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Predicting effects of operating condition variations on breakage rates in stirred media mills

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ABSTRACT

In order to model predictively the breakage kinetics in stirred media mills, it is required to determine the effect of operating conditions on the breakage rate. In this study specific breakage rates are measured for grinding experiments in wet-operated stirred media mills for various operating parameters. The experimental specific breakage rates are compared against two different models using yeast cells and limestone as test materials. A simple stressing energy model is able to predict the effect of changes in grinding bead size, grinding bead material, stirrer speed and mass concentration on the specific breakage rate. A more detailed model, which takes stressing energy distributions achieved by mill simulations and material breakage energy distributions from material tests as input, shows promising results. However, the latter is less robust and needs precise input data. Yeast cells prove to be a good test material for the purpose of model validation because of its well described breakage behaviour.

1. Introduction

Since fine grinding into the particle size range of micrometres and less has become steadily more important in various industries, the number of grinding and dispersing processes using stirred media mills has increased significantly. The mill chamber of a stirred media mill is filled with grinding media and a suspension with product particles (aggregates or primary particles that shall be milled or dispersed). A rotating stirrer moves the grinding media and the suspension. Stirred media mills are utilised with a wide range of operating conditions and various stirrer geometries due to their diversity of applications from pharmaceutical to the mining industry.

Typically, stirred media milling is incorporated as one unit operation in a longer process chain. For the dynamic simulation of the entire process chain there is a need for unit models that are capable of predicting the material's particle size distribution over processing time, considering changes in operating parameters and material composition. Population balance modelling allows the calculation of the evolution of particle size distribution in grinding processes. The specific breakage rate and the breakage distribution function are parameters of the population balance equations that can be obtained by experiments. However, the specific breakage rate obtained experimentally for one operating condition is not straightforwardly extendable to other operating conditions, since it is highly dependent on particle size, material properties and machine parameters. A model for the specific breakage rate shall distinguish between machine and material parameters to obtain predictive capabilities (Vogel and Peukert, 2002).

For dry impact comminution, Vogel and Peukert (2003) use the mass specific impact energy as machine parameter and derive a breakage probability with separated machine and material parameters. The stressing conditions in stirred media mills can be determined with semi-empirical models (e.g. Eskin et al., 2005; Kwade, 1999; Radziszewski, 2015; Afolabi et al., 2014). The shear based power model represents the stirred media mill as a viscometer and displays the impact of stirrer speed, geometry and viscosity on the power consumption of the mill (Radziszewski, 2015). The stressing energy model shows with the characteristic numbers stressing energy and stressing frequency, which are calculated by simple proportionalities, the influence of process parameters on the grinding result (Kwade, 1999). Eskin et al. (2005) estimate the mean velocity and frequency of grinding media

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oscillations via approximations of turbulent energy dissipations at the micro-scale. Based on this microhydrodynamic view of the particle stressing, Afolabi et al. (2014) define a process parameter depending milling intensity factor that correlates with the breakage kinetics of drug nanoparticles. Alternatively to the semi-empirical models, the stressing conditions in wet-operated stirred media mills can be obtained by the simulation of grinding media contacts via coupling of discrete element method (DEM) with computational fluid dynamics (CFD) (e.g. Jayasundara et al., 2011; Beinert et al., 2015, 2017) or smoothed particle hydrodynamics (SPH) (e.g. Cleary and Sinnott, 2015). Gers et al. (2010) characterize collision characteristics by determining collisional Stokes and Reynolds numbers from direct numerical simulations. Stressing energy distributions of DEM simulations in combination with material breakage behaviour have already been applied to ball mills for estimating the effect of operating parameters on the breakage rate (Concas et al., 2006; Tuzcu and Rajamani, 2011; de Carvalho and Tavares, 2013; Capece et al., 2014).

The aim of the present work is to predict the effect of varied operating and machine parameters in wet stirred media milling on the specific breakage rate. The stressing conditions in a stirred media mill are determined, using the semi-empirical model of Kwade on one hand, and using a CFD–DEM simulation approach on the other hand. The results of both approaches are compared to experimentally measured specific breakage rates of two materials: baker yeast cells and limestone. The use of yeast cells as a model system has following advantages: (1) the size reduction of inorganic particles occurs by several mechanisms such as body breakage, chipping or abrasion – each at different energy levels and with a different effect on the particle's size. In contrast to this, the cells can be seen as a binary system, i.e. having an intact or disrupted cell wall. (2) The cells show an almost monodisperse size distribution. (3) The burst energy is tunable in a certain range via the osmotic condition of the suspension (Arfsten et al., 2009). Previous studies suggest that repeated stressing at energies lower than the burst energy does not alter the ultimate burst energy significantly. Therefore, it is reasonable to omit damage accumulation for yeast cells. Other experimental model systems for stirred media mills have been proposed by Iwasaki et al. (2001, 2006) and Strobel et al. (2017). Iwasaki et al. determine the amount of deformation of copper particles through milling by measuring the diameter before and after milling. This allows calculating the effective transferred energy during milling. The stressing frequency could not be determined with this method. In a similar approach Strobel et al. also observe the deformation behaviour of copper and silica particles that have been stressed in a stirred media mill. Their elaborate characterization of the particles with a scanning electron microscope leads to estimations of stressing energy distributions and stressing numbers.

Limestone is frequently used to investigate the grinding process in stirred media mills (Kwade and Schwedes, 2002; He et al., 2006; Rácz, 2014). Unlike yeast cells, limestone exhibits a rather broad breakage energy distribution and shows naturally a breakage function.

2. Models

2.1. Stressing energy model

The stressing energy model of Kwade (1999) relates process parameters to the achievable product fineness using the characteristic parameters, stressing energy SE and stressing frequency SF, that describe the stressing conditions in wet-operated stirred media mills. This enables finding optimum operating parameters by performing a certain set of experiments. This article aims to extend the stressing energy model to the specific breakage rate.

The highest possible kinetic energy transferred to a product particle during a grinding bead contact, SE, is estimated using the tip speed of the stirrer v_t as highest possible relative velocity $v_{gb,rel}$ to:

$$SE = \frac{1}{2} m_{gb}\, v_{gb,rel}^2 = \frac{1}{12} \pi d_{gb}^3 \rho_{gb}\, v_t^2 \tag{1}$$

where m_{gb} is the mass, v_{gb} the relative velocity, d_{gb} the diameter and ρ_{gb} the density of a grinding bead.

The stressing frequency of the particles, SF, is proportional to the collision frequency of grinding media (number of bead-bead contacts per time and unit mill volume $\frac{n_c}{t}$), the amount of particles per unit mill volume n_p^* and to the probability P_{cap} that a particle gets captured.

$$SF \propto \frac{n_c}{t n_p^*} P_{cap} = \frac{n_c}{t n_p \left(1 - \varphi \left(1 - \epsilon\right)\right)} P_{cap} \tag{2}$$

Therein, t is the time and n_p^* is calculated by considering the number of particles per unit suspension volume n_p, the filling degree of the grinding media φ, and the porosity of the bulk of grinding media at rest ϵ.

The number of bead-bead contacts per time and unit mill volume is defined as to be proportional to the number of grinding beads per unit mill volume and the angular velocity of the stirrer ω_{cyc}:

$$\frac{n_c}{t} \propto \omega_{cyc} n_{gb} = \omega_{cyc} \frac{\varphi \left(1 - \epsilon\right)}{\frac{d_{gb}^3 \pi}{6}} \tag{3}$$

In order to obtain the capture probability an "active volume" between two grinding beads is defined by Kwade (1999) as shown in Fig. 1a.

$$V_{act} = \frac{\pi}{4} x^2 d_{gb} \tag{4}$$

The underlying assumption is that a particle positioned in this volume will be captured and stressed during a grinding bead contact. Assuming further that the particles are equally distributed in the suspension with the number concentration n_p, the average number of particles in the active volume can be calculated. In Fig. 1b the average number of particles in one active volume is shown for the investigated parameters of this work. The capture probability is interpreted to be the average amount of particles in the active volume for a homogeneous distribution of the particles in the liquid. This amount is limited to one, reflecting the assumption that only one particle will be captured and stressed with the highest stressing energy of the grinding beads, while other particles will be removed from the active volume or be stressed only insignificantly.

$$P_{cap} = \max \left(n_p V_{act}, 1\right) \tag{5}$$

In case of a non-homogeneous particle distribution, the predicted capture probability might be overestimated. Furthermore, particle displacements related to local fluid flow between approaching grinding beads are not taken into account. Nonetheless, the simplified equations have been proved to successfully describe the milling behaviour of stirred media mills in the micrometre particle range (Kwade and Schwedes, 2002).