

THE EFFECT OF DIFFERENT MILLING CONDITIONS ON PARTICLE SIZE DISTRIBUTION OF MILLED HERBAL MATERIAL

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1 INTRODUCTION

While size reduction has been explored with great interest for pharmaceutical drug powders, it has received relatively much less attention with herbal materials especially in extraction processes despite its ability to influence such operations. Size reduction operations are important



for attaining the desired particle size and size distribution and, hence determining the surface area for effective liberation of active components during extraction [1] as well as the extraction bed packing attributes. The efficiency in an extraction process as well as the proportion of active principles extracted is dependent on the feed particle size and the structure of the milled materials. Typically, milling of herbal material is carried out by either cut or impact mills. Conical screen milling, a relatively less explored milling technology, offers the advantage of preparing milled herbal material with tighter size distributions.

2 OBJECTIVE

The objective of this study was to investigate the use of conical screen mill for size reduction of botanicals prior to extraction, in comparison with conventional methods such as impact and cut milling.

3 EXPERIMENTAL

100 g batches of air-dried stripped and sliced magnolia bark (*Magnolia officinalis*, Zhejiang, China) were milled separately using a conical screen mill (Quadro® Comil®, Model 197S, USA) while impact and cut milling were performed using a FitzMill (Fitzpatrick, Comminutor M5A, USA) with blunt-edge and sharp-edge forward configuration respectively. The milling conditions employed are summarized in Table 1. Milling was performed in triplicates at impeller speeds of 2000, 3000 or 4000 rpm for all experiments.

Table 1. Milling conditions employed in the study

Code	Mill type	Impeller	Screen		
			Shape of aperture	Size of aperture (µm)	Open area (%)
H	Impact	Blunt-edge	Round	4750	51
K	Cut	Sharp-edge	Round	4750	51
C	Conical screen	Rectangle bar	Grater	4750	23
CS	Conical screen	Round bar with teeth	Grater	4750	23
CR	Conical screen	Rectangle bar	Round	4750	51
CJ	Conical screen	Round bar with teeth	Round	4750	51

Particle size distributions of milled materials were obtained using a stack of sieves from 125 µm to 4.00 mm in a $\sqrt{2}$ progression by vibratory agitator (Retsch, Model VS 1000, Germany) for 20 min at an amplitude of 2 mm. Cumulative distribution plots were used to determine the mass median diameter (MMD) and span calculated as $(D_{90} - D_{10}) / \text{MMD}$, where D_{90} and D_{10} is the 90th and 10th percentile of the cumulative % undersize distribution plot, respectively.

Control of particle size and size distribution of the feed material is important for the extraction process. As a rule, particles with mean diameters ranging approximately between 0.25 and 2 mm are used [2]. Fines were defined as particles below 250 µm while oversized particles were those above 2 mm. All experiments were triplicated and results analysed using ANOVA (SPSS v13, 2004, USA) at $\alpha = 0.05$.

Why is control of particle size important?

Production of high amount of fines leads to excessive dust generation and subsequent health and contamination risk issues in the production facility. Presence of an excessive amount of fines also leads to segregation and clogging of herbal material during conveyance through the extraction vessels in continuous processes which would contribute to variability in quality of the extract.

Oversized milled particles, on the other hand, would contribute to large mass transfer resistances leading to less efficient extraction processes and poor packing properties, affecting the production capacity.

4 RESULTS & DISCUSSION

- Milling of the magnolia bark failed when round bore screens were used with either impeller type in **conical screen milling**. This implies that **milling was mainly effected by the screens instead of the impeller**, contrary to what was reported in another study, whereby size reduction was mainly due to impeller action while the screen only acted as a classifier [3]. A **grater surface** was thus deemed necessary to provide shearing action for fracturing and slicing of the fibrous bark material.

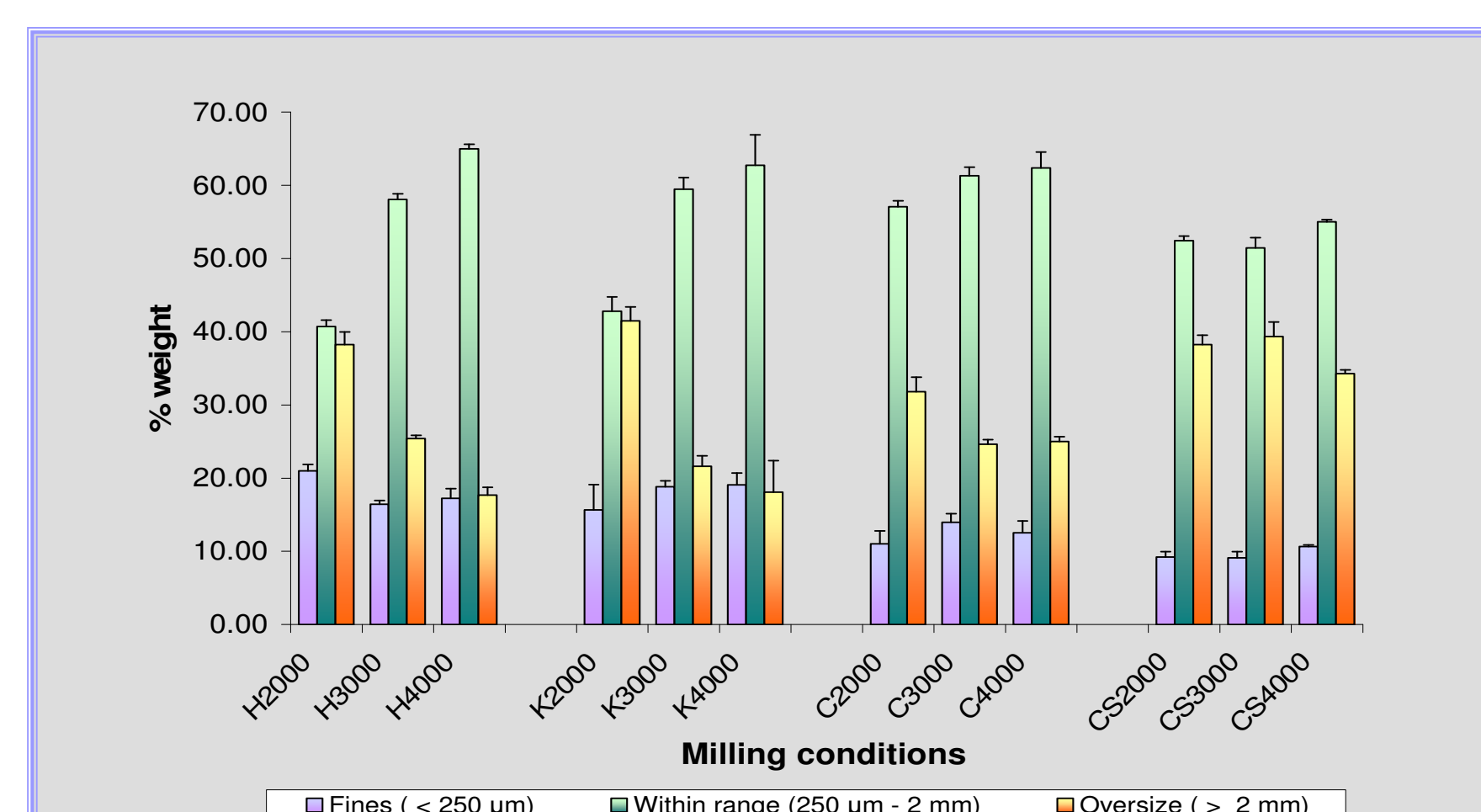


Figure 1. The effect of mill type and impeller speeds on production of fines, oversized and milled particles within the desired range. Numbers following code denote the impeller speed in rpm. E.g. H2000 = Impact milling at 2000 rpm.

- Use of the grater-bore screen in conical screen milling also resulted in significantly **lower production of fines and oversized particles** regardless of impeller speed and type compared to both impact and cut milling methods ($p < 0.05$), except at 4000 rpm.
- Intrinsic differences in the milling mechanisms.** **Impact and cut milling** depend on direct and rapid impact / shearing action of the impeller surface on the chamber walls to reduce the particle size of solids. Materials undergoing milling have a longer residence time in the milling chamber until they are reduced to the appropriate size before passing through the retention screen. In **conical screen milling**, the shearing action takes place at the grater surface itself. **Centrifugal acceleration** created by the rotating impeller sweeps product continuously outward through the screen to produce more defined particle size reduction, with minimal fines generated.

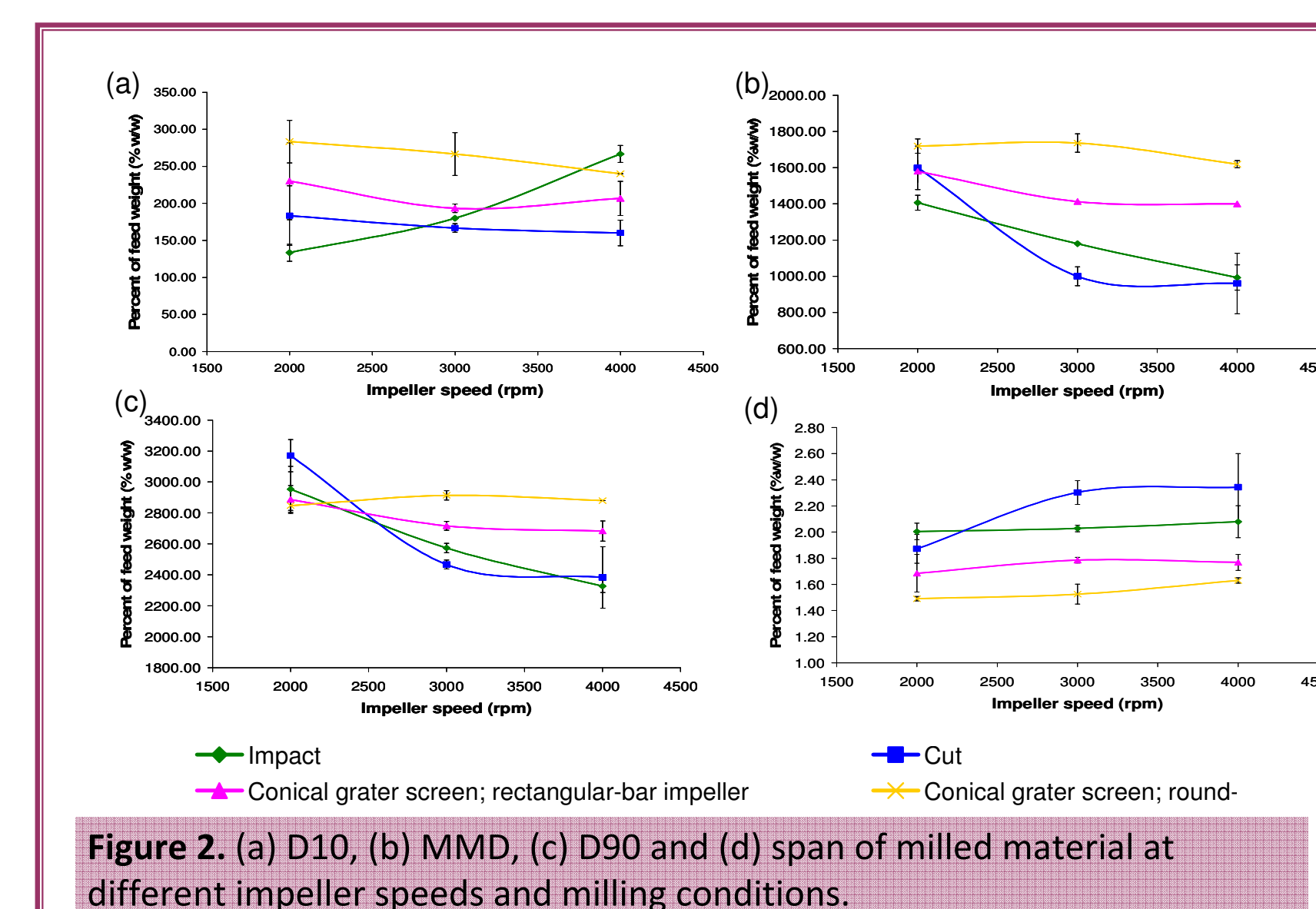


Figure 2. (a) D10, (b) MMD, (c) D90 and (d) span of milled material at different impeller speeds and milling conditions.

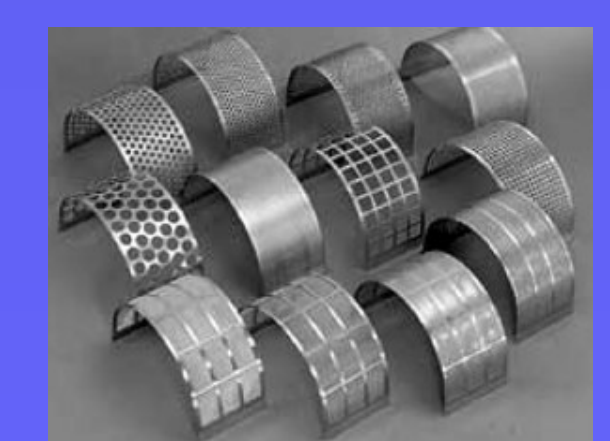
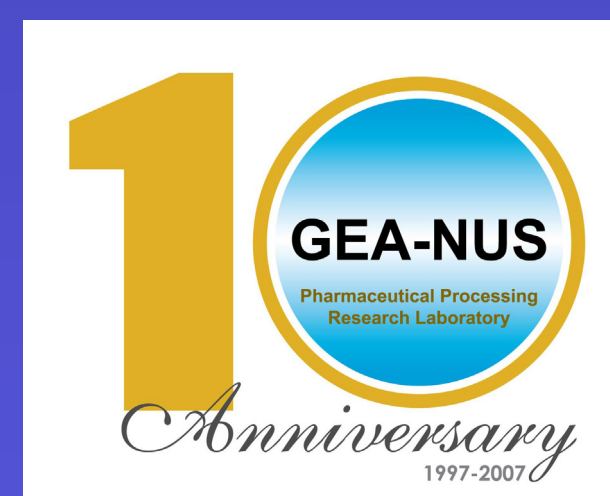
- The **round bar impeller with teeth** resulted in slightly lower fines generation and tighter particle size distribution than the rectangle bar impeller (Fig. 2(d)). Milled material by the former was coarser as indicated by the larger amounts of oversized particles and high MMD and D_{90} values (Fig. 2(b), (c)). The round bar impeller had less surface area in contact with the material as compared to the rectangular bar impeller, resulting in a lower degree of particle size reduction ($p < 0.05$).
- Increasing the rotor speed also led to less oversized materials as more energy was needed for size reduction. For the range of speeds studied, conical screen milled materials showed insignificant response in terms of the resultant particle size, implying that the process might benefit more from a second pass milling or varying the screen aperture size rather than increasing the milling speed.

5 CONCLUSION

The use of conical screen mill with rectangle bar impeller and grater-bore screen combination led to more consistent particle size reduction and more material within the desired range compared to conventional milling techniques at all speeds tested.



Quadro® Comil® 197S components



Fitzpatrick Comminutor M5A components

References

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