Optimizing Dry Ultrafine Grinding of Talc in Attritor Mill

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Abstract

In this work, the parameters affecting the dry grinding of talc in the attritor mill were investigated. The attritor was tested with its initial design without balls then with balls as additional grinding media to prepare a feed passing 45 microns. The prepared feed (-45 mm) was used to produce a product with 10 μm or less using statistical design in presence of balls. The presence of balls enhances product fineness. The significance of the studied factors is: stirrer speed>mill filling >grinding time. In addition, the d_{50} was correlated with studied factors. The mean particle size (d_{50}) as low as 6 μm was obtained at 460 rpm, 20% mill filling, and after 60 min grinding time.

Keywords: *dry grinding, ultra-fine grinding, attritor mill, statistical design, talc*

1. Introduction

Grinding is an essential process for reducing particle size and preparing a material with specific size range or separating different minerals based on the difference in their mechanical properties [1-3]. Yet, fine grinding is a challenge where the conventional ball milling seems uneconomical especially when sizes below 10 μm are targeted [4-9]. Recently, the ultra-fine grinding of minerals attracts attention of numerous industrial applications such as advanced ceramics, porcelain, cement, paper coating, plastic and pigments. Ultra-fine talc (<10 μm), in particular, has wide applications. In plastics, ultra-fine size platy talc is used to improve its mechanical and surface properties such as stretch resistance [10]. In paper industry talc is used as a filler to control pitch and sticky materials and in coating formulation. In oil paints talc is used as an extender and suspending agent [11].

Attritor mill is one of modified designs of stirred media mill that produces fine particles for chemical, pharmaceutical and mineral processing industries [12-13]. The stirred mills are much more efficient for fine grinding than conventional ball mills. By introducing the stirred media mills, the fine grinding becomes more economically favorable in terms of high grinding rate and low energy consumption [5, 14-16]. In attritor mill, particles are subjected to various forces such as impact, rotational, tumbling, and shear; therefore, the fine powders in micron range can be easily achieved.

Therefore, this work aims at studying dry ultra-fine grinding of talc to produce fraction with a d_{50} less than 10 *μ*m using attritor mill without or with alumina balls as additional grinding media. The main parameters affecting the grinding process such as grinding time, stirring speed, media size, and mill filling were investigated using either one-variable-at-a-time strategy or statistical design in terms of particle size (d_{50}) of the ground product. Moreover, the analysis of variance was used to determine the significance of the studied factors and derive a correlation of d_{50} of the ground product as a function of studied factors.

2. Experimental

2.1. Materials

Talc sample was supplied by El-Nasr Mining Co. It represents Bir Meseh, South of Shalatin locality, Eastern Desert, Egypt. Talc sample was crushed using a "Denver" jaw primary crusher in a closed circuit with a 6.63 mm screen. Representative batches were prepared by successive coning and quartering methods, one of which was finely ground to less than 200 mesh for XRD and chemical analysis.

2.2. Methods

2.2.1. Mineralogical and Chemical Analysis

Identification of the mineral composition of the considered sample was conducted by X-ray diffraction (XRD). Samples were analyzed by X-ray diffraction analysis pattern of the specimen, using the data of the powder pattern of ASTM. The samples for bulk mineral analysis as well as the pure mineral phases were finely ground, mounted randomly on an aluminum holder, and analyzed by X-ray diffraction. Different phases of the samples were identified by X-ray diffractometer (XRD, Brukeraxs D8 Advance, Germany) with Cu kα radiation (λ = 1.5406 Å) and secondary monochromator in the 2θrange from 2 to 75°

In addition, quantitative determination of the talc associated oxides was carried out using Panalytical XRF (Model advanced Axios, Netherlands). One gram of the sample was fired at 1000°C for 2 hours for calculating the loss on ignition (L.O.I). On the other hand, 10 g of the sample is pressed with 2 g wax as a binder in aluminum cup, and then exposed to X-rays as a disk.

2.2.2. Description of Attritor Mill

Vertical laboratory attritor mill (union process type 1S) consists of a stainless steel vessel of 9.5-liter capacity. It has a stainless steel shaft fitted with five arms made of stainless steel. The shaft is a belt-driven with a motor of 3Hp. The tank

Tab. 1. Factors and levels for Box Behnken experimental design
Tab. 1. Czynniki i poziomy dla eksperymentalnego projektu Boxa Behnkena

Variables	Symbol	-1	0	+1
Stirrer speed, rpm	A	270	365	460
Mill filling, %	B	20	30	40
Grinding time, min	C	30	45	60

Tab. 2. Chemical analysis of talc sample
Tab. 2. Analiza chemiczna próbki talku

Element	%	Element	%
SiO_2	46.33	SO_3	0.10
MgO	25.26	$Na_2O + K_2O$	0.12+0.015
CaO	9.48	P_2O_5	0.106
Al_2O_3	4.39	MnO	0.100
Fe_2O_3	2.21	F^-	0.193
TiO_2	0.27	L.O.I	11.40

Tab. 3. Box–Behnken design results in terms of d_{50}
Tab. 3. Wyniki obliczeń Boxa-Behnkena w zakresie d_{50}

Run	A: Stirrer speed, rpm	B: Mill filling, %	C: Grinding time, min.	d_{50}, µm
1	365	30	45	7.78
2	460	30	60	6.15
3	365	20	30	8.41
4	460	30	30	7.63
5	270	30	60	8.86
6	365	30	45	7.72
7	365	30	45	7.74
8	460	40	45	7.78
9	365	40	30	9.64
10	365	30	45	7.74
11	270	20	45	8.60
12	270	40	45	10
13	460	20	45	6.32
14	365	20	60	6.91
15	365	40	60	8.62
16	365	30	45	7.74
17	270	30	30	9.66

is jacketed for cooling and it includes a bottom discharge valve, and bar grid.

The grinding of talc is performed under dry conditions in the presence of 4 mm or 10 mm diameter alumina balls. The experiments were conducted in more details by varying different parameters; namely, grinding time (min), impeller speed (rpm), and mill filling by volume (%). The talc sample feed for attritor mill totally passed from 6.63 mm and its d_{50} equals 2.4 mm, Figure 1.

The attritor product at the end of each test was screened on 45 µm screen. The oversize fraction (+45 μm) was measured by screening, while the undersize fraction (-45 μm) was measured by COR-2000 Laser Particle Size Analyzer.

2.2.3. Statistical design

Box–Behnken experimental design [17-18] was used to investigate the significance of studied factors and for optimizing talc grinding process of -45 microns. The effects of stirring speed, mill filling and grinding time on the talc particle size (more specifically on d_{50} of the ground product) were studied. The design matrix consists of 15 randomized experimental runs. The levels of each factor are given in Table 1.

For this design, the optimal conditions were estimated using a second order polynomial function by which a correlation between studied factors and response (d_{50} of the ground product) was generated. Design-Expert 6.1 Software, Stat-Ease, Inc., Minneapolis, USA, was used for regression analysis of experimental data and to plot response surface. Analysis of variance (ANOVA) using F-test was used to estimate the significance of all terms in the polynomial equation as well as the statistical parameters within 95% confidence interval. The extent of fitting the experimental results to the polynomial model was expressed by the correlation coefficient, R2.

3. Results and Discussion

3.1. Mineral Characterization

The XRD analysis of talc sample, Figure 2, shows that it consists of talc $Mg_3(OH)_2Si_4O_{10}$ associated with chlorite ($(Mg,Fe)_5(Al,Si)_5O_{10}(OH)_8$), calcite ($CaCO_3$), quartz ($SiO_2$), and anatase ($TiO_2$). In addition, chemical analysis of sample, using XRF, is given in Table 2.

3.2. Grinding by Attritor Mill

3.2.1. Effect of ball size

The effect of ball size was studied at 35% mill filling, 270 rpm, and 30 min grinding time. Two ball sizes were tested as mono-size balls or as a mixture of the two balls sizes in different percentages. In the first set of experiments, all the balls are the same size (4 mm), the second set of experiments contains 50% of the ball charge from 4 mm balls and the other half consists of 10 mm, the third set of experiments uses 25% of the ball charge from 4 mm balls and 75% of the charge is 10 mm balls, while the fourth set consists of 100% of 10 mm balls.

Figure 3 represents the effect of ball size on weight % of fines. It is obvious that by increasing the percentage of 10 mm balls over 4 mm balls the product fineness increases. The weight % of -45 µm fraction increases by changing the ratio between the 4 mm balls to 10 mm balls. As the percentage of 10

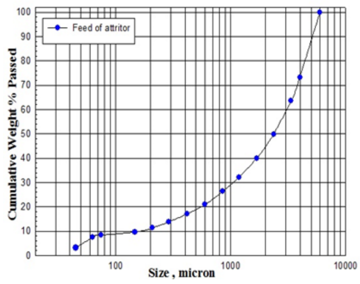

Fig. 1. Size analysis of Feed in Attritor mill
Rys. 1. Analiza wielkościowa paszy w młynie Attritor

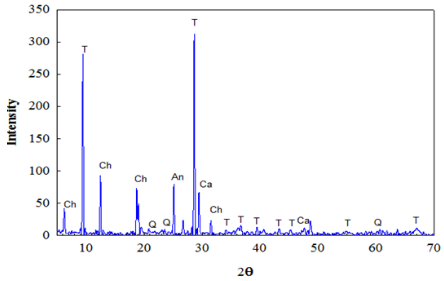

Fig. 2. XRD of talc sample
Rys. 2. XRD próbki talku

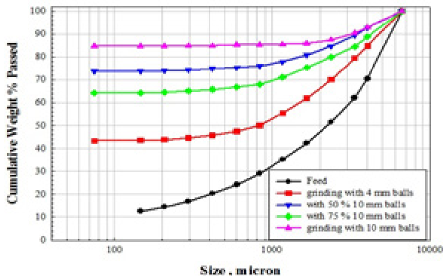

Fig. 3. Effect of different ball sizes on -45 µm wt% at 35% mill filling
Rys. 3. Wpływ różnych rozmiarów kulek na -45 µm % wag. przy 35% napełnieniu młyna

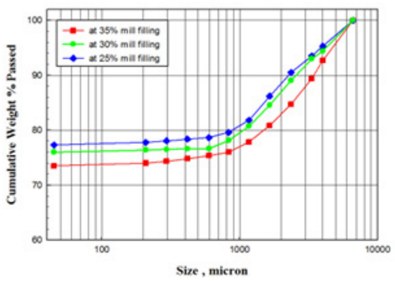

Fig. 4. Size distributions of product dry ground at different mill filling and balls size of (70% of 10 mm balls with 30% of 4 mm balls)
Rys. 4. Rozkład wielkości suchego produktu zmielonego przy różnym napełnieniu młyna i wielkości kulek (70% kulek 10 mm z 30% kulek 4 mm)

mm balls increases the weight percentage of fines increases. More clearly, the weight percentage of -45 µm changed from 50% at 100% 4 mm balls, passing through different 4 mm to 10 mm ratios, to about 85% at 100% 10 mm balls. This behavior can be attributed to the presence of large talc particles in the feed that requires large balls to be ground. The large balls is heavier and more efficient in breaking larger sizes. Whereas, the smaller balls is most probably needed in producing finer sizes [19-21].

3.2.2. Effect of Mill Filling

Different mill fillings (25%, 30%, and 35% by volume) at 270 rpm, and ball sizes (70% of 10 mm balls with 30% of 4 mm balls) at 30 min grinding time were tested. Figure 4 shows the effect of mill filling on the size distribution of the ground product as well as the percentage of -45 µm. It shows that the lower the mill filling is the higher the fines production. The weight % of -45 µm increases from 73% to 78% using 25% mill filling instead of 35% mill filling.

On the other hand, Figure 5 shows that the weight % of -45 µm increases using 100% of 10 mm ball at 25% mill filling to 85% compared to 78% in the case of using (70% of 10 mm with 30% of 4 mm balls). This means that the coarser ball size 10 mm is more effective than other ball sizes and mixtures. The load of 10 mm balls subjects the particles to higher stresses leading to their grinding.

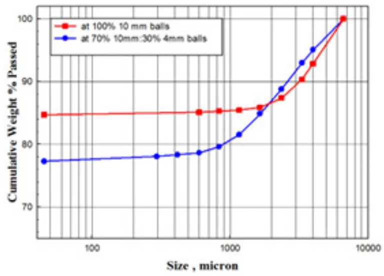

Fig. 5. Size distributions at different ball sizes at 25% mill filling
Rys. 5. Rozkłady wielkości przy różnych rozmiarach kulek przy 25% napełnieniu młyna

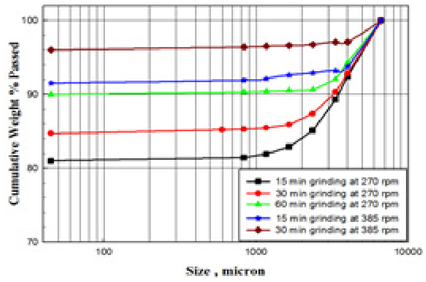

Fig. 6. Size distributions of ground products at 25% mill filling, different stirrer speeds and different times
Rys. 6. Rozkład wielkości zmielonych produktów przy 25% napełnieniu młyna, różnych prędkościach mieszania i różnych czasach

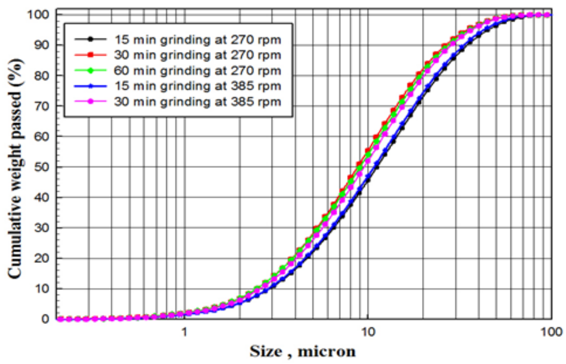

Fig. 7. Size distributions of -45 microns at different stirrer speeds and different times
Rys. 7. Rozkłady wielkości -45 mikronów przy różnych prędkościach mieszadła i różnych czasach

3.2.3. Effect of Stirrer Speed

Figure 6 shows the effect of stirrer speed at 25% mill filling, using 10 mm balls, at different grinding times. It is noticed that, for 15 min grinding time, the wt. % of -45 µm increases from 81% to 91.5% by increasing stirrer speed from 270 rpm to 385 rpm, respectively. While increasing the grinding time to 30 min, the weight % of -45 µm reaches 84.6% at 270 rpm and 95.6% at 385 rpm. It is clear from the size distribution of products that the grinding fineness increases by increasing stirrer speed. It has been shown that d_{50} in a fraction -45 µm at 385 rpm and 30 min grinding is 10.3 µm.

It can be concluded that the fineness of grinding products significantly increases with increasing both stirrer speed and time. The d_{50} of the ground products ranges from 9 to 12 µm, Fig.7. Therefore, to grind the all the sample to -45 um with d_{50} less than 10 µm, the optimization of the process using statistical design was studied.

3.3. Statistical design

The experimental design is used to optimize the dry grinding in attritor mill in terms of mill filling, stirrer speed, and grinding time. Table 3 shows Box-Behnken design results in terms of d_{50} of the products.

The standard deviation and R-Squared for d_{50} are 0.032, 0.9995, respectively. These values indicate the well-fitting of the experimental results to the polynomial model equation. The correlation between d_{50} and the studied facors is derived as follows:

$$d_{50},\ \mu m = +17.33 - (7.97X10^{-3} *A) - (0.2*B) - (0.14*C) + (4.0X10^{-3}*B2) +(1.13X10^{-3}*C2) - (8.8X10^{-5}*A*C) + (8.0X10-4*B*C)$$

where,
A : stirrer speed, rpm
B: Mill filling, %
C: grinding time, min

3.3.1. Effect of mill filling and stirring speed

Figure 8 shows the response surfaces for d_{50} at different values of mill filling and stirring speed at 30 min grinding time. It is noticed that by increasing stirring speed to 460 rpm

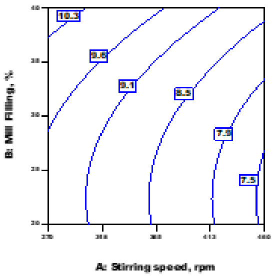

Fig. 8. Effect of stirrer speed and mill filling on d50 of the product at 30 min grinding time
Rys. 8. Wpływ prędkości obrotowej mieszadła i wypełnienia młyna na d50 produktu przy czasie mielenia 30 min

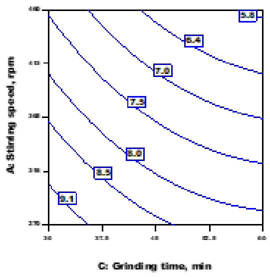

Fig. 9. Effect of stirring speed and grinding time on d50 at 20% mill filling
Rys. 9. Wpływ szybkości mieszania i czasu mielenia na d50 przy 20% wypełnieniu młyna

and decreasing mill filling to 20%, d_{50} decreases. The lowest d_{50} is 7.5 µm at 460 rpm stirring speed, 20% mill filling, and 30 minutes grinding time. Increasing the mill filling produces larger talc particles. This may be attributed to the lower collision probability between the media and talc particles because many hits will be among the media balls rather than with particles.

3.3.2. Effect of grinding time

Figure 9 shows the d50 as function of grinding time and stirring speed at 20% mill filling. The higher the grinding time is the lower the d_{50}. The combined effect of grinding time and stirring speed reduces the particle size from 9.4 µm to 5.8 µm. The smallest d_{50} is achieved at 460 rpm, 20% mill filling, and after 60 min grinding time.

4. Conclusions

Dry grinding of coarse talc feed in an attritor mill with/without balls, as grinding media, to produce fraction completely passes -45 μm was investigated. The results showed that a product with d_{50} ranges from 9-12 μm was obtained by grinding of 2.4 mm feed, with the larger ball size, the higher impeller speed, and lower mill filling. After preparing the fraction of -45 um fraction, its grinding is further studied by statistical design to investigate the significance of the studied factors.

In addition, a functional relationship between three grinding variables (i.e., mill filling, stirrer speed, and grinding time) and the product mean size d_{50} was obtained. It showed that the higher the speed and grinding time and the lower the filling is the higher the product fineness. A product as low as 6 μm can be achieved at optimum conditions (i.e., 460 rpm, 20% mill filling, and after 60 min grinding time).

Literatura – References

1. Chen J., Pan Z., Wang Y., 2018. Preparation of submicron-sized quasi-spherical silica particles via ultrafine grinding with chemical dissolution assistance, Powder Technology, 339: 585-594.
2. El-Midany A. A.; Selim A.Q.; Ibrahim S.S., 2011. Effect of Celestite-Calcite Mineralogy on Their Separation by Attrition Scrubbing, Particulate Science and Technology, 29: 272–284.
3. Sun C., Liu R., Ni K, Wu T, Luo X, Liang B, Zhang M., 2016. Reduction of particle size based on superfine grinding: Effects on structure, rheological and gelling properties of whey protein concentrate, Journal of Food Engineering, 186: 69-76
4. Chatterje, K.K., 2009, "Uses of industrial minerals, rock and fresh water", Chapter 43. Talc-Steatite-Soapstone, Nova Science Publishers, Inc., New York. pp. 425-437
5. Botin, J.A. (2009). Sustainable management of mining operations. SME: Society of Mining, Metallurgy and Exploration, Inc.
6. Roufail, R. (2011). The effect of stirred mill operation on particles breakage mechanism and their morphological features. Master's thesis, University of Waterloo.
7. Underle, U., Woodall, P., Duffy, M., Johnson, N.W., (1997). Stirred mill technology for regrinding McArthur river and Mount Isa zinc/lead ores. In: Proceedings of XX IMPC––Aachen, 21–26 September, 71–78.
8. Burgess, F., McGuire, I., Willoughby, R. (2001). Operation of sand mill detritors at Pasminco operations. In: Fine Particle Processing and Tailing Summit, July, Perth, Australia.
9. Mio, H., Kano, J., and Saito, F., 2004, "Scale-up method of planetary ball mill", Chemical engineering science, 59 (24) 5909-5916
10. El-Midany, A.A., Ibrahim, S.S. 2010, The effect of mineral surface nature on the mechanical properties of mineral-filled polypropylene composites", Polymer Bulletin, 64, 387-399.
11. Jayasundara, C.T., Yang, R.Y., Guo, B.Y., Yu, A.B., Govender, I., Mainza, A., and Rubenstein, J., 2011, "CFD–DEM modeling of particle flow in IsaMills–Comparison between simulations and PEPT measurements", Minerals Engineering, 24 (3) 181-187.
12. Jimbo, G., 1992, "Chemical Engineering Analysis of Fine Grinding Phenomena and Process", Chem. Eng. Japan, Vol. 25, p. 117.
13. Jankovic, A., 2003. Variables affecting the fine grinding of minerals using stirred mills, Minerals Engineering, 16, 337-345.
14. Choi, W.S., 1996, "Grinding Rate Improvement Using a Composite Grinding Ball Size for an Ultra-fine Grinding Mill", J. Soc. Powder Technol., Japan, 33:747.
15. Cayirli S., 2016. Dry grinding of talc in a stirred ball mill, E3S Web Conferences, Mineral Engineering Conference (MEC2016), Vol.8, article no. 01005.
16. Schilling, R.E., Yang, M., 2000, "Attritor grinding mills and new developments", Union process, Inc., Akron, Ohio.
17. Box G.E.P., Behnken D.W., 1960. Technometrics 2, 195 -202.
18. Box, G. E. P., W. G. Hunter, and J. S. Hunter, 1978. Statistics for Experimenters: An Introduction to Design, Data Analysis, and Model Building. Wiley, New York.
19. Schnatz, R., 2004. Optimization of continuous ball mills used for finish-grinding of cement by varying the L/D ratio, ball charge filling ratio, ball size and residence time, International Journal of Mineral Processing, 74S, S55-S63.
20. Austin, L.G., Shoji, K., Luckie, P.T., The effect of ball size on mill performance, Powder Technology, 14 (1976) 71 - 79
21. Bwalya, M.M., Moys, M.H., Finnie, G.J., Mulenga, F.K., 2014. Exploring ball size distribution in coal grinding mills, Powder Technology, 257, 68-73

Optymalizacja ultradrobnego mielenia talku na sucho w młynie Attritor

W niniejszej pracy zbadano parametry wpływające na mielenie talku na sucho w młynie Attritor. Attritor został przetestowany w początkowej wersji bez kulek, a następnie z kulkami jako dodatkowym medium mielącym w celu przygotowania nadawy przechodzącej przez 45 mikronów. Przygotowany wsad (-45 mm) został wykorzystany do wytworzenia produktu o uziarnieniu 10 µm lub mniejszym przy użyciu statystycznego projektu w obecności kulek. Obecność kulek zwiększa stopień rozdrobnienia produktu. Znaczenie badanych czynników to: prędkość mieszadła > wypełnienie młyna > czas mielenia. Ponadto d_{50} był skorelowany z badanymi czynnikami. Średni rozmiar cząstek (d_{50}) tak niski jak 6 µm uzyskano przy 460 obr/min, 20% wypełnieniu młyna i po 60 min mielenia.

Słowa kluczowe: *mielenie na sucho, mielenie ultradrobne, młyn attritor, projektowanie statystyczne, talk*