

INDICATION OF GRAIN ENLARGEMENT IN DRY GRINDING PROCESS USING A ROD MILL, OBSERVED AT INCREASED MILLING TIME

INDIKASI PEMBESARAN BUTIR PADA PROSES PENGGILINGAN KERING PENGGERUS BATANG YANG DIAMATI BERDASARKAN PERTAMBAHAN WAKTU GILING

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ABSTRACT

The aim of the study is to determine whether size distribution and grain properties of the milling product can be controlled by rod size, milling time or a combination of both. Milling experiments had been carried out using rod of 9.8, 23.8, and 46.0 cm respectively as well as milling time of 30, 60, 210, 360 and 600 minutes. Grain properties were studied by sieve analysis and binocular microscope. The entire rod sizes and 30-minutes milling time yielded grain enlargement as a result of van der Waals forces among colliding particles with the help of dampness as a binding media. The longer the milling time, the more coalesce the grains. It is also known that the smaller the particles, the lesser the particle density but the brighter the grain appearance. For all combinations, grain enlargement took place within size of -60 +100# showing the highest weight proportion in the period of 286.54 minutes.

Keywords: grain property, size distribution, rod size, milling time, grain enlargement

SARI

Tujuan studi ini adalah menentukan apakah distribusi ukuran dan sifat butir produk penggilingan dapat dikendalikan oleh ukuran batang penggiling, waktu giling ataupun kombinasi keduanya. Beberapa percobaan telah dilakukan menggunakan batang penggiling dengan panjang masing-masing 9,80; 23,80; dan 46,00 cm dan waktu giling 30, 60, 210, 360 dan 600 menit. Sifat butir diuji menggunakan ayakan dan mikroskop binokuler. Semua ukuran batang penggiling dan waktu giling menghasilkan pembesaran butir akibat adanya gaya-gaya van der Waals di antara permukaan partikel yang bertabrakan dengan kelembapan sebagai jembatan pengikatnya. Semakin lama waktu giling, semakin banyak butiran yang menggumpal namun semakin kecil ukuran partikel penyusunnya, serta semakin menurun kerapatannya, dan semakin cerah butiran aslinya. Dari semua kombinasi yang dilakukan, pembesaran butir terjadi hanya pada ukuran butir -60+100# yang proporsi berat tertingginya berubah secara berulang pada periode waktu 286,54 menit.

Kata kunci: sifat butir, distribusi ukuran, ukuran batang, waktu giling, pembesaran butir

INTRODUCTION

Naturally, minerals own physical and chemical characteristics. To extract the desired content or to optimize the utilization of the minerals commercially both characteristics need to be analyzed (Gupta and Yan, 2006). Grinding is one of the processing stages that is frequently applied to yield the mineral in certain size (King, 2001; Lamec, 2005; Gupta and Yan, 2006). When the entire parameters controlling reduction process within a mill can be fully understood, then the improvement of both the efficiency and reducing the stages of existing processing process will certainly be able to be developed. Nevertheless, physical treatment is generally involved in the entire reduction processes; and so does efficiency improvement. A certain chemical substance is frequently used. Those will depend on the ground material characteristic, involved process stages, and the required product specification (Inglethorpe, 1993; Bergstrom, 2002; Wang and Forssberg, 2013).

Since material application can be improved by engineering its compiler particles, not only nanoscience having been developed extensively but the impact of milling process on the grain properties and its application have also been studied intensively (Castellanos, 2005; Bai, 2005; Drzymala, 2007). Though the sintering and the agglomeration are two relatively different theories that disclose the grain growth phenomena (Survacy, 2008; Elsey et al., 2010; McKenna, 2010; Olevsky, 2011), they are also revealed interchangeably by a few researchers in asserting a grain growth took place in a mineral processing (Pietsch, 1991; Nichols et al., 2002; Castellanos, 2005; Bai, 2005; Lekkas et al., 2014). Rod mill is a tumbling mill characterized by the use of rods as grinding media and frequently used in primary grinding (Gupta and Yan, 2006). The rod is more demanded than the ball due to its higher reducibility. As a result, the grain size distribution can be more controlled (Sedarta et al., 2014).

Limestone is a sedimentary rock that is abundantly found in almost continent. It contains significant quantity of calcium carbonate (CaCO_3). The remaining constituents may include other carbonate minerals such as dolomite ($\text{CaMg}(\text{CO}_3)_2$) and less commonly aragonite (CaCO_3). In 2013, world lime production was 350 million tons (USGS, 2014). Producing limestone in specific characteristic can involve a few stages of processing and purification such as crushing, grinding, sizing, washing and

burning. The stages intend to meet the needs of optimizing the utilization. Since the ground limestone is demanded in certain characteristics, e.g. fine to ultrafine grain size with a certain density, abrasiveness, moisture, solubility, or chemical composition; processing to get fine particle becomes an important aspect. The aim of this study is to determine whether grain properties such as size distribution, density, shape, and humidity can be controlled through the rod size, milling time or combination of both.

METHODOLOGY

This study presents a comparison of the limestone characteristic ground within a horizontal hollow cylinder sized laboratory-scale (See Figure 1) using 3 different rod sizes and milling time of 30, 60, 210, 360 and 600 minutes, respectively. Such the rods are made of cast steel performing diameter of 2.30 cm with length of 9.80, 23.80 and 46.00 cm, respectively. The materials used for the experiment was Penen limestone from North Sumatera. It was crushed by a hammer mill equipped with a sieve of aperture sized 5 mm. A standard sieve to gain a bulk sized -10+20# sifted the crushed limestone. The bulk was split up into 16 parts to gain 15 samples at certain weight. Based on picnometer test the limestone bulk density was 1.3 g/cm^3 .

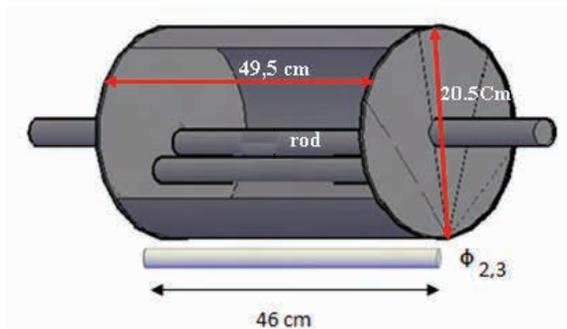


Figure 1. Mill specification

For each experiment, the mill was rotated at 57 rpm or equal to 61.01% of the critical speed. As the weight of media and feed is 5,000 g, the feed weight should be appropriated with the weight of media. The mill loading ratio comprises of media to feed weight = 3.08 - 3.90; media to feed volume = 0.41 - 0.52; and mill charge = 7.30 - 8.16%.

Based on the above ratios, the potential and kinetic energy of the grinding media is three times higher than that of the feed, while the feed volume is two and a half times more than that of the media. Consequently, due to mill rotation, the grinding media was much more capable to move than that of the feed. The rod surface is covered by the entire feed so that it will more intensify the reduction processes. By considering the critical speed, the entire load tends to exist at the bottom side. The lower the toe position, the closer the packing and locking of the media so that the cascading speed is relatively less compared to the mill speed (Lamec, 2005).

RESULTS AND DISCUSSION

Based on the fraction weight for all rod size (Table 1), the produced grain can generally be divided into 4 different groups, namely:

- (1) Undiminished particles, i.e. size of -10+20# and -20+40#;
- (2) Enlarged grains; i.e. size of -40+60# and -60+80#;
- (3) Reducible particles; i.e. grains sized -80+100# and -100+120#;
- (4) Seed sticking on to another grain surface; i.e. seed sized -120+140#, -140+200, and -200#.

Although the limestone had been ground for 600 minutes, the third rod sizes had not been capable of reducing the all particles (Figure 2.A). The amount, however, was limited and the proportion tends to be changed repeatedly. This tendency also took place at the size -20+40# (Figure 2.B),

but the amount is, except for milling time 30 minute, relatively higher than that of size -10+20#. It is assumed that few materials tend to exist on the top of the material heap. The natural particle with the biggest size tends to be sunk due to load movement while new grain with lower density will shift to the top of load. Mobius et al. (2001) stated that in the aggregate movement, the larger particles tended to be floated due to the Brazil-nut effects. Sedarta and Munthe (2013) also affirmed that reduction process took place only on the largest size grain available among grinding media.

The grain size of -40+60# and -60+80# tended to increase when the milling time was ≤ 60 minutes, but the size of -40+60# decreased until the milling time up to 210 minutes and was relatively unchanged until milling time was ≤ 360 minutes. The grain size of -40+60# was increased afterwards. It seems that the longer the milling time, the higher the percentage of grain weight.

The weight percentage of the grain sized -60+80# was relatively different for every rods size. The medium and long rods rather increase whereas the short rod decreases the weight percent when milling time is up to 60 – 210 minutes. When the milling time exceeded 360 minutes, it intended to re-increase the grain size of -60+80#. The amount of the ground particle is augmented due to increase of the milling time. The increase at the short and the medium rod are higher than that of the long rod (Figure 2.D).

It is assumable that the rods had shattered the entire original particle at least into two fragments in a relatively same size so that the fragments

Table 1. Weigh percent of the grain for each experiment

Grain size (µm)	Millig time for Rod-A (minutes)					Milling time for Rod-B (minutes)					Milling time for Rod-C (minutes)				
	30	60	210	360	600	30	60	210	360	600	30	60	210	360	600
841	0.64	0.05	0.03	0.05	0.07	0.53	0.02	0.06	0.07	0.10	0.52	0.02	0.03	0.07	0.12
420	41.08	2.22	0.02	0.14	0.51	41.34	11.88	0.12	0.12	0.39	59.25	17.94	0.04	0.16	3.75
250	20.31	18.81	0.28	7.22	26.36	19.03	27.11	5.11	1.36	29.36	13.84	31.71	0.75	5.86	51.10
178	12.33	29.17	20.71	39.53	51.42	10.61	26.32	51.28	33.65	52.58	9.25	19.48	41.31	22.04	37.75
150	10.58	31.69	49.64	37.76	20.58	10.15	20.29	31.07	43.14	16.28	4.86	11.99	43.93	34.73	6.94
125	8.29	12.29	24.63	13.28	0.71	9.62	10.09	10.49	17.48	0.78	4.37	9.60	11.53	25.42	0.25
103	3.50	3.04	3.44	1.06	0.05	3.65	1.61	1.05	3.46	0.12	2.88	3.94	1.11	9.73	0.07
73	0.73	0.66	0.05	0.06	0.04	0.92	0.32	0.09	0.15	0.02	0.95	1.22	0.07	1.09	0.02
64	2.53	2.06	1.22	0.90	0.27	4.14	2.36	0.74	0.57	0.37	4.06	4.08	1.21	0.91	0.01

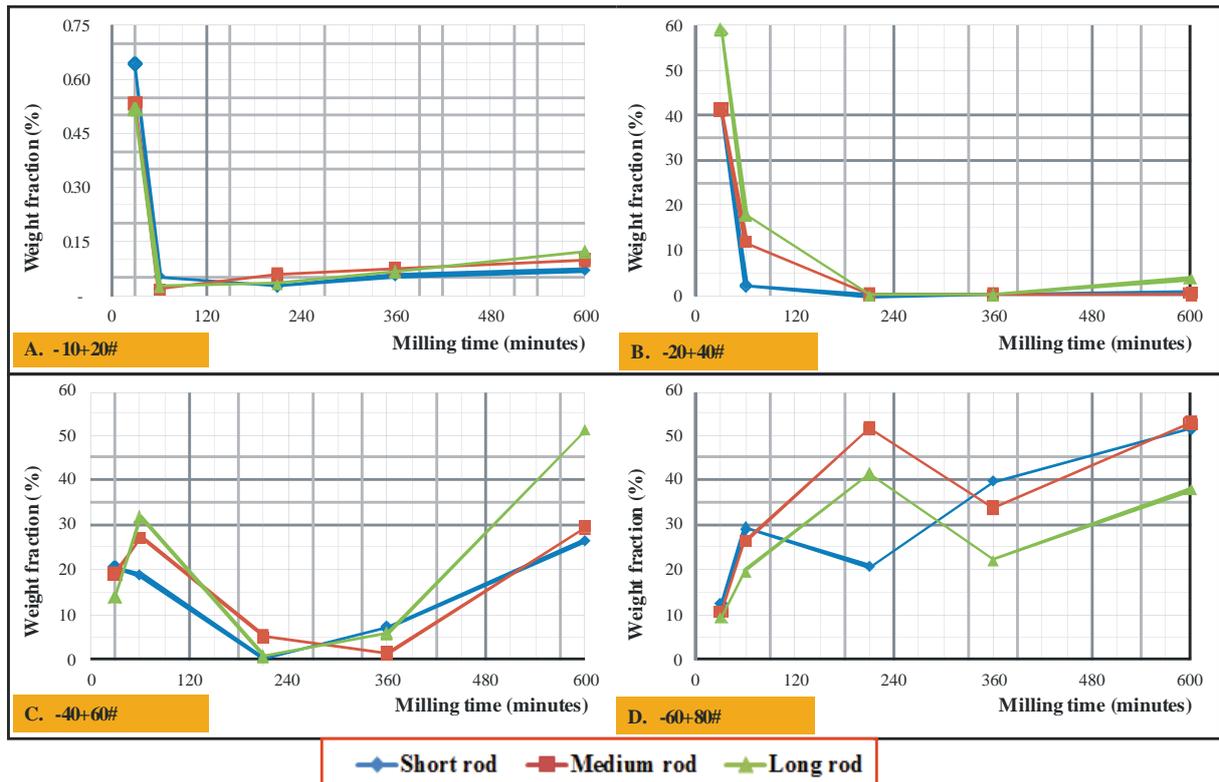


Figure 2. Weight fraction of the grains sized bigger than 80#

hardly increase the percentage size of -120+140#, -140+200# and -200#. The size -140+200# and -200# were so little in weight percentage that both of them were combinable (Table 1 and Figure 3). When milling time more than 60 minutes; referring to Figure 2A and 2B, the original particle size would have been reduced entirely. For milling time more than 120 minutes, it took place in -40+60# raw materials (Figure 2C). The entire rods decreased the material size of -40+60# and increased the material size of -60+80#. The process was also carried out to -60+80# particle with milling time of 360 minutes. When the particles size of -60+80# was entirely deducted, the rapidness of grain enlargement had been equal to the rate of grain size reduction.

The third rod sizes had relatively equal tendency in yielding grain sizes of -80#+120. The percentage was increased until milling time around 210 minutes then be decreased along with the increase of milling time (Figure 3). The change of the weight percentage for size -100+120# has similar tendency as for size -80+100#. The short rods, unfortunately, was the most increased the

size -80+100# until milling time reached 210 minutes. When 360-minute milling time was applied, the third rod size produced a grain enlargement at similar tendency. The longer the milling time the lesser the percentage of size -60+80#. Those occur due to the long milling process. As the reduction process is primarily carried out on the largest particles, so the longer the milling time, the smaller the original particle sizes.

However, the finest particle tends to adhere on the surface of larger particle. As a result, the fractional weight of the finest particle is difficult to increase. Except for the 360-minute milling time with long and medium rod size, the entire percent weight for -120+140# increases less. Similar phenomena also take place to percent weight for both -140+200# and -200# of which the tendency is higher than that of the percentage of -120+140#. It is assumed that the rate of reduction process and grain growth took place after 360-minute milling time. Furthermore, the reduction process causes grain agglomeration. Particle size diminution did not occur anymore. However, the function of rod collision is only for reducing particle bonding.

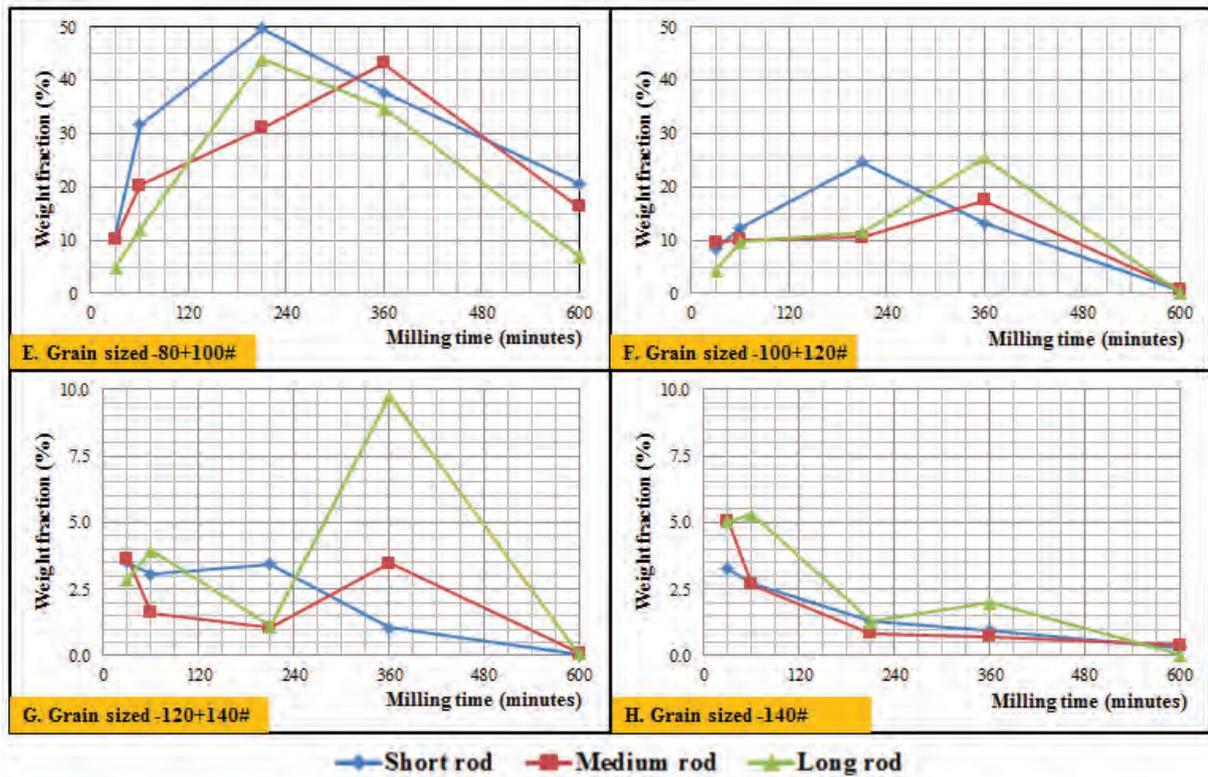


Figure 3. The weight fraction of the grain sized less than 80#

Based on the curve of cumulative grain size distribution (Figure 4) and the variation of the gradation curvature coefficients (Table 2 and 3), it can be understood that rods of all size yielded a grain by which the size distribution is relatively indistinctive.

Tabel 2. Coefficient of Curvature, Cc

Rod size	Milling time (minutes)				
	30	60	210	360	600
Short	0.89	1.02	1.04	1.01	1.03
Medium	0.87	1.02	1.01	1.04	1.03
Long	1.11	1.05	1.01	1.02	0.92

Tabel 3. Coefficient of Uniformity, Cu

Rod size	Milling time (minutes)				
	30	60	210	360	600
Short	2.30	1.45	1.30	1.33	1.26
Medium	2.43	1.53	1.33	1.33	1.25
Long	2.70	1.91	1.29	1.45	1.31

Those were disproportionately graded ($C_c \approx 1.0$ while $C_u < 4.0$). The grain size at cumulative 60% is neither two times larger than that of cumulative 30% nor four times larger than that of cumulative 10%, though the C_c was equal to 1 (Schofield and Wroth, 1968). It is more implied that the grain size at cumulative 30% is equal to the product of the grain size cumulative 10% and 60%. The grain weight percentage at cumulative 30% is higher than that of the cumulative 10% and cumulative 60%. The grain size distribution is dominated by only a certain size, namely, -40+60# and -60+80# (see Table 1 and Figure 2). When the milling time was less than 30-minutes, the long rods yielded coarser grain than that of the short and medium rods. Unfortunately; when the milling time more than 210 minutes, it tended to yield relatively finer grains than the others.

Based on Figure 4 and Table 4, the lowest reduction ratio, i.e., $RR_{80} (= 2.31)$ occurred at the long rod while the highest $RR_{80} (5.56)$ took place at the short rod. The third rod had similar reducibility tendency. RR_{80} would only be increased when the milling time ≤ 360 minutes. Furthermore, it would be decreased in accordance with the increase of milling time. Followings are the

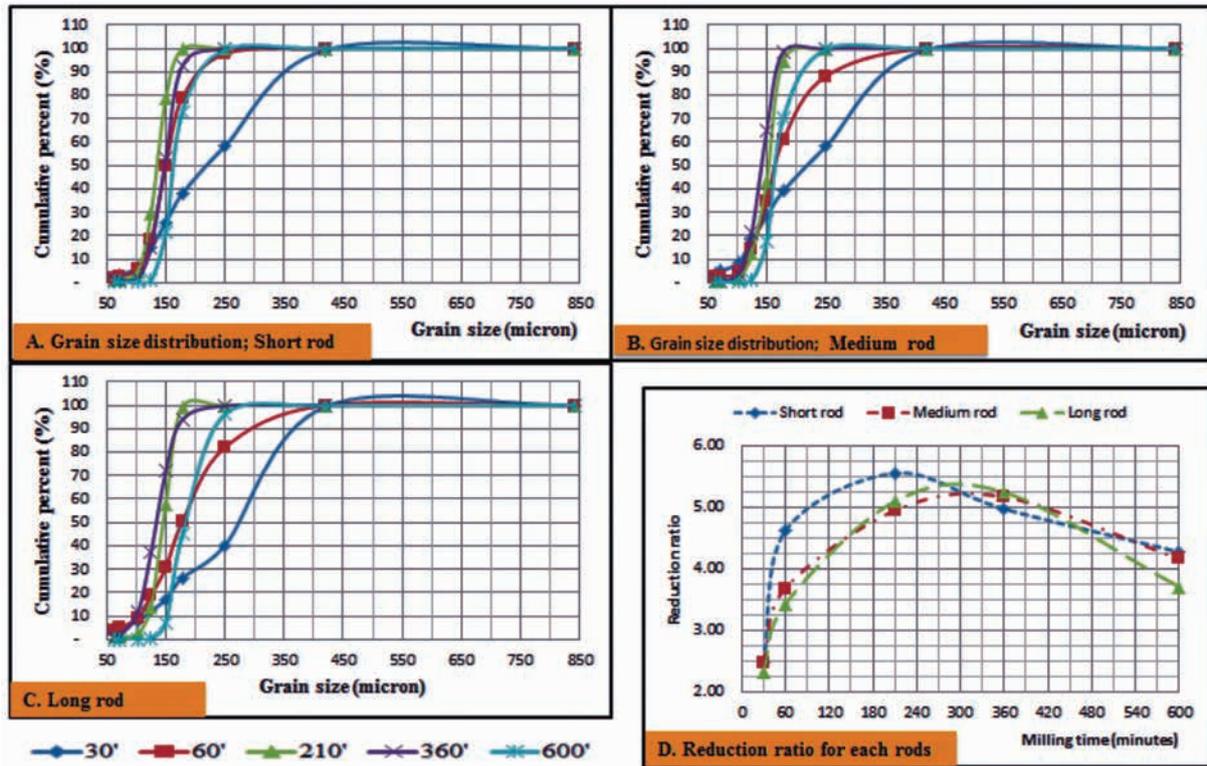


Figure 4. The cumulative grain size distribution and reduction ratio

Tabel 4. Reduction ratio, RR₈₀

Rod size	Milling time (minutes)				
	30	60	210	360	600
Short	2.47	4.62	5.56	4.97	4.27
Medium	2.47	3.68	4.95	5.17	4.16
Long	2.31	3.43	5.10	5.25	3.70

proposed empiric equation when approached by cubic spline interpolation:

$$f_1(x) = 1E-07x^3 - 0.0002x^2 + 0.0461x + 1.7071 \dots \quad (1)$$

$$f_2(x) = 5E-08x^3 - 7E-05x^2 + 0.0278x + 1.9356 \dots \quad (2)$$

$$f_3(x) = 5E-08x^3 - 8E-05x^2 + 0.0311x + 1.6068 \dots \quad (3)$$

where:

f_1, f_2, f_3 are RR₈₀ for short, medium, and long rods, respectively
 x is milling time (minutes)

$f_1, f_2,$ and f_3 have a R² = 0.8536, 0.9603, and 0.9852 respectively

By optimizing the entire function $f_1(x), f_2(x),$ and $f_3(x)$ in which is each $f''(x) =$ must be equal to zero and is achieved by $x_1 = 127.47, x_2 = 286.54$ and $x_3 = 255.65$ minutes. Those determined milling time is not too different from using a ball mill (Sedarta et al., 2014).

Microscope observation shows that the shapes of feed pulverized grain are not changed. The crushed limestone sized -10+20# and particles-forming grain size of -40+60# are relatively indistinctive for 30-minute milling time. If the milling time goes to 360 minutes, the grain is finer than that of the 30-minutes ground So does for 600-minute ground. The particles are agglomerated showing relatively smaller compared to the initial form.

To understand such a fact in more detail, wet sieving analysis was conducted. A few samples was not only washed by fresh water within a breaker glass but it was also examined chemically. The washing test was carried out by shaking the bulk for 5 minutes and poured it into a series standard

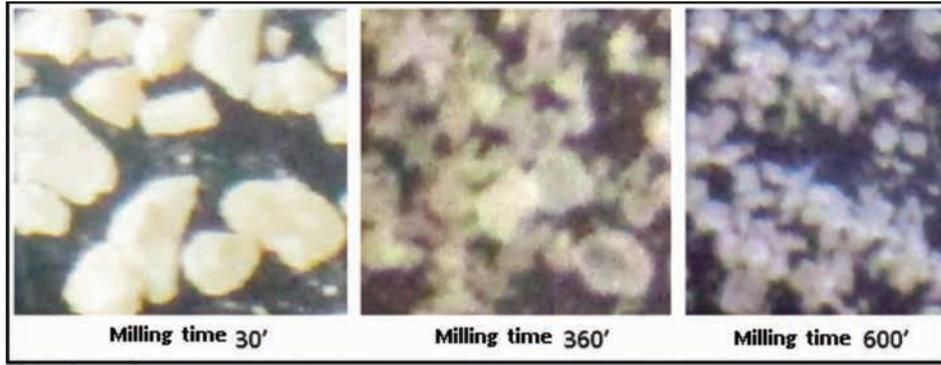


Figure 5. Grain shape appearances observed under a binocular microscope (10X magnification)

sieve to gain the retained particles -80+100#, -100+120#, and -120#. The undersize materials (-120#) were considered as wet grains. Drying the retained particles intends to gain the net weight of the particles. Based on the obtained weight (Figure 6), it is understandable that size -60+80# comprises finer particles which adhere to the coarser ones. The bonding among particles is not strong enough. It would be loose when immersed into stirred water. Based on the screening and washing observation, the longer the milling time, the stronger the particle bonding. Each rod

sizes and milling time have a different weight when shaken in less than a minute. From grain solubility tests using water, alkaline, acid and the combination of the three as media, it can also be understood that both the original particles and the agglomerated grains underwent a change of their chemical property (Figure 7). The grain solubility was increased along with milling time but decreases when milling time ≥ 360 minutes.

Pietsch (1991) stated that adhesion phenomena became more important with decreasing par-

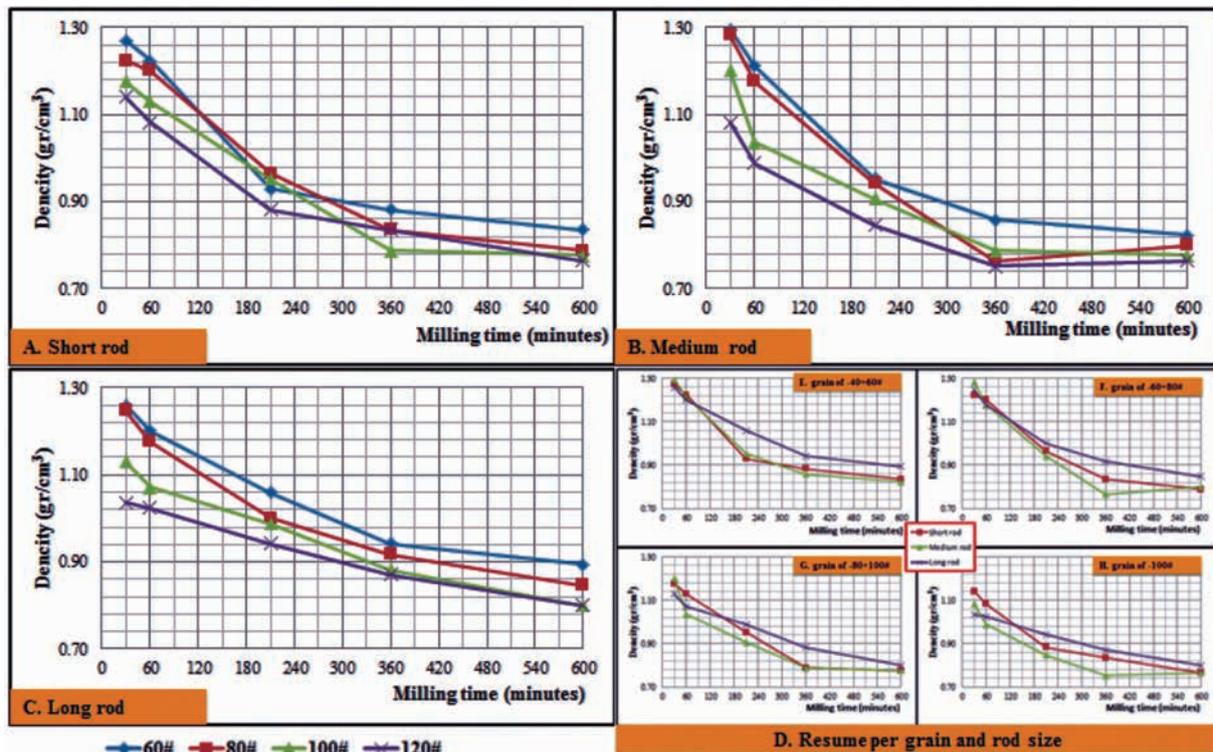


Figure 6. Grain density for each rod and grain sizes

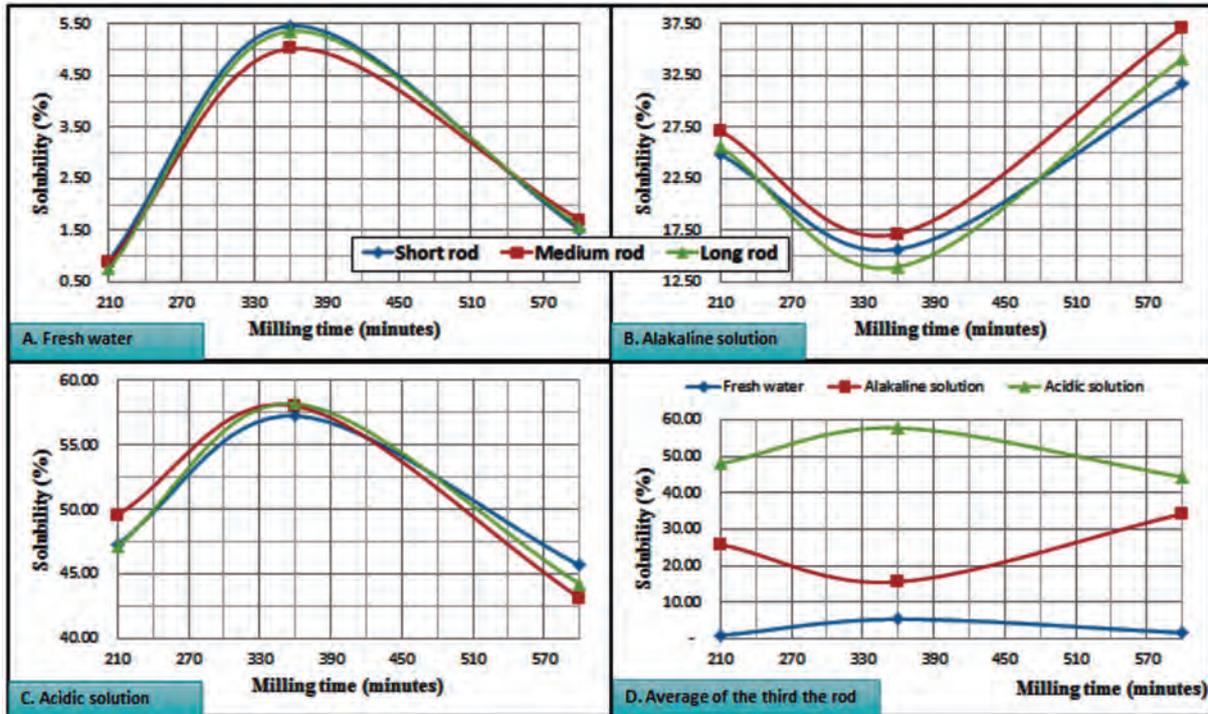


Figure 7. Grain solubility in fresh water, alkaline, and acid

tle size, it caused aggregation, agglomeration, coating, and/or caking. Critical particle size was approximately 100 μm, but it was also possible that many coarser particulate matters could be affected if sufficient fraction of fine particles was present or if specific binding mechanism became effective. The smaller the particle size, the more increase the interfacial contact among particle (McKenna, 2010). The longer the contact phase, the more strength the bonding and the longer the milling time, the larger the granule size followed by decreasing the relative density.

Based on density data for each rod and milling time (Table 5), it can be understood that the highest density is found at size of -40+60# (≈ 250 μm)

using a medium rods while the lowest one is found at size of -120+140# using shortest and medium rods and the particle density for all rod size tends to decrease along with the increase of the milling time. Based on the observation under microscope and chemical test, it is concludable that the shape and the properties of the grain tend to be changed along with the increase of milling time. The longer the milling time, the more coalesces the particles and the smaller the arranged particle size. Washing or stirring the particles size -40+80# with fresh water in a container will cause the grain lost. When the content of the container pours into a series sieve, there will be a lot of particles in variation sizes. The longer the milling time, the smaller the particles size will be.

Table 5. Grain density for each rod size and milling time

Grain size (μm)	Milling time (minutes)					Milling time (minutes)					Milling time (minutes)				
	30	60	210	360	600	30	60	210	360	600	30	60	210	360	600
	The shortest rod					The medium rod					The longest rod				
250	1.27	1.22	0.92	0.88	0.84	1.29	1.21	0.95	0.86	0.82	1.26	1.20	1.06	0.94	0.89
178	1.22	1.20	0.96	0.84	0.79	1.28	1.18	0.94	0.76	0.80	1.25	1.18	1.00	0.92	0.85
150	1.18	1.13	0.95	0.79	0.78	1.20	1.04	0.91	0.79	0.78	1.13	1.07	0.99	0.88	0.80
125	1.14	1.08	0.88	0.84	0.76	1.08	0.99	0.85	0.75	0.76	1.04	1.02	0.94	0.87	0.80

From the experiments it may be found the unusual related to the modern comminution theorem proposed by King (2001). A Mineral was diminished repeatedly that it should be shattered into a few fragments with variable of size. The longer the reduction time, the finer the particle size will be. In fact, varying 600-minute milling time with grinding media size and milling ratio do not increase the percentage of finer grain (less than 100#). The entire amount is not more than 10%. The percentage of the particle weight only increase for size -60+80# than that of -100+120#, -120+140# and -200#.

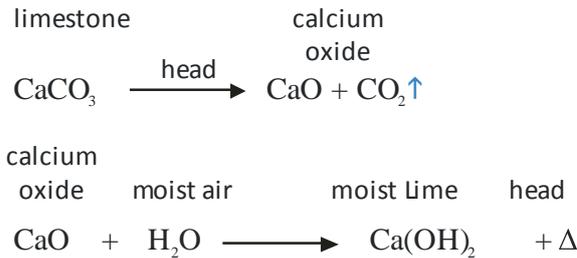
Referring to the change of particle density (Table 5), it seems that the longer the milling time the more decrease the density. It means that longer milling time will cause smaller particle size. As a result particle surface will be wider. Hence, the interfacial force becomes higher than that of the gravitational one (Snow et al., 1997; Tomas, 2007) as indicated by increasing the angle of repose (Sedarta, 2012). Based on above statements, it can be concluded that dry milling for limestone particles size of -10+20# can cause grain enlargement. The enlargement phenomena occur when the milling time has carried out more than 30 minutes yielding the size of -40+60# and -60+80.

Considering microscope observation, it shows that the -60+80# grain comprises of particles sized -60# indicating that the longer the milling time, the smaller the particle size and the finer the particles-forming grain. The main particles do not only undergo brighter color alteration and more transparent but also their solubility change repeatedly. Those indicate that -60+80# grain had been compiled by fine dense particle.

Because the enlarged grains did not yield a new grain intently compacted, the enlarged grain cannot be considered as a sintering as previously stated by Sedarta and Munthe (2013). The enlarged grains tended to lose easily when stirring within a fresh water, although it did not separate when being screened with a standard sieve. It may more appropriate as agglomeration than sintering though Lekkas (2014) revealed that sintering was also part of agglomeration.

Considering the critical mill speed is equal to 60% while the recommended one is 65-80% (Snow et al., 1997; Gupta and Yan, 2005), it means that the abrasion is more dominant than the impact (Snow et al., 1997; King, 2001; Gupta and Yan, 2006).

Based on the experiment grain size (less than 120#; $\approx 125 \mu\text{m}$) and the chart of particle interaction proposed by Tomas (2007), it can be inferred that the grain unification is mechanically caused by van der Waals force helped by moisture condition as a medium at approximately tensile strength of 0.1 – 50 N/mm². Those agglomeration particles are more strengthened by not only milling action but also increasing temperature during the increase milling time (Castellanos, 2005; McKenna, 2010). The phenomena may also be explained by referring to the equilibrium of the calcium carbonate (CaCO₃) proposed by AusStab (2010):



Colliding among rod and rod, rods and liner plate will increase the temperature within the crushing chamber. Referring to the Bond reduction energy (Gupta and Yan, 2006), the energy within mill can be calculated as follows:

$$\begin{aligned} E &= 11.20 \left(\frac{10}{\sqrt{182.16}} - \frac{10}{\sqrt{841}} \right) \\ &= 4.44 \text{ kWh/t} = 4.44 \text{ Wh/kg} \end{aligned}$$

The heat really exists and will be known when the milling has completely finished. The product within mill can only take after a few minutes off. Increasing the temperature will cause the limestone to be CaO and CO₂. CO₂ will vaporize and associate with the air. CaO will be covered by a moist air results in wetting the particle surface. The moisture then functions as a bridge among coalescing particles (Figure 8).

Considering the analyzed data and discussion above, there may be a few important proposed conclusions for developing particle engineering. Since the rod size has a different reducibility but does not influence grain distribution as well as its density, it can be developed to meet any determined production rate. The required density and other grain properties may be provided either by appropriate milling time or by re-engineering the milling chamber equipped by a heater.

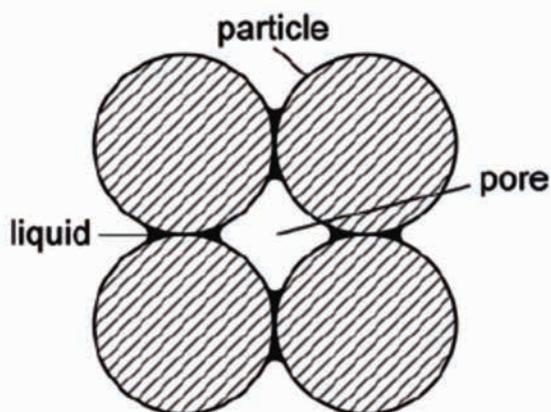


Figure 8. Agglomeration mechanism

CONCLUSIONS

Grain enlargement phenomenon takes place during milling process is an agglomeration. The enlargement is caused by van der Waals force among colliding particles which assisted by wet condition on particle surfaces as a binding bridge. The bonding particles tends to be more strength in accordance with the increased milling time. It has occurred since 30-minute milling time. The longer the milling time the more enlarged the grain. Milling time around 127.47 – 286.54 minutes are enlargement rate as well as reduction one.

Bounding particles form a new shape that is up to 4 times the initial size. Grain solubility also repeatedly changes every 360-minutes milling time. Milling time affects not only the particle size distribution but also the properties of the new grain. When milling time is more than 120 minutes, both rod size and milling time do not influence the grain size distribution.

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