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Statistical Analysis of Dry Grinding of Mica in Planetary Mill

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ABSTRACT

A huge amount of energy can be used for fine particle breakage using the planetary mill resulting in high-cost consumption. Understanding how these operating parameters could affect the dry grinding mechanism in a planetary mill is still not sufficiently discussed. The effect of different operating parameters of planetary mills in the dry grinding of mica was investigated using statistical analysis. A laboratory scale of the planetary mill was used by varying the operating parameters such as grinding time (minutes), rotational speed (rpm), and percentage of grinding media (%). A full factorial design was used involving 48 experiments, and the grinding process' efficiency was evaluated using the cut size of particles (d_{50}) obtained from the particle size distribution analysis. The analysis was supported by morphological analysis by SEM image and structural distortion by XRD test. The statistical analysis showed a good correlation with the R^2 value of 0.874 with the standard deviation of 0.852. It was found that the optimum parameters for grinding time, grinding speed, and grinding media were 20 minutes, 400 rpm, and 30% media charged, respectively, with the d_{50} value of 7.44 µm. This study provides further insight into the mica breakage operating parameters in a planetary mill.

Keywords: Dry grinding, fine grinding, mica grinding, planetary mill, statistical analysis

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INTRODUCTION

Mica is an important mineral widely utilized in applying fine particles in which the bulk of domestic output is being processed into small-size mica by a wet or dry grinding process (Andrić et al., 2013). Mica was usually used to improve strength and thermal properties (Abd, 2016). For example, Roshanaei et al. (2020) studied rubber reinforcement in addition to silica and mica particles, and the results showed that the mica-containing composites have increased in strength. Another research investigated the relationship between rheology and the qualitative appearance of dried, mica-based paint coatings used in the aerospace industry (Anderson et al., 2020). The mica usually being used as paint filler for its natural glossy appearance.

Mica fine particles are produced by fine grinding, which is the main unit process in the mineral industry. The involved processes are impact or compression caused by pressures applied almost regularly to the particle surface, chipping caused by oblique forces, abrasion caused by forces operating parallel to the surfaces, and other mechanisms (Usman, 2015). Grinding procedures such as wet grinding (product range from 95 to 45 µm) and dry grinding (product in the range of 1.2mm to 150µm) are being utilized in the industries (Pérez-Maqueda et al. 2004). However, the production of this fine grinding process requires a large amount of energy, has high overall cost consumption, and has low production rates. Furthermore, due to the following factors, it is difficult to fulfill the rigorous requirements for fineness, purity, shape, and crystallinity:

(a) Because particle strength increases dramatically as particle size decreases, particle breakage intensity must be high.
(b) Particles of brittle materials deform plastically below the tough brittle transition size, depending on the materials' types. Therefore, the breakage of these particles becomes difficult.
(c) The adhesion force in the fine particles can cause agglomeration. The stringent demand from the industries according to the specific application has made the grinding industry able to move ahead to produce custom-made minerals that can suit the specific application for the industrial product.

The industries have been finding ways to decrease the amount of energy needed in the fine grinding process by using several approaches. Generally, the efficiency of the grinding operations can be increased by developing new technologies which require a lot of cost and time or by optimizing the currently available grinding process. Planetary mill, stirred mill, jet mill, vibration mill, and peripheral mill are considered high-intensity grinding mills that could exhibit mechanochemical effects due to high power bulk density compared to ball mill (Palaniandy & Jamil, 2009). In the industry, the planetary mill is one type of popular equipment that has been used in producing a finely ground product and for the mechanical activation process (Atanov et al., 2020; Ajaka & Akinbinu, 2011; Li & Hitch, 2017; Pribytkov et al., 2019; Guzzo et al., 2014). In the planetary mill, the product (powders) depends on the low or high energy impact and the number of energy impacts received throughout the process. The huge amount of energy can be used for fine particle breakage, and the excess energy will be used for crystal structural changes besides transformation to the energy. These are the few reasons why mills are chosen for the fine grinding process. Other than that, the abrasion between the container and balls also impacts the product quality. The planetary mill has been used widely in industries and therefore has received lots of attention from researchers on its operation mechanism and performance (Atanov et al., 2020; Feng et al., 2004; Real & Gotor, 2019; El-Mofty et al., 2020; Burmeister et al., 2018; Ashrafizadeh & Ashrafizaadeh, 2012). The planetary mill has also been used to reduce the size of the mica for a variety of industrial uses, including insulators, pearlescent pigments, polymers, aeronautical devices, condensers, and plastic fillers (Barlow & Manning, 1999). In manufacturing these advanced materials, micronized mica can be utilized as fillers to enhance the product quality (Cheng et al., 1999).

A recent study investigated particle impact energy in a planetary mill by varying the size of particles and numbers (Hirosawa et al., 2021). The behavior of particles and the grinding medium were simulated using the discrete element method (DEM). The results indicate that the grinding medium, which was the grinding balls size, must be appropriately selected in response to particle size and number changes so that the particles can acquire a significant amount of impact energy during the grinding. The effect of planetary operating parameters such as grinding time, rotational speed, the ratio of media to powder, mill filling, and media size was investigated in talc grinding (El-Mofty et al., 2020). The study suggested that the ratio of media to powder and grinding time were the most significant parameters. An optimum ball size was selected as impropriate size of balls could produce a larger particle size. Simultaneously, a reduction of the mill filling and an increase in the mill speed produce finer output. The effect of the operating parameters is important to develop more advanced technologies and computer simulations. For example, in recent research, the controlled grinding process of the planetary mill was investigated and developed with the function of automatic control in a closed-loop system using a linear-quadratic controller (Atanov et al., 2020). Another study involves the study of the optimum ratio of the gyration diameter to mill tube diameter (G/D ratio) of the planetary mill (Cho et al., 2006). Despite these developing technologies, the understanding of how these operating parameters could affect the dry grinding mechanism in a planetary mill is still not sufficiently discussed.

In this study, the effect of operating parameters of planetary mills such as grinding period, rotational speed grinding, and percentage of grinding media during the mica grinding was evaluated in size particles. In addition, the structure of the mica products was discussed, and optimum conditions for the mica grinding in the planetary mill were determined. It provides further insight into understanding a planetary mill's mica breakage operating parameters.

METHODOLOGY

The sample used for this experiment was mica powder obtained from a local kaolin company in Perak, Malaysia, with an average particle size of 13.8 µm. The sampling process was done using the cone and quartering method for 10 kg of sample. The cone and quartering method reduces the bulk sample size without introducing systematic bias by forming a cone and dividing it into quarters. Two quarters will be discarded while the other two are combined and used as a sample. The feed of the sample was characterized in terms of the particle size distribution, X-ray Fluorescence (XRF), X-ray diffraction (XRD), and Field Emission Scanning Electron Microscope (FESEM) before the grinding process. This particle size analysis was performed using a particle size analyzer, Malvern model Mastersizer E Ver. 1.2, which can measure the size in the range of 0.1µm to 1000µm for 0.5 g–3.0 g of a sample. X-ray Fluorescence (XRF) was used to determine the chemical composition present in the sample tested in terms of oxide minerals. 25 g sample of raw material was sent for Rigaku X-ray Spectrometer RIX 3000. The purpose of the XRD test for this study was to determine the structural distortion due to the grinding in the tested sample. The pattern of the XRD was obtained using the XRD Model D8 Advance. The DIFFRAC.EVA software was used to interpret the pattern from XRD data to determine the intensity of each high peak.

In this study, FESEM Model ZEISS SUPRA 35VP was used. Three or four photomicrographs for every sample were selected from a different location under a certain magnification. Delaminate layer and a thin film of sample, morphology, and structural shape were observed under magnification.

The grinding process was done using a 250 ml pot in a planetary mill with a fixed feed rate which was 50 g, as shown in Figure 1. The maximum feed size for the planetary mill was 10 mm, and the sample can be grounded up to 1 µm. The grinding pot was made up

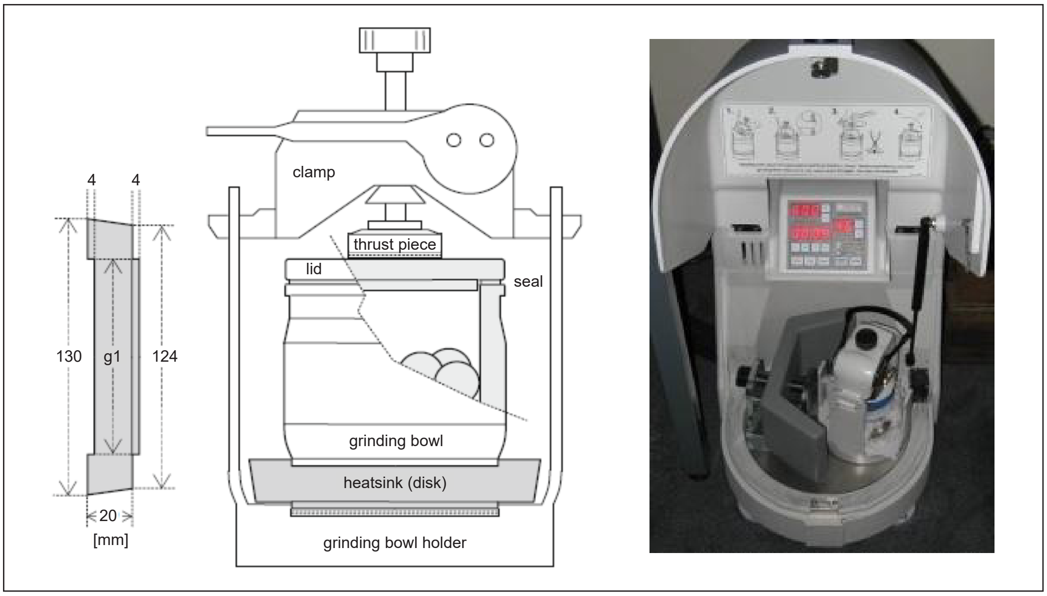

Figure 1. Planetary mill

of stainless steel and agate, which had been used as a grinding media type in this study. The rotational speed of the grinding bowl was in the range of 100 rpm to 500 rpm. A full randomized factorial design with 48 experiments with various combinations of the grinding time, rotational speed, and percentage media content was used, as shown in Table 1. The statistical software Minitab 16 was used for the statistical analysis. Experimental runs of general full factorial design (FFD) and the results of the factorial design were analyzed based on d_{50}, which was determined from the particle size distribution graph obtained from the particle size distribution testing. The standard deviation and R^2 for each response were observed.

Table 1
The operating parameters involved in this study

No	Grinding period (min)	Rotational speed (rpm)	Percentage of grinding media (%)	No	Grinding period (min)	Rotational speed (rpm)	Percentage of grinding media (%)
1	40	400	30	**25**	10	100	30
2	60	100	20	**26**	40	200	40
3	10	200	40	**27**	60	400	20
4	20	400	30	**28**	60	100	40
5	60	200	20	**29**	40	100	20
6	40	200	30	**30**	60	200	30
7	20	500	40	**31**	10	400	20
8	40	200	20	**32**	40	500	40
9	60	100	30	**33**	20	200	20
10	60	500	20	**34**	10	200	30
11	10	100	40	**35**	40	500	20
12	20	100	20	**36**	60	500	30
13	10	100	20	**37**	10	500	20
14	20	400	40	**38**	40	400	40
15	20	500	30	**39**	40	100	30
16	20	500	20	**40**	60	200	40
17	10	400	30	**41**	40	500	30
18	60	500	40	**42**	10	400	40
19	20	100	30	**43**	40	400	20
20	20	400	20	**44**	40	100	40
21	10	500	30	**45**	10	500	40
22	20	100	40	**46**	60	400	30
23	20	200	30	**47**	20	200	40
24	60	400	40	**48**	10	200	20

RESULTS AND DISCUSSIONS

Characterization of Sample

Table 2 shows the percentage of the chemical composition found in the raw sample of mica using XRF. The predominant constituents of the sample were SiO_2 and Al_2O_3 with

55% and 34%, respectively, while other elements were less than 1%. A high percentage of SiO_2 low percentage of Al_2O_3 and Fe_2O_3 contents had decreased the grind ability process (Paine, 2019).

Table 2
Percentage of chemical composition present in the mica

Composition	Weight (%)	Composition	Weight (%
SiO_2	54.86	CaO	0.035
Al_2O_3	34.37	P_2O_5	0.034
K_2O	7.23	ZrO_2	0.033
Fe_2O_3	1.15	NiO	0.021
MgO	1.15	ThO	0.011
TiO_2	0.83	CuO	0.01
Rb_2O	0.059	ZnO	0.007
Cr_2O_3	0.053	Nb_2O_5	0.006
		VO_2	Trace

Figure 2 shows the photomicrograph of mica with 1000 times and 10, 000 times magnification. It can be seen that the mica was in the form of layered structural and irregular shapes.

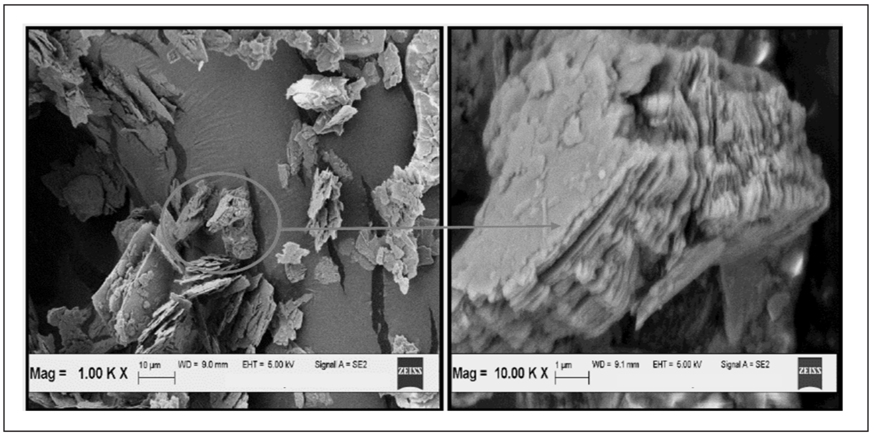

Figure 2. Photomicrograph of mica with 1000 times and 10, 000 times magnification

ANOVA STATISTICAL ANALYSIS

The statistical analysis (ANOVA) was presented in Table 3, and the final model equation in the genuine factor represented the d_{50} value shown in Equation 1.

$$d_{50} = 13.9 - (0.0144 * grinding\ time) - (0.00723 * speed) \qquad [1]$$

Table 3
ANOVA analysis to predict d_{50} of the product

Source	Seq SS	Adj SS	Adj MS	F	P	Significance	% Contribution
Grinding Time	8.6395	8.6395	2.8798	3.97	0.025	Significant	8.33
Speed	63.2325	63.2325	21.0775	29.05	0.000	Significant	60.99
Grinding Media	0.7116	0.7116	0.3558	0.49	0.620		0.69
Grinding Time * Speed	8.1197	0.81197	0.9022	1.24	0.330		7.83
Grinding time * Grinding media	3.0088	3.0088	0.5015	0.69	0.660		2.90
Speed * Grinding Media	6.9004	6.9004	1.1501	1.59	0.208		6.66
Error	13.0580	13.0580	0.7254				12.59
Total	103.6740						100

Based on the value presented in Table 3, it was found that the grinding time and speed were significant parameters in determining the d_{50} of the product for having a P-value less than 0.05. On the other hand, the grinding media percentage did not significantly affect having a P-value of more than 0.05. The coefficient of determination (R^2) was 0.8 with a standard deviation of 0.85. The average absolute percentage error (AAPE) was 6.09, while the average error was 0.21. The actual and predicted data of the d_{50} as shown in Figure 3. However, it was found that the contribution for the error term is 12.59, which is higher than the contribution of one factor. Therefore, this model does not cover as much of the surface roughness variance. The possible reason for this is the measurement error in the mill material discharge system, which could contribute to the cut size determined from the particle size distribution results. Further investigation of the discharge system should be conducted in the next study. However, in this study, the reliability of the trends was confirmed by SEM and the XRD analysis.

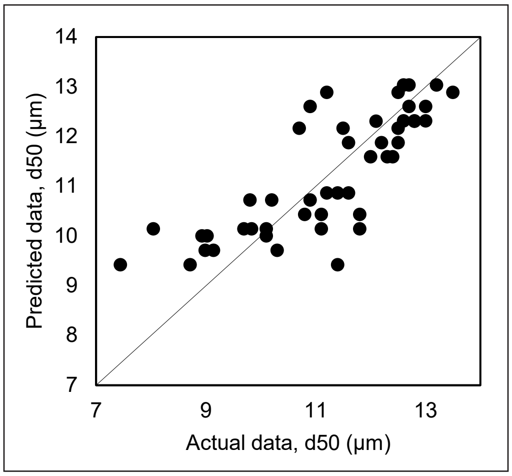

Figure 3. Actual and predicted data of d_{50}

The Effect of Grinding Time

Figure 4 shows the plot of the grinding time to the cut size (d_{50}). The size of the particles was decreased with the increase of the grinding time from 10 to 20 minutes. Increasing time had allowed more impact on the particles. Figure 5 shows the breakage mechanism of the ground mica in the planetary mill. The particles with irregular shapes had undergone the breakage mechanism and delaminated into a layered structure. It was due to the collision between the grinding media-feed-grinding media. Feed that stuck between the grinding media during a collision would undergo the deformation process and crack, determining the particle structure (Liu et al., 2016). With increasing grinding time, the coarse particles with angular morphology decreased, while the fine particles with granular morphology increased, resulting in an improvement of sphericity in the particle size distribution. The collision process depended on the feed's mechanical behavior, stable feed phase, and levels of emphasis during the grinding process.

However, increasing the time from 20 minutes to 60 minutes had increased the size of the particles due to the agglomeration. During the grinding process, the size of particles reached a critical point in which the milling equilibrium was reached. It is the stage where the cut size shows insignificant changes or remains constant. At this state, larger agglomerates were formed by stronger chemical bonds and van der Waals forces (Lee et al., 2020). Figures 6 and 7 show the morphological analysis of the grinding time effect

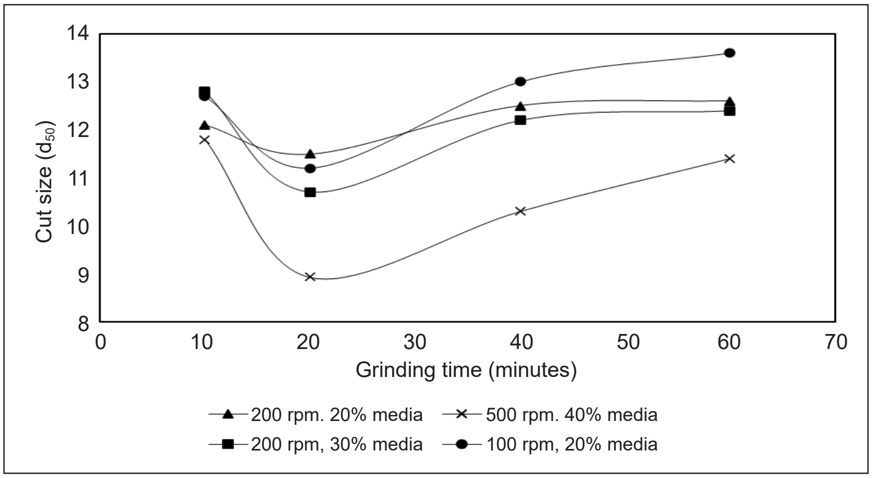

Figure 4. The effect of grinding time to cut size, d_{50} (μm)

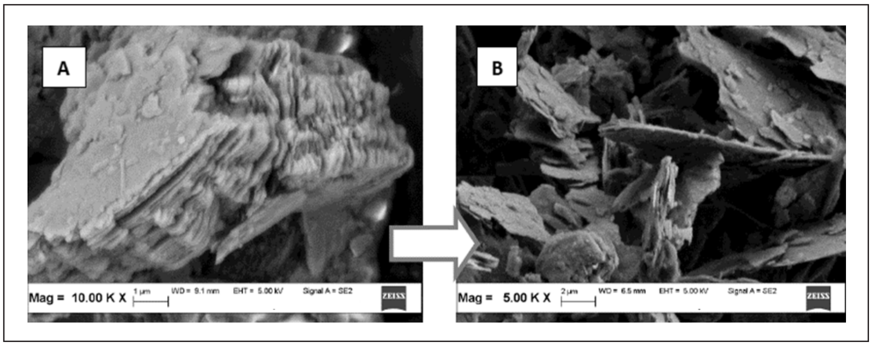

Figure 5. Breakage mechanism applied on mica (A) and delaminated into a layered structure (B)

on the agglomeration of the mica. A longer grinding period tended to cause an increase in amorphism (Zhao et al., 2021; Arbain et al., 2011). The agglomeration process started because of the high surface energy that increases the adhesion forces between the particles. Therefore, longer grinding time produced bigger particles size in this agglomeration phenomenon. Another possible reason for agglomeration is the solid residue produced during grinding, cemented by highly reactive amorphous material that serves as a coating. The accumulation of these small particles on the agglomerates' surface could reduce the milling efficiency as broken particles reassemble to form large particles. This condition should be avoided in the production of fineness particles, for example, in the filler industries, as it could not disperse well in the rubber matrix and would give a poor property.

Increasing the grinding time may also change the structure of the mica particle. It can be shown by a structural distortion test using XRD, as shown in Figure 8 and Table 4. In Figure 3, the XRD patterns had different grinding times ranging from 10 min to 60 min. Due to their significant distortion, two peaks were observed based on the XRD pattern. It can be observed clearly at the first peak, 2θ angles of 17.82º. Based on Table 4, the intensity peak before the grinding was 337.0727. Then, the peak increased until the value achieved 1385.071 for 10 and 20 minutes of the grinding period, but after 40 min of grinding, the intensity was reduced to 322.5305. A great intensity reduction was observed during 40 min of the grinding. After that, it continuously decreased until the period of grinding achieved 60 min. The grinding process transformed the crystallinity of all the ground mica from crystalline to partially amorphous. Another peak was also observed at 2θ angles of

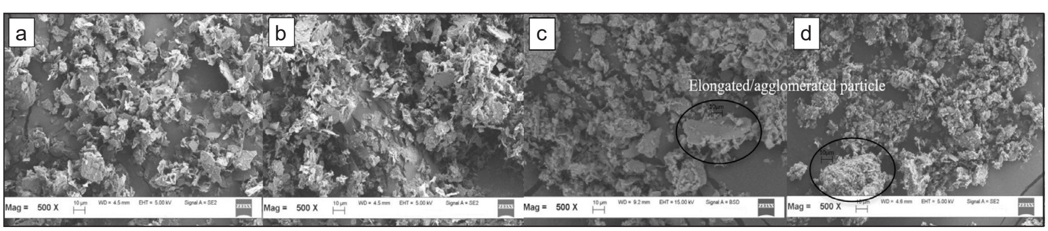

Figure 6. Photomicrographs of ground mica at a) 10 minutes, b) 20 minutes, c) 40 minutes, and d) 60 minutes

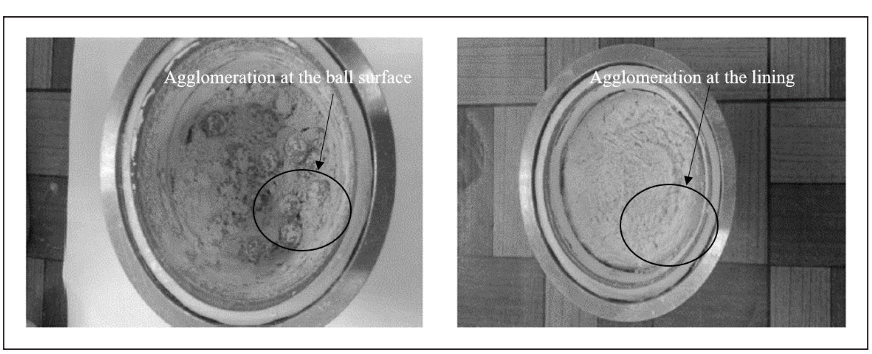

Figure 7. Agglomeration of mica powder at the lining and surface of the ball

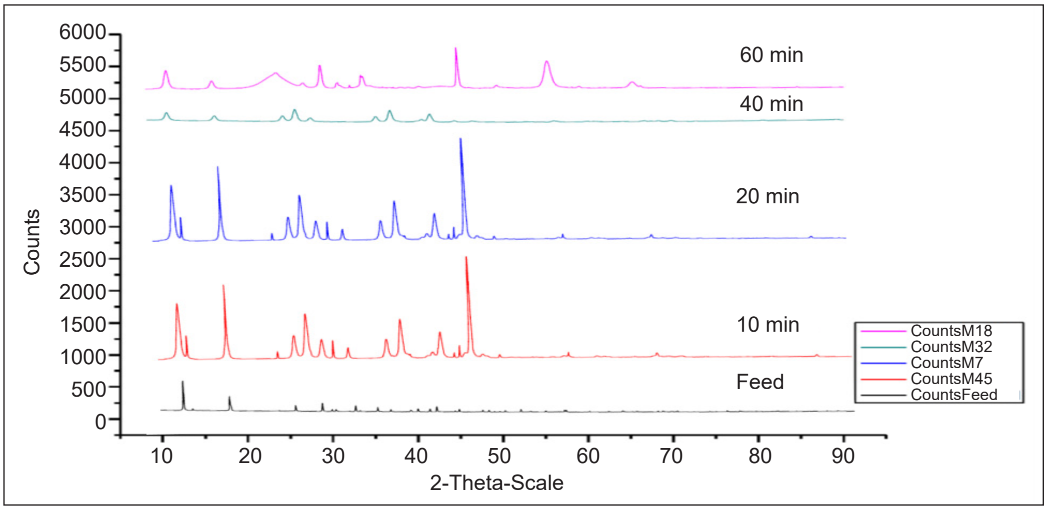

Figure 8. XRD pattern with the rotational speed of 500 rpm and 40% of percentage grinding media at the different grinding time

Table 4
Peak intensity and d_{50} value for the various grinding periods

Sample	Raw sample	45	7	32	18
Grinding time, minutes		10	20	40	60
Rotational speed, rpm		500	500	500	500
Grinding media, %		40	40	40	40
d_{50}, µm	13.8	11.8	9.02	10.3	11.4
2-Theta Scale	17.82	17.82	17.82	17.82	17.82
Intensity, calc	337.0727	1385.071	1385.071	322.5305	298.682
2-Theta Scale	45.53	45.53	45.53	45.53	45.53
Intensity, calc	157.332	1755.751	1755.751	162.8971	755.2411

45.53º. At this peak, intensity tended to increase with the grinding time. However, after 40 minutes, the intensity value reduced, and at 60 minutes of the grinding, the peak intensity increased again. Within 10 minutes of the grinding operation, a significant change in the crystal structure of mica was observed.

The Effect of Grinding Speed

Figure 9 shows the plot of the grinding speed to d_{50}. A significant size reduction was observed when the mill rotational speed increased from 400 rpm to 500 rpm. Micronized mica below 10 µm was obtained as the mill rotational speed was more than 200 rpm. This observable fact was due to the rate of impulses. As the mill speed increased, the rate of impulses and acceleration also increased. The size reduction per unit time also increased due to more repetitive stress. The increase in mill rotational speed would cause the frequency

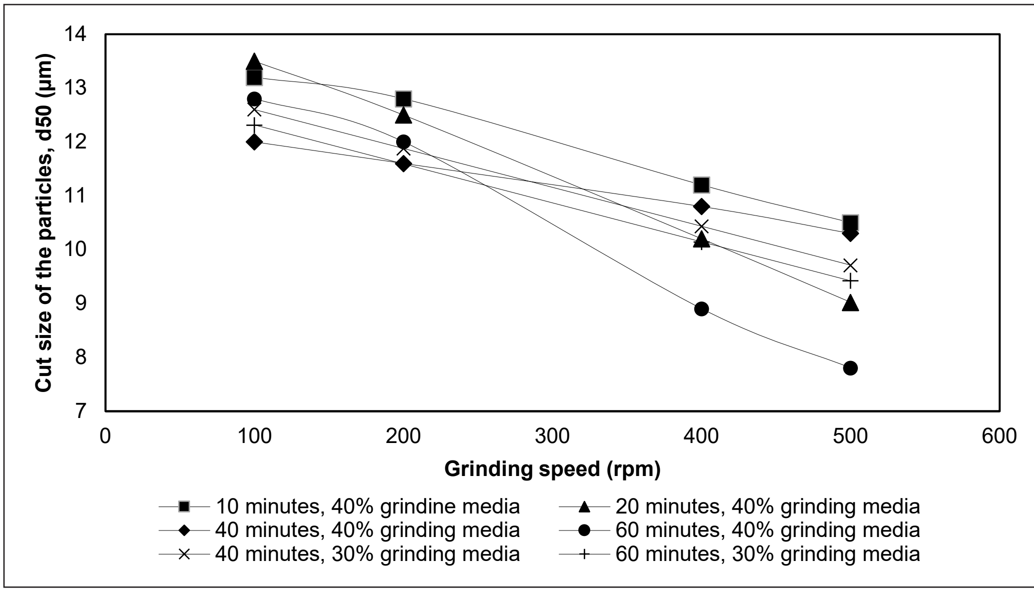

Figure 9. The effect of grinding speed on the cut size of the particles (d_{50})

of collision between the grinding media to increase. This phenomenon may contribute to the breakage of the particle at coarse size because the frequency of collision between the grinding media and the coarse particle was higher than the collision between the grinding media and fine particles. There are two probable reasons for this phenomenon. The first one is that it might be explained by the heat generated by the higher operational speed that might lead to the particles being in a brittle stage. The other reason is that the increasing speed might behave like an abrasive compound that breaks the particles (Real & Gato, 2019; Sato et al., 2010).

The optimization of the dry grinding of mica was predicted using the Minitab 16. In this case, it was found that the optimum parameters for grinding speed, grinding time, and grinding media were 20 minutes, 400 rpm, and 30% charged, respectively, with the d_{50} value of 7.44 µm.

CONCLUSION

Mica with predominant constituents of 55% SiO_2 and 34% Al_2O_3 was grounded in the planetary mill. The effect of dry grinding of mica on the d_{50} of the ground product in a planetary mill was investigated. Statistical analysis was done, and it was found that the grinding process of mica depended on the various operating variables such as grinding speed and grinding time while grinding media charge only gives a small contribution during mica grinding. During the extreme condition when the operating variables were at the maximum level, such as 60 min of the grinding period with 500 rpm rotational speed of grinding and 40% of the grinding media, it was observed that the fine particles of mica tended to agglomerate, contributing to the inefficient of the grinding process. The coefficient of determination (R^2) was 0.874 with a standard deviation of 0.852, which indicated good agreement between the actual and predicted data of the $d_{50.}$ It was suggested that an appropriate grinding aid for mica grinding was determined and used in a future study to avoid particle agglomeration issues and improve process efficiency.

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