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PARTICLE SIZE REDUCTION IN THE BALL MILL

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ABSTRACT

The size distribution produced by the ball milling of various crystalline and non-crystalline materials, showed that initially there was a fairly even distribution over the size range up to 355μm. However, as milling proceeded two distribution modes developed; one at about 90μm (the persistent mode) and one at about 250μm (the transitory mode). Unlike the work of Heywood (1) on coal, further grinding did not produce a gradual elimination of the coarse mode with corresponding increase in the persistent mode at 90μm. Less than 2%w/w of material was always found between the 170-180μm size, indicating that this was a critical dimension in the grinding process. A linear relationship was found between sample weight and milling time required to grind 50% of the sample down to 150μm. The slopes of these lines were steep for rock salt and sucrose (crystalline materials) indicating that milling was

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rapid, whilst with acacia the rate was half as fast and slower still for the other two non-crystalline materials, cinnamon bark and gentian root. In terms of milling efficiency defined as the amount of material milled to 150μm per minute, it was found that there was no optimum load size; doubling the sample size effectively doubled the milling time. However, the most critical factor affecting the milling process, was the weight and nature of the grinding medium. It was found that heavy porcelain was more efficient than glass spheres and that the optimum weight was 401.8g; the weight required to approximately half fill the ball mill.

INTRODUCTION

The ball mill is extensively used in pharmacy for the comminution of drugs and excipients, but suprisingly with this type of mill little is known about the mechanism of size reduction. Heywood(1) found that with coal, as milling proceeded a bimodal size distribution initially developed which eventually reduced to a unimodal distribution as grinding increased. However, despite the numerous and varied applications of particle size reduction in the ball mill, there is little and adequate pharmaceutical literature on the subject as illustrated by Parrott(2) in his review article. Garlick(3) reported that there were six variables influencing the operation of a ball mill. These were the speed of the mill, quantity and size of the balls, quantity and consistency of the material and initial particle

size. Although no experimental data was presented, Garlick suggested that correct adjustment of these six variables would influence the efficiency of milling and produce less wear and tear on the mill. The economic advantages of using a smaller ball load were discussed by Fahrenwald(4). Using a 12 inch ball mill it was shown that a 29% ball load was more efficient than a 49% load, since there was less overgrinding as well as an increase in material recovery and a reduction in power consumption.

Since ball milling is such an important pharmaceutical operation and there appears to be little known about the factors affecting this milling process, the aim of this work was to compare the milling of various materials under standardised conditions. The variables investigated were; the weight of the balls in the mill, the nature of the grinding media and the weight of material added to the mill.

MATERIALS AND METHODS

Commercial rocksalt, acacia, cinnamon bark, gentian root and sucrose were graded such that all the materials passed through a 2mm aperture sieve but were retained on a 500μm screen.

MILLING

Milling was performed in a 1 litre porcelain ball mill pot (Pascall Engineering Ltd., Crawley, U.K.) of outside diameter

120mm, overall length 146mm and containing 401.8g of porcelain cylinders, 1cm long by 1cm diameter, average weight 5.62g, which together with the voids approximately half filled the pot. The mill was powered by a 1/6 H.P. electric vibration motor operated at a constant velocity of 1450 revolutions per minute. 50g of material was placed in the mill pot and the contents were milled for 3 minutes. The milled material was seperated from the porcelain cylinders by screening through a 2mm sieve. A sieve analysis was performed as described below and then the material was replaced back into the mill and milling continued for 5,8,10,15,20 and 30 minutes total milling time. Subsequent samples of 25,100 and 150g of rocksalt were milled and sieve analysed as above and 50g samples using 204.5 and 101.2g of porcelain cylinders were also subjected to this process. For acacia, cinnamon bark, gentian root and sucrose the milling time was increased up to a maximum of 120 minutes and the sampling times were chosen according to the rate of breakdown of the material. Milling was continued until the size of the sample approximated to that of rocksalt (70-80μm). A final 50g sample of sucrose was milled and analysed using 204.5g of glass spheres instead of the porcelain cylinders.

SIEVE ANALYSIS

A sieve analysis was carried out using the following sieves: 500,355,250,180,178,125,90,63 and 45μm. The sample was poured into

the top 500μm sieve and the nest of sieves secured to the inclined table of a sieve shaker ("Pascall Inclyno", Pascall Engineering, Crawley, U.K.) by means of an adjustable clamping plate. The sieve table gyrated at 2.5 cycles/min. and simultaneously moved up and down at 300 jolts/min. A standard sieving time of 5 minutes was selected and the amount of material on each sieve was weighed and recorded. The material on each sieve was then transferred back to the ball mill for further grinding.

RESULTS AND DISCUSSION

Graphs of % material on sieve against sieve size were plotted (figure 1). Initially the material was distributed uniformily on each of the sieves up to 355μm. No initial size distribution showing a single mode corresponding to a relatively coarse size was evident, as found by Heywood(1) for coal. The process of comminution must overcome the forces which hold solid particles together. These forces are basically chemical and comminution is a mechanism by which the applied mechanical energy is transformed into chemical energy. Size reduction thus begins with a crack fracture and it is only when the crack has spread so that it has reached the boundary of the particle at all parts of its perimeter that comminution is observed. Cracks generally follow planes of weakness consequent upon the existence of dislocations in the material. The basic theory of crack propagation is attributed to Griffiths(5). He argued that for a crack to propagate two conditions must be fulfilled, namely (a) it must be energetically

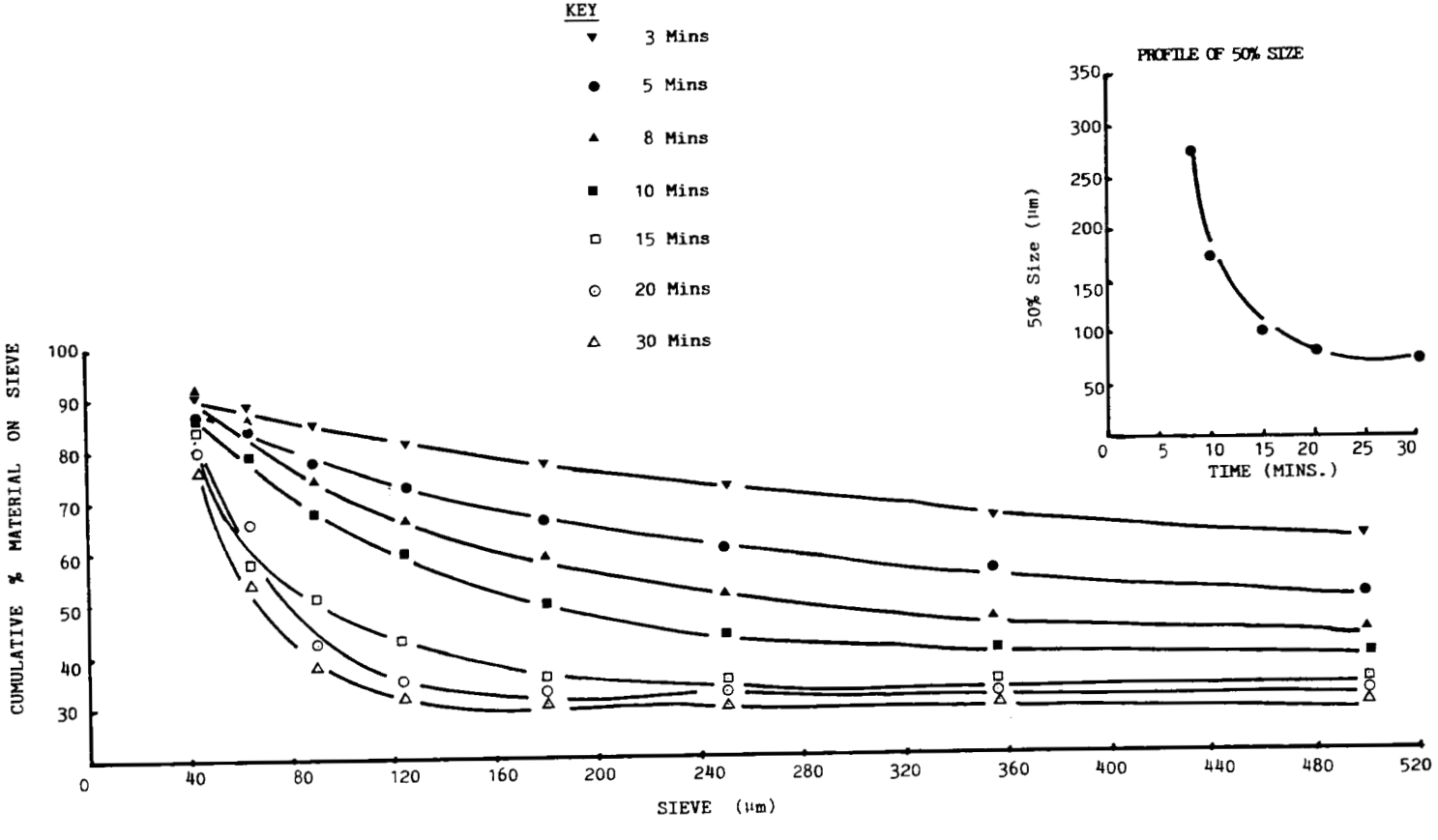

FIGURE 1

Graph of % Material on Sieve against Sieve Size for 50g Cinnamon Bark at Different Milling Times Using 401.8g of Porcelain Cylinders.

desirable and (b) there must be a mechanism by which the energy transformations can take place. Typical profiles (figure 1) showed that as milling proceeded two distribution modes developed; one at about 90μm (the persistent mode) and one broad mode at about 250μm (the transitory mode). The initial small number of large particles, being subjected to increasing mechanical energy, broke to become more numerous and smaller in size. However, the fine particles because of their smaller number of dislocations, did not greatly alter in size, but the number produced increased appreciably. Griffiths(5) proposed that crystalline materials fracture along crystal cleavage planes, whereas non-crystalline materials fracture at random. As milling proceeds material breaks down into a few large particles and a number of fine particles with relatively few of intermediate size. The size of the larger particles is related to the size reduction process and the size of the finer particles is dependant on the structure of the material. Unlike the results of Heywood(1) with coal, no gradual decrease in size of the coarse transitory mode with increased milling time was found with the pharmaceutical materials investigated. Thus (figure 1), although the initial stages of grinding did eventually produce a two component distribution, subsequent grinding did not in all cases completely reduce the magnitude of the coarse component and continually increase that of the fine component, as found by Heywood. A characteristic and predominant factor in all the sieve analyses was the existence of very little material of an intermediate size, between 170-180μm. In all cases less than 2% by

weight was always found on these sieves. This particle size would seem to be a critical dimension. Particles very much larger than 180μm fracture from the particle periphery to produce a high proportion of very small particles. Eventually the core particle breaks in two to produce two particles of a size less than 170-180μm. The net result as can be seen from figure 1, is that there are very few particles of an intermediate size corresponding to 170-180μm. This phenomenon is characteristic of all types of material investigated.

Graphs of cumulative % material on sieve against sieve size were also drawn and from these graphs the mass median size was interpolated. This value against milling time was plotted and in all cases (figure 2) the 50% size reduced dramatically after the initial stages of milling, but remained fairly constant thereafter. In order to summarise all of the results and to see an overall picture of the effects of the different factors influencing milling, 150μm was taken as a representative size of the milled material and the total milling time taken for the 50% size to reach 150μm was evaluated (table 1). The effect of varying the weight of sample added to the ball mill was suggested to be an important factor in the milling process. The 25g samples, irrespective of the nature of the material itself, were milled to the required size in the shortest time (table 1). This time was approximately doubled for the 50g samples. A further doubling of time was found for the 100g samples and generally there were

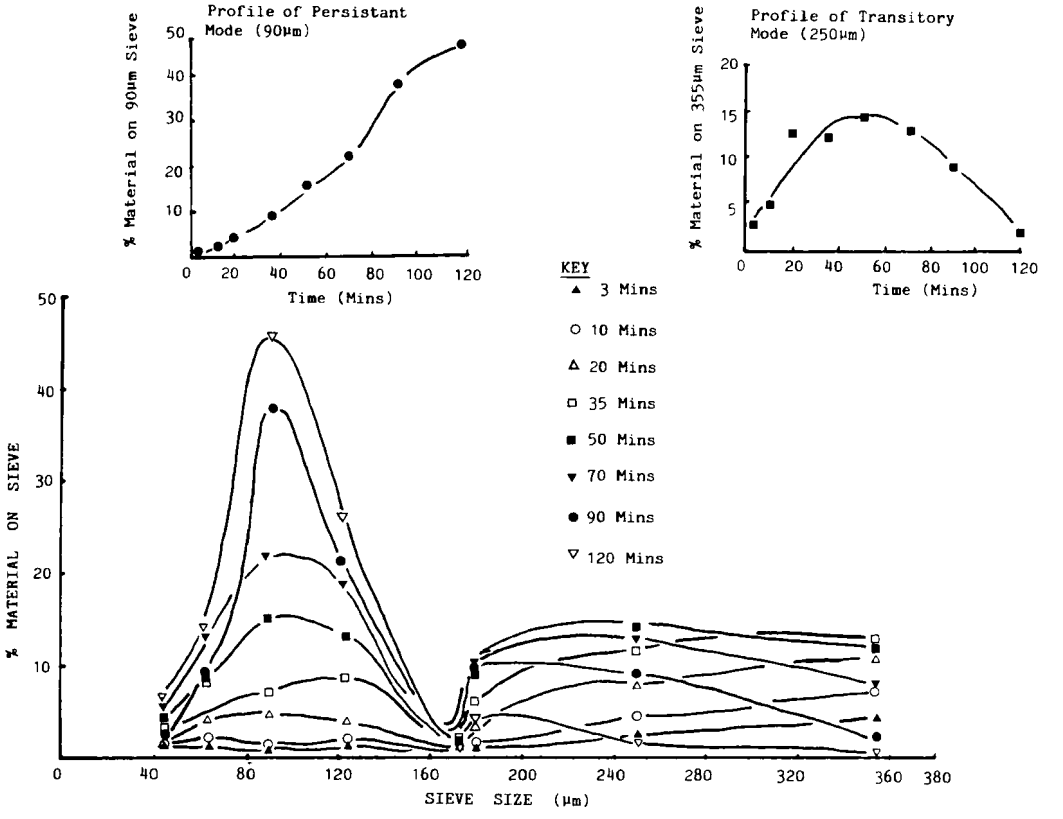

FIGURE 2

Graph of Cumulative % Material on Sieve against Sieve Size for 50g Rocksalt at Different Total Milling Times Using 401.8g of Porcelain Cylinders.

linear relationships between sample size and milling time required to grind 50% of the sample down to 150µm (figure 3). The slopes of the graphs were steep for rocksalt and sucrose, indicating that milling was rapid, whilst with acacia the rate was half as fast and very slow for cinnamon bark and gentian root. Rocksalt and sucrose are examples of crystalline materials which break by fracture along the crystal cleavage planes. Cinnamon bark and gentian root however, cannot crack propagate and thus size

Table 1. Total Milling Time (min.) Required to Mill 50% of Sample Down to 150um.

Sample Size	Grinding Media	Rock Salt	Acacia	Cinn. Bark	Gentian Root	Sucrose
25g	401.82g*	5	11.5	34	22	4.5
50g	401.82g*	8.5	20.5	65	>30	8
100g	401.82g*	17.5	50	120	≫30	20
150g	401.82g*	27	84	>120	⋙30	42
50g	204.50g*	14	52	128		18
50g	101.18g*	>20	110	≫120		54
50g	204.50g**					120

• Porcelain cylinders ** Glass beads

reduction is achieved by tearing the bark or root cells. Obviously, greater mechanical energy is required to do this and therefore with these non-crystalline materials, a slower milling rate is observed. Figure 3 indicates that sample loading is not critical, since although doubling the sample size, doubles the time of milling, the efficiency in terms of grammes of material milled per minute remains constant. Thus there would appear to be no optimum load size which produced the most efficient milling of the materials.

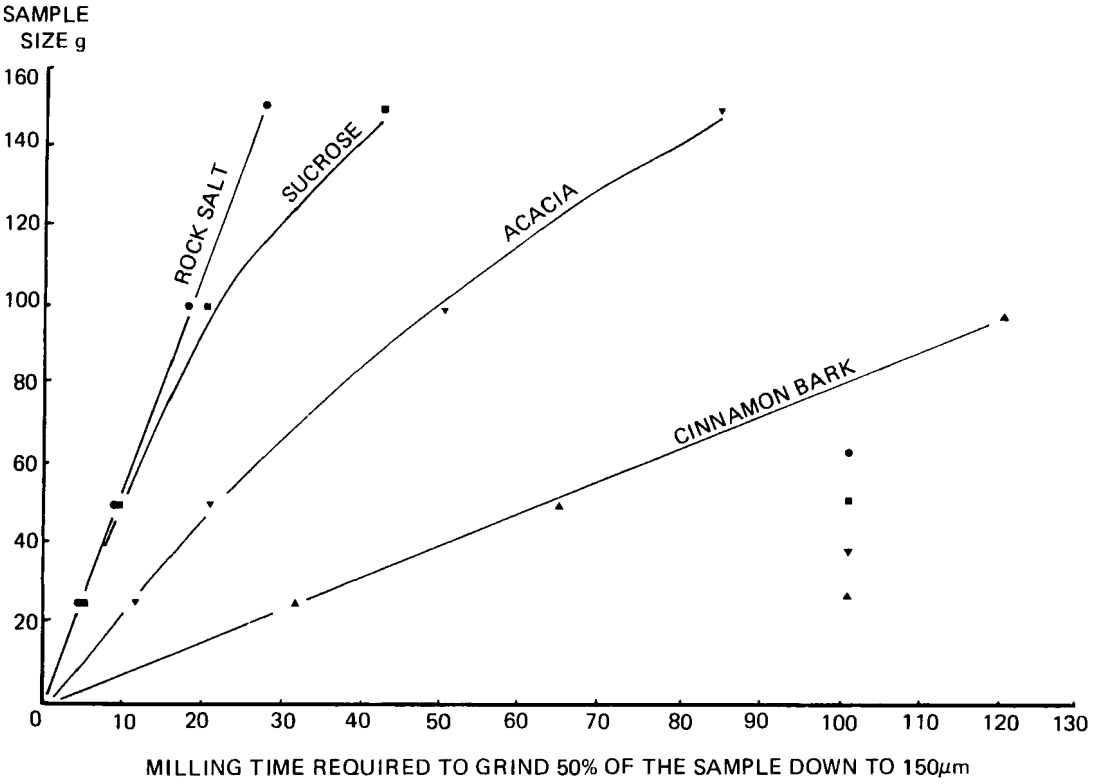

FIGURE 3
Graph of Sample Size Against Milling Time Required to Grind 50% of the Sample Down to 150μm.

Examination of table 1 shows that for most efficient milling, irrespective of the properties of the sample, the optimum weight of porcelain cylinders was 401.8g. This quantity approximately half filled the ball mill pot. Using one half and one quarter of this weight of porcelain cylinders significantly increased the total grinding time necessary to reach the specified sample size. Table 1 also shows that by changing the grinding media from porcelain to glass beads drastically reduced milling efficiency. Sucrose was used as the material in this study, because it

exhibited the fastest milling time needed to reduce the sample down to 150μm. This milling time was increased more than twice by changing to glass beads.

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