BAHAGIAN PENYELIDIKAN & PEMBANGUNAN
CANSELORI
UNIVERSITI SAINS MALAYSIA

Laporan Akhir Projek Penyelidikan Jangka Pendek

1. **Nama Penyelidik**	:	Prof. Madya Dr. Khairun Azizi Mohd. Azizli
Nama Penyelidik Lain	:	En. Samayamutthirian Palaniandy
2. **Pusat Pengajian**	:	Pusat Pengajian Kejuruteraan Bahan & Sumber Mineral
3. **Tajuk Projek**	:	**Fine Silica Particles as a Feedstock for Various Advance Materials Application**

4. **(a) Penemuan Projek / Abstrak**
(Perlu disediakan makluman di antara 100-200 perkataan di dalam Bahasa Melayu dan Bahasa Inggeris ini kemudiannya akan dimuatkan ke dalam Laporan Tahunan Bahagian Penyelidikan & Pembangunan sebagai satu cara untuk menyampaikan dapatan projek tuan/puan kepada pihak universiti)

ABSTRAK

Pengisar planet adalah pengisar intensif tenaga yang digunakan dalam pengisaran halus. Kesan perubahan parameter pengoperasian ke atas saiz partikel produk, penggunaan kuasa dan penjanaan tenaga haba di dalam pot pengisaran telah dikaji. Analisis statistik telah digunakan untuk mengesahkan parameter pengoperasian yang paling signifikan bagi proses pengisaran halus. Perubahan dalam struktur partikel semasa pengisaran halus telah dikaji dengan menggunakan kaedah belauan sinar-X. Model pengisar bebola campuran sempurna dan pengisar bebola sapuan udara telah dipadankan dengan data pensaizan pengisar planet dan parameter operasinya menggunakan fungsi-fungsi pemecahan Piawai JKSimMet, Broadbent dan Callcott serta Broadbent dan Callcott yang telah diubahsuai.

Keadaan pengisaran optimum dicapai dengan partikel 58.8µm yang disuap pada kadar 0.0375kg/jam dengan 20% media pengisaran. Pemecahan maksimum partikel diperolehi pada 125 putaran seminit dengan menggunakan media pengisaran keluli pelbagai saiz. Penggunaan kuasa adalah diantara 0.9Kw hingga 1.1Kw dan dipengaruhi oleh semua parameter pengoperasian kecuali saiz media pengisaran. Penjanaan tenaga haba diantara 3.4 Kj sehingga 7.1 Kj pula dipengaruhi oleh semua parameter pengoperasian. Perubahan struktur semasa proses pengisaran lebih signifikan apabila masa pengisaran bertambah dan saiz partikel bertambah kecil. Fasa hablur didapati bertukar menjadi amorfus semasa proses pengisaran. Keamatan maksimum hablur adalah 45.3% selepas 2.5 jam pengisaran dan ianya hilang selepas 192 jam pengisaran. Proses pengisaran halus di dalam pengisar planet boleh diwakili oleh model pengisar bebola sempurna dengan fungsi pemecahan Broadbent dan Callcott yang diubahsuai. Kadar pemecahan partikel yang diperolehi adalah 2.05 untuk knot 1, 3.09 untuk knot 2 dan 3.55 untuk knot 3.

ABSTRACT

The planetary mill is an energy intensive grinding mill utilized in fine grinding. The effect of operational parameters on the product particle size, energy consumption and the heat generation in the grinding pot was studied. The significant effect of the operational parameters on the fine grinding process was verified via statistical analysis. The structural changes of silica particles during fine grinding were studied using x-ray diffraction method. The perfect mixing ball mill and the air swept ball mill models were fitted with the planetary mill's sizing data and operational parameters using the standard JKSimMet , Broadbent and Callcott and modified version of Broadbent and Callcoat breakage functions in the model fitting process.

The optimum condition was achieved with 58.8μm particles fed at 0.0375 kg/h with 20% media charge. Maximum particle breakage was obtained at 125 rpm with a mixture of various sizes of steel balls. It was also noted, that the power consumption which ranged from 0.9Kw to 1.1Kw was influenced by all the operational parameters except for grinding media size. Furthermore, the heat generation in the grinding pot, ranging from 3.4Kj to 7.1Kj was affected by all the operational parameters. The structural changes during the fine grinding process were more significant with increased grinding time and smaller particle size. The crystalline phase of silica particle was found to transform to amorphous phase during fine grinding process. The maximum intensity of crystalline silica was 45.3% after 2.5 hours and it disappeared after 192 hours grinding. The fine grinding process in planetary mill could be described by the perfect mixing ball mill model and the modified version of Broadbent and Callcott breakage function. The particle breakage rates obtained for knot 1 was 2.05, followed by 3.09 for knot 2 and 3.55 for knot 3.

(b) Senarai Kata Kunci Yang Digunakan Di Dalam Abstrak

Bahasa Inggeris	**Bahasa Melayu**
fine grinding	Pengisaran halus
silica	silika
model fitting	pemadanan model
breakage	pemecahan
crystalline phase	fasa hablur

5. Laporan Projek

EXPERIMENTAL

The study was conducted to investigate the effect of operational parameters of a planetary mill on the product particle size and structural changes, energy consumption and the heat generation in the grinding pot during fine grinding process.

Material
High purity silica (97%-99%SiO_2) samples from local mineral industry were used as feed materials:

Sample	Type	Size range, mm
1	Tailing sand	-2.350 mm + 0.212 mm
2	Natural beach sand	-0.850 mm + 0.053 mm
3	Natural beach sand	-0.150 mm + 0.038 mm
4	Natural beach sand	-0.060 mm + 0.038 mm
5	Natural beach sand	-0.060 mm + 0.038 mm

CHARACTERIZATION STUDIES

Feed Sample

Particle Size Analysis

Full size analysis of feed samples was conducted following the $\sqrt{2}$ series. The cumulative percentage passing versus size graph was plotted to determine the size distribution.

Products

Particle Size Analysis
The particle size distribution of ground the products was measured by scattering of laser beam; in this case the Malvern particle size analyzer with a size measurement range of 0.1μm to 1000μm was used. Whilst, the structural changes of silica particles during fine grinding was studied using x-ray diffraction method.

RESULTS AND DISCUSSION

Particle size analysis of the feed material

Figure 1 shows the particle size distribution of the feed samples.

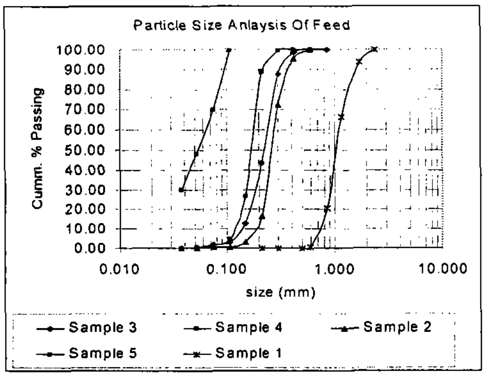

Figure 1: Particle size analysis of feed samples

From Fig. 1, the median particle size (d_{50}) of each feed sample was obtained. Table 1 shows the value of median particle size of each feed sample.

Sample	d_{50},mm
1	1.085
2	0.2335
3	0.2730
4	0.1675
5	0.0558

Table 1: Median particle size (d_{50}) of feed samples

The results showed that the most significant operating parameters affecting fine grinding in a planetary mill were feed rate, grinding media charge, mill speed, median particle of feed, grinding media size and the type of grinding media.

Feed rate

Feed rate was one of the important factors affecting the fineness of the ground products. Figure 2 shows the effect on d_{50} of ground silica when the feed rate of the planetary mill changed for different content of grinding media.

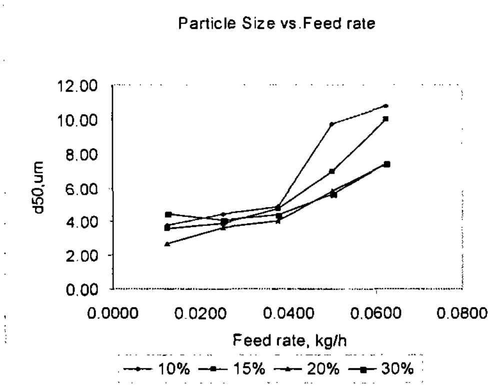

Figure 2: Median particle size at various feed rates

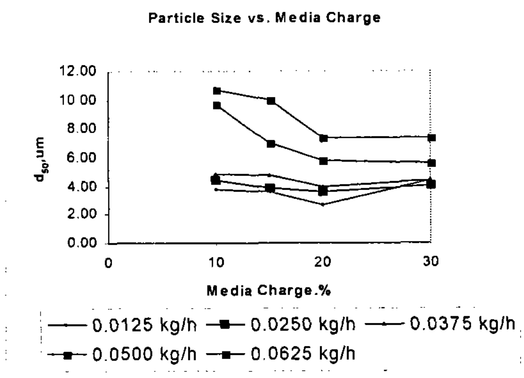

Figure 3: Median particle size at different content of grinding media

From Fig.2, it could be seen that as the feed rate increased, the d_{50} also increased. A drastic change in d_{50} value could be observed when the feed rate was more than 0.0375kg/h. These phenomena could be due to a decreased in the breakage rate when the feed rate increased. Considering a fixed grinding media content, the rate of breakage was clearly zero when there was no feed material. As the feed material increased, some of the balls gave ball to powder impact and the rate of breakage increased proportionally to the amount of the powder. However, as more powder was added, eventually the breakage reached a flat maximum. Further feed added to the planetary mill was not acted upon, and the cushioning action of excess powder eventually reduced the rates of breakage. This phenomena increased the particle size of the ground products[1,3].

Grinding media charge

Figure 3 shows the effect of d_{50} of ground silica when the grinding media charge varies. The result showed that, as the percentage of grinding media increased from 10% to 20%, the value of d_{50} decreased but for 30% grinding media charge, the d_{50} increased. The effect of d_{50} shown in Fig. 3 could be interpreted as resulting from an increased in the breakage rate. Increasing the grinding media charge would increase the maximum breakage by providing proportionally more ball-powder-ball impact region. However, overfilling the mill tends to reduce the tumbling action and the breakage rate dropped proportionally. The maximum breakage was obtained at a filling of about 20% grinding media.

Mill Speed

Mill speed was another factor that influenced the product fineness. Fig. 4 shows the effect on d_{50} when the mill speed varies for different feed size. From Fig. 4 it could be seen that as the mill speed increased the d_{50} decreased rapidly, especially when the mill speed increased from 60rpm to 80rpm. When the mill speed was more than 100rpm, the fineness of the ground product was in the submicron range. These phenomena could be due to the rate of impulses. As the mill speed increased, the rate of impulses also increased. The acceleration determined the rate of stress. In planetary mill, particles of the charge are stressed in the material bed. As a result of repetitive stress and high number of loading cycle, the number of material bed increased and this was due to the size reduction of particles. As the mill speed increased the number of impulses and acceleration also increased, so the size reduction per unit time also increased due to more repetitive stress.

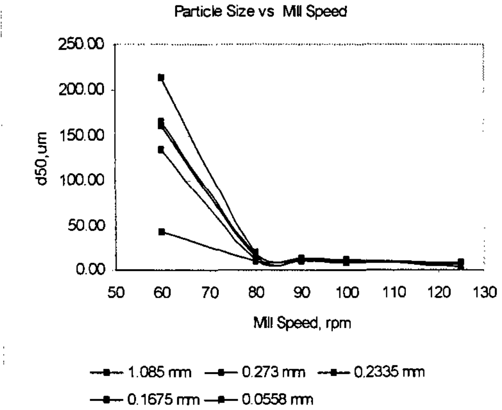

Figure 4: Median particle size at different mill speed

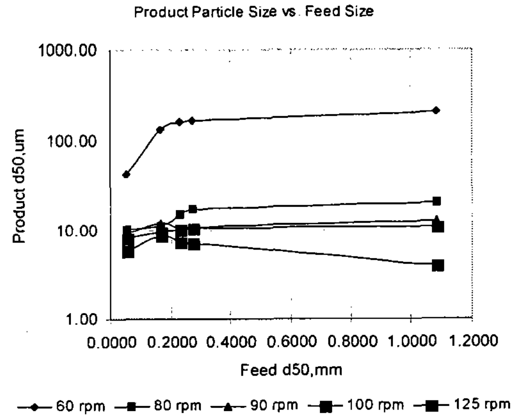

Figure 5: Median particle size at different feed size

Feed size

Figure 5 shows the effect on the product fineness when the median size of the feed changes. From Fig.5, it could be seen that as the median particle size of the feed increased, the d_{50} value also increased. This phenomenon could be related to the rate of breakage of the particles. The rate of breakage were low for larger feed particles because

- The particles were so big that the force required to break them is only achieved by relatively more rotations.
- The particles were too big to be nipped by a glancing blow.

Ball Diameter

Figure 6 shows the effect on the product fineness when the diameter of the grinding media varies. From Fig. 6, it was observed that the d_{50} value increased with an increase in the diameter of the grinding media. A mixture of ball sizes gave the fineness ground product. The maximum particle size which could be broken efficiently by a given ball size increased for larger ball diameter. The required force of a properly nipped impact was more important than the total number of impact. Smaller balls broke smaller particles more efficiently because there were many more ball-ball collisions per unit mill volume per unit time for small balls and the impact force was big enough to break small particles[4]. A mixture of balls should be more efficient than small ball size because the larger balls would break larger particles efficiently and vice-versa. So the product d_{50} size increased with the grinding media sizes.

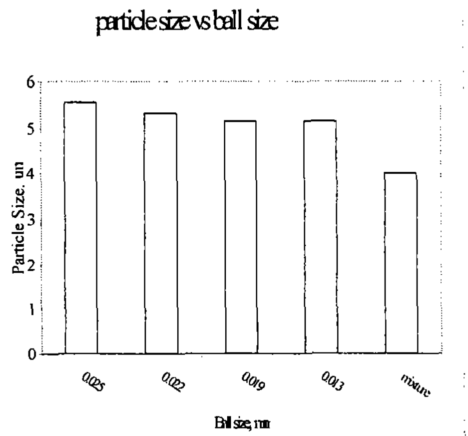

Figure 6: Median particle size at different ball size

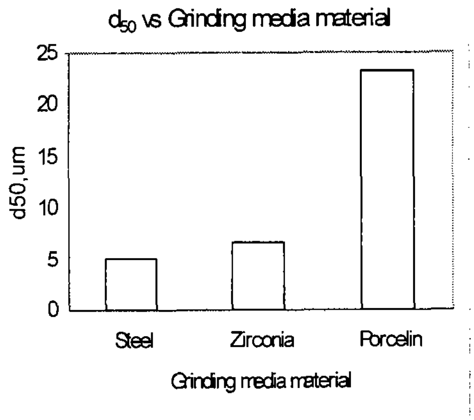

Figure 7: Median particle size of different ball type.

Grinding media material

Figure 7 shows the effect on the product fineness when using different types grinding media. In this case, the grinding media chosen was steel, zirconia and porcelain. Fig. 7 showed that the steel ball gave the highest rate of breakage compared to other grinding media materials because the density of the steel grinding media was higher compared to other materials. Higher density grinding media would give higher breakage because the stress would be high so the products would be finer when using the steel balls.

CONCLUSION

- The optimum condition was achieved with 58.8μm particles fed at 0.0375 kg/h with 20% media charge. Maximum particle breakage was obtained at 125 rpm with a mixture of various sizes of steel balls.
- The power consumption which ranged from 0.9Kw to 1.1Kw was influenced by all the operational parameters except for grinding media size. Furthermore, the heat generation in the grinding pot, ranging from 3.4Kj to 7.1Kj was affected by all the operational parameters.
- The structural changes during the fine grinding process were more significant with increased grinding time and smaller particle size. The crystalline phase of silica particle was found to transform to amorphous phase during fine grinding process. The maximum intensity of crystalline silica was 45.3% after 2.5 hours and it disappeared after 192 hours grinding.
- The fine grinding process in planetary mill could be described by the perfect mixing ball mill model and the modified version of Broadbent and Callcott breakage function. The particle breakage rates obtained for knot 1 was 2.05, followed by 3.09 for knot 2 and 3.55 for knot 3.

6. Output dan Faedah Projek

(a) Penerbitan

1. Samayamutthirian Palaniandy & Khairun Azizi Mohd Azizli (2002) "Structural Defects in Silica during High Intensity Grinding Process", Malaysian Journal of Science. Vol. 21A, pp.147-150

2. Samayamutthiarian Palaniandy & Khairun Azizi Mohd. Azizli. (2003) "Fine Grinding- A Complex Process". Proceedings of the 3rd International Conference on Recent Advances in Materials, Minerals and Environment, RAMM 2003. pp. 21-25. Penang . Malaysia

3. Samayamutthirian Palaniandy & Khairun Azizi Mohd Azizli (2003) "Model Fitting of Fine Grinding Process in Planetary Mill". *Proceedings of 2ndAsian Particulate Technology Conference.* Penang, Malaysia

4. Samayamutthirian Palaniandy & Khairun Azizi Mohd Azizli. (2003) "Morphology of Particles - Jet Mill vs. Oscillating Mill". *Proceedings of 12th Scientific Conference Electron Microscopy Society Of Malaysia.* Langkawi.

5. Samayamutthirian Palaniandy & Khairun Azizi Mohd Azizli (2002) "Morphology of Ultra Fine Silica Particles by Impact Grinding". *Proceedings of 11th Scientific Conference Electron Microscopy Society of Malaysia.* Johor Bahru

6. Samayamutthirian Palaniandy & Khairun Azizi Mohd Azizli (2002) "Ultrafine Particles Morphology!! Do Grinding Mechanism Influences it? *Proceedings of 11th Scientific Conference Electron Microscopy Society of Malaysia.* Johor Bahru

(b) Faedah-faedah Lain Seperti Perkembangan Produk, Prospek Komersialisasi dan Pendaftaran Paten

- Malaysian manufacturing sectors are using high quality imported fine silica particles from other countries such as Japan although Malaysia has a large amount of silica deposits. Further processing and value adding of local silica utilizing fine grinding process will upgrade the local mineral industry through import substitution.

- Optimum production of manufactured fine silica particles in Malaysia according to the needs of the manufacturing industries in terms of particle size, shape as well as degree of crystalline could be achieved.

- Direct collaboration with local industry such as TOR Minerals in Perak.

(c) Latihan Gunatenaga Manusia

1. Pelajar Ph.D: Satu orang - Sedang menjalankan kajian
2. Pelajar MSc : Satu orang – Telah memperolehi MSc
3. Pelajar – Prasiswazah : Dua orang

7. Peralatan Yang Telah Dibeli

Tiada. Menggunakan peralatan yang sedia ada di Pusat Pengajian

UNTUK KEGUNAAN JABATAN PENYELIDIKAN UNIVERSITI

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