Powder Technology 278 (2015) 84–93

Contents lists available at ScienceDirect

Powder Technology

journal homepage: www.elsevier.com/locate/powtec

Effects of mill design and process parameters in milling dry extrudates

Aditya Vanarase [a], Rizwan Aslam [b], Sarang Oka [b], Fernando Muzzio [b,*]

[a] Bristol-Meyers Squibb, 1 Squibb Drive, New Brunswick, NJ 08903, United States
[b] Department of Chemical & Biochemical Engineering, Rutgers, The State University of New Jersey, NJ, United States

A R T I C L E I N F O

Article history:
Received 30 November 2014
Received in revised form 29 January 2015
Accepted 13 February 2015
Available online 20 February 2015

Keywords:
Milling
Cone mill
Hammer mill
Process parameters
Particle size distribution

A B S T R A C T

An experimental study was performed to characterize two continuous mills for their ability to mill alumina–magnesia extrudates. The effect of mill parameters, namely, the screen aperture size, and impeller speed on the particle size distribution of the milled product was quantified for a conical screen mill and a hammer mill. In general, the conical screen mill was found to be more sensitive to changes in impeller speed compared to the hammer mill. The effect of impeller speed in case of the hammer mill was non-monotonic while the increasing speeds led to reduction in particle size in case of the cone mill, for the same screen aperture size. The effect of aperture screen size was observed to play a dominant role in dictating particle size distribution of the product material for both mills. In case of the cone mill, grated type screens exhibited higher milling capacity than round screens with equivalent apertures.

Lastly, a study comparing the statistical particle size distribution parameters was performed for process design purposes. It was deduced that, if the desired particle size is greater, the comil provides a narrower particle size distributions than the hammer mill; whereas if the desired particle size is smaller, both mills exhibit similar poly-dispersity. The study provided insight into fundamental breakage mechanisms for both mill classes. Breakage in the hammer mill occurs primarily due to the impact of the hammers and large particles may often leak through the mill without sufficient breakage. Breakage in the comil is more gradual as the impeller sweeps a wide area generally ensuring sufficient breakage of particles before they exit the milling chamber.

1. Introduction

Granular milling processes are used throughout industry. For example, in the pharmaceutical industry, milling is used at various manufacturing stages, including wet and/or dry milling after crystallization of drugs to reduce the particle size or achieve the desired PSD [1], de-lumping of dry powder ingredients to de-agglomerate and improve power handling, milling of ribbons after roller compaction, and de-lumping of the wet mass in wet granulation [2,3]. Other industries make similar or even more extensive use of milling steps. For example, in catalyst manufacturing, ingredients are often de-lumped, wet granulated, extruded, dried, and milled again. Similar sequences involving multiple milling steps are common in food processing, cosmetics manufacturing, minerals processing, etc.

In order to integrate mills effectively into continuous manufacturing processes, developing predictive understanding of continuous mills is necessary. This particular study is aimed at understanding the size reduction behavior in continuous mills. Alumina–magnesia dry extrudates, a ceramic material used in the manufacture of catalyst supports, was used as the model material. In this study, a methodology to optimize the particle size distribution under process constraints such as milling capacity and/or PSD requirements is presented.

Often times in the manufacturing environment, the processing rate of the milling operation needs to be changed depending on the upstream/downstream process requirements, while maintaining the PSD of the milled material within the desired specifications. In such cases, the material response to operational parameters of the mill needs to be well understood.

Typical industrial continuous mills include hammer mills, conical mills, pin mills, knife mills and reciprocating mills. Population balance models haven been applied for modeling the milling performance in hammer mills [3,4] and conical mills [5]. Hammer mills can also be modeled based on the damage mechanics of the material [6,7]. The population balance models built on the batch milling case have been applied for the equivalent continuous mill by assuming perfect mixing conditions and measuring residence times [8,9]. However, studies comparing the performance of different mills for the same materials remain scarce, and quantitative methodologies for comparing the resulting PSDs need further development.

In this paper, we attempt to address both of these gaps. This study is focused on a comparison between two commonly used continuous mills—a conical mill and a hammer mill. The relative effects of

* Corresponding author. Tel.: +1 848 445 335.
E-mail address: fjmuzzio@yahoo.com (F. Muzzio).

http://dx.doi.org/10.1016/j.powtec.2015.02.021
0032-5910/

operational parameters are examined and quantitative criteria for comparing PSDs are introduced.

2. Equipment

2.1. Conical mill (Comil—Quadro)

The conical mill used in this study was a Quadro Comil Model-197-S (Fig. 1(a)). Following recommendations of the equipment manufacturer, the conical impeller was positioned as close as possible to the screen (Fig. 1(b)). Two types of screens (Fig. 2(a)), one with round holes, and another with a combination of round and grated holes, were used in this study. The Comil operates by applying intense shear in a narrow region between an impeller and the screen, which creates frictional contacts and drives particles through the perforations of the screen. The choice of screen is very important because the perforation size and spacing greatly affect the PSD. The material used in this study is very brittle and undergoes fragmentation (resembling many dry granulated and some wet granulated materials following drying). In order to impose the least amount of shear on the material, an impeller with a round cross section (Fig. 2(b)) was used.

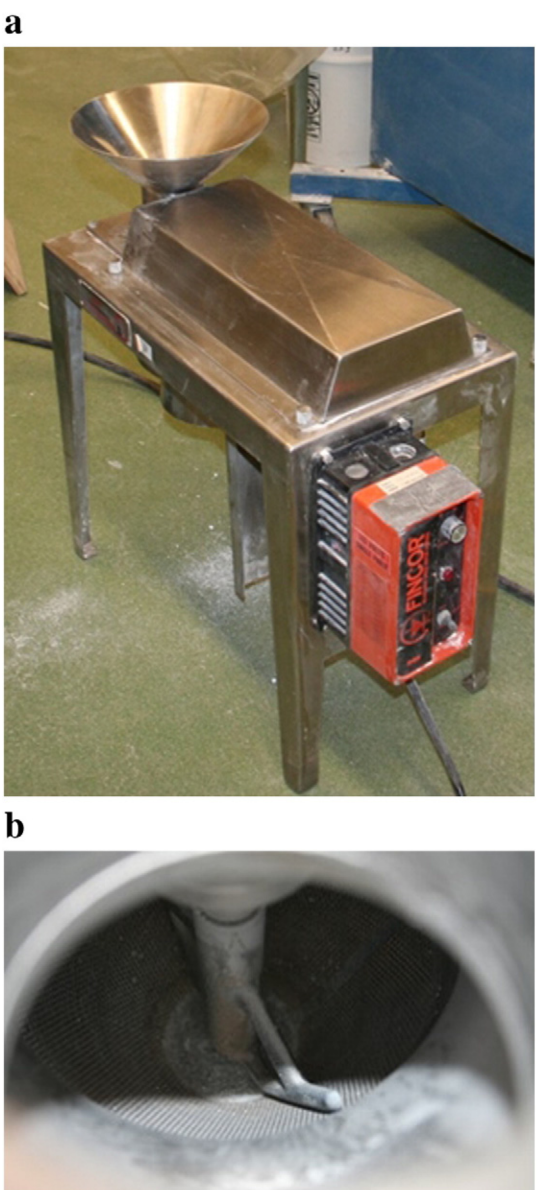

Fig. 1. (a) Conical mill (Comil—Quadro Model # 197); (b) milling chamber with conical round impeller.

2.2. Hammer mill (Fitzpatrick)

The hammer mill used in this study is manufactured by Fitzpatrick (Fig. 3(a)). The blades of the hammer mill pulverize particles along their rotational path. The shape of hammers is shown in Fig. 3(d). As shown in Fig. 1(b), the tips of the blades create a narrow region of intense shear near the surface of the screen. The screen size in this setup has less importance because the shearing is mostly being done by the blades. The screens used in this mill are of a 'C' shape (Fig. 3(c)).

3. Materials

The material used in this study, called 'Material 1', is a mixture of aluminum and magnesium oxides that has been mixed as a wet paste and then calcined at high temperatures, and is used in the manufacture of catalyst supports. The initial material was in the form of extrudate pellets as shown in Fig. 4(a). The pellets were 0.5 cm in diameter and variable in lengths. The length varied between ~0.5 and 3 cm.

4. Particle size distribution measurement method

A vibrational sieve shaker, 'Endcotts Octagon 2000', shown in Fig. 5, was used for sieving. The milled powder was sieved into 9 size bins. The sieves had 8″ diameter and U.S. standard sieve numbers of 10, 14, 18, 30, 40, 60, 120 and 230 were used in the PSD measurement. The sieving procedure used can be described as follows. All the trays were cleaned and dried before the sieve analysis. A sample of 200 g of material was loaded on the top sieve. The sieve tray assembly was then placed on the shaker table and secured with the shaker tables clamp. The shaker table was set to power 8, and run for 15 min. After sieving, the mass of the powder on individual trays was weighed. A bar chart (Fig. 6(a)) of mass fraction vs. size bins was thus created. Particle diameters (d10, d50, or d90) were read from the cumulative PSD graph using linear interpolation.

5. Experimental conditions

The experimental conditions examined for the Comil and hammer mill are presented in Tables 1 and 2 respectively. The range of impeller speeds used in both mills was similar. The milling chamber of hammer mill is larger than that of the Comil, which makes the tip speeds in the hammer mill greater than those in the Comil for equivalent impeller RPMs.

6. Results

6.1. Effect of operational parameters on the flow rate and impeller passes in the Comil

The PSD of the milled material is dependent on its residence time in the mill. The residence time of the material in the mill was estimated by measuring the material hold-up in the mill and dividing it by the mass flow rate. Out of the two possible feeding modes (chocked feeding and starved feeding), the Comil was operated under chocked feeding condition. Under the starved feeding condition, flow rate of the incoming material is constant, and hold-up (mass) in mill varies as a function of the operational parameters (screen, impeller speed); whereas under the chocked feeding