

This is a repository copy of Comminution and amorphisation of Diaqua-bis(Omeprazolate)-Magnesium Dihydrate: An analysis of the energies involved.

White Rose Research Online URL for this paper: <a href="https://eprints.whiterose.ac.uk/187551/">https://eprints.whiterose.ac.uk/187551/</a>

Version: Accepted Version

## Article:

Abouhakim, H, Alizadeh Behjani, M, Quayle, MJ et al. (4 more authors) (2022) Comminution and amorphisation of Diaqua-bis(Omeprazolate)-Magnesium Dihydrate: An analysis of the energies involved. Powder Technology, 405. 117415. ISSN 0032-5910

https://doi.org/10.1016/j.powtec.2022.117415

© 2022, Elsevier. This manuscript version is made available under the CC-BY-NC-ND 4.0 license http://creativecommons.org/licenses/by-nc-nd/4.0/.

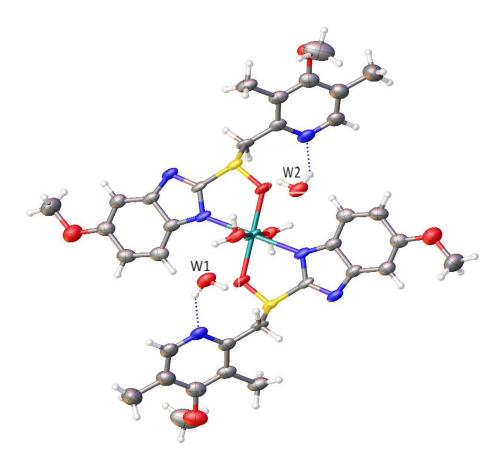
## Reuse

This article is distributed under the terms of the Creative Commons Attribution-NonCommercial-NoDerivs (CC BY-NC-ND) licence. This licence only allows you to download this work and share it with others as long as you credit the authors, but you can't change the article in any way or use it commercially. More information and the full terms of the licence here: https://creativecommons.org/licenses/

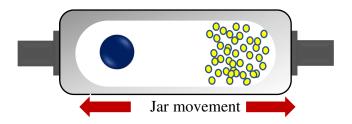
## Takedown

If you consider content in White Rose Research Online to be in breach of UK law, please notify us by emailing eprints@whiterose.ac.uk including the URL of the record and the reason for the withdrawal request.

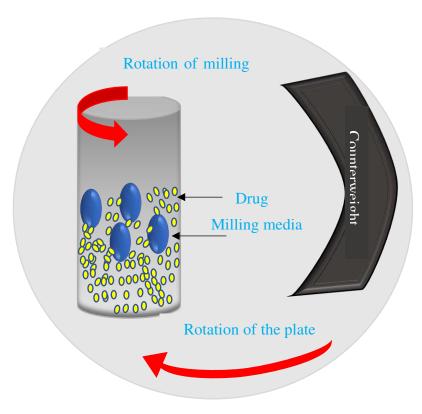




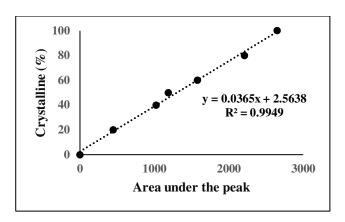
**Figure 1:** Molecular structure of diaqua-bis (Omeprazolate)-magnesium dihydrate (DABOMD). intermolecular hydrogen bonding between the molecules in the lattice shown in blue dotted lines Mg: green, S: yellow, N: blue, O: red, C: grey, H: white. Reproduced with the permission of the International Union of Crystallography (IUCr) [18]



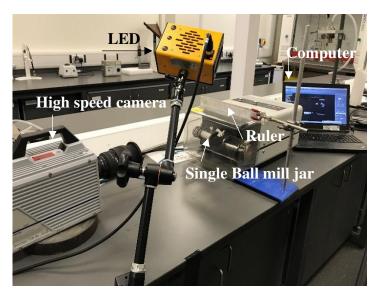
**Figure 2:** Schematic shows ball and powder motion in the single ball mill (modified from [9])



**Figure 3:** Schematic of a planetary ball mill ( (Reproduced with the permission of American Chemical Society (ACS) [1]



**Figure 4:** Calibrated Curve of the area under the curve of different ratios of standard crystalline and amorphous DABOMD measured by XRPD



**Figure 5:** Experimental setup for measuring the speed of the milling jar in the single ball mill using high-speed Camera

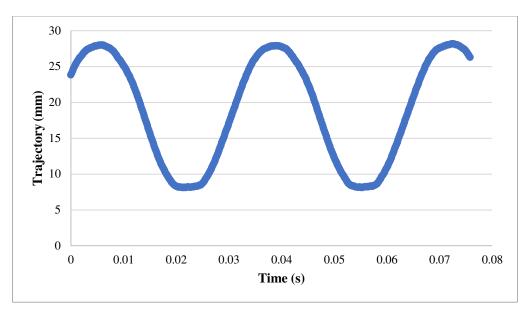
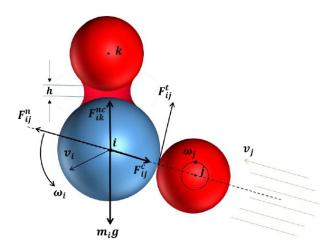
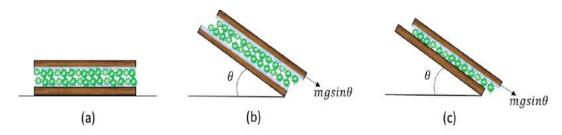


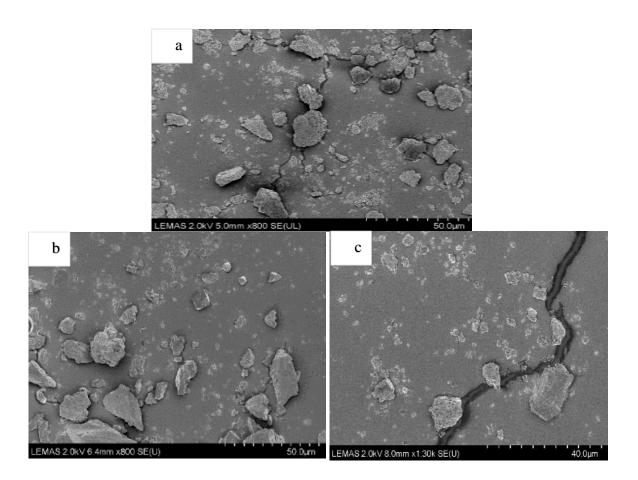
Figure 6: Trajectory of the vibratory mill jar



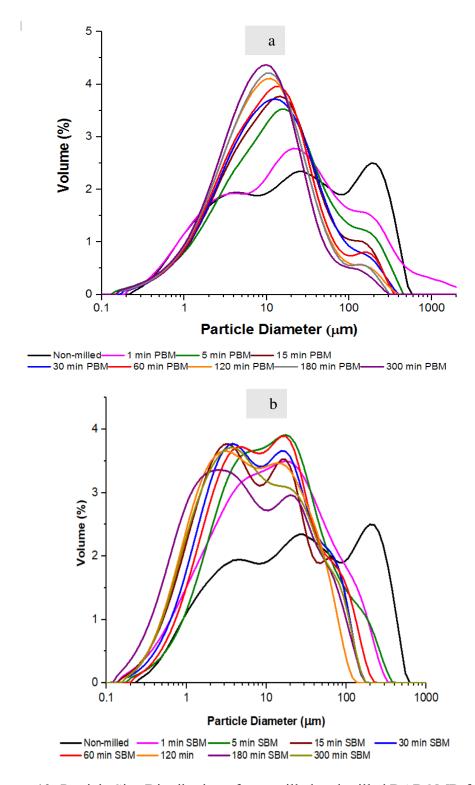
**Figure 7**: Schematic of forces applied to three arbitrarily interacting particles (reprinted from [14])



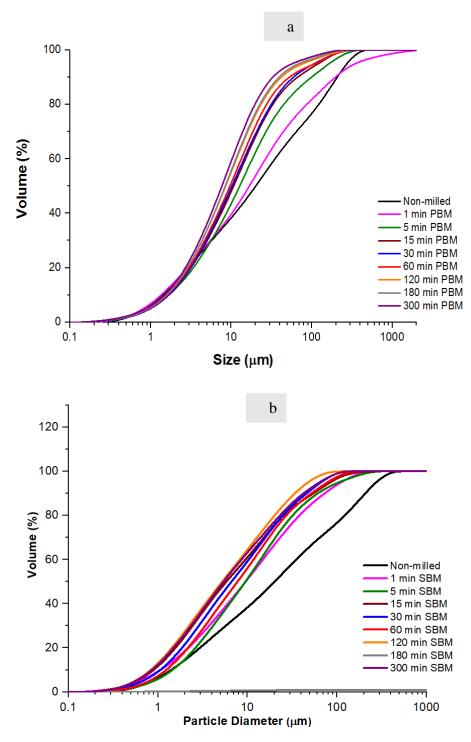
**Figure 8:** Schematic diagram representing the coefficient of sliding friction, with (a) is the surface at rest, (b) particle-particle sliding friction and (c) particle-wall sliding friction (reprinted from [14])



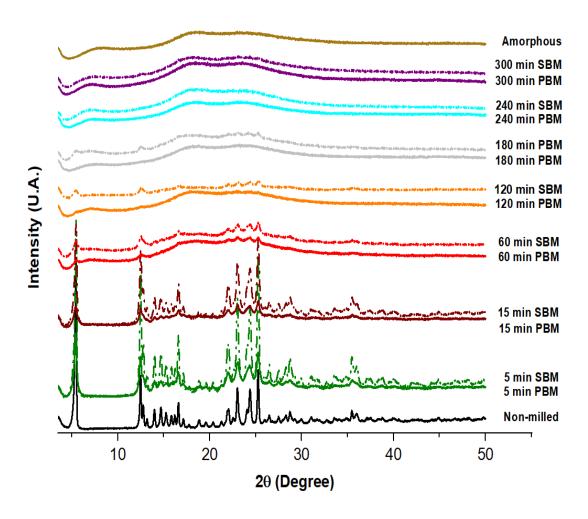
**Figure 9**: SEM images show the Morphology of DABOMD, a-Non-milled (×800), b- 300 min planetary ball milled (×800), c-300 min single ball milled (×800).



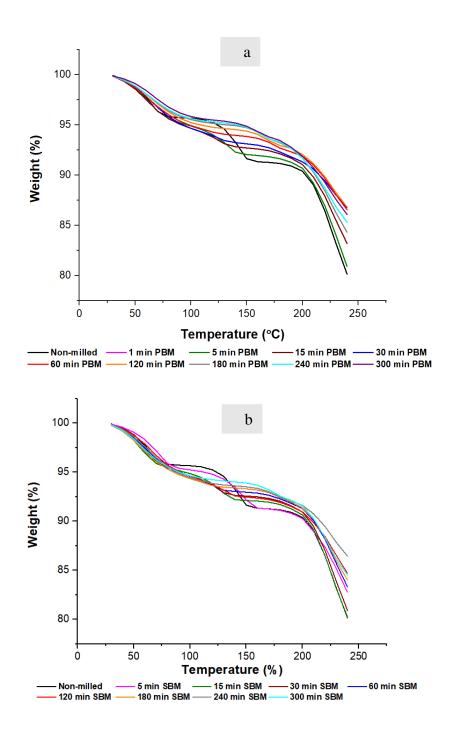
**Figure 10:** Particle Size Distribution of non-milled and milled DABOMD from 1min to 300 min. a) Planetary ball milled (PBM) ( (Reproduced with the permission of American Chemical Society (ACS) [1] . b) Single ball milled (SBM)



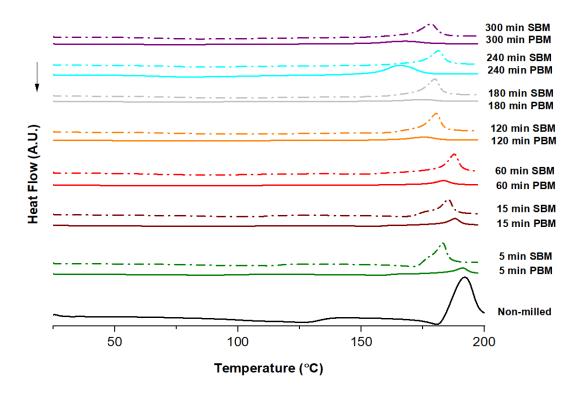
**Figure 11**: Cumulative Particle Size Distribution of non-milled milled DABOMD from 1min to 300 min. Planetary ball milled (PBM) ( (Reproduced with the permission of American Chemical Society (ACS) [1]. b) Single ball milled (SBM)



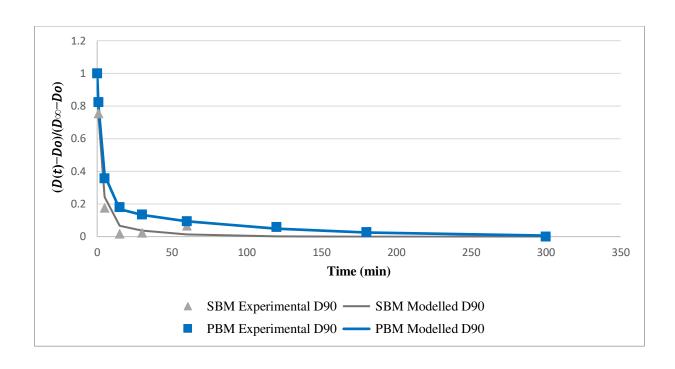
**Figure 12:** XRPD of DABOMD before milling and after milling from 1 min to 300min in Planetary ball milled (PBM) and in Single ball milled (SBM)



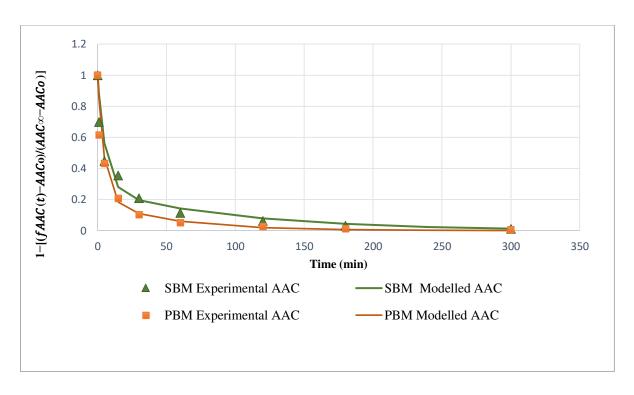
**Figure 13:** TGA Profile of the non-milled and milled DABOMD from 25 to 250 °C and a rate 5°C/min. a) Planetary ball milled (PBM) ( (Reproduced with the permission of American Chemical Society (ACS) [1] b) Single ball milled (SBM)



**Figure 14:** DSC Profile of DABOMD for the non-milled and milled DABOMD from 25 to 250 °C and a rate **5**°C/*min* of Planetary ball milled (PBM) and Single ball milled (SBM)



**Figure 15:** The kinetic of comminution of DABOMD represented by  $D_{90}$  for the Planetary ball milled (PBM) and Single ball milled (SBM) DABOMD



**Figure 16:** The kinetic of amorphisation of DABOMD derived from XRPD analysis of Planetary ball milled (PBM) and Single ball milled (SBM) DABOMD

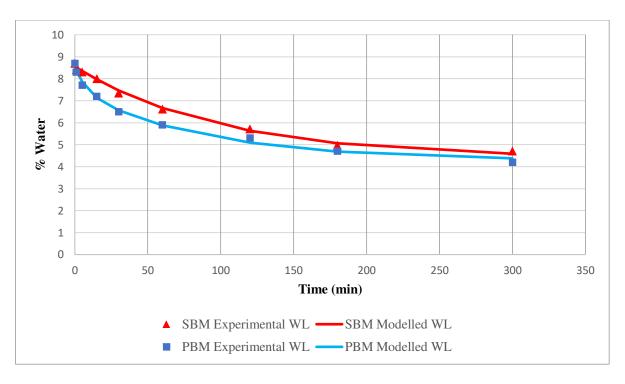
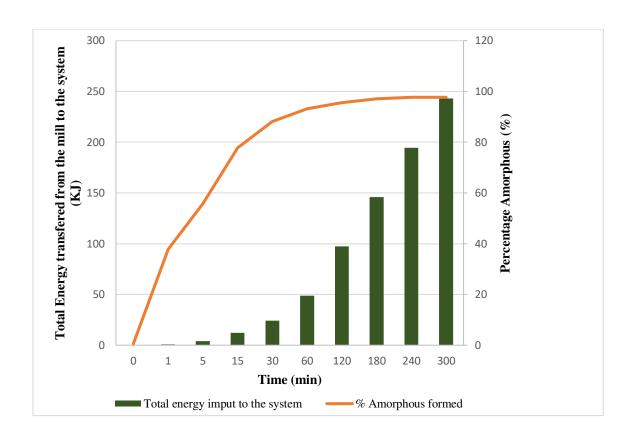
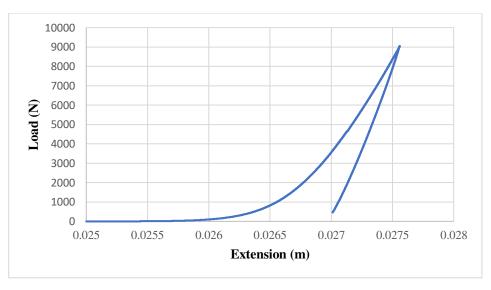


Figure 17: The kinetic water loss DABOMD in planetary ball mill and single ball mill



**Figure 18:** Total energy transferred to the system in a planetary ball mill and the amount of generated amorphous



**Figure 19:** Deformation profile illustrating load versus extension graph for compression test

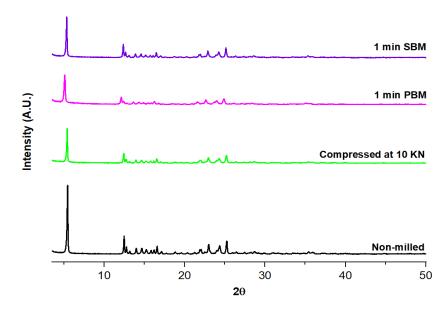


Figure 20: XRPD profile of DABOMD after compression

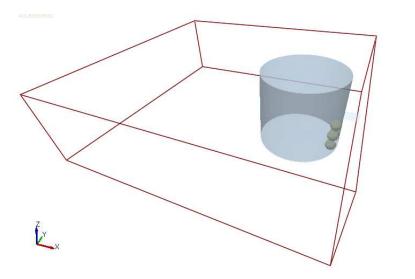




Figure 21: DEM Simulation of Planetary Ball Mill

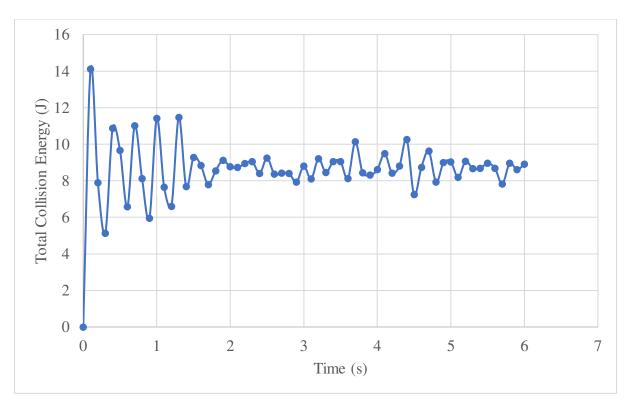


Figure 22: Total collision energy in planetary ball mill

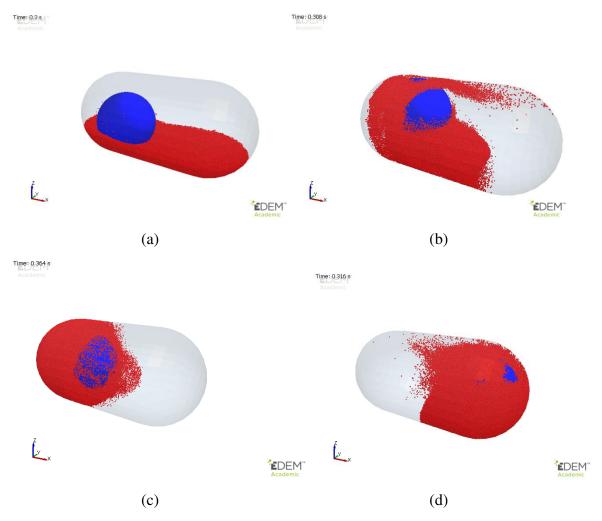


Figure 23: DEM simulation of single ball mill at different times, (a) before vibration, (b) 0.008s, (c) 0.064 and (d) 0.16s

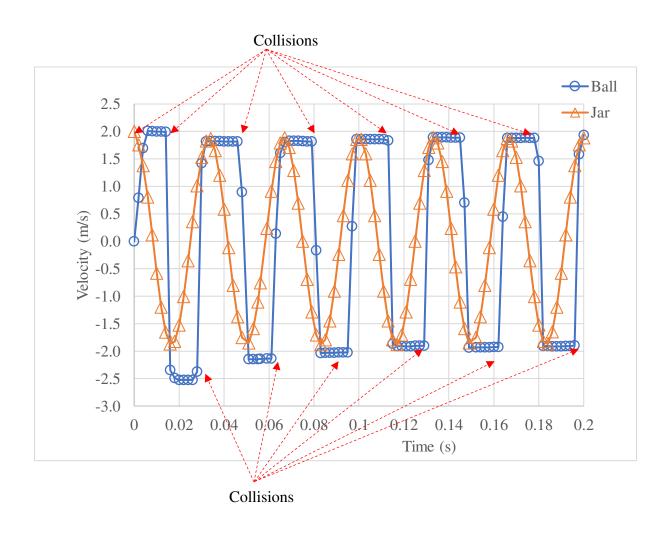


Figure 24: Velocities of jar and ball in SBM from DEM simulation

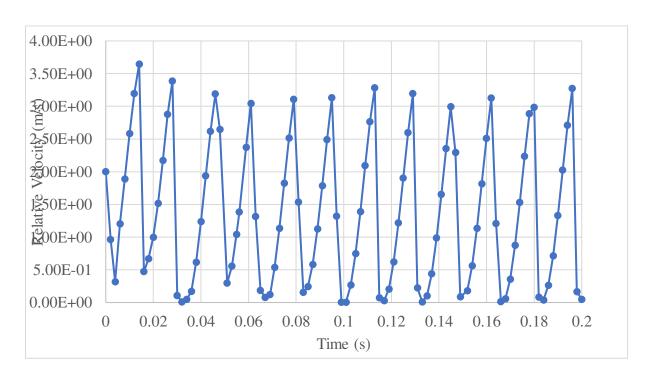


Figure 255: Relative velocities of ball and jar

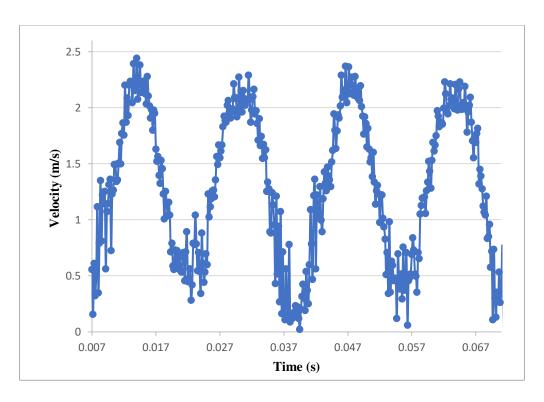
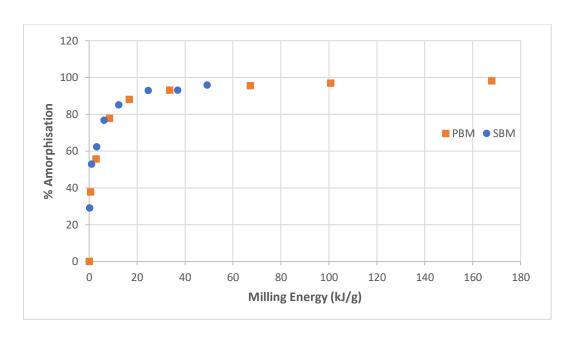


Figure 26: Velocity profile of the jar from high speed camera recording in SBM



**Figure 27:** Correlation between amorphisation of DABOMD and energy level in different mills