-structure-property-relations and to find optimized stressing conditions for the mechanochemical synthesis of the thiophosphates Li3PS4. In order to understand the effect of the main operating parameters, the different stressing conditions within the different high energy ball mills have been simulated using DEM. By this the stress energies and stress frequencies within the different mills was quantified and compared. Moreover, the effective power was calculated as product of stress energy and stress frequency. It could be shown that the conversion ratio is overall a function of the specific normal power input. At a constant conversion ratio, the required stress number is a clear function of the stress intensity (see Figure 1).

In fact, the process was successfully improved in terms of process efficiency, process time and additional possibilities to design the product properties by varying the stress energy of the grinding media. The highest stress energies at maximum rotational speeds enabled the fastest synthesis in less than 5 hours, which is a significant reduction of process time, compared to previously reported mechanochemical processes which run for at least one day or longer. The optimized process not only shows a minimum of required conversion time but also proves a minimum of specific energy to complete the reaction. Moreover, this specific synthesis combines a competitive ionic conductivity with the positive effects of fine particle sizes and the highest product yield in comparison to the remaining investigated parameter sets. While mechanochemical processes usually cause amorphization of particles, milling with high stress energies led to the partly formation of crystalline β-Li3PS4. Therefore, besides process time, efficiency and particle properties, choice of process parameters also effects crystallinity, enabling many possibilities for material and product design in a single mechanochemical process.

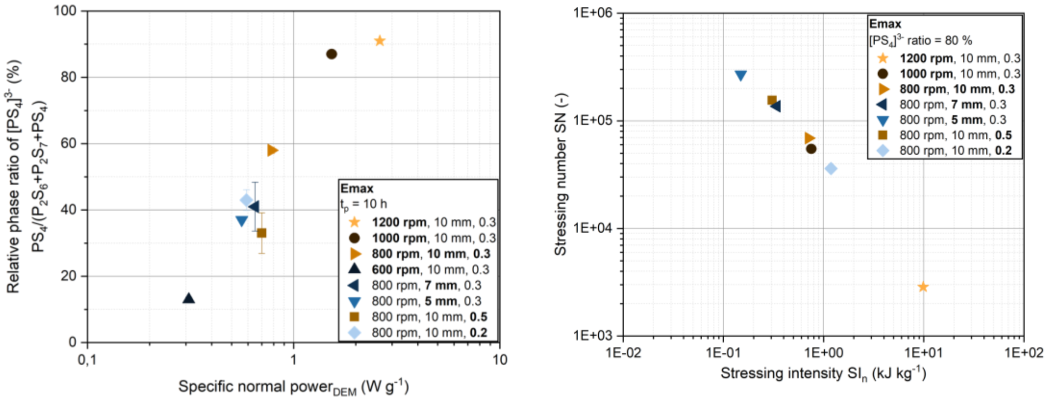

Figure 1. Conversion ratio (relative phase ratio) as function of specific normal power (left) and stress number as function of stress intensity for a conversion ratio of 80%

Based on the reductions in overall process time down to a few hours in laboratory scale, the mechanochemical process was scaled-up using a 2 Liter stirred media mill. Using the stirred media mill which was operated in multi-passage mode in a dry room more than 90% conversion could be achieved after a residence time of about one hour.

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Attrition and leaching: two synergetic processes for passivation limited hydrometallurgical processes

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Keywords: attrition, leaching, hydrometallurgy, surface passivation.

Hydrometallurgical processes seemingly as different in appearance as the extraction of copper from a chalcopyrite ore in a sulfuric acid solution and the mineral carbonation of magnesium silicates in CO_2-enriched water have in fact a major common denominator. Indeed, both situations are limited in rate and / or extent of reaction by the formation of diffusion limiting layers on the surface of reactive particles [1,2]. This situation is by no means an isolated case and can be found in many hydrometallurgical contexts.

This recurring problem can be solved in one and the same process, which consists in conducting the leaching step inside an attrition chamber. The coupling between attrition and leaching provides the leaching process with continuously refreshed reactive surfaces, which yields both increased leaching kinetics and high extraction rates. The objective of this paper is to exemplify the simplicity and universality of this mechanically improved leaching process, especially in comparison with alternative solutions to control the formation of surface layers by chemical methods.

In this paper, two significantly different ore processing situations are considered. The first one is the aqueous mineral carbonation of ferronickel slags, an industrial waste produced by the New Caledonian pyrometallurgical industry. The second is the acid leaching of chalcopyrite from the Swedish Aïtik mine. Both processes use a leaching step, that of magnesium in the case of the slags, and that of copper in the case of chalcopyrite. In the first case, the rate limiting mechanism is the formation of a Si-rich amorphous layer on the surface of the Mg-Si rich slag particles. In the second case, the passivation is mainly due to the precipitation of elemental sulfur on the surface of the chalcopyrite particles. Particle leaching being a surface area driven process, even when ground very finely, the leaching yield of both feedstocks under the most favorable thermodynamic conditions does not exceed 20-30%, leaving behind 70% or more unrecovered Mg or Cu.

In both cases however, notwithstanding the different time scales for both processes due to the difference in their intrinsic kinetics, the combination of leaching and attrition frees the process from the passivation limit and leads to a giant leap in extraction yield (Figure 1). Inside a 300 mL attrition-leaching reactor, which is basically the fusion of an autoclave and a stirred bead mill (1mm Yttria stabilized zirconia grinding beads; aqueous slurry with 10wt% minus 100µm ferronickel slags, P_{CO2} = 10 bar, T=150°C), Mg extraction yields as high as 70% are obtained in the case of nickel slag particles leaching. Using a 4L attrition-leaching reactor for chalcopyrite (1mm glass grinding beads; 3.25 wt% chalcopyrite slurry with 17.8M H_2SO_4, pH=1.3, Eh=700mV vs. SHE, T=42°C), the Cu yield jumps to 80% without reaching a plateau.

By investigating different configurations of the process for both cases, namely leaching only, attrition followed by leaching, and concomitant attrition and leaching, the paper demonstrates the beneficial synergy between both processes. This result, as it applies to two intrinsically different hydrometallurgical processes, leads to the claim that the coupled attrition-leaching process has wide applicability. Based on analysis of the products using a wide array of analytical techniques, the paper also sheds light on the mechanisms of the coupled attrition-leaching process for passivation limited hydrometallurgical processes.

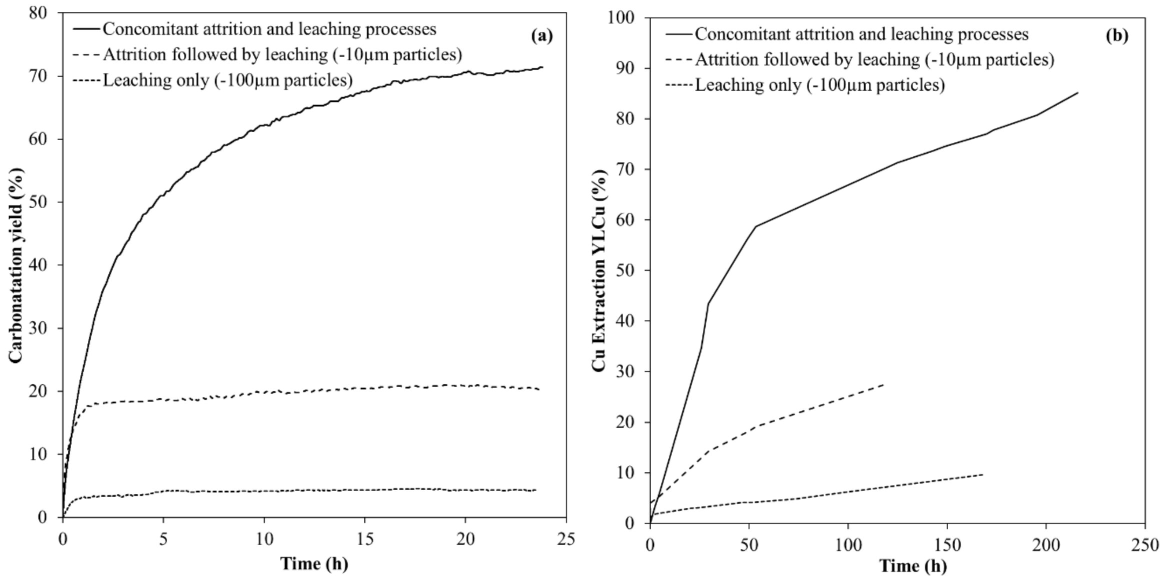

Figure 1. Illustration of the synergetic effect of attrition and leaching on (a) the carbonation yield for ferronickel slags and (b) the copper extraction yield for chalcopyrite.

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Development of a Guide Ring Classifier for Fine Classification

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Keywords: classification, flow guidance

A novel design for a guide ring classifier is shown. The main idea behind the design is to achieve efficient classification by significant improvement of both the particle routing and the air flow into the classification zone. Computational Fluid Dynamics (CFD) were used to a great extent in the design process. Preliminary tests have shown good agreement between the predicted and measured performance parameters on the one hand as well as a high classification efficiency on the other hand.

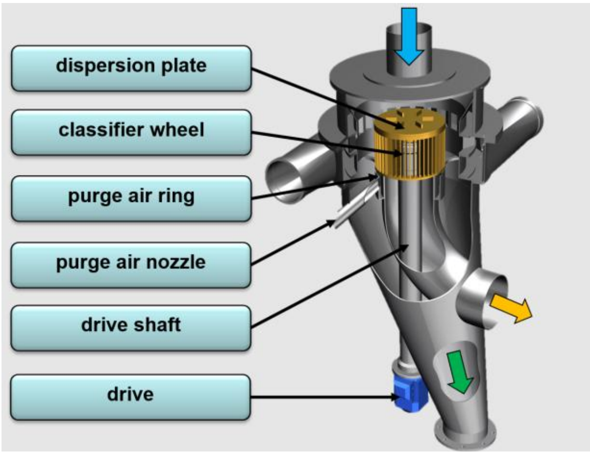

Figure 1: Guide Ring Classifier

The operating principle is depicted in Figure 1: Guide Ring ClassifierFigure 1: The ground product (blue arrow) from an upstream grinding system is transported gravimetrically into the classifier housing. Once the product stream hits the dispersion plate, it is deflected towards the outer circumference of the classifier wheel. It will then enter the classification zone around the classifier wheel from above. At the same time, the process gas (usually air) enters the grinding zone via the two horizontal circular tubes. The product is then classified at the classifier wheel into the coarse and the fine fraction. The coarse fraction is transported gravimetrically out of the classifier (green arrow). The fine fractions is being transported pneumatically through the classifier outlet (yellow arrow).

The fact that the process gas and the product enter the classification zone via different routes offers the possibility to control both of these streams to a certain extent. For good classification results, both the radial and tangential velocity component of the process gas have to correspond to the desired cut condition at the outer circumference of the classifier wheel. The efficiency of the classification process is strongly dependent on the flow situation in the classification zone, i.e., in the immediate vicinity of the outer circumference of the dynamic classifier wheel [1] [2] [3] [4] [5].

The guide ring was designed to achieve this favorable flow condition in combination with a classifier wheel designed for superfine cuts. The design principle of both the classifier wheel and the guide ring correspond to the design of the NEA PMX [6], and the NEA DCX [7].

The design process was conducted using Computational Fluid Dynamics (CFD). A first design concept was used as input geometry, which was then analyzed and improved with focus on flow guidance and energy efficiency.

Results from lab scale tests show good agreement with the predicted performance. The presentation will show the development and design process as well as detailed results.

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Conceptual model structure of transport in mills

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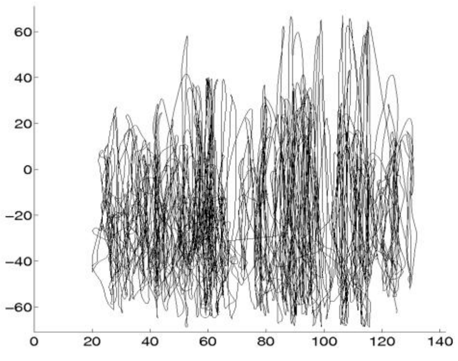
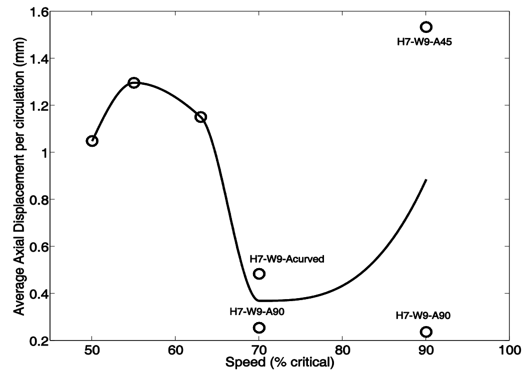
Keywords: Mill mass transport, particle transport, tumbling mills, grinding mills.

It is understood that rocks do not instantaneously transport along a tumbling grinding mill and are consequently not perfectly mixed, but this is not captures in mill modelling. A lack of understanding of the transport phenomenon and the difficulty of modelling realistic transport in a complex tumbling system, contribute this. Visual evidence of segregation in mills and lag time of mill response to changes in feed support incorporating transport in dynamic models to underpin dynamic simulation for design and for model-based control.

Insights on the motion in mills have been obtained from a range of experimental and industrial tests. The work of Powell [1] identified axial motion in a small laboratory mill when analysing the motion with bi-planar X-ray filming. The continuation work by Govender [2] provided improved data and quantified the axial transport that occurs even in batch milling mode, as illustrated in Figure 1. This demonstrates that particles can drift back and forth in the mill, leading to diffusive transport, which also allows back-mixing. Continued work in this area has advanced to the use of PEPT filming, using radioactive tracers in both batch [3] and continuously fed [4] laboratory mills. Based on this research Govender [5] refs has developed a diffusion theory on the cascading flow of particles in a tumbling batch mill. This work has provided the insights and basis for understanding the non-intuitive drivers of particle transport in mills.



Axial view of tracked particle



Axial displacement per circulation with mill speed

Figure 1. Measured axial motion in the laboratory mill [2]

Tracer tests reported in the literature are limited to the slurry using liquid tracers. This does not provide insights to the transport of the rocks and balls in a mill. The work on pilot tracer tests [6] provides the first data upon which to base the development of a model of rock transport in a continuously operating mill. In analysing this data, it was found that the measured transport rates and distribution along the mill in the tests conducted for in-mill segregation demonstrated the dominance of the surface cascading flow phenomena.

The conceptual model is built off 4-sub-processes (as this stage).

- Advection – bulk flow along the mill. This corresponds to the transport rate of the finer material, taken as 1-10 mm in the first implementation. This can be calculated based on superficial residence and thus transport rate of the measured mass of this size range in the mill load. When estimating this, it aligned with the water flow velocity, providing a pointer to calibrating this in future modelling.
- Bulk diffusion. This is classic diffusion within the granular, stirred media, with particles shifting into available spaces that are formed during the mixing process. It is inferred from the experimental data to be a low rate.
- Cascade diffusion created by the flow of particles down the cascading surface of the descending charge. This is strongly size-dependent, with larger particles having a larger cascade angle and thus displacement, plus the fraction exposed to cascade is greater for larger particles.
- Discharge grate. This hinders discharge of particles, allowing zero above grate size and increasing to a maximum for small particles below about 10% of grate aperture. The hinderance to flow forces particles to crowd and then backflow through diffusion.

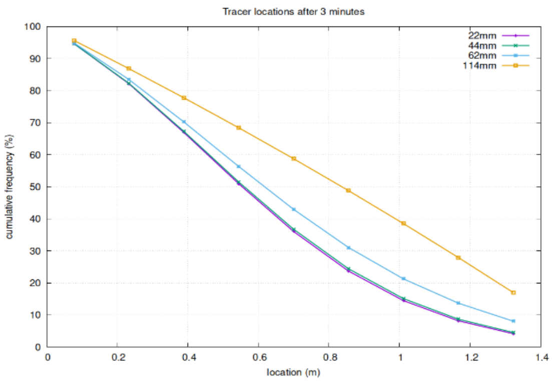


Figure 2. Modelled distribution of particles along a mill, after being added as a batch.

A simulation outcome (without discharge grate) is illustrated in Figure 2. This critical factor of cascade diffusion may well unlock the ability to predict the transport and segregation by size of particle in a tumbling mill. The ideas presented here are being used to build a transport and segregation model for tumbling mills.

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Smart Screening for Optimum Efficiency

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Keywords: Screening efficiency, statistical resonance,

Vibrating screens are widely applied in mechanical processing to split a disperse particle flow in two or more size classes. The screening efficiency is often used to characterize the quality of the partition. It is influenced by a large number of material, machine and operational parameters. Fluctuating material parameters, in particular those of natural materials, can usually be controlled with substantial effort only. Even the results gained with such effort are often not satisfying.

The options for an operator to react timely on a temporary deterioration of screening efficiency are usually limited to throughput control. Therefore, so-called smart screening machines where developed which allow the online adjustment of further parameters, such as oscillating frequency, direction and the pattern of oscillation from circular via oval to linear. The question is, how to find the best parameter combination with the least effort.

While a number of modelling tools were published so far which allow an insight into various aspects of vibrating screening, calibration is often a challenge and experimental investigations are still indispensable. At the Institute of Mineral Processing Machines and Recycling Systems Technology (IART) of the TU Bergakademie Freiberg a test rig was installed in close collaboration with Haver & Boecker Niagara, which allows a rapid online determination of screening efficiency and thus the optimum machine parameter set by varying a number of parameters systematically, potentially even automated.

The material is guided in a closed circuit with weigh scales measuring cyclically the screen overflow and underflow. The relationship between those two values allows conclusions on the screening efficiency acc. to [1]. The underflow and overflow materials are put in layers on a collecting conveyor and lifted via a pendulum bucket elevator back to the receiving hopper. The dosing of the feed to the screening is done volumetrically with good accuracy. While certain pre-selected parameters, including those mentioned above as well as the screen slope, may be modified systematically within a certain range in continuous operation of the circuit, the combination with the highest screening efficiency can be determined.

This approach allows to determine the best parameter combination with much less effort than the batch tests common so far. A wider range of machine parameter combinations can be investigated in a shorter period of time. They may as well be used as starting parameter field for machine learning (ML) or artificial intelligence (AI) approaches. It is expected that respective smart screens will gain their place also directly in the industry, wherever a fluctuation of material parameters adversely influence the performance of vibratory screen applications.

The screening system developed was tested using a screen machine HAVER T-Class D50 with 0.5 m² screen area and two independently controlled eccentric drives. The test material dry quartzite gravel 2…8 mm was fed with a specific throughput 14 t/(m²h) on a wire mesh deck with 5 mm aperture and a wire thickness of 1.6 mm in circular motion. Frequency, stroke and screen deck slope were varied (1,000 … 1,300 RPM; 1.8 … 2.4 mm, 10 … 18°).

It could be shown, that for the various parameter combinations tested, the best screening efficiency was reached at an acceleration of $k_v \approx 2.75$ g, see Figure 1. This result was attained with various combinations of frequency and stroke, all leading, however, to the same optimum acceleration. This was quite surprising, since the common understanding is, that the optimum acceleration for a single particle motion is around $k_v \approx 3.3$ g, in so-called statistical resonance [2]. Often, vibrating screens are even operated at acceleration levels of $k_v > 4$ g to compensate for particle-particle interactions or even sometimes substantially higher, if material is difficult to screen.

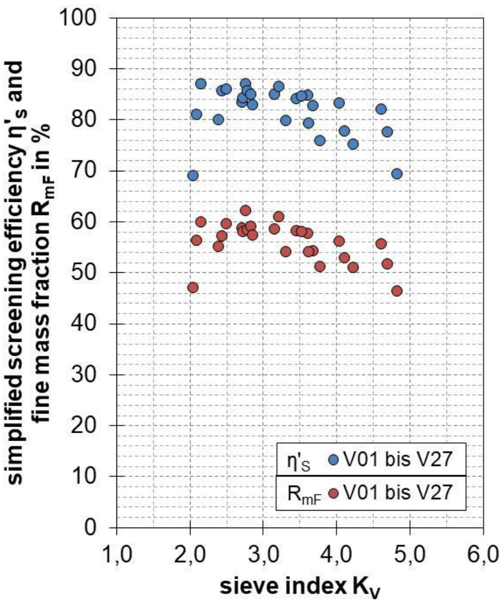

Figure 1. Sieving result as a function of sieve index k_v for all tests V01-V27 [3]

A reduction in the k_v value, however, would reduce mechanical stresses to the machine components, hence wear and increase the live time of the screen deck. Certainly, this value depends substantially also on other factors. Nevertheless, with the new test rig, various parameter combinations can be tested quickly, to be included in smart machine control models. Thus, future comminution-classification circuits can be better adjusted to chang-ing feed material or other ambient conditions.

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Particle fractionation using a crossflow with superimposed electrical field

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Keywords: Classification, Suspension, Crossflow Filtration, CFD-Simulation.

In the course of the technical development of production, its processing and recycling of particulate materials, the demand for increasingly smaller, highly specific particle systems in suspensions is growing. The physical properties of a particle collective are directly related not only to the particle size, but also the particle shape, surface properties and chemical composition of the individual particles. Suspensions often form highly complex particle collectives which contain these characteristics in various distributions. In well-known separation processes, the particles are either classified according to a certain grain size or sorted by a physical characteristic. However, in many applications in which finely dispersed multicomponent mixtures of particles with sizes < 10 µm with different properties are to be separated in industrially relevant quantities, the use of a single separating characteristic is often no longer sufficient. This work aims to investigate and develop a new method for continuous, multidimensional fractionation of suspensions. The method is based on crossflow filtration and extends it by an electrical field [1 – 4].

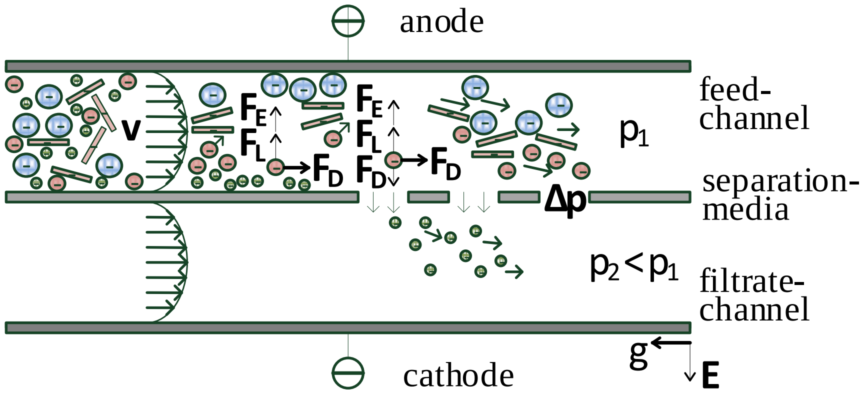

Figure 1. Scheme of the separation process.

In the process two parallel main flows, both in laminar regime, are realised in parallel channels: one containing a feed suspension (feed channel) and the other a particle-free liquid (filtrate channel). Based on the size-dependent hydrodynamic forces acting in a laminar flow on the particles, bigger particles are transported into the core flow faster than the smaller particles. The smaller particles are less influenced by this effect and remain longer in the wall boundary layer. To separate them, a separation medium with pores bigger than the particle size is adjusted between the two channels. A very small differential pressure between the two channels allows a suction of a small volume of the suspension flow containing only small particles in the filtrate channel. By adding an electrical field, which is acting orthogonally to the main flow, the particles are subjected to an additional electrophoretic force. The magnitude and direction of this force are determined by the particle properties, in particular the electrophoretic mobility and the electrical field strength. The multidimensional fractionation of a suspension according to the separation characteristics, such as particle size, shape and electrophoretic mobility, can be achieved by the combination of the lift force F_L, drag force F_D and electrophoretic force F_E (Fig. 1).

A test setup is developed and used for experimental study of the separation efficiency. Additionally, a numerical study with computational fluid dynamics (CFD) is performed. Based on CFD, the optimal geometry of the channels and the separation medium are chosen, as well as the operation parameters for a hydrodynamic classification of particles < 10 µm are determined. Moreover, to describe the cut size, the motion of the particles in the laminar boundary layer is investigated with Lagrangian particle-tracking in CFD (one-way coupling). For this, we implemented the electrophoretic force in CFD. Fig. 2 illustrates the results of a CFD simulation for a single-gap geometry.

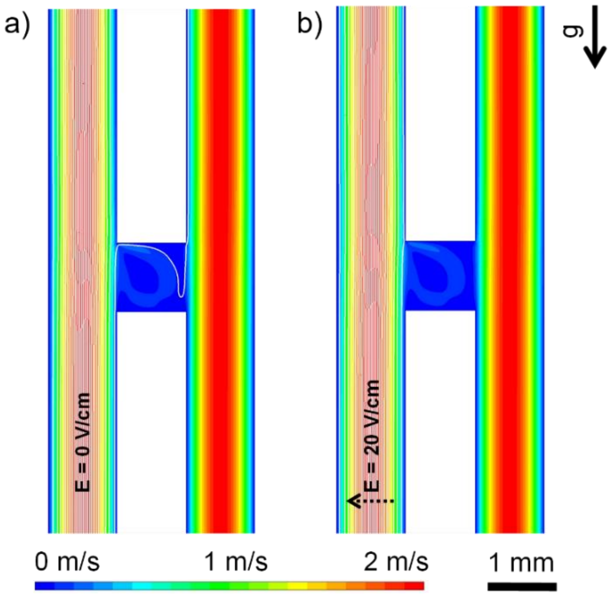

Figure 2. CFD-Simulation of the flow channel with particle trajectories (diameter 5 µm, negative charge of -3.2 $(\mu m \cdot s^{-1})/(V \cdot cm^{-1})$) for the case without (a) and with electrical field (field strength of 20 $\cdot cm^{-1}$) (b).

In a) a constant differential pressure between the suspension-carrying channel (left) and the filtrate-carrying channel (right) leads to an exchange volume flow, which moves particles from the boundary layer into the filtrate channel. In the second case (Fig.2, b)) by the same flow conditions and particle properties, the acting electrophoretic force leads to a deflection of the particle trajectories according to the direction of the field strength vector $\vec{E}$ and particle charge.

The separation behaviour of different materials with different particle size distributions, shape and electrical charge is investigated. The effect of influencing parameters such as particle concentration, pH value or additives are determined using various methods. The results of CFD simulations and experiments are used for the optimization of the fractionation process to increase the separation efficiency and perform the scale-up.

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About dynamic modeling of nanoparticle classification in tubular bowl centrifuges

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Keywords: Dynamic modeling, classification, nanoparticles, real-time simulation

Solid bowl centrifuges are sedimenting centrifuges and widely applied in almost all parts of the process industry for different tasks such as solid-liquid separation, mechanical dewatering, degritting, sorting and classification. A special type of this apparatus class is the tubular bowl centrifuge, which operates semi-continuously and achieves very high g-forces of up to 80,000, which can be achieved by the slim rotor design. This allows the separation and classification of nanoparticles in larger quantities on a technical scale, which depends on the properties of the particulate matter and the process conditions. Up to now, theoretical models to describe the classification in tubular bowl centrifuges have achieved only insufficient quality in predicting the grade efficiency. Reasons are the complexity of the flow pattern, the not considered real sedimentation behavior of particulate matter and the sediment build-up, which influences the residence time behavior of the tubular bowl centrifuge.

This work focuses on the detailed dynamic modeling of the process behavior of tubular bowl centrifuges under the prerequisite of real-time capability to permit process simulations for the entire process time of several hours in a few seconds of simulation time. It is crucial to represent the dynamic behavior, which is caused by the sediment build-up of separated particles and which significantly influences the residence time behavior in the apparatus. Only the detailed dynamic modeling allows further application possibilities like the model-predictive control or the implementation into flowsheet simulation tools to simulate the upstream and downstream process of, for example, products from a precipitation reaction.

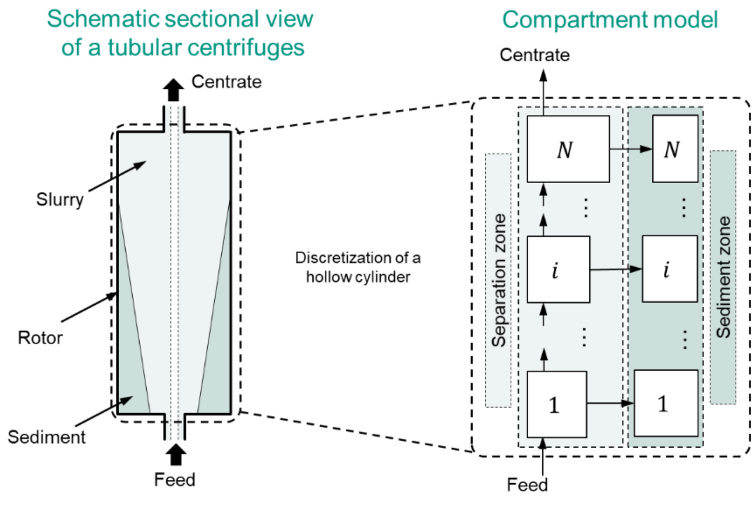

Figure 1: Schematic representation of the compartment model of a tubular bowl centrifuge.

The presented dynamic model for tubular bowl centrifuges is based on a compartment approach (see Fig. 1), which allows to connect the settling behavior and the sediment build-up at the inner rotor wall. Both, the settling behavior and the sediment build-up depend mainly on the particle properties such as particle size distribution, particle shape and slurry composition as well as on the flow conditions and requires a temporal and spatial discretization along the axis of the centrifuge rotor, which is considered by the interconnection of different compartments. The developed approach enables for the first-time the theoretical investigation of the temporal change of grade efficiency for semi-continuously operating tubular bowl centrifuges. The applicability and the accuracy of the dynamic model and the existing influencing variables (volumetric flow, rotor speed) are shown for different experimental and simulative scenarios. In addition to the applicability and validation, however, current limitations of the model are also discussed extensively.

Ultra fine classification with superheated steam

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Keywords: classification, Steam classification, dispersion, closed loop operation

With steam jet mills, it is possible to achieve very fine grinding results. The reason for these possibilities are connected on one hand to the high energy input and on the other hand to the finer classification due to the thermodynamic properties of steam. Motivating for this new development was to decouple the energy intensive grinding process from the fine classification. For this reason, the presented research will show a new classification development by changing the classification media from air to superheated steam. The advantages and challenges of superheated steam are transferred from the well-known grinding with steam to the classifying process in order to achieve a significantly finer separation cut compared to state-of-the-art air classifying processes.

A plant of this concept is operated in closed loop. Normally closed loop systems for gas has to be cooled, due to the difference in energy flows between added and removed energy. In the new development the energy overhead is compensated by the evaporation of water. The total system is out balanced by the choice of the process parameters. An additional steam generator is not necessary.

Model-based control of centrifugal classification for intelligent Direct Recycling of Li-ion battery electrodes

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Keywords: Dynamic Modelling, Model-Based Control, Digitalization, Direct Battery Recycling.

The process industry is currently undergoing various changes, to a large extent induced by the growing request for more resource-efficient production and product cycles and motivated by a range of economic and ecological reasons. Thereby, the focus lies not only on installing new plants for novel processes, but also on optimal operation of existing production plants, enabling optimal exploitation of energy and materials. However, when processes are very complex, conventional practices are often not suitable to operate the process satisfactorily close to the optimum. The same challenge arises when specific product properties are demanded, but the process behavior or feed properties change during operation. Accurate setting of operational parameters is required, which must be realized through a sensible control strategy. In such complex cases, a model-based control is a potentially sound solution.

At the same time, mobility is about to undergo a fundamental transformation towards electric vehicles. This will certainly entail a growing demand for materials needed in batteries (Li, Mn, Co for example) as well as increase the urgency to develop recycling processes for end-of-life batteries. There are recycling approaches, but further development is necessary to achieve thoroughly safe, efficient, and profitable processes for environment, economy and functionality of recyclate-based batteries.

The presented investigation concentrates on centrifugation as a potential step in a battery recycling process chain. The well-known unit operation is examined as the key step – separation of electrode constituents for further selective treatment – in a newly developed direct recycling approach for Li-ion batteries. Exemplary processed materials are, e.g., LFP or NMC, which have to be separated from conductive Carbon Black to receive specific reactivation treatment. The partially nano-sized particulate material enters a centrifuge to classify/ fractionate the constituents. In order to assure the separation of only one species from the watery dispersion, the operational parameters must be carefully set. Additionally, the separation conditions in centrifuges may vary over operation time due to the progressive sediment build-up inside the rotor. If this is not counteracted, centrate properties are time-dependent and the desired fractionation output is not attained. To achieve complete fractionation over the entire operation time, continuous control of the operating parameters, that are rotational speed and volumetric flow rate, is realized in form of a model-based control predefining the settings, combined with monitoring via on-line measurements.

The basis of this control approach is a real-time centrifuge simulation that describes particle separation and sediment build-up in the centrifuge rotor, covering the dynamic behavior and thus providing predictions of the process output (centrate particle size distribution, for example).

The model-based strategy aims to facilitate the control of the time-dependent output through a model covering the complexity of the centrifugation process. This way, it shall be possible to continuously accomplish the desired particle classification while, at the same time, given constraints like machinery limits are respected and varying conditions such as fluctuating inlet properties and outer disturbances are coped with.

However, with a broad variety of electrode materials in the market and even more under development, the necessity for a flexible, "learning" process control is definite. To cope with different materials and be able to extend the spectrum in future application, an approach for smart determination of the materials' properties concerning centrifugation is being developed. It is based on on-line sensors on the one hand and optimization algorithms on the other, finally in a smart combination supplying reliable material data for sensible model-based control.

Modelling and Dynamic Flowsheet Simulation of a Cutting Mill for Use in Lithium-Ion Battery Recycling

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Keywords: Cutting Mill, Modelling, Lithium-Ion Battery Recycling, Dynamic Flowsheet Simulation

The recycling of lithium-ion batteries is becoming more important due to the rising demand on raw materials mainly through the increase of battery electric vehicles. Recycling helps to cover the increased need of valuable materials such as cobalt or lithium and to reduce the environmental impacts of raw material mining. Different processes exist to recycle lithium-ion batteries, including mechanical recycling, in which different comminution and separation processes are connected. In the mechanical process route considered here, at first the batteries are shredded under inert gas atmosphere. After this the electrolyte will be evaporated and the dried materials are fed into a zig-zag-separator to separate the heavy components. A second crushing step in a cutting mill is used to increase the yield of black mass which can then be screened. The last step is a second air classification with a zig-zag-separator to remove the separator from the copper and aluminum foils [1, 2].

With the use of dynamic flowsheet simulations complex processes and process chains can be simulated. For this simulation, the different sub processes are connected by using material and energy streams [3]. In the context of mechanical recycling the different battery materials have to be simulated for several connected comminution and separation processes. So changes in the boundary conditions as well as parameter changes of the first processes, for example a varying feed mass flow or varied process parameter of the shredder, influence the following processes. Therefore, dynamic flowsheet simulations are very suitable to simulate this process.

With the mechanical recycling as much valuable battery materials as possible should be separated with as little as possible contamination of other materials. Therefore, the mechanical recycling includes the second comminution step to decompose the inclusions of black mass and to remove the remained black mass on the current collector foils to get higher recycling rates. For this process step a cutting mill can be used [1]. The development of process models for the cutting mill and the dynamic simulation of these models helps to make predictions of the particle sizes of the different materials at the end of the process and the percentage of inclusions and remaining black mass on the current collector foils. This information can be used to determine the yield and impurities of the black mass in the following screening step. With the use of dynamic flowsheet simulations, the change of these output parameters can be simulated time-dependent under varying process parameters [3]. So the influence of different process and material parameters can be depicted and also the influence of process fluctuations can be considered. Therefore, when using online measurement technology, it is also possible to use appropriate models for process control. Due to the fact, that not many physical models for the processes within a cutting mill exist, empirical modelling was started.

Among the process parameters considered, which have an influence on the cutting process, are the rotational speed of the blades, the feed mass flow and the processing time. Also the processed materials are important for modelling. One challenge of this modelling are the multicomponent feed materials. The first model approach is based on cylindrical battery cells and one goal is to investigate the different results for the battery materials and find relationships to their material properties.

Apart from the different process and material parameters, the construction of the mill also influences the process. It can be assumed that the volume of the mill as well as the number of blades influence the number of cutting processes at each particle. Additionally, the screen mesh size at the bottom of the mill can be varied. The screen size influences the mass load and also the composition of the materials within the mill. Materials which are easier to cut or have a smaller feed particle sizes will leave the mill faster than other materials. In addition to this, the screen size influences the particle sizes of the different materials at the end of the process due to longer residence times for smaller mesh sizes. Based on experimental data, the particle size distribution for different screen mesh sizes and materials can be calculated. For this the experimental data were approximated with a Weibull-Distribution.

This equation was used as a basis to create a first model to predict the particle size distribution for different mesh sizes, which have not been determined experimentally. As shown in figure 1, the modelled particle size distribution is a good approximation for the experimental data. For the experimental and model data the resulting particle sizes of the materials decrease with smaller screen sizes due to the longer residence time. Comparing Figure (a) and (b), it can also be seen for two exemplary materials that different distributions occur for the materials. This is due to the different material properties whose influence is to be investigated in more detail.

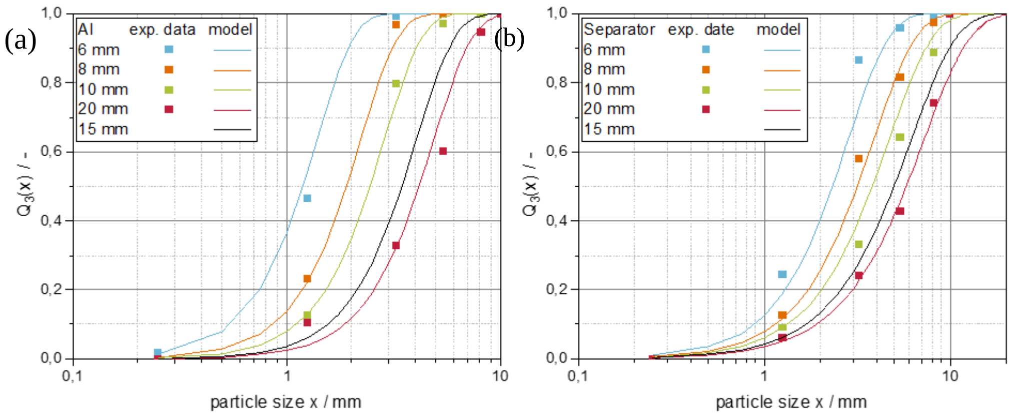

Figure 1. Experimental and model data for the particle size distribution of (a) aluminum and (b) the separator

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Recycling of refrigerators: Linking design decisions and liberation behaviour

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Keywords: recycling, pre-treatment, crushing, liberation.

Recycling is part of the circular economy contributing to the sustainable and secure supply of raw materials. Most consumer products, from cars to domestic appliances, consist of multi-material structures which form complex wastes after their use phase. Therefore and with the focus on sustainability, the design decisions of product manufacturers have a significant influence on recyclability. So far, methods exist to evaluate the performance of metallurgical recycling systems through process simulation [1]. However, there is a lack of methods to estimate the impact of design decisions on the liberation behaviour during crushing.

In a case study, a large-scale recycling campaign examined 100 refrigerators from the household sector in a commercial primary waste treatment facility. Initially, the fridges were analysed regarding e.g. initial mass, size, outer appearance, and manufacturer in order to be categorized into three fractions. Then the conventional pre-treatment steps were documented such as depollution, removal of glass shelfs, printed circuit boards and compressors. Finally, the mechanical processing started beginning with a two-stage crushing and subsequent separation in an air classifier (density sorting), magnetic and eddy current separator.

The main products of the mechanical processing are a PU rich, a ferrous, a non-ferrous and a plastics fraction. The last three of those were analysed in more detail in order to quantify the degree of liberation as well as the separation efficiency. Liberation was evaluated at particle level in terms of unliberated connections between different materials (material mixed particles) as well as connections of different components consisting of the same material (component mixed particles) as illustrated in figure 1. This data allows deducing the liberation efficiency, which affects sorting and final product qualities.

Furthermore, the liberation efficiency of different connection types (e.g. screwing, gluing, coating, snap-fitting) was identified. Coupling these insights with a material compatibility assessment for subsequent recovery processes [2], design recommendations have been derived for liberation-oriented choice of connection types and specific material combinations. For example, steel in the PU rich fraction causes problems during conveying and pelletizing and is therefore regarded as hazardous pollutant for subsequent processing. In contrast to that, aluminium in the ferrous fraction deceases slightly the product quality but can be removed easily during subsequent metallurgical refinement.

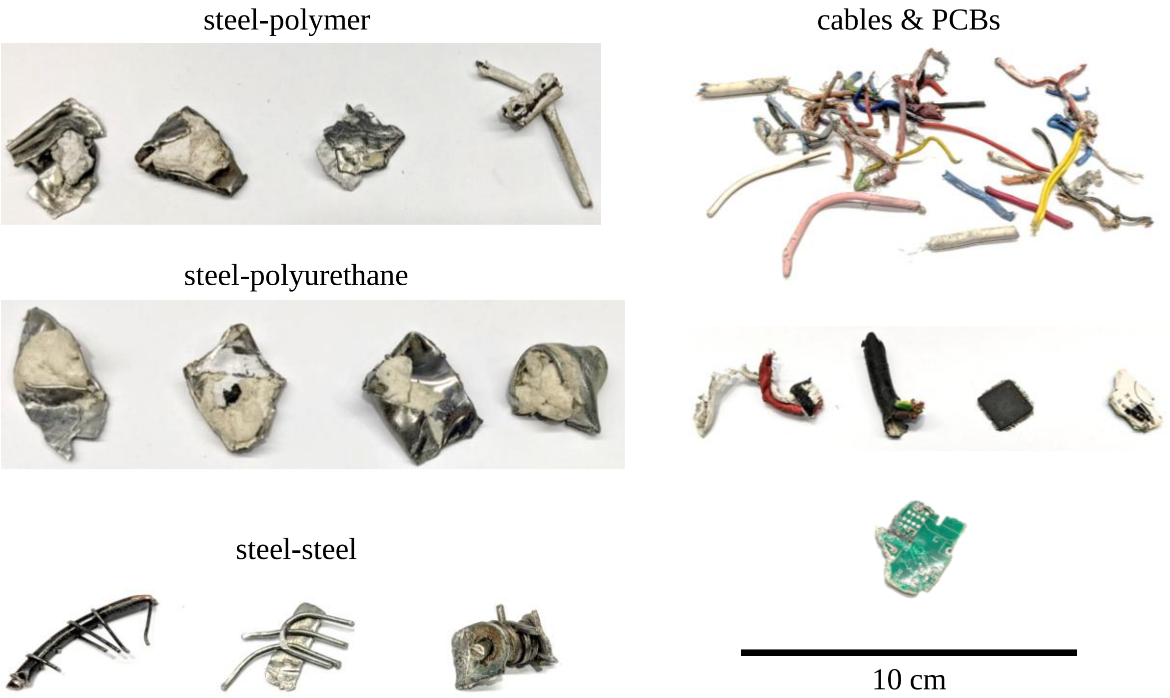

Figure 1. unliberated particles of different materials (steel-polymer, steel-PU, cables & printed circuit boards (PCBs)) as well as unliberated particles of the same material (steel-steel)

As an ongoing research topic, finite element method (FEM) simulations supplement these experimental investigations to enable the analysis of various multi-material designs. In these simulations, composites of metal and fibre-reinforced plastics are modelled in a crushing process and compared to laboratory scale experiments (figure 2) regarding their liberation potential during crushing and their overall recyclability. In the future, this methodical approach will allow assessing the effects of the product design on recyclability already in the design stage assisting the development of recycling-friendly products.

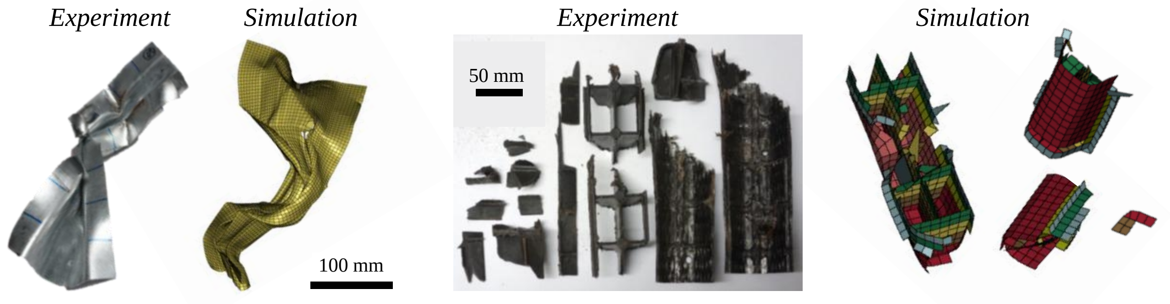

Figure 2. Comparison of experiment and FEM simulation of a crushing process (left: steel structure; right: fibre-reinforced plastics with rib structure)

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An experimental milling study on UO_2 powder: influence of the drum size and some milling parameters

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Keywords: ball milling, UO_2, nuclear fuel, scale-up.

In the framework of the nuclear fuel fabrication, the scale-up of the powder-grinding step is studied. This crucial step of nuclear fuel manufacturing is performed in order to obtain a suitable particle size and an optimal mixture as several components are considered.

A scaling factor was determined by Orozco et al. using a discrete element method to implement the particle dynamics and a particle breakage model [1]. It corresponds to a dimensionless parameter and is assumed to predict the powder particle breakage during a milling process in the case of coarse particles and without any balls (autogeneous milling).

Model checking experiments are carried out to assess the ability of the so-called scaling factor to describe ball milling on UO_2 fine powder. Different parameters are taken into account for these experiments. More precisely, two drum diameters (135 mm and 240 mm), different rotation speeds between 4.7 and 8.4 rad.s^{-1} are tested. Furthermore, the filling degree, defined by the ratio of the filling height to the drum radius, is varied between 0.6 and 1. Orthocylindric milling media (25 x 25 mm) are used for each batch test carried out for a duration of 120 min. For each experiment, process milling is stopped at predefined times to characterize milled powder evolution on samples. Specific surface area and particle size distribution measurements, as well as SEM observations are performed. A repeatability test is also undertaken in order to confirm the robustness of the applied method.

Results show an evolution of the specific surface area into two steps corresponding to fragmentation and agglomeration phenomena, in good agreement with literature [2, 3]. These mechanisms are confirmed by laser diffraction analysis using wet dispersion for the fragmentation and dry dispersion for the agglomeration (Figure 1).

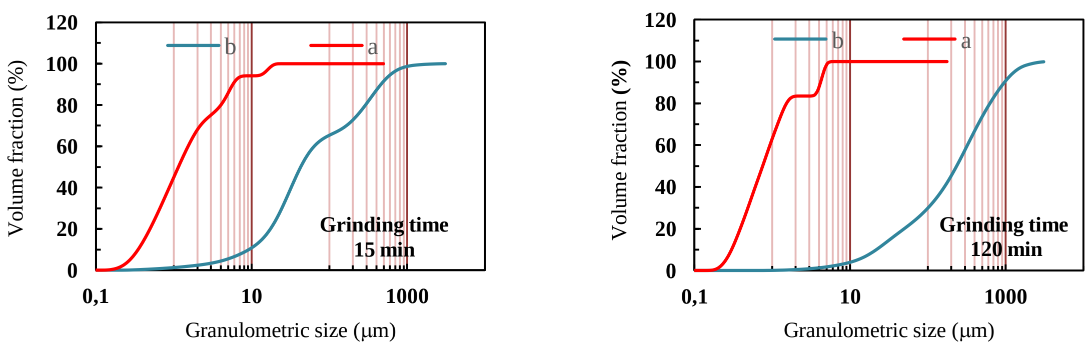

Figure 1. Granulometric analysis using wet (a) or dry (b) dispersion for a milling experiment considering a rotation speed of 6.9 rad.s^{-1}, a filling rate of 1 and a drum diameter of 135 mm for grinding times of 15 and 120 min respectively

Furthermore, at the beginning of the milling process, curves show a linear increase in the specific surface area over time [2, 4, 5]. This part of the curve is used in order to fit experimental data and determine an average grinding rate attributed to the agglomerate fragmentation. These grinding rates are used to interpret our experimental data regarding the results of the numerical model [1]. In our case, the scaling factor is not suitable to describe the fragmentation process in all conditions. Based on our data, we propose an adjustment of two coefficients of the general law studied (Figure 2).

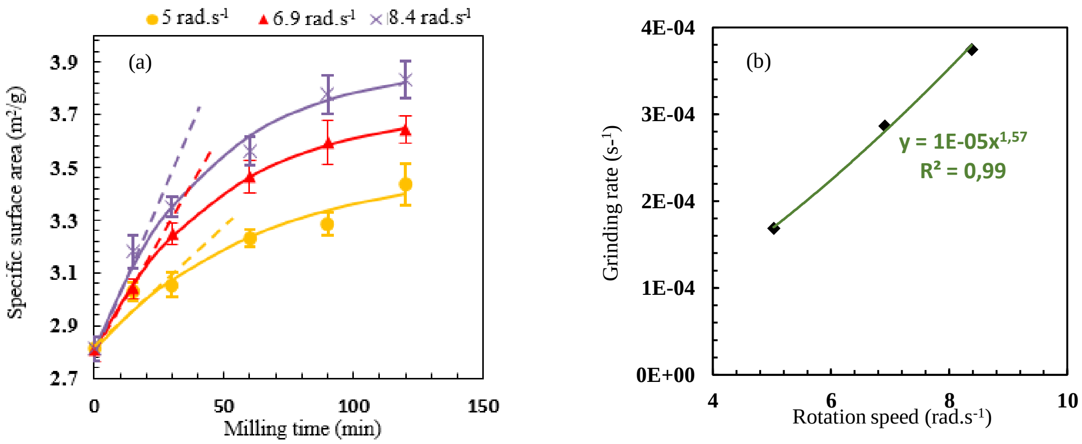

Figure 2. Specific surface area evolution (a) and grinding rate (b) for three experiments considering three different rotation speeds 5.0; 6.9 and 8.4 $rad.s^{-1}$ (constant drum diameter of 135 mm and same constant filling degree of 0.6).

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Quantifying Electrification Requirement of Mobile Crushing Units in Swedish SME Quarries

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Keywords: Electrification, Mobile Crushing, Power measurement,

The European aggregate industry faces the same challenges as other large industries in meeting the Agenda 2030 goals of net-zero emissions, as stated by the Sustainability Development Goal 13 [1]. In Sweden, the aim is to achieve this by 2045, where the intermediate goal is to have accomplished a 50 % reduction by 2030. As for most industries, the main focus is to investigate how to phase out fossil fuels and fossil-driven vehicles which can contribute with approximately 65 % of the greenhouse gas emissions for a medium size aggregates plant, for a smaller, off the grid, sites this will be higher. The other large contributors to greenhouse gas emission includes the explosives and electrical consumption of the equipment. How much the actual contribution from diesel to greenhouse gas emission depends on the selection and configuration of the heavy machinery, configuration of the process and the geological location of the site.

In quarries, the fossil-driven vehicles consist of heavy machinery such as load carriers, front-loader, drills, and mobile crushers. Mobile crushers are installed with an asynchronous motor and in most cases an auxiliary diesel generator to power the electrical motor. The diesel generator can consume up to 100 litres/hour during operation. Figure 1 illustrates the current required for the different types of mobile crusher types tested in this study. Total of 6 mobile crusher were tested to identify loading condition during start up, operation and during an emergency shutdown.

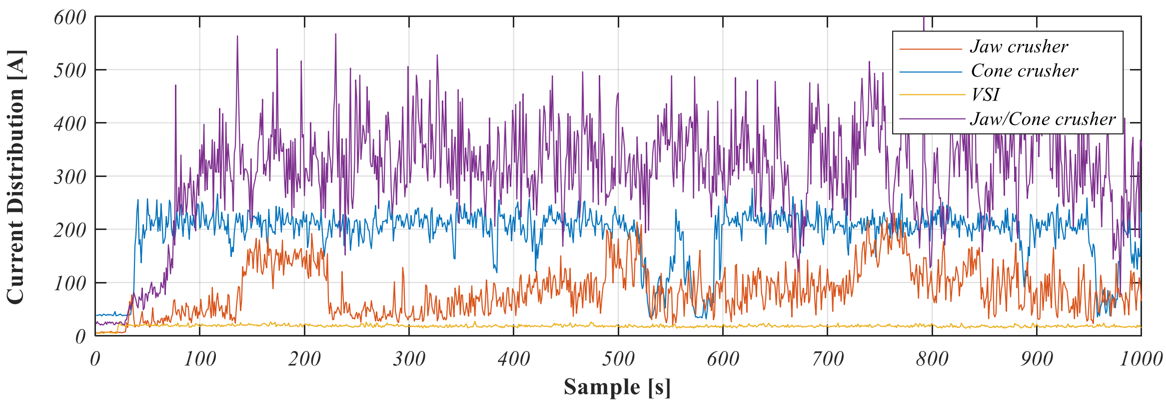

Figure 1. Current distribution for the 4 different crusher configurations

Most mobile crushers can be operated using either a diesel-powered generator or electricity directly from the grid or another power source. However, many Swedish SME quarries are not necessarily connected to the local electrical grid. In those cases, it has not been considered to be economically feasible due to large investment cost. In Sweden approximately 85 % of all quarries produce less than 50.000 tons a year, with a total production representing 27 % of the overall production [2]. Other electrification initiative includes hybrid wheel loaders, cable-connected excavator, autonomous and battery-powered load carrier.

Considerable fluctuation in the power draw was observed during each experimental run, the largest contributors to variation in current and power is misaligned feed, segregation and low filling level in the crushing chamber. With an unchoked crusher the material flow tends to segregate when it hits the top bearing housing and cause uneven filling in the chamber. Additional with an unchoked crusher there is less interparticle breakage occurs in the first crushing zones. This further excides the uneven force distribution that act on the mantle. Additionally, adding a Li-ion battery of only 2-5 kWh could e.g. reduce the peak current, where a battery up to 20 kWh could significantly reduce high load fluctuations on the power grid and hence reduce the power level. By reducing fluctuation and momentary peaks, the transformer could also be scaled down to better match the application, making it more economical to transition from fossil-driven mobile crushing to a crusher connected to the local power grid and in turn significant reducing the environmental impacts of the production to meet the Agenda 2030 goals.

This paper aims to describe the work done at Chalmers Rock Processing Systems and Stonepower AB within the first phase of Swedish Aggregates Producers Association project, Sustainable Smart Electrification of Small Quarries. The work aims to map out the crushers' electrical power requirements under different operating conditions in different quarries. This is to create a requirement specification for the development of a modular solution for more cost-effective battery-transformers package for a more economical feasible investment plan for smaller quarries.

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Artificial intelligence in comminution

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Keywords: Artificial Intelligence, Particle Technology, Genetic Programming, Simulations

Artificial intelligence (AI) has experienced a boom in recent years due to technical breakthroughs in software, e.g., deep neural networks, exponential gains in hardware performance, and data acquisition and generation. AI is capable to identify inherent patterns in data sets which would be too large or too complex for conventional analysis or manual investigation. Increasingly, it can be used for applications with moderate amounts of data. Furthermore, the need for required experimental and numerical investigations can be greatly reduced through the use of predictive models (generalized AI models trained on existing data that are quick to apply). Furthermore, methods exist to transform these black-box models into more transparent models, e.g. reverse engineering approaches, hybrid modeling or genetic programming. [1]

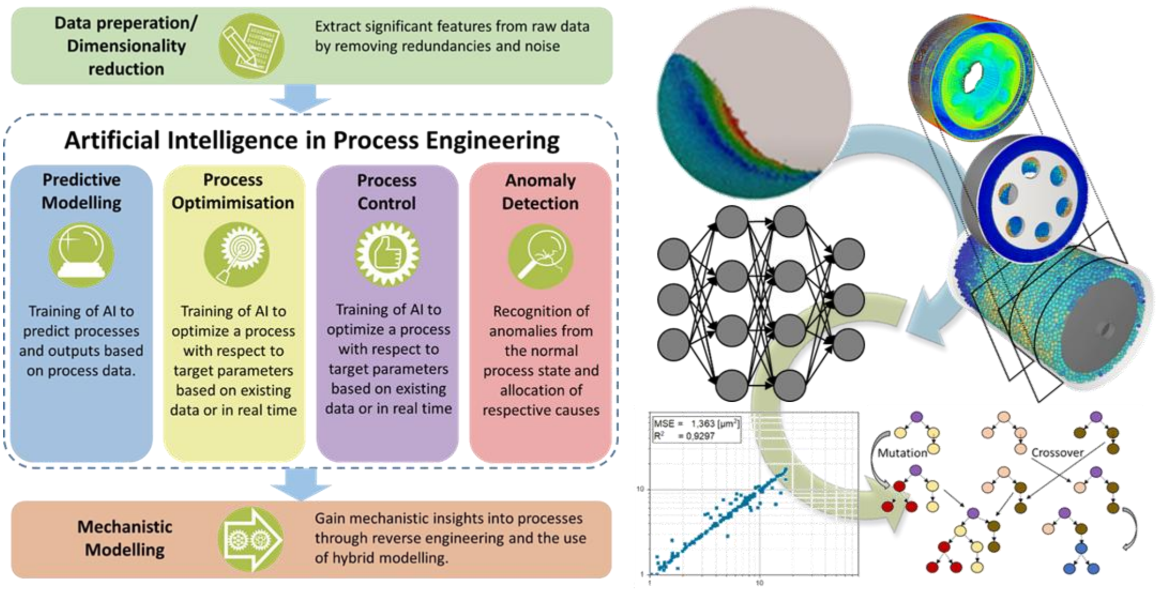

Figure 1. Application domains of AI in process engineering and depiction of an applied procedure in particle technology in comminution

The core fields are predictive modelling, in which a model is trained on the basis of existing process data in order to make predictions about future process developments. Derived from this are the optimization and control of processes as well as the detection of anomalies or errors in processes. The training of the AI models usually takes place on the basis of existing process data. Existing databases, specially conducted experiments or simulations are also used to generate data and evaluate the trained models. Before these core fields, process data is sometimes "pre-processed" using artificial intelligence methods prior to the actual training in order to facilitate the subsequent modelling effort by means of pattern extraction and compression resulting in a reduction of complexity. The training for the purpose of empirical process modelling can optionally be followed by mechanistic modelling, in which the non-transparent "black box" models of artificial intelligence are broken down into more transparent "white box" models using a wide variety of methods such as reverse engineering strategies or the derivation of mathematical formulas by means of "genetic programming". The use of hybrid models or "grey box" models, combining AI methods with known physical or mechanistic models, can also be used to simplify this process.

This contribution aims to illustrate the application of AI based on practical academic use cases such as CFD-DEM simulations of mills or experimental investigations in the field of comminution. The use of limited amounts of data can be considered as the current major challenge in the field of AI. By focusing on academic use cases of predictive modelling based on laboratory experiments with comminution devices and computer simulations in limited quantity, the practical use of AI even with small data sets as well as the transition of black-box models into more transparent models via genetic programming can be effectively demonstrated for comminution processes.

[1] Christoph Thon; Benedikt Finke; Arno Kwade; Carsten Schilde, Advanced Intelligent Systems 2021, 3(6)

Machine learning based impact crusher monitoring

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Keywords: impact crusher, machine learning, data processing, software based sensor

Following the idea of more efficient production in all industrial areas, we can state that efficiency means higher performance combined with lower costs [1]. Due to the high energy consumption, this topic is particularly relevant for the area of comminution in mineral processing. Automation systems play an important role in increasing the productivity of processing plants [2]. A particular challenge there pose the sometimes rapidly fluctuating characteristics of the natural feed material, requiring flexible systems which can deal with respective process dynamics.

Looking at the first crushing stages in a mineral processing plant, the main focus is usually to produce the highest amount of material within a target fraction, getting the best yields or obtaining a higher throughput combined with more energy efficient production [3]. Information on changing granular parameters of the crusher product is usually obtained only by weigh scales measuring the mass flow behind downstream classifying machines, such as screens. Deployment of smart automation systems is becoming of immense interest to detect, for instance, the result of a comminution process immediately behind the machine or even already in a crusher [4]. Control dynamics could be substantially increased. Benefits like lower product loss, higher process stability and longer maintenance cycles can be reaped [5].

While different crusher types can be found in the first crushing stages of mineral processing plants, horizontal shaft impact crusher (HSI) are seen as the ones offering particular potential for smart operation benefits [6], considering the particularly high stochastics of the crushing process in that crusher type.

A key technology for making crushers “smarter” is the application of software-based sensors. Those sensors generate information that are hard to measure directly. Based on a large database of the process, valuable information can be extracted. This extraction can be achieved by using machine learning methods to generate valuable information from data that a human isn’t necessarily able to detect.

In the pilot scale experiments, the application of a software-based sensor was tested on an industrial scale horizontal shaft impact crusher (HSI). Having equipped the machine with a multitude of acceleration sensors (as seen in Figure 1), an extensive trial plan was carried out. Based on its results, it was possible to obtain a well-founded dataset for this specific HSI. Following the common steps in machine learning, the dataset will be visualized and analyzed followed by the training and testing of two different machine learning models. All with the goal to achieve a functional software-based sensor for detecting the granulometric properties of the crusher product.

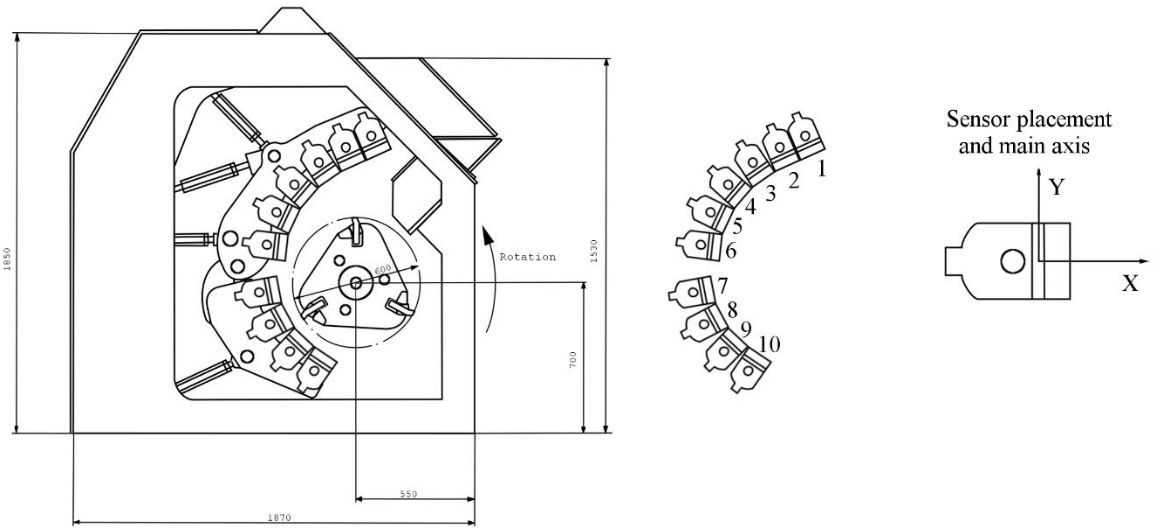

Figure 1. HSI with sensor placement and main axis.

As a result, it was possible to predict the granulometric properties of the HSI product instantaneously, i.e. before the product has physically left the crusher, with an error less than 1,5 %. In addition, the compared machine learning models will be ranked by there learning curves in regards to a practical deployment during assumed plant installation.

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Advancement in ore characterisation using Precision Rolls Crusher

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Keywords: Ore characterisation, minimum comminution energy, size specific energy, comminution circuit benchmarking

The application of Size Specific Energy (SSE) approach that refers to the amount of energy required to generate material below a specific marker size is an evolving topic to assess the efficiency of comminution circuits [2]. This topic is gaining significance due to its proven ability in benchmarking individual comminution equipment and circuits relative to the best-achieved site performance and against lab-scale equipment when operating on the same ore type [1, 3]. However, a suitable lab-scale characterisation device to measure the SSE of ore samples has been lacking. An ideal device should break particles singly to effectively measure practical minimum comminution energy across a wide particle size range. Practical minimum comminution energy relates to the energy required for the body fracture of individual particles. Single-particle breakage is generally regarded as the most efficient way of breakage as it does not include losses due to interparticle interactions such as friction and inherent equipment-related inefficiencies [4]. The widely used single particle breakage tests such as Drop Weight Test and Rotary Breakage Tester have shortcomings to determine practical minimum SSE as these methods are limited in a number of ways: inaccurate input and measure of low energy impact; insufficient generation of fines (sub 100 μm) for use in calculating the SSE, inability to apply appropriate energies to small particles (below 5 mm); and they cannot achieve the ideal of primary fracture only.

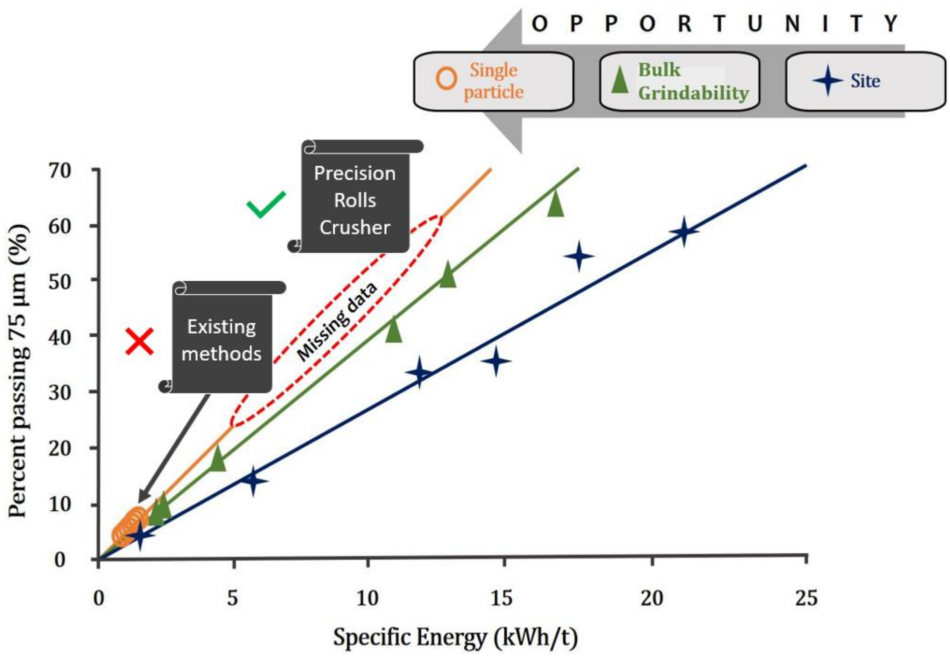

Figure 1. Hypothetical comparison of specific energy measured on-site versus specific energy measured using bulk grindability tests and single-particle breakage tests for the same generation of -75µm materi-al (modified from [5])

This paper explores development of a novel ore characterisation method for the precise quantification of practical minimum comminution energy. A monolayer of particles with narrow size distribution is broken using a precision rolls crusher (PRC), an ore characterisation device developed to overcome the shortcomings of conventional tests. For energy measurement, PRC uses two shaft-mounted rotary torque transducers. These transducers acquire precise torque and angular speed data in response to a breakage event, which is converted into energy. The test progressively crushes feed particles down to a final product size in many controlled breakage stages. The generation of fines in each breakage stage is then plotted as a function of input energy (Figure 1). The gradient of this plot provides the size specific energy (SSE) in kWh/t of material generated below a predefined marker size; -75µm in this case. PRC has the potential to evolve as a geometallurgical ore characterisation test – it is simple, fast, requires small sample mass (200-300 g), covers a wide particle size range (10 mm to ~100 µm), and provides ideal SSE measurements that can be used for the benchmarking on site production equipment (Figure 1). Additionally, the progeny size provides the primary breakage appearance function for direct implementation in mechanistic comminution models.

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SAG mills - changes in response between conventional ball load levels and high steel load operating regimes

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Keywords: SAG mills, Ball load, Grind curves, Size Specific Energy.

AG/SAG mills are commonly used in comminution circuits for plants around the world. AG/SAG mills are used either as single stage mills in closed circuit with classifiers [1] or as primary mills in circuits with two or more grinding stages [2]. AG mills are preferred for mines that have sufficient competent rocks in the feed or in situation were contamination with steel media must be avoided. SAG mills are applied where there is insufficient rock in the feed or supplementary media is required to operate the plant at higher throughputs [3]. The percentage of steel media for SAG mills is depended on the process requirements which include throughput and the product size. The ball load in conversion SAG mills range from 4 to 15% [4] by volume depending on the proportion of competent rocks in the mill and the operating setpoints and the maximum power the mill can draw. However, high steel load SAG mills or Run-of-Mine (RoM) ball mills are operated with ball loads ranging from 16 to 40%. The high steel SAG mills or RoM ball mills combine attributes concerning feed preparation from SAG mill and high steel media from ball mills. The lack of sufficient competent ore in the coarser fraction, suitable for use as grinding media provides a motivation to operate the mills with steel loads that are significantly higher than the typical SAG mill range of 4 – 15%, which is a range for high steel SAG mill or RoM ball mode. Where ore is friable, High steel SAG mills/ RoM ball mills are utilized instead of conventional ball mills to reduce the costs associated with pre-crushing in preparing the ore for grinding. Addition of steel balls also help in stabilising the load in the mill where friable ores are processed.

A study at pilot plant scale was carried out using the grind curve methodology was applied to assess the performance of the mill [5]. The grind curve methodology allows to study the influence of key variables over a wide range to obtain a comprehensive understanding of the mill response.

Figure 1 shows the relationship between power/throughput and charge volumetric filling. The change in the size specific energies (SSE) with different degrees of mill filling and ball filling is illustrated in Figure 2**Erreur ! Source du renvoi introuvable.Erreur ! Source du renvoi introuvable.**. The size specific energy is the amount of energy used to produce a material finer than the reference size. The trends in Figure 2**Erreur ! Source du renvoi introuvable.** show the relationship between size specific energy (SSE) determined for the new -75µm material produced and volumetric fraction of steel balls in the total charge. A consistent trend is observed whereby the SSE increases as the fraction of steel balls in the charge increases for each of the four ball filling levels (15% – 26%). The increase in SSE occurs at between 0.5 and 0.6 volumetric fraction of balls in the charge, the turning point increasing as the total ball charge increases. This indicates that for the range of ball fillings and total volumetric filling tested it is advantageous to limit the fraction of balls in the charge to below 0.5 to 0.6 to lower the specific energy performance of the RoM ball mill. Further insights on the how steel balls ranges in various regimes influence mill performance are discussed.

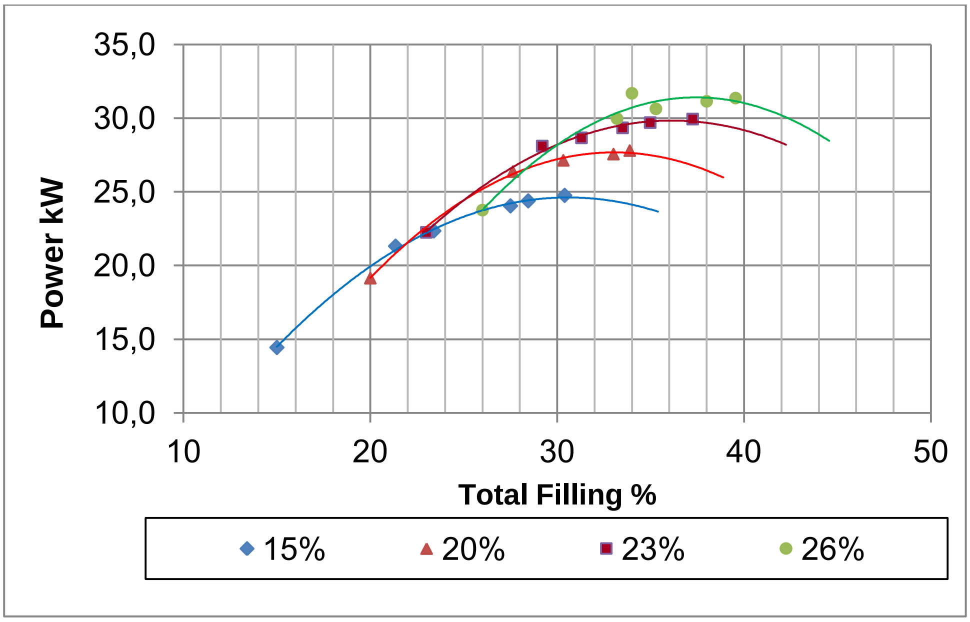

Figure 1. Mill power curves at different ball filling levels.

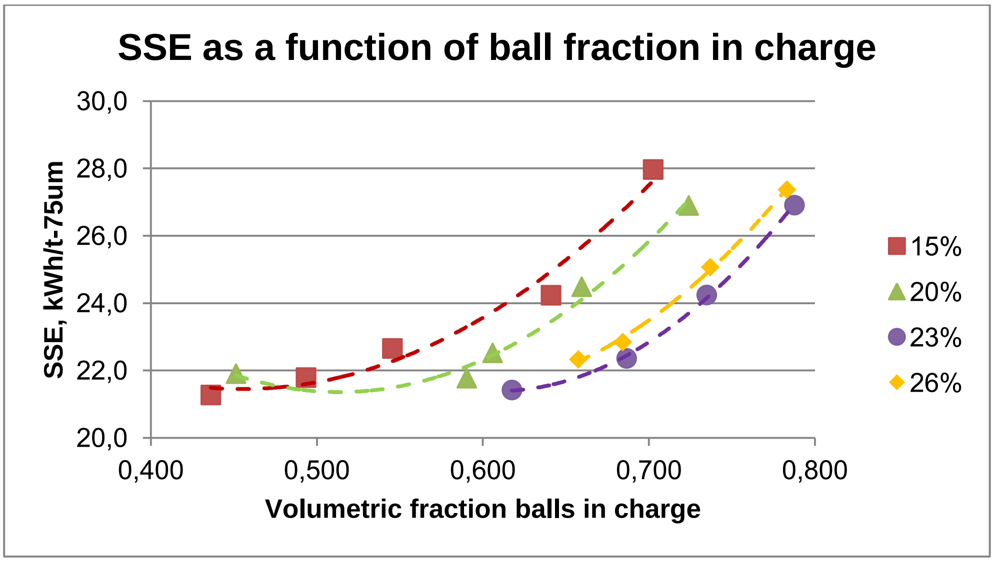

Figure 2. Size specific energy as a fraction of ball fraction in charge

References in the text should be given by numbers in square brackets [1]. The list of references (11 pt) follows the last line of the text with one extra line inserted.

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Interaction between crushing and milling in an SAB circuit

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Keywords: Downscaling, Single-Stage Ball Mill, SAB

The performance of milling circuits is generally influenced by circuit configuration, ore feed characteristics and throughput. Operations are sometimes required to downscale capacity due to changes in demand that occur during the life of mine. This normally poses a challenge because most circuits are designed based on work plans that consider life of mine to define nominal throughputs. Comminution circuits comprising semi-autogenous (SAG) mills in primary duty and ball mills in secondary role often present design challenges in determining profitable operational throughputs. A SAG mill and ball mill (SAB) circuit is designed to operate over a wide range of conditions as an integrated circuit. Operating at a reduced throughput requires careful consideration to maintain operation in the economical region. As a result, there is need to optimise the original circuit design and operation in order to maintain profitable operation. The changes required range from non-invasive adjustments to the original circuit such as ball load changes, to major procedures such as mothballing major inline equipment. This paper discusses a methodology to investigate options for downscaling an operational SAB circuit due to sustained low demand. The case study highlights the merits of either making relatively minor design and operational adjustments to the existing SAB configuration to achieve favourable cost per ton; or converting the existing circuit to a single-stage ball mill configuration that operates more profitably at the reduced throughput rates. The first alternative presents a relatively attractive option that involves operating the original SAG mill in AG mode at the reduced throughput. The single-stage ball mill proposition involved mothballing the SAG mill and crushing the run of mine feed to a finer size in order to produce suitable feed for the single-stage ball mill. Concomitant simulations were performed on the crushing circuit to investigate changes required to deliver a series of finer feeds of different size distributions to the milling section. A series of simulations are discussed where simulated crushed feed size distributions are used to characterise the interaction between crushing and the alternative configurations of the original milling circuit considered. The simulations were designed to investigate whether the reconfigured SAB circuit in either AG mode or single-stage ball mill mode could be operated profitably at reduced tonnage while delivering the desired product quality. Typical key product quality parameters monitored during simulation were the P80 (mm) and the portion of material below 106µm (%) in the circuit product. Key indicators of operational viability monitored were the circulating load and scats production.

The breakage characteristics of different liberation classes during primary breakage

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Keywords: primary breakage, liberation, mineral deportment, coarse waste rejection.

Coarse waste rejection is a processing strategy that saves grinding energy and increases circuit capacity by separating and removing gangue particles as early as possible in the flowsheet. This approach avoids grinding the entire ore stream to the final required grind size. One potential method of achieving coarse waste rejection would be via coarse particle flotation. The development of flotation technologies for processing coarse particles has been highlighted as one of the leading areas where significant improvements in mine operational efficiency are possible. Teetered bed flotation devices have been demonstrated to effectively recover coarser partly-liberated particles and require a lower energy input than conventional flotation cells [1,2,3,4]. Studies with the HydroFloat® (one of the industry-leading teetered bed flotation devices) showed its ability to recover coarse sulphide mineral particles (850 x 500 µm) containing as little as 1% of the exposed grain surface [5].

Although coarse gangue rejection is attractive for mining operations, integrating this technology into comminution circuits poses technical questions that require attention. Questions such as optimal circuit configuration, impact on upstream and downstream processes, the requirement of a coarse, narrowly sized feed distribution, and potential impacts on capital and operational costs need to be addressed. Besides that, one of the most critical aspects when using coarse particle flotation technologies is understanding the effects of breakage on mineral liberation [6]. The coarse particle flotation performance is likely governed by the degree of liberation of the feed to the flotation device. A difficulty with respect to characterising multi-component particle breakage is the presence of residual energy which causes secondary fragmentation of the products after the initial or primary fracture event. To optimise the effectiveness of coarse gangue rejection using teeter bed flotation, there is a need to understand better how mineral deportment and liberation are affected by comminution and classification processes. To be able to decouple the effects of breakage and classification within a breakage device, there is a need to measure how different liberation classes respond to primary breakage events where the secondary breakage is minimised.

This work will introduce a methodology developed to understand the relationship between the primary breakage mechanism and its effects on mineral deportment and liberation and how this methodology can be used to test an ore's amenability to coarse waste rejection.

The ore selected for the test work is a coarse-grained copper ore from an Australian mine composed mainly of chalcopyrite, pyrite, silicates, and calcium carbonates. The material was classified into discrete size fractions and was subject to a density classification to create distinct liberation classes. The density is used as a proxy for selecting liberation classes based on the assumption that different liberation classes will have a distinct density. The difference in density is because of differences in their composition – a low-density class would be composed mainly of gangue minerals, whilst a high-density class would be composed mainly of valuable minerals.

The initial result shows that the methodology used was able to create three distinct density groups: a low-density group with specific gravities ranging from 2.56 to 2.88, a high-density group with specific gravities ranging from 3.71 to 4.83, and a medium-density group with specific gravities ranging from 3.06 to 3.13. Element to mineral conversion from semi-quantitative X-ray fluorescence (XRF) results showed that the high-density group is predominantly composed of chalcopyrite and iron sulphides, and the low-density group is predominantly composed of calcium carbonates.

The hypothesis that there are intrinsic differences in the breakage response for the different components of a mineral was tested. This work is conducted using the Short Impact Load Cell (SILC) device located at the Julius Kruttschnitt Mineral Research Centre (JKMRC) to measure the minimum-fracture energy that a single particle suffered during a primary fracture event. The progeny distribution from the breakage events was analysed using X-ray microtomography and MLA to determine the breakage functions and quantify the differences in mineral liberation and deportment achieved for the different density groups after a single breakage event.

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Influence of an internal deflector wheel on the transport behavior in stirred media mills

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Keywords: Dyssol, population balance, stirred media mill, internal deflector wheel, dynamic process simulation, residence time distribution.

Dynamic process simulation gains importance in control and regulation of mechanical processes [1] such as grinding in stirred media mills [2]. Furthermore, for stirred media mills the combination of complex models, necessary to simulate the device, increases the understanding of the process itself and especially of its dynamics [3-9]. For instance, changes in particle size distribution influence the grinding efficiency, particle transport and grinding media movement [8, 9]. One option to simulate these dynamics is applying population balances which are based on breakage function, breakage rate and particle transport of the stirred media mill [10]. This allows real-time simulations of the processes.

More and more advanced products need precise process control to achieve desired product requirements. Therefore, the process itself and its modelling have been improved in the past decade. One example of such improvements, often applied in industrial operation, is an installed internal deflector wheel, which influences the grinding media and particle transport within the mill [8, 9]. Originally, it was installed to avoid grinding media compression at the outlet. However, some particles above the cut size are held back in the mill, changing the holdup mass. Due to the forces acting on the particles above the cut size of the internal deflector, the particles are not transported directly by the fluid flow. Therefore, current methods based on the residence time distribution of the fluid, most often measured by conductivity [11], are not valid. A new method has been established which is based on step function of two materials with similar material characteristics, such as density and size. Based on experimental investigation of the residence time distribution, the particle transport has been back-calculated. It was found that the cut size varies by the rotational speed and the number of sticks attached to the internal deflector wheel. Depending on the cut size, the holdup in the mill changes, influencing the viscosity, stress-energy distribution of the grinding media, the contact probability and breakage function.

In addition, the deflector wheel influences the grinding media transport [8], leading to different local filling degrees. A global filling degree of 70 % leads to local filling degrees from 30 % up to 95 %. The compromise of grinding bead velocity and contact probability defines the breakage rate according to Fragnière et al. [3]. The presented model for the breakage rate is based on grinding media distributions measured with radiometric density [8].

The effect of process and material parameters on the resulting grinding media distribution, viscosity, breakage rate and particle size distribution were simulated for a stirred media mill with an internal deflector wheel via flowsheet simulation. In Fig. 2 (a), the local mass concentration is presented for each cell. In case of the suspension holdup, the mill is only divided into 6 cells, since the mass concentration can only be measured between the discs. Then it is obvious that the internal deflector wheel has a larger impact on coarser particles. After 120 s, the holdup distribution is still changing due to the forces acting on the particles. After 700 s, the mass concentration stays constant, if the mill is operated in continuous mode, since the particle size runs into a steady state. In Fig. 2 (b), the local filling degree of the grinding media is presented for each cell. In this case, the mill is divided into 12 cells, since the grinding media filling degree varies before and behind a disc. Coarser grinding media have a higher drag and are not transported as far to the mill inlet as smaller grinding media. The interplay of the particle dynamics and the installed deflector wheel play an important role in the resulting particle size distribution. Furthermore, there is an equilibrium of the drag forces acting against the flow and the forces acting from the internal deflector wheel on the particles, depending on the cut-size of the deflector wheel. On the one hand, the simulations confirm that the internal deflector wheel influences the fines positively. At around 90 µm as feed particle size, the product particle size stagnates at 50 µm. On the other hand, the internal deflector wheel increases the mill holdup, changing the mass concentration at the mill outlet and therefore, as well the product quality. Next to a dynamic holdup depending on the particle size, the viscosity effects the grinding media distribution and velocity, effecting the particle size itself.

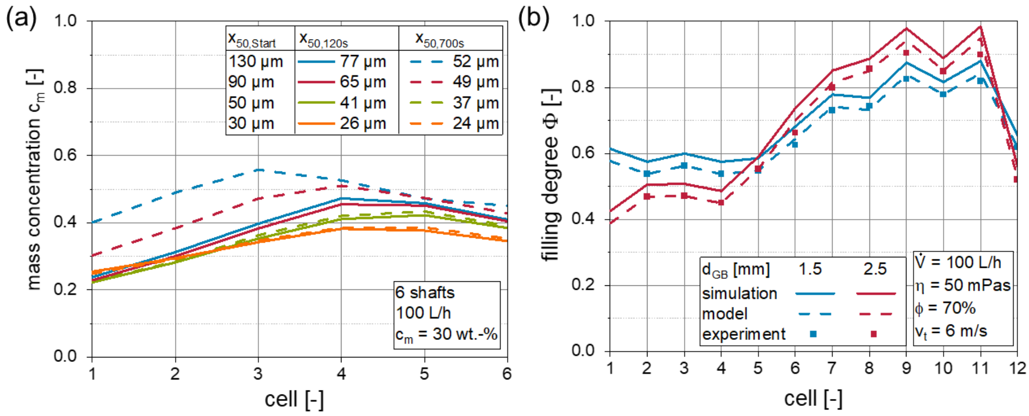

Figure 2. Simulation of the influence of an internal deflector wheel on the (a) local mass concentration and (b) local filling degree

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Nanoparticle Fractionation: UV/vis Soft Sensor Development for Real-Time Process Monitoring

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Keywords: solid–liquid separation, multidimensional particle features, tubular centrifuge, process monitoring, soft sensor, UV/vis spectroscopy, chemometrics

The precise adjustment of product-relevant properties by efficient separation processes is an important aspect in solids process engineering. Consequently, process development requires advanced characterization with respect to multiple separation criteria such as material (solid density, particle surface functionalization, ...) and morphology factors (particle size, elongation, flatness, ...). In other words, beneficial product properties are defined by precise specifications e.g. regarding particle size and material composition, which requires the innovative and efficient processing of well-characterized particle suspensions. High centrifugal accelerations and throughput rates of tubular centrifuges enable such solid-liquid size separation and fractionation of nanoparticles on a bench scale. With this type of centrifuge working in a semi-continuous mode, however, an online observation of the separation outcome is needed for optimization purposes. With the aim of a precise real-time composition analysis in the centrifuge downstream, a UV/vis soft sensor is developed and presented in this work. The monitoring principle for sorting a feed suspension of polymer and metal oxide nanoparticles according to their size and density is shown in Fig. 1. Using multi-component spectroscopic analysis, an observed UV/vis signal is used for a model-based prediction of the material specific solids volume fraction in the centrifuge overflow. A mandatory calibration routine and high signal stability enable the soft sensor setup to accurately predict product composition under variable operating conditions.

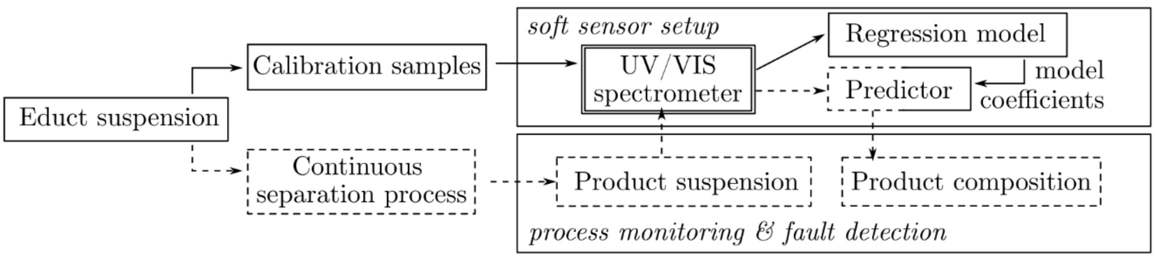

Figure 1. Soft sensor principle for real-time process monitoring and fault detection of a continuous separation process.

The presented work introduces a software-based UV/vis sensor and its effective utilization regarding real-time separation process analytics of multi-component suspensions. Furthermore, the setup provides insight into the underlying process dynamics and helps to optimize the outcome of separation tasks in tubular centrifuges on the submicron- and nanoscale.

Energy effectiveness in microwave-assisted size reduction methods

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Keywords: Microwave Fragmentation, Mine to Mill, Energy efficiency, Weakening over Microwave Energy

Mining companies have been searching for innovative solutions to improve the energy efficiency of the comminution and processing operations. To this end, experts have regarded microwave-assisted size reduction methods as a promising leading-edge technology towards a more sustainable mine-to-mill process. The majority of existing literature on microwave pre-treatment of rocks has focused on utilizing the technology based on mineralogical aspects of rocks. Koleini and Barani [1] reviewed the existing literature on microwave heating applications in mineral processing. The published literature highlighted that microwave treatment could have grinding, drying, heating, roasting, or smelting applications in mineral treatment and metal recovery processes. Furthermore, the potential of implementing the technology in the carbothermic reduction of oxide minerals, pre-treatment of ores and concentrates, carbon regeneration, and waste management have been emphasized. Although the existing literature has studied and shown the potential implementation of microwave treatment for comminution and processing of ores, the energy-based considerations remained un-tackled. However, in a comprehensive study, Hassani et al. [2] and Shadi et al. [3] offered an energy-based analysis method to be used in fragmentation. They investigated the effects of integrated energy and rocks/minerals strength parameters on the effectiveness of microwave-assisted fragmentation. It was found that microwave-induced degradation was directly proportional to the energy efficiency of microwave heating. This novel energy-based analysis was employed in the current study to evaluate the feasibility of microwave-assisted size reduction through a series of experimental investigations. Multiple strength degradation tests, together with calorimetric measurements, were performed on treated and untreated rock samples.

The results show that it is possible to improve the failures nucleate and its progress in rocks in an effective manner. The primary factor in approaching the feasible zone of microwave-induced degradation and size reduction is maximizing heat over microwave energy (HOME). The different maximum HOME percentages for basalt and kimberlite (92% and 80%, respectively) in various distances from the waveguide corroborates the vital impacts of a good cavity design, e.g., optimum placement distance from the waveguide (Figure 1), on microwave heat absorption. High power, low energy microwave exposure alongside a good cavity design will result in maximizing HOME and weakening over microwave energy (WOME) simultaneously. Taking advantage of better sample positioning, it was shown that WOME values could be improved up to 6%. However, further improvements can be made in cavity design and sample placement to raise WOME values to 20%, which is the practically feasible range favoured by industry.

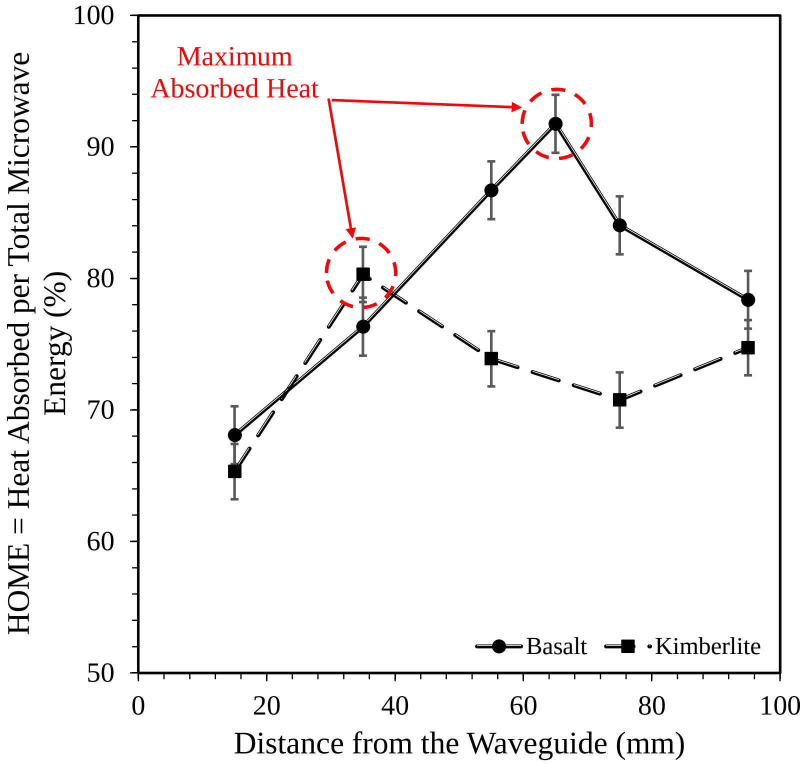

Figure 1 HOME variation in various placements from the waveguide for kimberlite and basalt slabs

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POSTERS

Mechanical grafting of fluorescein onto lignocellulosic biomasses in a ball mill to design biobased sensor

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Keywords: Mechanochemistry, ball mill, grafting, fluorophore, lignocellulosic biomasses

Mechanochemistry is a promising green process in which chemical reactions outcome by the use of mechanical constraints. The starting materials are generally under the solid state, and the energy necessary to the creation of the chemical bond is provided by an intense milling in a twin screw extruder or in a ball mill [1]. These mechanochemical reactions have the advantage of being solvent-free and easily implementable in comparison to the traditional wet chemical reactions. In the past 20 years, mechanochemistry received an increasing interest for applications in various fields including inorganic materials synthesis, organic compounds synthesis, metals recycling, crystal engineering, supramolecular aspects, etc [2]. Recently, mechanochemical processes have been explored as an efficient pretreatment prior to lignocellulosic biomass conversion [3], by disrupting the crystalline structure of cellulose and enhancing catalytic accessibility of cellulose and hemicellulose during hydrolysis [4]. Indeed, intense comminution of plant biomass in a ball mill not only significantly affects the crystalinity but also leads to the breakage of covalent bonds linking constitutive macromolecules, generating free radicals that can recombine with other molecules present within the grinder thus modifying the chemical composition of the starting materials [5]. In presence of a reactive, these free radicals could be exploited to graft some specific molecules and functionalize the lignocellulosic biomass for various applications.
In previous work we demonstrated that flax fibers can be chemically grafted with a pH-sensitive fluorophore. The fibers, mixed with a polymetric matrix, brought functional properties to the material allowing the design of specific sensor by 3D printing that can react to pH variation [6]. As a continuation of this work, we explore in this study, the possibility to exploit the free radicals formation during ultrafine milling to mechanically graft fluorescein on hemp core.

Fine powder of hemp core (d_{50}= 160.6 μm) from premilling steps with a kniffe mill and an impact mill were milled in a vibratory ball mill (MM400, Retsch®) in presence of fluorescein, in a ratio 1:500, corresponding to the optimal ratio for chemical grafting in 100 mM carbonate buffer at pH 10.0.
Powders (M=2g) were milled in a bowl of 50 mL with a single ball of 25 mm (mass m_b) at a frequency f=20 Hz during 60 min. The same fine powder of hemp core was also milled in the same milling conditions but without fluorescein as a control.
In the milling configuration used, the specific energy provided by the ball to the material at each impact can be assimilated to its kinematics energy and determinated according to the following equation [7].

with A is the stroke amplitude of the jar (A=7.2 mm), f the frequency, m_b the of the ball and M the mass of the powder.

After the milling the powders (control and grafted powder) were washed several times with carbonate buffer at pH 10.0 (2x), distillated water (3x) and ethanol (3x). Absence of residual fluorescein in the last washing solution was confirmed by spectral analysis (absorption at 490nm). The fluorescence of the washed milled powders were evaluated with a fluorescence microscope by looking at the emitted light intensity (λ_{em}>515nm) recovered under blue light excitation (λ_{ex}=450-490 nm) with an acquisition time of 10 ms. Results, presented in figure 1, cleary evidenced the grafting of the fluorescein onto the hemp core milled in the presence of the fluorescein. The main particle descriptors, d10, d50 and d90 of the mechanically grafted hemp were equal to 4.7µm, 37.4 µm, and 142.5 µm respectively, making them suitable as a functional charge in a biobased composite materials [8], and paving the way to design in a simple and effcicient way biobased sensor by 4D printing.

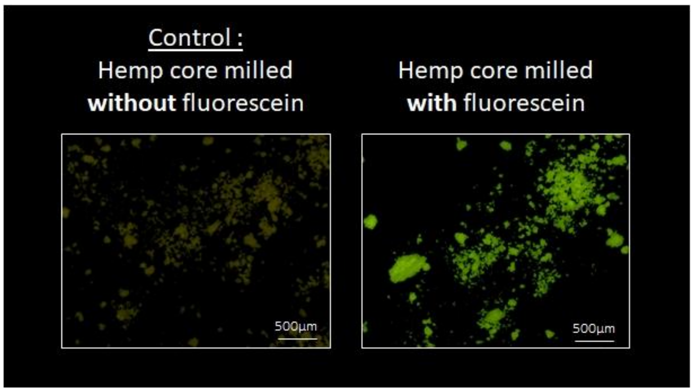

Figure 1. Hemp core powder milled without fluorescein (control) and mechanically grafted hemp core powder observed with a fluorescent microscope (λ_{ex}=450-490 nm , λ_{em}>520nm, the contrast in both RGB images was similarly adjusted with values enabling the visualization of the non-fluorescent particles in the control).

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Production of high value-added molecules from red pitaya using stirred media milling

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Keywords: wet media milling, fine comminution, red pitaya peel, betacyanin

The increasing production of food and agricultural waste worldwide imposes several burdens on the environment, depletion of finite landfill space and irreversible environmental problem like global warming. In order to lessen this on-going threat, recent studies focus on the valorization of various agricultural waste products and value-added trends. Bioactive compounds mining is one of the applications being focused, thus extraction of these high value molecules requires the design of novel green processes which could produce a high yield of target compounds with less harmful and toxic solvent waste generated.

One of the objectives of this work is focus on physical process of cellular destruction in order to valorize the residues of a biomass used in the agri-food sector. The biomass of interest is the peel of exotic fruit pitaya which occupies a growing niche in Europe's exotic fruit market at present [1]. It is considered to be a new, promising fruit species and being cultivated in tropical and subtropical countries, including Reunion Island where all the ecological requirements are met, [2] due to its high commercial value as functional fruit. It has antimicrobial and prebiotic potential [3] and high amount of flavonoids and phenolic compounds [4], most especially the natural food pigment betacyanin [5] rarely found in any edible products. These compounds have antioxidant and anti-inflammatory properties, which may be helpful in preventing some oxidative stress-related disorders [6].

The significant part of the work focuses on the optimization of stirred-bead mill (SBM) parameters complemented by modelling of cell lysis and extraction kinetics in order to achieve a high yield of target molecules. The stirred media mill LabStar, NETZSCH uses wet media milling technique commonly used in pharmaceutical industry [7] and biorefinery of microalgae [8]. The work explores the efficiency of SBM in deconstruction of various lignocellulosic biomass with the combination of attrition and impact stresses [9] in releasing betacyanin and other bioactive compounds of interest. Operating parameters such as grinding time, bead filling ratio, size, and properties as well as grinding operation mode and suspension formulation are the working conditions optimized.

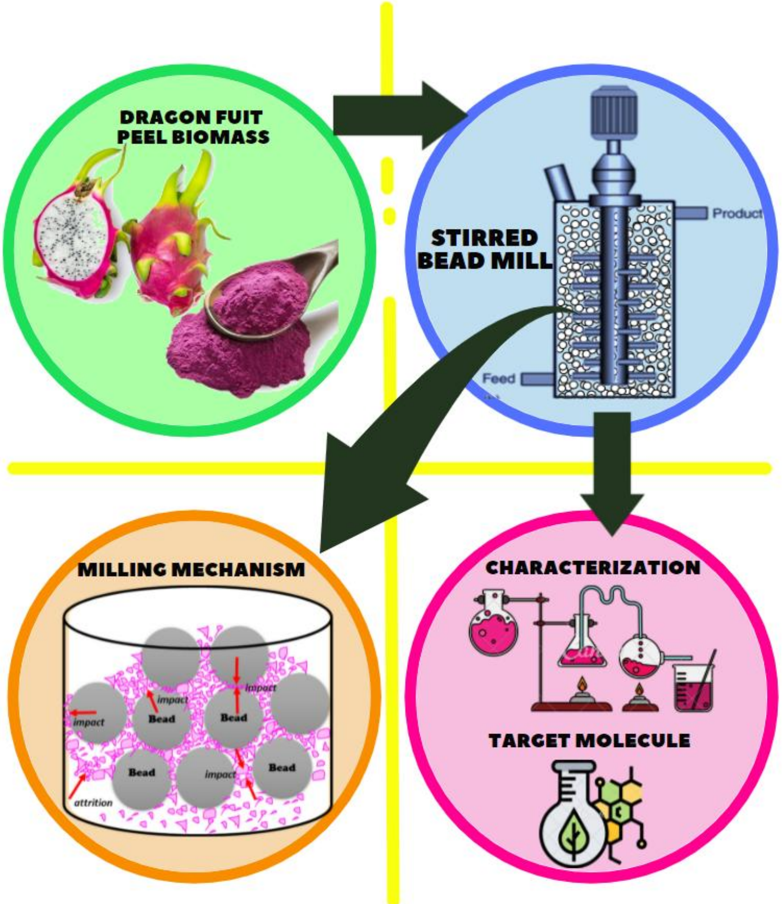

Figure 1. Extraction of the natural dye betacyanin and functional compounds from the peel of pitaya (*Hylocereus undatus*) using physical process of wet media milling

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Production of glass flake reinforced PBT-PC composites via co-comminution in a stirred media mill

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Keywords: composites, polymer, glass flakes, co-comminution

Specialty polymers have become indispensable materials in our today's world and are being used in an increasing area of specialty applications such as aviation and spacecraft. Often, multiple polymers are combined to form a blend to enhance desired properties and create a purpose-specific material. For example, PBT-PC blends are well known and reported as they combine the stiffness of PBT and pair it with the dynamic strength of PC [1, 2]. Often, additional filler materials are incorporated into these polymer blends at the macroscopic level, creating complex structural materials. Fillers are often used as cost-efficient additives to further elevate desired properties such as strength, stiffness, corrosion resistance or even the crystallization behavior. Common fillers can be distinguished not only by their chemical composition, but also by their shape. Most used additives are spherical particulate fillers or fibers [3, 4]. Recently, flake-like fillers have gained some interest as filling materials in composites due to their improvements in terms of out-of-plane flex moduli and improved strength properties at the end of the production chain. In this work, polybutylene terephthalate (PBT) and polycarbonate (PC) are comminuted to obtain a homogeneous polymer blend that is subsequently co-comminuted in a stirred media mill with different glasses to obtain a glass-reinforced polymer blend composite. Typically, different forms of silica, such as glass fibers or fumed silica beads, are used to reinforce polymers [3, 4]. To the best of our knowledge, no group has considered using SiO_2-GeO_2 or GeO_2 glass flakes as a filler in PBT-PC blends even though GeO_2 glass is widely considered to be a classic network glass composed of tetrahedral building blocks similar to SiO_2 glass [5]. Therefore, this work is focused on the influence these glasses have on the thermal properties of the resulting composites. For this purpose, SiO_2-, SiO_2-GeO_2- and GeO_2 glass are prepared via melt quenching technique and further processed in a stirred media mill to yield flake-like particles with high aspect ratios that are added to PBT-PC blends in different mass fractions. Crystallinity and thermal properties of the composites are investigated by means of x-ray diffraction and differential scanning calorimetry, respectively. Glass-specific differences on the resulting thermal properties and crystallinity are highlighted and showcased.

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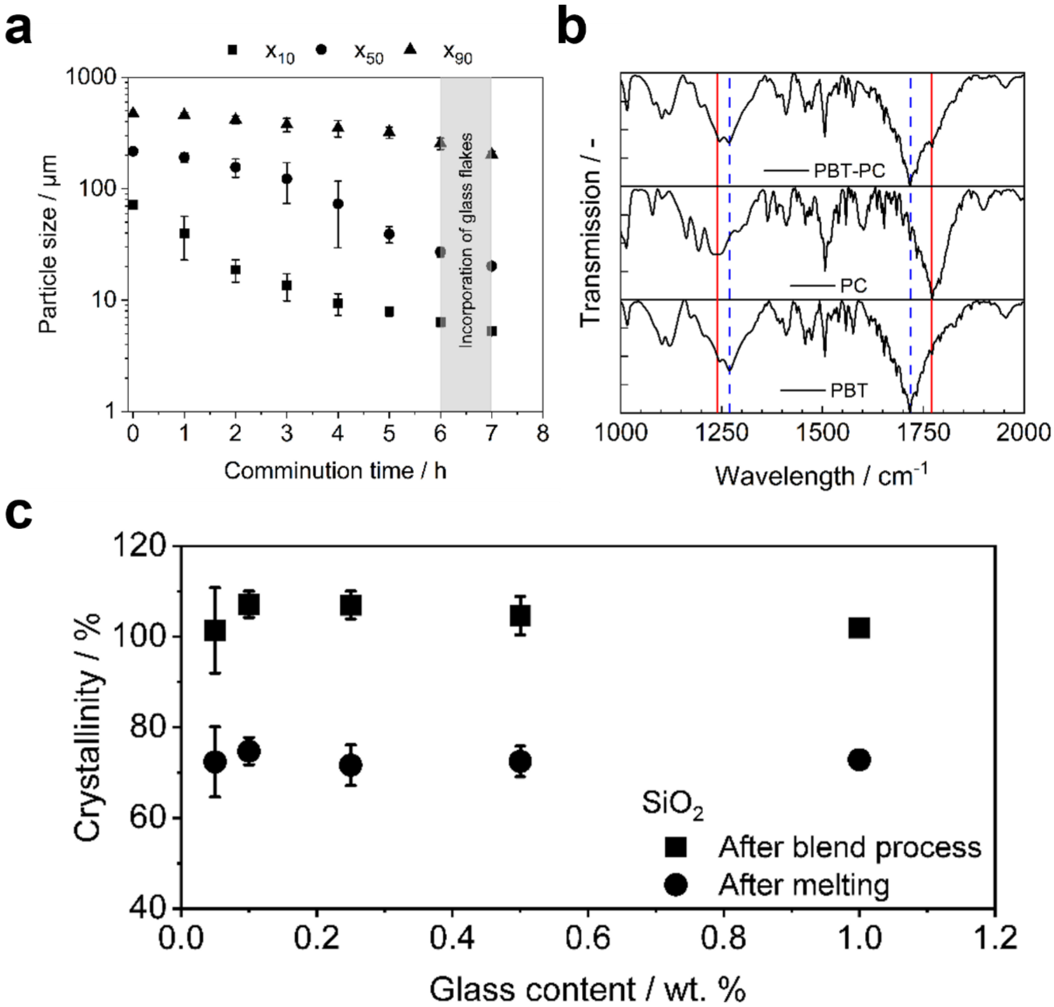

Figure 1. Comminution kinetics of the PBT-PC composite and incorporation of glass flakes (a); FTIR spectra of the PBT-PC composite to verify the existence of both polymers on particle level (b) and influence of SiO_2 glass flakes on the crystallinity of the PBT-PC composite (c).

Comminution and CFD-DEM simulations of peas in a hammer mill

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Keywords: Hammer mill, CFD-DEM Simulation, comminution of peas, simulation of breakage behavior.

In order to make important nutrients accessible in animal husbandry [1,2], the comminution of a wide variety of fodder is an important process step that also has a significant influence on the health of the animals [3,4]. Consequently, there is a target particle size in animal husbandry that can be well digested by the animals and prevents the formation of stomach ulcers [5]. Hammer mills are often used for this process step because of the most healthy structures of the products [1] and their simple design to allow their easy adaptability for different products in the feed industry. The parameterization of hammer mills is most often based on experience and empirical models [6]. In order to understand the effect of the process parameters in hammer mills better and to develop a new model based on mechanistic events, grinding of peas is compared to CFD-DEM simulations carried out in Fluent coupled to Rocky ESSS. The aim is to identify the influence of different process parameters on the velocity distribution, stress conditions and particle size distribution in the hammer mill.

In literature, there are several parameters stated which influence the stress conditions inside the hammer mill [7,8]. These investigations are adapted to the geometry in Figure 1 and expanded by CFD-DEM simulations to investigate the stress conditions. The geometry consists of three important parts. The mill chamber, the rotor, and the sieve geometry. The airflow enters the mill with 15 m/s due to air suction at the outlet. To analyze the effect of the airflow, CFD-DEM simulations have been carried out with not only 15 m /s, but also 0.1 m/s. The material is fed at the top of the mill and is comminuted by the contact of the material with the hammers and the impact plates. Ideally, the materials leaves the mill after two effective grinding events through the sieve geometry.

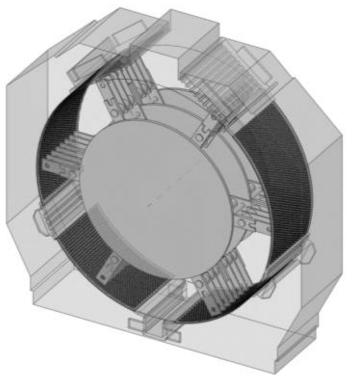

Figure 1. Design of the simulated hammer mill; (light grey) grinding chamber, (medium grey) rotor, (dark grey) sieve geometry

In Figure 2, the influence of the sieve geometry and inlet velocity on the residence time distribution as well as the impact energy are displayed for the simulation of the hammer mill. The fluid velocity of 15 m/s reduces the mean residence time of the particles inside the mill, because of the enhanced particle transport through the mill (Figure 2 (a)). The sieve geometry shows two contradicting effects. If a higher fluid velocity is applied, the particles are not only stressed a second time on the impact plate, but also on the sieve itself. This increases the number of contacts and breakage events and therefore, the particles can leave the mill more easily than the particles unaffected by the sieve geometry. This statement is underlined by the impact energies in Figure 2 (b) where 15 m/s fluid velocity combined with the sieve geometry shows the highest number of large impact energies. Without the sieve geometry lots of high energy impacts are lost and the residence time of the particles increases. At low fluid velocities the same effect can be observed for the influence of the sieve geometry.

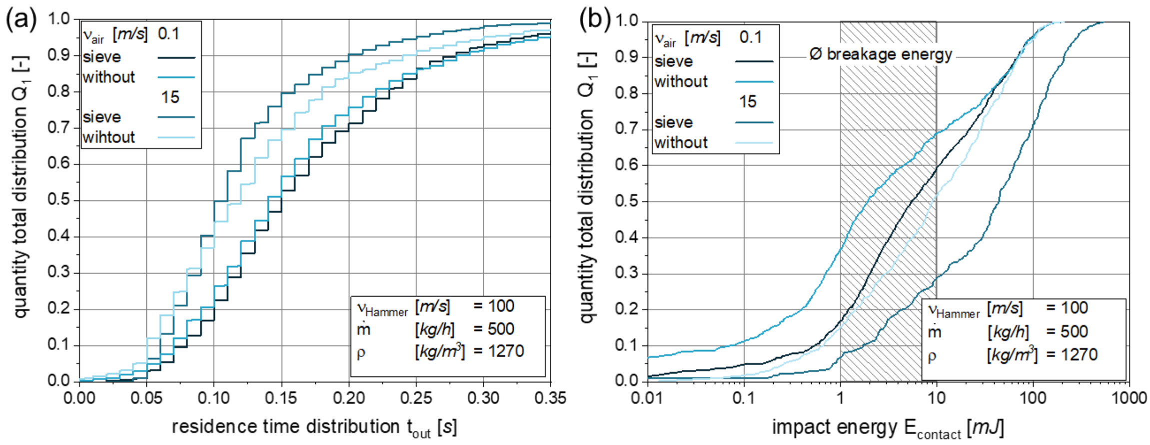

Figure 2. Influence of the sieve geometry on (a) the residence time distribution and (b) the impact energy.

The particle size distribution of the product is mainly influenced by the two observed effects, impact energy and residence time distribution. To display the particle size distribution in the hammer mill simulation, experiments of peas are carried out on an impact tester. Combining the information of impact energy and particle size distribution. This way the breakage function and breakage point are determined which is included in the CFD-DEM simulation and the particle size distribution is predicted. The predicted particle size is compared to the experiments carried out in a pilot scale hammer mill.

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Large Grinding Media makes the Difference

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Keywords: Wet grinding, Pregrinding, High speed mixer, Agitator bead mill

In the production of printing inks, the demands for ever lower prices combined with higher quality characteristics, such as color strength, brilliance, gloss and transparency, are increasingly contradictory. Added to this is the desire to be able to produce these inks with a higher degree of automation and maximum operational reliability in an absolutely reproducible manner.

Around 15 years ago, experienced operators were still of the opinion that large grinding media had to be used for grinding printing inks in order to meet the broad range of requirements and, at the same time, avoid production losses, e.g. due to screen blockages. However, due to the increased quality requirements, the use of grinding media with diameters <1.0 mm has almost become a standard today. In order to be able to ensure operationally reliable processes regardless of the quality of the raw materials, very good predispersion of the pigments and well-matched formulations are essential.

An essential aspect is that pre-grinding with large grinding media can guarantee the operational reliability of the process, despite fluctuation in the quality of the pigments. The desire for the simplest possible process design with as little equipment as possible is understandable from the point of view of the investment. However, this contrasts with the desire for ever higher degrees of automation, reproducibility and the potential for enormous energy savings and capacity increases.

On the basis of test results, the article shows that an automatic, emission-free and reproducible pigment task is possible by using an inline disperser. In combination with a pregrinding unit, the quality of the product in the pre-dispersion can be adjusted in such a way that very small grinding media can be used always in the fine grinding stage, regardless of the pigment quality. This enables energy and time savings of more than 100%.

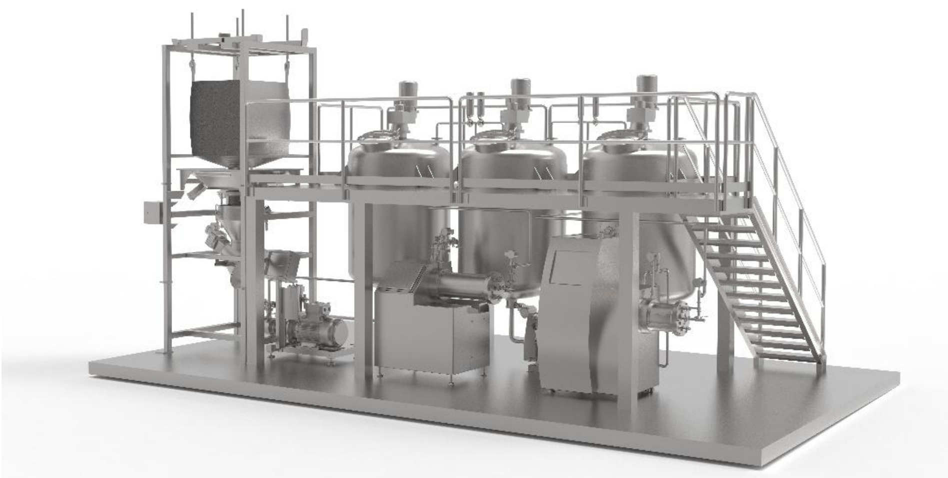

Figure 1. Production plant with modular design

Mechanochemical effects of simultaneous milling and carbonation on industrial steel slags

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Keywords: milling, carbonation, steel slags, soil additive.

As the carbon dioxide (CO_2) concentration in the atmosphere continues to increase despite ongoing efforts to decarbonate entire industries, it has become clear that reaching the climate targets set out in the most recent COP26 (Glasgow, 2021) has become impossible without developing negative emission technologies (NETs) in order to remove CO_2 from the atmosphere and store it quasi-permanently. An example of such long-term storage is the natural weathering of silicate containing rocks, which is responsible for the sequestration of 100 million tons of CO_2 per year [1]. Seifritz already had the idea in 1990 to exploit this phenomenon by pumping CO_2 through a slurry of pulverized rocks, but he also immediately recognized the large energy requirements needed for the mining and the grinding of the material on the one hand and the limitations of the kinetics of the sequestration reaction on the other hand [1].

The first challenge can be avoided by using silicate containing material that is quite fine already and available at relatively large scale such as industrial steel slags (rich in Ca, Fe, Si, Mg and Al) and is moreover produced at point sources for carbon dioxide emissions (steel plants) [2]. Furthermore, steel slags are currently already used as fertilizer: as they are applied to the soil, they weather naturally, providing ecological benefits such as increased crop growth and supply of nutrients [3-4]. Previous research suggest that these benefits can be improved by weathering the material intensively before application [5].

To tackle the second challenge and speed up the kinetics of the carbonation, one could look at the traditional approaches of increasing the CO_2 partial pressure or increasing the temperature, but more innovative methods include the use of ultrasound which has already proven to increase the carbonation yield in slurry carbonation by removing the passivating Si-rich layer on the surface of the slag particles [6]. The current work, however, focuses on the effects of mechanical activation through the use of ball milling on the carbonation reaction under thin film conditions. While the long term effects of milling such as an increase in specific surface area naturally improve the kinetics and the conversion of the sequestration [7], the potential of the short term effects such as lattice vibrations, the creation of fresh surfaces and internal stresses for further improvement of the kinetics [8] remain relatively unknown as most studies systematically perform the carbonation after the milling step [9-10]. Therefore, this research performs carbonation reactions without, after and while milling in order to decouple the short and long term effects of mechanical activation and quantify their respective contributions on the acceleration of the carbonation. In addition, milling parameters such as ball-to-powder ratio, ball size, rotation speed and milling time are varied as well to optimize the procedure.

Based on the findings of the current work, future research may include the investigation of other milling devices in order to get a better idea of what kind of forces (impact, shear and/or compression) are most useful for the acceleration of the reaction. This is crucial if the current lab-scale process is to be scaled up for industrial use.

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Comminution of lithium bearing pegmatites in a Hugger crusher based on a free crushing method

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Keywords: free crushing, microcrack, Hugger crusher, lithium bearing pegmatites.

Comminution in mineral processing is very energy intensive. For example in ball milling, energy efficiency is below 1 %. Valuable minerals are lost as ultrafine particles, which are generated when the ore minerals are crushed by arbitrarily directed loading during comminution. In addition, the fracture surfaces of mineral particles have physically and chemically changed because of wear and oxidation, making their further processing difficult and limiting application of dry and energy-efficient processes.

The “Hugger crusher” brings a new concept of comminution, which allows a new way to load, crush and mechanically separate solid materials. The Hugger comminution technique is a promising technology to be used for developing more sustainable dry processing, froth flotation, chemical leaching, and bioleaching methods. The controlled formation of microcracks in mineral particles enables more sustainable comminution and classification of ore minerals with less water, energy, and chemicals.

The formation of microcracks during the Hugger crushing is based on a phenomenon called “free crushing”. In the free crushing, the rate of feed is such that the crushed material passes freely through the double converging crushing unit without a contact between liberated particles. During the free crushing and slow compression, microcracks are formed at the natural boundaries of mineral crystals. This prevents the formation of ultrafine particles and reduces energy consumption.[1][2][3]

Our aim in this study is to analyse the effects of composition and textures of different ore types on the liberalisation and classification of minerals during the Hugger comminution. Our other aim is to study the potential of Hugger crushers in the dry processing on the benefication of different type of ores. In this study, we will focus on the ores containing metals and minerals that are critical for the European Union. One example of these type of ores are lithium bearing pegmatites. Pegmatites can contain spodumene, potassium feldspar, quartz, albite, and muscovite. Spodumene ($LiAl(SiO_3)_2$) is the source of lithium in pegmatites.

In this study, rock samples of spodumene pegmatite were first splitted with hydraulic press and then sorted by hand to ensure the comparability between sample batches before crushing tests. The crushing tests showed that the jaw crusher produced more ultrafine particles (<38 µm) than the Hugger crusher or the roll crusher, which can be seen in Figure 1. Microscopic examinations showed that particles produced in the Hugger crusher and in the roll crusher were fully liberated, sharp edged and almost dustless. Instead, liberated particles produced in the jaw crusher were heavily worn and rounded, which explains the abundant formation of ultrafine particles.

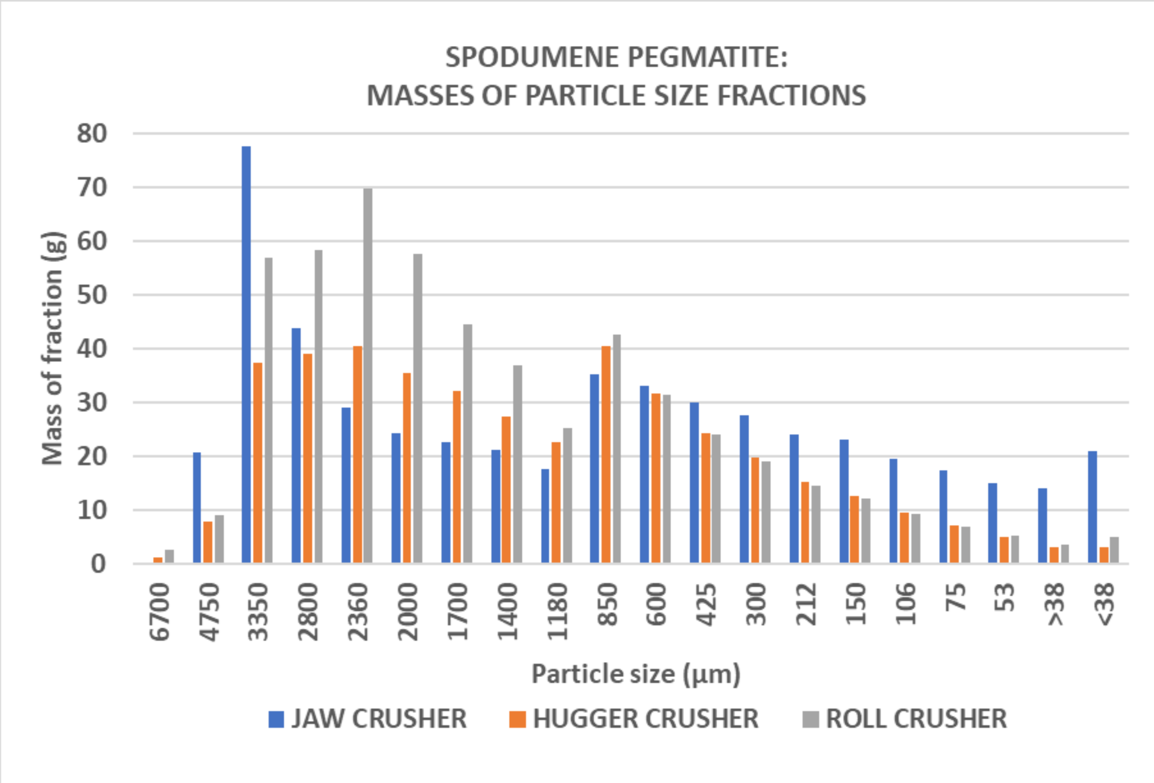

Figure 1. Masses of particle size fractions of spodumene pegmatite crushed with the jaw crusher, Hugger crusher, and roll crusher. The jaw crusher produced significantly more ultrafine particles compared to other crushing methods.

Lithium and aluminium concentrations were analysed from every particle size fraction with Microwave Plasma Atomic Emission Spectrometer (MP-AES). Analyses showed that lithium and aluminium concentrations of <38 µm size particles were same as >38 µm size particles. These metals are lost if the fine slime is disposed to waste. Another problem arises with other silicate minerals in pegmatites, which tend to enrich together with spodumene in froth flotation.

Ultrafine particles are undesired in froth flotation of spodumene, or any other flotation processes. Desliming is often needed to avoid the harmful effects of fine slime in froth flotation process. By optimizing the parameters of the Hugger comminution technique, it could be possible to produce cleaner mineral particles and reduce the amount of ultrafine particles in the froth flotation. This requires more detailed mineralogical studies on the formation of microcracks and liberation of mineral particles, as well as leaching and froth flotation tests. However, there is a lot of research work needed to validate this technique before its wider application in industry.

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Towards a new industrial way of micronizing problematic materials down to nanometric size

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Keywords: Micronization, Cryogenic suspension, Liquid nitrogen, ultra-fine powders.

Most industrial sectors use powders to develop end products, and they are becoming more demanding in terms of particle size distribution. Moreover, the rise of ultra-fine powders (submicronic or even nanometric sizes) is more and more significant. However, some materials are not that easy to micronize: they are either too ductile, soft, or too reactive. To meet the needs while considering the case of these materials difficult to micronize, the CEA has developed for several years a new technology by cryo-micronization using dense suspension made with nitrogen liquid and material to micronize (Figure 1).

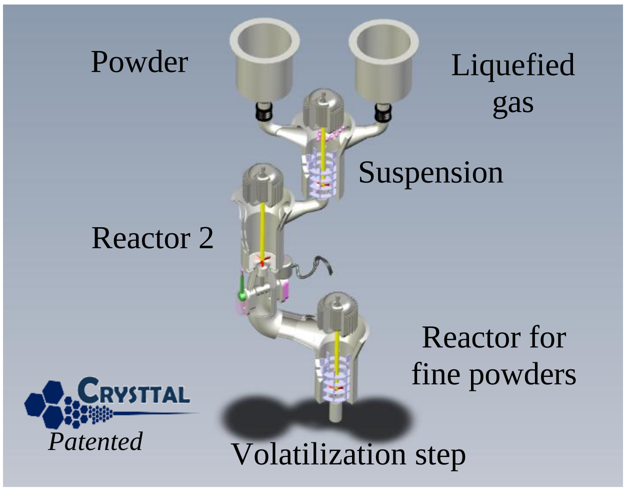

Figure 1. Princip of cryogenic suspension milling by CRYSTTAL [1].

Significant and promising results have been obtained on a wide range of products from heat-sensitive pharmaceutical active ingredients to compounds that can constitute future all-solid battery elements that are very reactive to humidity.

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[1] M. Brothier and S. Vaudez, patent WO 2017 / 076944A1 Device for mixing powders by cryogenic fluid.