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Combination of Conventional Ball Mill and Stirred Mill to Obtain Ultra-Fine Talc

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Abstract

In this study, it was investigated that talc ore could ground to very fine sizes using a combination of conventional ball mill and vertical stirred mill. Firstly, the conventional ball mill parameters such as mill speed (% of critical speed), material filling ratio (J_b), ball filling ratio (f_c), ball size distribution (10-20-30-40 mm, %), grinding aid ratio (% of powder) and grinding time (min.) were optimized using classic experimental design. As a result of the optimization, fine talc product was obtained with d_{90}=93.52 µm particle size. In the second stage, the ball mill product was re-milled using a stirred mill. As a result of study, a final ultra-fine talc powder was obtained after 60 minutes grinding time with d_{50}=1.85 µm particle size and 14058 cm^2/g total surface area. It has been seen that the conventional ball mill+stirred mill combination effective a process to obtain talc powder that suitable for industrial use.

Keywords: Talc, Conventional ball mill, Stirred ball mill, Ultra-fine product

Bilyeli Değirmen ve Karıştırmalı Değirmen Kombinasyonu ile Çok İnce Talk Üretimi

Öz

Bu çalışmada, konvansiyonel bilyeli değirmen ve dik karıştırmalı değirmen kombinasyonu kullanılarak talk cevherinin çok ince boyutlara öğütülebileceği araştırılmıştır. İlk olarak, değirmen hızı (kritik hızın %'si), malzeme doluluk oranı (Jb), bilye doluluk oranı (fc), bilye boyut dağılımı (10-20-30-40 mm, %), öğütme yardımcısı gibi geleneksel bilyeli değirmen parametreleri klasik deneysel tasarım kullanılarak optimize

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edilmiştir. Optimizasyon sonucunda d_{90}=93.52 µm partikül boyutuna sahip ince talk ürünü elde edilmiştir. Çalışmanın ikinci aşamasında ise bilyeli değirmen ürünü dik karıştırmalı değirmen kullanılarak yeniden öğütülmüştür. Çalışma sonucunda, 60 dakika öğütme ile d_{50} değeri1.85 µm olan ve 14058 cm^2/g toplam yüzey alanına sahip çok ince talk ürünü elde edilmiştir. Çalışmanın sonunda konvansiyonel bilyeli değirmen+karıştırmalı değirmen kombinasyonunun endüstriyel kullanıma uygun çok ince talk eldesi için etkili bir teknoloji olduğu ortaya konulmuştur.

Anahtar Kelimeler: Talk, Konvansiyonel bilyeli değirmen, Karıştırmalı değirmen, Çok ince ürün

1. INTRODUCTION

Grinding is a very important activity in many industries such as the chemical, pharmaceutical and material industries. [1]. Grinding process is intensively used in industrial raw materials (calcite, talc, barite, kaolin, quartz etc.) in mining sector to obtain fine/ultra-fine powder products. In practice, two grinding technologies are predominantly involved in the industry. The first of these is the conventional ball mills and the second is the stirred mills. Conventional ball mills are still predominantly preferred in the fine and ultra-fine grinding sector in the world. The most important reasons for this are knowledge of its technology, making domestic production and having a high capacity. The view of a dry micronized grinding line is given in Figure 1.

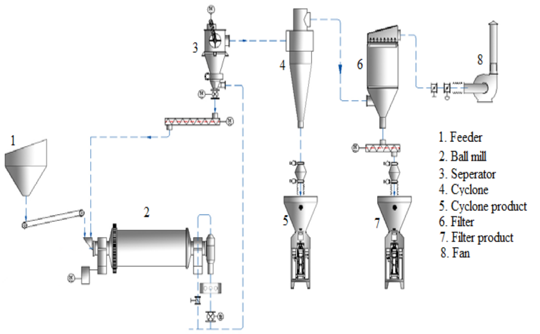


Figure 1. A simple micronize grinding flowsheet [2]

If 80 percent of the product size is below 75 µm the ball mill efficiency drops sharply. For this reason, it is accepted that the ball mill grinding limit in the range of 40–45 µm. [3]. Wang and Forssberg [4] emphasize that the ball mill is used for products below 100 µm in the relationship between feed, product sizes and energy consumption of different mill types. In addition, there are many academic studies on fine and very fine grinding in ball mills. These studies show that ball milling is still indispensable in powder production technology in the world [5-13].

In ball mill experimental studies performed using the classical or traditional experiment design. In this method, one parameter at a time is changed and one level of other independent parameters is kept constant. As seen in Figure 2, in a product development or experimental design made with the classical methodology to solve a production problem, the parameters for the experiment are determined and the experiment is started. In the experiment, the parameter X1 is changed, the result of the experiment is measured and the effect on the experiment is tried to be determined, while other parameters are kept constant. In the experimental study conducted with the classical methodology, external factors (uncontrollable) affecting the experiment are not taken into account too much [14,15].

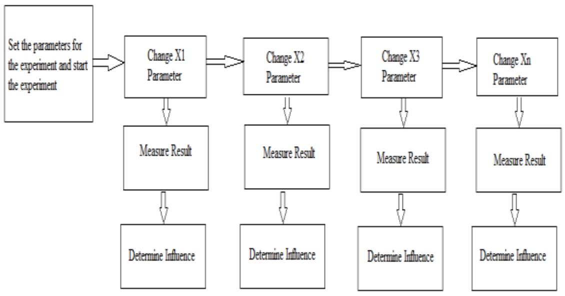


Figure 2. Experiment design and measurement with classical methodology [14].

In recent years, the use of stirred mills in various industrial branches has become increasingly widespread. In the mining sector, in parallel with the increase in the need for fine grinding and the increase in equipment sizes, the stirred ball mills constitute an alternative to the traditional size reduction equipment used in the facilities. The most important reason for this situation that the amount of energy released in unit volume in the equipment is very high compared to traditional size reduction equipment and grinding to fine sizes can be performed economically [16]. Stirred ball mills are used for very fine and submicron grinding, especially in mining, chemistry, ceramics, pharmaceutical industries, paint, microelectronic industry etc. It is included in the literature in many academic studies about the stirred mill [17-23]. The use of stirred ball mills in the very fine grinding sector continues to increase. Although these mills have a lower capacity compared to conventional ball mills, they are preferred because of their ability to produce uniform products in very fine sizes (d_{50}=2-3 µm and even in submicron sizes.

Talc is a raw material widely used in the industry due to its many physical and chemical properties. It is a layered, aqueous magnesium silicate with the chemical formula [$Mg_3(Si_2O_5)_2(OH)_2$] and consisting of 63.5% SiO_2, 31.7% MgO and 4.8% MgO [24]. Talc is widely used in different industries such as ceramics, chemistry, paint and food all over the world. due to its very special mineralogical and chemical structure are used in the cosmetics and pharmacology industry. The talc reserve of Türkiye is 1,158,000 tons, of which 106,000 tons are visible [25]. Grinding of talc ore to very fine sizes is one of the requirements for its use in industry. For this reason, it is clear that it is necessary to take advantage of the possibilities offered by technology in grinding the talc mineral into very fine sizes.

To obtain ultra-fine talc powder, two different grinding systems were combined in this study. For this purpose, first the dry grinding parameters of ball mill were optimized and secondly, obtained fine talc powder product was subjected to a regrinding process by vertical stirred ball mill.

2. MATERIAL AND METHOD

The talc ore used in this study was obtained from Mikron'S Co. (Niğde, Turkiye). Ore supplied from the factory in crushed form (-2 mm). The chemical composition of the ore is given in Table 1. Due to the classical composition of talc ore, the studied ore mainly consists of SiO_2 (63.15%) and MgO (33.33%) compounds. The iron oxide content, on the other hand, is not at a very low level, but not at a very high value. Sieve analysis of the ore supplied from the factory as -2 mm is given in Figure 3. As can be understood from here, the d_{80} dimension of the ore to be used for grinding is 1.3 mm.

Table 1. Chemical composition of the ore sample

Element	%
SiO_2	63.15
MgO	33.33
CaO	0.79
Fe_2O_3	3.12
Al_2O_3	0.03

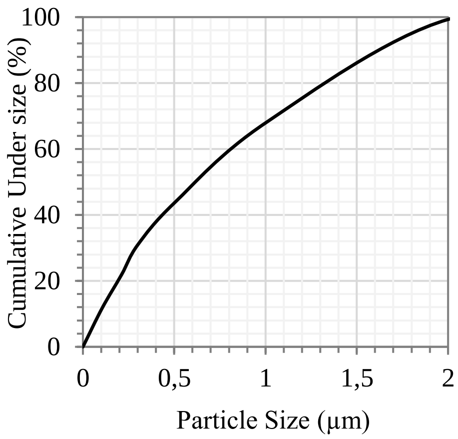


Figure 3. Particle size distribution (PSD) of the crushed sample

In the grinding tests, a 200*200 mm sized laboratory-type stainless steel ball mill was used (Figure 4a). Balls of four different diameters (10,20,30,40 mm) with a density of 8000 kg/m^3 were preferred as the grinding medium (Figure 5a). In conventional ball mill tests, grinding operation speed (% of critical speed), talc filling ratio (fc), ball filling ratio (Jb), ball size distribution, grinding aid ratio and grinding time (min) parameters were optimized. In this optimization, the grinding products were sieved through a 106 micron (150 mesh) sieve and the under-sieve ratios were taken as basis. Critical speed (N_c), ball filling ratio (J_b), and powder filling ratio (f_c) were calculated using Equations 1, 2, and 3, respectively. The ball mill grinding experiments were performed as a batch operation.

N_c = [42.3 / (D - d) $^{0.5}$] rpm (*D* = mill diameter and *d* = ball diameter; in meters) (1)

J_b = [(mass of balls / ball density) / mill volume)] × (1 / 0.6) (2)

f_c = [(mass of powder / powder density) / mill volume)] × (1 / 0.6) (3)

In the secondary grinding process, a 170*150 mm polyethylene-based stirred mill was used (Figure 4b). d_{Al} =3.5-4 mm alumina balls were used as grinding medium (Figure 5b). Dimensional analyzes of the stirred ball mill product were determined using a dry laser sizer (Sympatec GmbH Co., Germany).

The ball mill grinding test conditions are given in Table 2. The ball mill grinding experiments were performed as a batch operation. In micronized grinding plants, it was aimed that the final product has a uniform structure, that is, close to each other in size. Therefore, some calculations are made for the uniform product indicator. The most used ones are given below. As the value of the numerical results obtained from here is lowered, it is assumed that the product has a narrower particle (product) size distribution.

$(d_{90}-d_{10})/d_{50}$ [26, 27, 28] (1)
d_{80}/d_{20} [26, 29] (2)
d_{50}/d_{20} [30] (3)
d_{90}/d_{10} [31] (4)

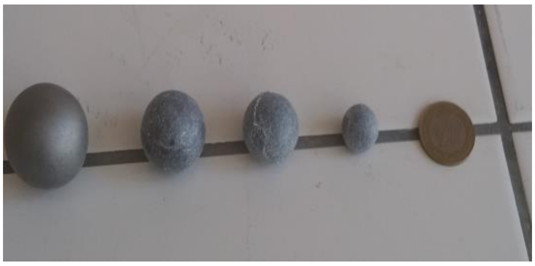
(a)

(b)

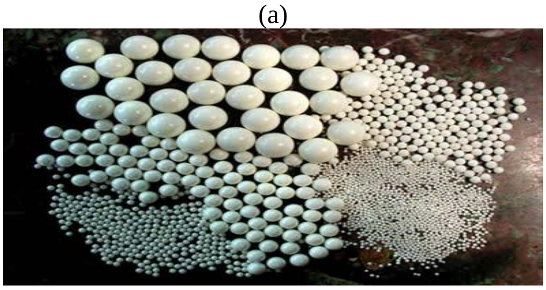
Figure 4. Ball mill (a) and stirred mill (b) used in the study

Table 2. Ball mill grinding test conditions

Parameter	Option
Mill speed (% of N_c)	50
	60
	70
	80
	90
Ball filling ratio (Jb)	0.20
	0.25
	0.30
	0.40
	0.45
Ball size distribution (10-20-30-40 mm, %)	Group I:0-0-50-50
	Group II:0-20-30-50
	Group III:20-20-30-30
	Group IV:30-30-20-20
	Group V:50-50-0-0
Powder filling ratio (fc)	**0.10**
	0.11
	0.12
	0.15
	0.20
Grinding aid ratio (% of powder)	**0**
	0.025
	0.050
	0.075
	0.10
Grinding time (min.)	**15**
	20
	25
	30



(a)



(b)

Figure 5. Balls of four different diameters using ball mill (a) and balls of stirred mill grindings (b)

3. RESULTS AND DISCUSSIONS

In this study, the critical speed of 50%, 60%, 70%, 80% and 90% was used in the ball mill working speeds. The grinding test conditions of mill speed (% of N_c) are given in Table 2. As a result of the experiment, the values given in Figure 6 were reached. As it can be understood from here, the best result was obtained at 90% of the critical speed. At this value, 52.66% of the material passed through a 106 μm sieve. It is thought that the reason for the best results to be obtained at this high speed is the need for a cataract effect in the grinding of talc ore with a high silica content. The speed was high but the ore contained about 63.15% SiO_2.

In this context, the ball fill ratio (Jb) is respectively 0.20 4.5 kg, 0.30 6 kg, 0.35 9 kg, 0.42 12 kg, respectively; 0.45 of 14 kg balls values were studied. The grinding test conditions of ball filling ratio (Jb) are given in Table 2. The results given in Figure 7. As can be understood from these results, it was determined that the best ball fill rate was Jb=0.42, 12 kg ball. It is seen that the rate of passing through a 106 μm sieve of the product obtained at this value is 69.40%.

The size distribution of balls for 1, 2, 3 and 4 cm balls are respectively as %; 0, 0, 50, 50 for the first group, 0, 20, 30, 50 for the second group, 20, 20, 30, 30 for the third group, 30, 30, 20, 20 for the fourth group, 50, 50, 0, 0 for the fifth group, values were used in the optimization. The test was made under Table 2 conditions. As a result of the experiment, the results given in Figure 8 were reached. According to this, the best ball size distribution was obtained in the 4th Group, namely 30% 1 cm, 30% 2 cm, 20% 3 cm, 20% 4 cm. The rate of passing through a 106 μm sieve of the product obtained as a result of grinding was found to be 73.40%.

The most important parameter affecting the capacity and grinding in ball mill grinding is the material fill rate. The operating conditions given in Table 2 were used for grinding talc in a conventional ball mill. In this context, the talc occupancy rate (fc) is respectively; the values of 0.10 for 1 kg, 0.11 for 1.15 kg, 0.12 for 1.250 kg, 0.15 for 3 kg and 0.20 for 4 kg were studied. The results obtain can be seen in Figure 9. It showed that the best talc occupancy rate was fc=0.10 that is 1.0 kg talc ore. The passing rate of the product obtained at this value through a 106 μm sieve was found to be 73.40%.

We know that one of the important parameters in studies to optimize micronized grinding is the assistance to grinding. For this reason, although the milled material has hydrophobic properties, it was desired to observe whether the grinding aid has an effect on the micronized grinding of the talc ore studied. Due to its harmless and water-soluble properties, poly-acrylic acid (CH2CHCOOH) was used as a grinding aid chemical in this study. The test conditions are given in Table 2. As a result of the experiment, Figure 10 was obtained. According to these results, it has been seen that the grinding aid does not have a function in talc grinding.

The grinding time is a critical parameter in fine grinding because of pertaining to the residence time of the material in the mill in conventional ball mills. The grinding time should be selected and studied based on the size of the product to be taken more. The operating conditions given in Table 2 were used in the study of this parameter. In this context, the grinding time was studied as 15, 20, 25 and 30 minutes, respectively. The results given in Figure 11 were reached. According to this, the best grinding time was determined as 30 minutes. In this result, almost all of the material, which is the purpose of grinding talc in the ball mill, is intended to pass through a 106-micron sieve. After 30 minutes of grinding, approximately 100% of the material passed through the said sieve size.

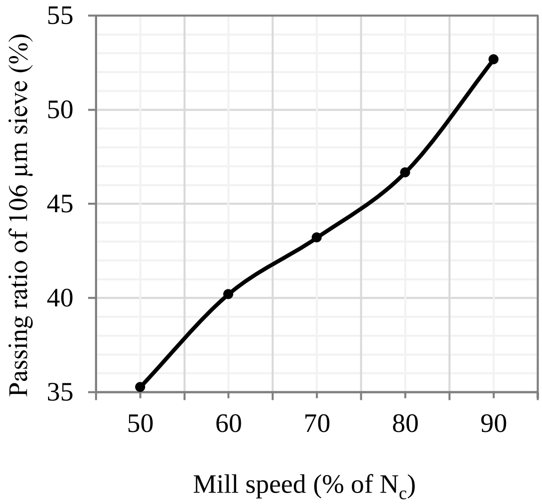


Figure 6. Passing ratios of 106-micron sieve of products obtained in different mill speed

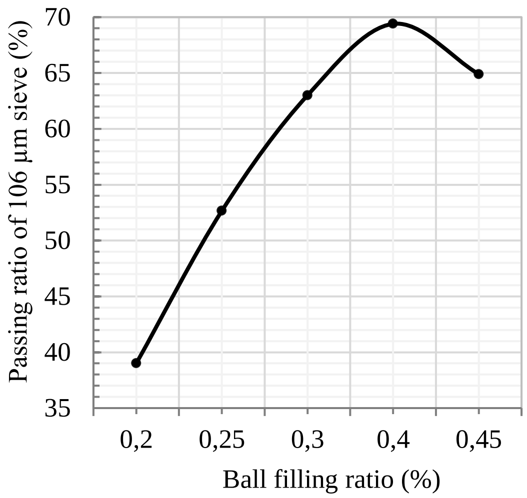


Figure 7. Passing ratios of 106 µm sieve of products obtained in different ball filling ratios

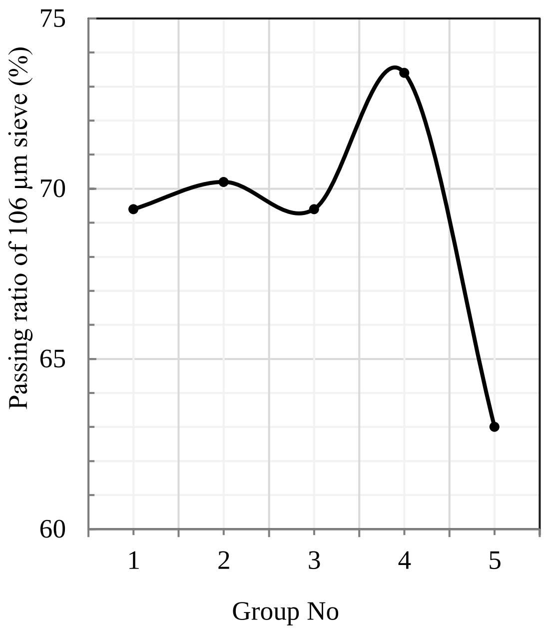


Figure 8. Passing ratios of 106 µm sieve of products obtained in different ball size distribution

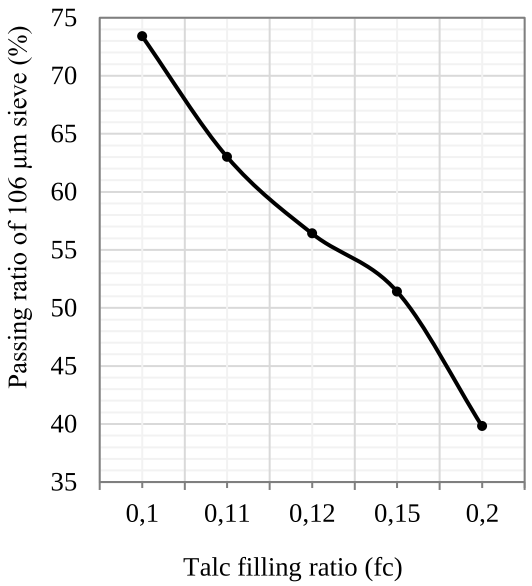


Figure 9. Passing ratios of 106 µm sieve of products obtained in different talc filling ratio

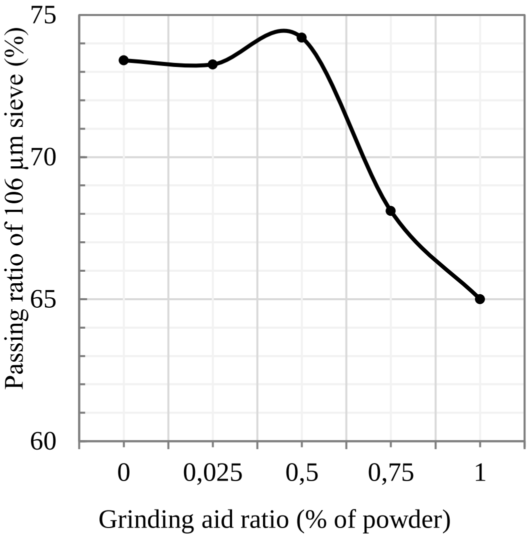


Figure 10. Passing ratios of 106 µm sieve of products obtained in different grinding aid ratio

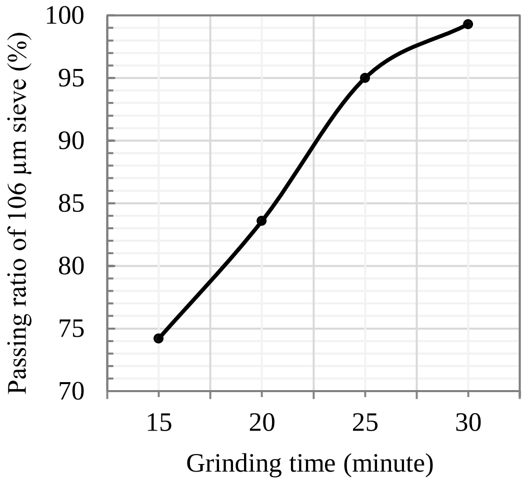


Figure 11. Passing ratios of 106 µm sieve of products obtained in different grinding time

Fine sizes (<100 µm) product obtained at conventional ball mill product was used in the stirred mill. Figure 12 shows cumulative undersize of the fine material. The product had d_{50}=17.92 µm and d_{90}=93.52 µm particle sizes. The powder was then ground in a laboratory dry stirred mill under the conditions specified in Table 3.

The d_{50} value, which is defined as the medium size of industrial raw material products ground to very fine sizes, is very important. The results are provided in Figure 13a and b based on the cumulative undersize and d_{50} values for various specified grinding times (5, 10, 15, 20, 30 and 60 min), respectively. In micronized grinding processes, the concept of d_{50} (median particle size) is commonly used in the evaluation of product fineness. In this study, after 60 minutes grinding time of the fine talc product in the vertical stirred ball mill, the product with a d_{50} value of 1.85 µm microns was reached.

It is necessary to know the total surface areas of the filler minerals used in industrial used. In this study, the total surface area values of ground six talc products are given in Figure 14a. These results were obtained from the Sympatec particle size distribution measuring instrument. Naturally, the highest surface area (14058 cm2/g) was reached after 60 minutes of grinding. The PSD values of the products milled at different times in the vertical mill are presented in Figure 14b. Table 4 shows some specific particle size, SF, and span values of feed material and final products. The PSD of the talc powder at 60 min grinding time was very narrow (span 2.62), with average particle size of 1.85 µm, while more than 90% of particles were less than 5.53 µm.

After crushing process, a flowsheet was developed that can be used in grinding of talc ore to micronize sizes (Figure 15). As can be seen from the figure, a ball mill was used in the primary grinding unit, and its product was fed to the vertical stirred mill to obtain very fine talc ore.

Filo et. al., (1994) [32] studied the grinding of talc ore using tumbling and planetary ball mills. They observed that size reduction of talc samples was observed to predominate in grinding using a moderate energy type tumbling ball mill and the resultant fine particles of ground talc were found to agglomerate with increasing grinding time. Elbendaria et al. (2013) [33] studied wet grinding of talc with a vertical attritor mill and after optimization, they achieved a product size of 12 µm and 3.8 μm, as d_{90} and d_{50} values, after grinding for 180 minutes. Katırcığlu Bayel [34] worked about modeling and optimization of some grinding parameters on dry grinding in stirred media mill using talc ore. According to the study resuls, the optimal conditions for maximizing the breakage rate were obtained at 599 rpm for the mixer speed, 0.07 for the solids ratio, 65.58% for the ball fill ratio, and maximum ball size distribution.

Table 3. Stirred mill grinding conditions

Parameters	Variables
Mill type	Stirred
Mill capacity	3000 ml
Mill material	Polythene
Shaft material	Stainless steel
Material of balls	Alumina
Ball density	3.65 g/cm^3
Diameter of the balls	1-3 mm
Ball mass per milling run	4500 gr
Ball to powder ratio run	9:1
Sample mass	500 gr
Rotation	750 rpm
Grinding time	5,10,15,20,30,60 min.

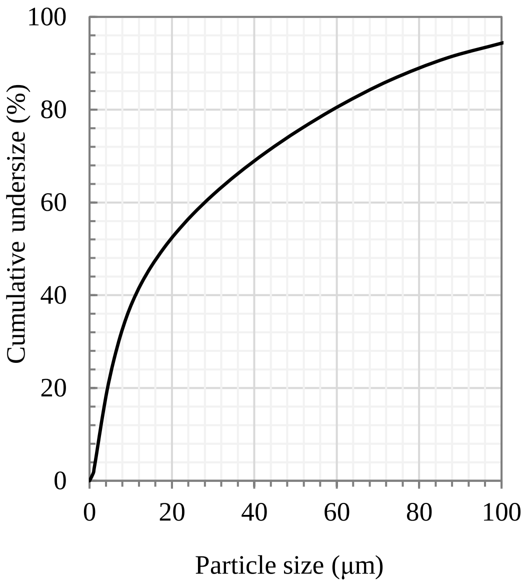


Figure 12. Particle size distribution of the feed powder of stirred mill

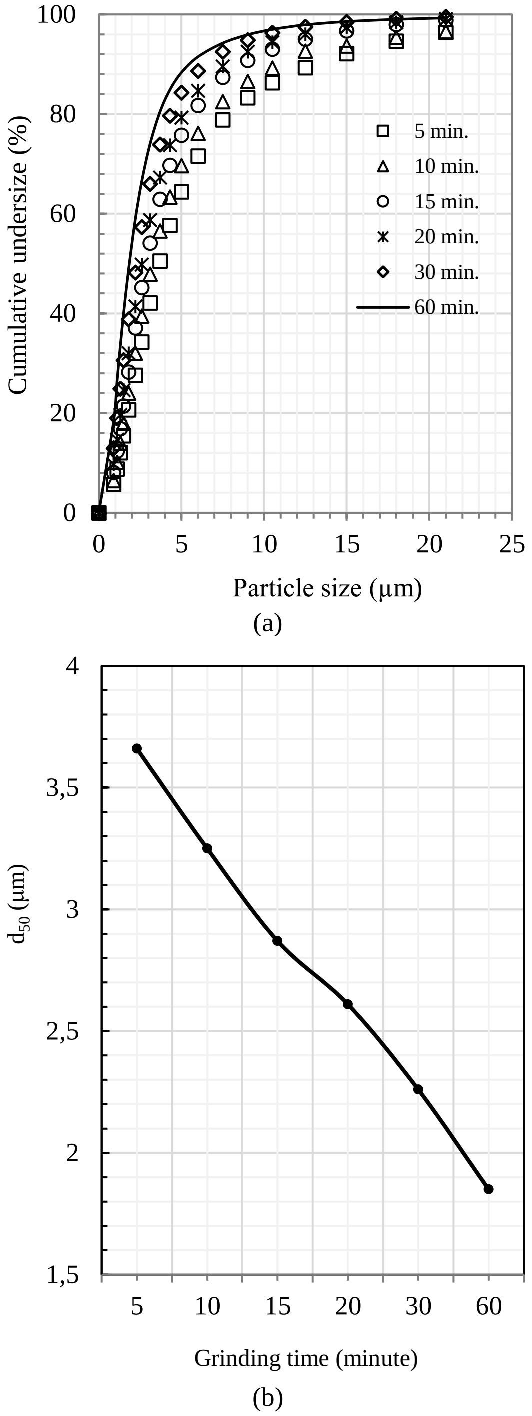


Figure 13. Particle size distribution of the ultrafine ground sample (a), d_{50} values of the ultrafine grinding sample (b)

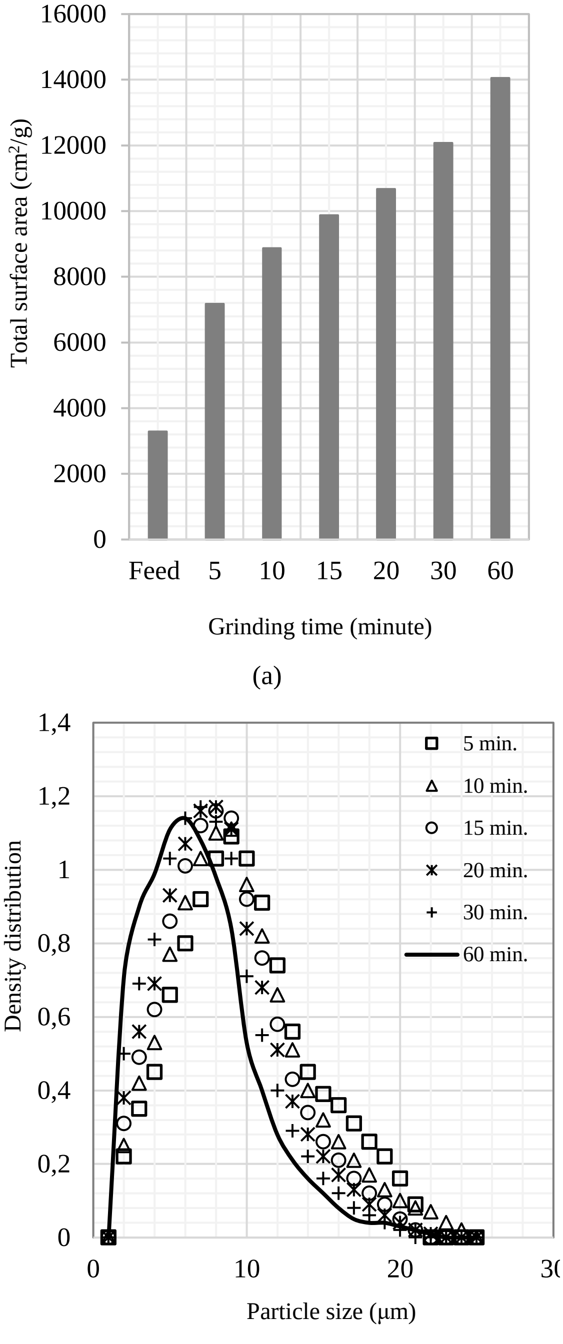


Figure 14. The total surface area values of ultrafine talc products (a) particle density distribution of ground products (b)

Table 4. Some width of PSD values of feed material and products

Grinding time (min)	SF (d_{50}/d_{20})	d_{90}/d_{10}	d_{80}/d_{20}	$[(d_{90}-d_{10})/d_{50})]$
Feed powder	4.17	34.35	14.19	4.52
5	1.74	12.23	4.50	3.67
10	2.03	10.23	4.50	3.10
15	1.94	8.75	4.05	2.67
20	1.95	8.47	4.00	2.61
30	1.97	8.04	3.74	2.63
60	1.76	7.68	3.52	2.60

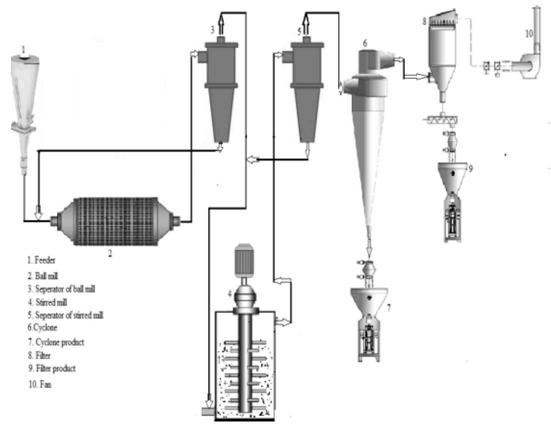


Figure 15. Recommended ultra-fine grinding processing flowsheet of talc ore

4. CONCLUSION

A laboratory scale ball mill+stirred mill combination was used for ultrafine grinding of talc ore. The experiments were carried out firstly, followed by a stirred mill study at the optimum conditions obtained from ball mill.

- The best results obtained at optimum conditions in the ball mill experiments are as follows: 90% of N_c for mill speed, 0.40 for ball filling ratio, 30-30-20-20 for ball size distribution (10-20-30-40 mm), 0.10 for powder filling ratio (f_c), 0 g/t grinding aid ratio and 30 minute for grinding time. A product was obtained with average particle size (d_{50}) of 17.92 µm.

- The fine product was subjected to a regrinding process by a stirred mill. The PSD of the talc powder at 60 min. grinding time was very narrow (SF=1.76, span=2.60), with d_{50}=1.85 µm.

5. ACKNOWLEDGMENTS

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