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Energy balance and design equation for the milling of low-melting organic solids

Michael T. Searfass *

Occidental Chemical Corporation, Corporate Engineering, 1000 Tidal Road, Deer Park, TX 77536, United States

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Abstract

To model the behavior of a three-phase stream involving a change of state, an energy balance is derived and then reduced into a practical design equation for a hammer mill. A combination of experimental data, known physical properties, and classical thermodynamic relationships are used to relate processing rate to energy input to the mill. The design equation is applicable to the processing of three-phase streams containing a low-melting organic compound and an evaporating cryogenic liquid, such as nitrogen, which is used as a coolant to preserve the state of the organic material.
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1. Introduction

Engineers frequently encounter unit operations for processing solid materials, but may lack the necessary empirical tools to define, scale-up, and optimize the performance of such units. As organic chemical process development and manufacturing methods are refined, expertise in solids processing becomes ever more critical to the engineer who may be accustomed primarily to liquid and gas phase processes. Solids processing is usually multiphase, involving the solid and at least air or an inert gas, so some preliminary knowledge about the process thermodynamics and design of relevant unit operations can be invaluable.

In many applications of organic fine chemicals, the morphology and particle size of the final compound is as important as its molecular structure for accomplishing the desired end use. Regardless of whether the compound is a pharmaceutical, agricultural, or other specialty chemical, typically short process development deadlines demand more concentration on optimizing reaction yield and maximizing the purity of isolated intermediates than on the final macroscopic form of the technical material. Particle size reduction is often left to formulation chemists or engineers to resolve later, especially if size control relies upon mechanical equipment somewhat outside the expertise of those engaged in reaction step development for the active compound. Consequently, an important opportunity to gather useful and scalable data for particle size reduction can be lost if manufacturing methods are not tested early in the development process, and with particular emphasis upon the unique physico-chemical properties of the solid.

A common unit operation employed for particle size reduction in both pilot plant and chemical manufacturing operations is the hammer mill. Shown schematically in Fig. 1, this device consists of a solids feeding system, milling chamber or casing that houses a heavy flywheel, discharge screen, and motor. The flywheel hub contains many flat sided bars, arranged like the spokes of a wheel and often hinged at their bases, that rotate at high speed and break apart incoming solid particles on impact. The particles remain inside the mill casing and continue to be pulverized by the hammers and contact with each other until they are sufficiently small to pass through the mesh

* Tel.: +1 281 476 2221; fax: +1 281 476 2246.
E-mail address: MTS9297@sbcglobal.net

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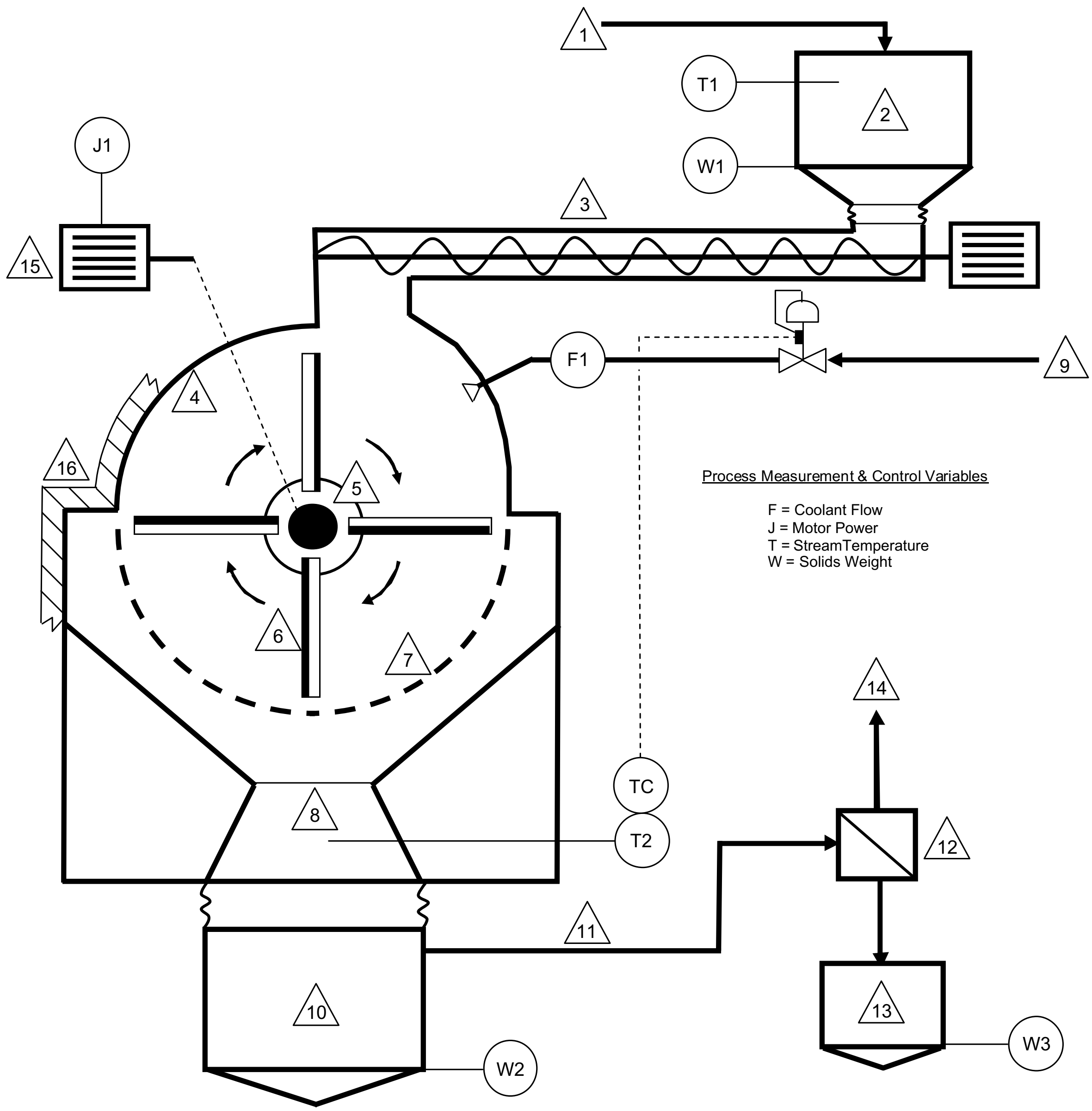

Fig. 1. Schematic diagram of milling process. Raw solids (1) enter through a feed hopper (2) and are charged via a screw conveyor (3) into the mill housing (4). The flywheel (5) equipped with multiple hammers (6) spins at high speed to break apart solid particles that, when sufficiently reduced in size, exit through a screen (7) and into a discharge chute (8). Inert coolant (9), such as liquid nitrogen, is simultaneously sprayed at a controlled flow rate into the mill based on temperature measured in the discharge chute. Finished solids are collected in a weighed hopper (10) and vaporized coolant (11) passes through a filter (12) that separates entrained fines (13) from clean vent gas (14). The mill drive motor (15) is equipped with a current or power monitor, and the mill housing (16) is insulated to both minimize heat loss and conserve coolant.

of the discharge screen. The smallest discharge screen employed, and therefore the smallest diameter particle size achievable, is dependent upon energy input to the mill and hardness of the particles.

Significant heat can accumulate due to the work done by the mill on the particles, the impact of particle against particle, and friction between particles and interior surfaces of the casing. When processing low-melting organic solids, this heat can be sufficient to soften or melt the material, thereby causing unwanted agglomeration or plugging of the discharge screen. Also, even a moderate increase in temperature can change the crystal structure of the material, consequently affecting its activity in the final formulation. Undesired structural alteration is of particular concern for organic compounds composed of two or more unstable polymorphs [1]. One effective method to remove heat during milling is to inject a cryogenic fluid, such as liquid nitrogen, into the mill along with the feed solid. Heat transferred from the solid vaporizes some or all of the liquid phase and maintains the milled solid at a suitably low temperature.

2. Defining the process

Many attempts have been made to relate particle size reduction to the energy input to a mill, but these

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