Parameters affecting wet ultra-fine grinding of talc ore

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Abstract—Fine and ultra-fine grinding have several applications in many industrial fields such as advanced ceramics, porcelain, cement, paper coating, plastic and pigments. The stirred mills are much more efficient for fine grinding and regrinding than conventional tumbling mills. Conventional mills require long retention time and tremendous energy input for micron size production. This work aims at studying the parameters affecting wet ultra-fine grinding of Egyptian talc from Shalatin locality of the Eastern Desert to produce ultra-fine product less than 10 microns. Attritor mill (Union process type 1S) is used to achieve the desired size that is utilized as a filler material for different industrial applications such as paints, plastics, paper coating, and other advanced applications. Crushed talc less than 6630 microns used as a feed. The studied parameters were media size, stirrer speed, solid content in slurry percentage by volume, and media to talc percentage by volume. The results showed that in wet grinding, about 96 % by weight with maximum size reduction d90 and d50 were 12 µm and 3.8 µm at 180 min overall grinding time. Particle size enlargement occurred after the time aforementioned. The scanning electron microscope of ultra-fine grinding showed that distortion of platy structure occurred after 180 min. Therefore, in order to keep the platy structure and crystallinity of talc in order to be used as filler material in different industrial applications, it is recommended not to grind talc for more than this time. The submicron ultra-fine grinding products of Egyptian talc could be used in different industrial filler applications such as paints, ceramic and paper coating.

Index Terms— Ultra-Fine grinding, Stirred media mills, Attritor mill, Talc ore, Ultrafine talc properties, Ultrafine talc applications, and Functional fillers.

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1 INTRODUCTION

In recent years ultrafine grinding in the submicron range become essential due to the development of the new functional materials such as new ceramics and electronic materials for various industrial fields [1, 2]. The most famous ultrafine grinders are jet mill and agitated mills such as stirred media mills [1]. Jet mill first developed for producing particles diameter less than 100 microns and having a high degree of purity. Jet mills are extensively used in the pharmaceutical industry due to their ability to produce fine particles without the wear of mechanical parts [3]. Although jet mill has several advantages but it is still an energy intensive process as only 2-5%of the energy supplied is used to create new surfaces Mebtoul et al [4]. Nakach et al [5] disclosed the opposed fluidized bed jet mill disadvantages such as poor particle size and its distribution, expensive, low capacity, mechanically complex and requires regular maintenance. Another type of mills used to produce ultra-fine particle size, are the stirred media mills, which are similar to ball mills except they contain an agitator, which supplies the necessary energy to the grounded particles instead of rotation of the vessel. The agitator allows the media to collide with a muchhigher force than is possible in the conventional ball mill [3]. The importance of stirred media mills increases steadily, because of an increasing demand for ultra-fine particles. In many cases, the grinding by a stirred ball mill (attritor mill) to submicron range has been achieved commercially. Because of their easy operation, simple construction, high grinding rate and low energy consumption compared with the other fine grinding machines, stirred ball mills recently have received more and more attention [1, 2]. Stirred mill technology was applied in the ultra-fine grinding process in the pigment industry and it has wide applications in other industrial fields such as pharmaceuticals, ceramics, and chemical industries [6, 7]. It consists of a water cooled grinding tank, charged with media and agitated with a central impeller. Impact and attrition grinding occurs by collision of the media with tank walls and collision of media with itself [8]. In stirred mill the power providing (input) is directly used for driving of agitating media which is the key of grinding efficiency. The most advantage is that the grinding does not take place against the tank walls. So it enables little wear on the walls, and leading to longer service life of the vessel. Attritor mill could be used in wet or dry process. In wet grinding the impact action is created by the constant grinding media impinging due to its irregular movement. Shearing action is created as a result of spinning of the media in different rotation due to its random movement and, therefore, exerting shearing forces

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on the adjacent slurry. As a result, both liquid shearing force and media impact force are present. Such combined shearing and impact forces results in a great size reduction as well as good dispersion. Meanwhile in dry milling the process is achieved by expanded moving bed of grinding media. This condition is described as kinematic porosity figure 1. The particles are subjected to various forces such as impact, rotational, tumbling, and shear; therefore, micron range fine powders can be easily obtained. Additionally, a combination of these forces creates more spherical particles than other impact-type milling equipment [9]

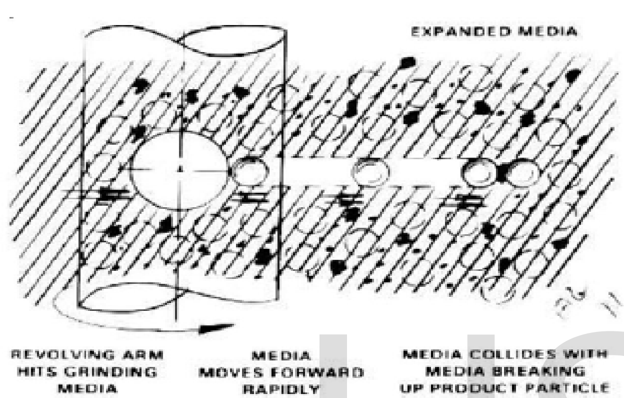

Fig. 1: Media collisions with agitator arms and ground particles

There are two different classes of stirred mills that can be referred to as slow milling speed or high speed. The first class includes tower mill or Vertimill and conventional pin mills where a relatively slow impeller speed and coarser media size results in the fluid having a limited effect on the interaction of the media with itself, this class is effective at coarse and hard feed grinding. The second class includes the Netzsch / IsaMill and the stirred media deteritor. This class is more effective for ultrafine milling where the impeller speed is high enough to be effective to fluidize the media so that it can take on the flow pattern of a viscous fluid [10].

2. EXPERIMENTAL TECHNIQUE

2.1. Materials

Talc sample representing Bir Meseh, South of Shalatin locality of the Eastern Desert, has been supplied by El-Nasr Mining Co.

2.2. Talc Feed Preparation for Ultra- FINE GRINDING

Talc ore has been crushed in closed circuit using "Denver" primary jaw crusher with 6.63 mm screen. Representative batches, have been prepared by successive coning and quartering methods. A representative sample has been finely ground using grinding mortar to less than 200 meshes for XRD and chemical analysis.

2.3. Mineralogical, Morphological and Chemical Analyses

Identification of the mineral composition of the considered samples has been conducted by X-ray diffraction (XRD, Bruker axs D8 Advance, Germany). The morphology of talc ground product was measured using scan electron microscope (FESEM; QUANTAFEG 250, Holland). Complete chemical analysis of the samples was conducted by Panalytical XRF (Model advanced Axios, Netherlands)

2.4. Ultra-fine grinding Experiments:

Grinding of talc ore has been conducted in a vertical laboratory attritor mill (Union process type 1S). It consists of a stainless steel vessel of 9.5 liters' capacity. It has a stainless steel shaft fitted with five arms with maximum speed 560 r.p.m. The tank is jacketed for cooling and it includes a bottom discharge valve, and bar grid. The crushed talc less than 6630 microns was put in the tank contains alumina balls and water at low attritor speed and after finishing, the tank was covered and stirring speed was adjusted, and grinding time was recorded. The studied parameters were grinding time, media size, stirrer speed, solid / slurry percentage by volume, and media to talc percentage by volume. At the end of each test, the attritor mill was discharged and sieved on 45-micron laboratory screen. The oversize fraction was dried, weighed, and its particle size was determined using a series of different sieve sizes. The undersize fraction was dried, weighed and its particle size was measured using COR-2000 LASER PARTICLE SIZE ANALYZER. A regrinding process for the products was carried out to achieve the desired size.

3. RESULTS AND DISCUSSION

3.1. Mineralogical and Chemical Analyses

Figure 2 shows the X-ray diffraction of the talc ore sample. It has been shown that the sample composed of talc mineral $Mg_3(OH)_2Si_4O_{10}$ associated with Chlorite $(Mg.\ Fe)_5(Al, Si)_5O_{10}(OH)_8$, Calcite $CaCO_3$, Quartz SiO_2, and Anatase TiO_2.

Table 1 shows the results of complete chemical analysis of the talc sample using X-ray fluorescence analysis technique (XRF). It has been seen that the major species of talc are 46 % SiO_2, 25 % MgO, 9 % CaO and 2.2 % Fe_2O_3.

3.2. Feed Preparation for Ultra-fine grinding of Talc

Figure 3 shows the size distribution of crushed talc in closed circuit with 6.63 mm screen. It has been shown that the d80 and d50 were 4442 µm and 2400 µm respectively. It was used as a feed in wet grinding process.

TABLE1

CHEMICAL ANALYSIS OF TALC SAMPLE

Species	%	Species	%
SiO_2	46.329	P_2O_5	0.106
MgO	25.259	MnO	0.100
CaO	9.480	CuO	0.007
Al_2O_3	4.390	ZnO	0.008
Fe_2O_3	2.207	SrO	0.014
TiO_2	0.267	ZrO	0.008
SO_3	0.101	F^-	0.193
Na_2O	0.116	L.O.I	11.4
K_2O	0.015	**Total**	**100**

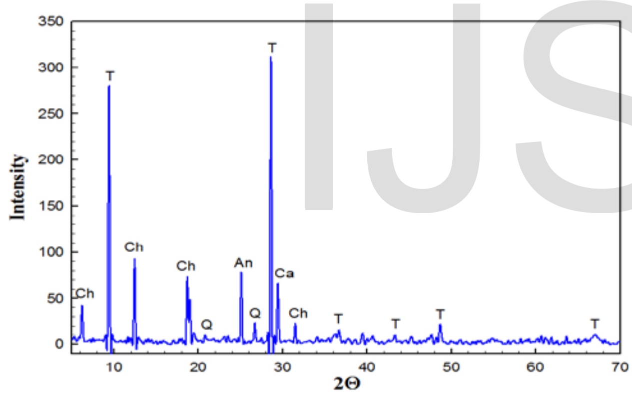

Fig2: XRD of talc sample

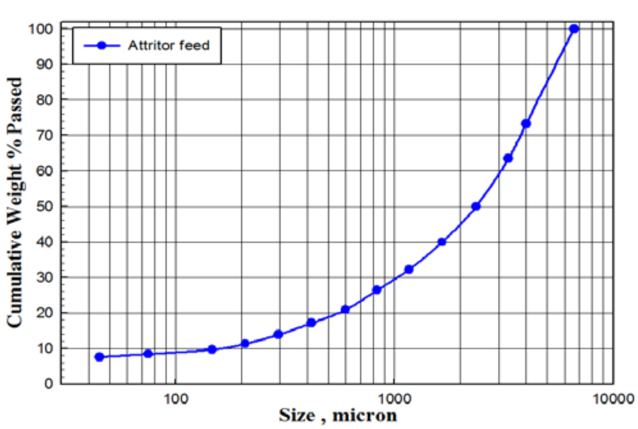

Fig3: Size distribution of crushed talc

3.3. WET ULTRA-FINE GRINDING OF CRUSHED TALC

Two steps of ultra-fine grinding were performed in order to achieve the desired size (<10 micron). The first grinding step includes grinding coarse feed less than 6630 microns. The second grinding step as regrinding process was carried out on the fraction less than 45 micron. The grinding parameters in the two steps were media size, stirrer speed, and solid content in slurry percentage by volume and media to talc percentage by volume.

3.3.1. EFFECT OF MEDIA SIZE

The grinding of talc was carried out by using two different sizes of balls (4 mm and 10 mm), at 60 % solid content in slurry, 175 r.p.m, and different grinding times. Figure 4 shows the results of grinding using (a) (3.6 Kg) 4 mm & (b) (3.6 Kg) 10 mm alumina balls. It has been found out that the efficiency of grinding using 10 mm ball size is better than using 4 mm balls especially in short time grinding. This may be due to that the initial feed size is larger than 6000 microns and it needs larger ball sizes to break the larger sizes. Jankovic [11] also reported that coarser grinding media were more efficient for the coarser feed sizes but less effective with the finer feeds. Increasing the grinding time increases the percentage of fines rapidly at short grinding time (15 min). However, by increasing the grinding time has less significant effect, i.e. 55% passed at 15 min, compared to 72% at 60 min. The above observation can be explained by the fact that as the particle size decreases, the number of particles increases, and the number of contact points between balls and particles decreases. Thus, the ratio of ball to particle decreases and the grinding rate decreases.

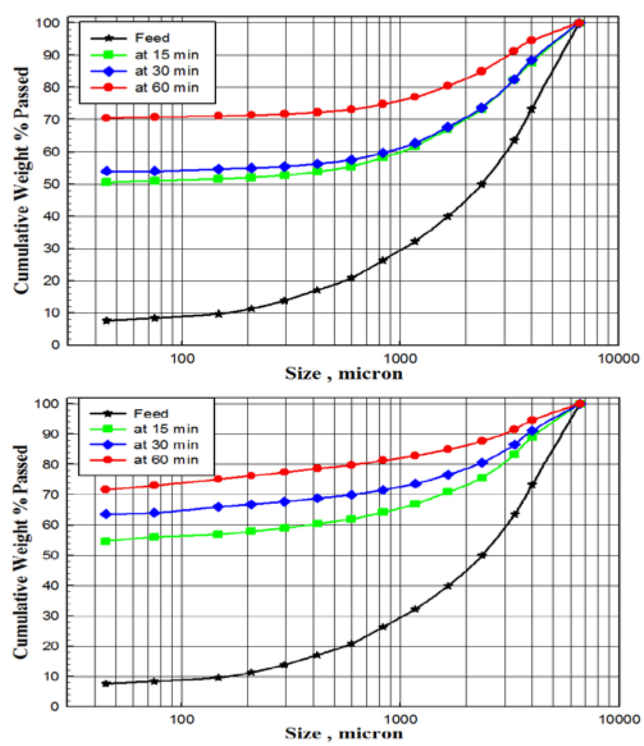

Fig. 4 : Size distribution of product ground at different time
(a) By using 4 mm alumina balls (b) By using 10 mm alumina balls

3.3.2. Effect of stirrer speed (r.p.m)

The effect of stirrer speed has been investigated using 10 mm balls (3.6 kg), 60 % solid content, and 60 min grinding time. As Figure 5 illustrates, by increasing the stirrer speed, the grinding rate increases, where the percentage passed from 45 micron increased from 71% to 80% at 175 and 385 r.p.m respectively. By increasing the rotational speed, the impact energy and the number of impacts of ball–ball, ball–container, ball–powder, and powder–powder was increased [12, 13].

The results have also been interpreted according to [11] who reported that higher stirrer speed and coarser media had a positive and stronger effect than fine media on grinding efficiency.

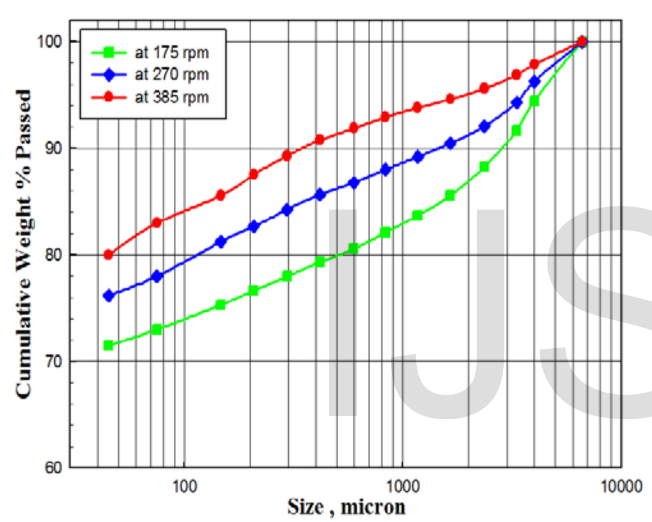

Fig5: Size distribution of products ground at different stirrer speed

3.3.3. Effect of Slurry Solid Content

Figure 6 (a, b) illustrate the results of grinding with different solid in slurry content at stirrer speed 385 r.p.m during 60 min grinding. It has been noticed that at solid in slurry content ranging from 30 up to 50 %, more than 92 % by weight was less than 45 microns, having a peak of 96 % at 40 % solids. Meanwhile, increasing solid content above 50 % decreases the weight percentageof fraction less than 45 microns (80% at 60% solids). Increasing the slurry density will improve mill performance up to a certain limit above which a decrease in the mill efficiency will be expected together with slurry flow problems due to increased viscosity, [11].
The d90 and d50 for fraction less than 45 at 40 % solid / slurry were 33.16 micron and 10.5 micron respectively Figure

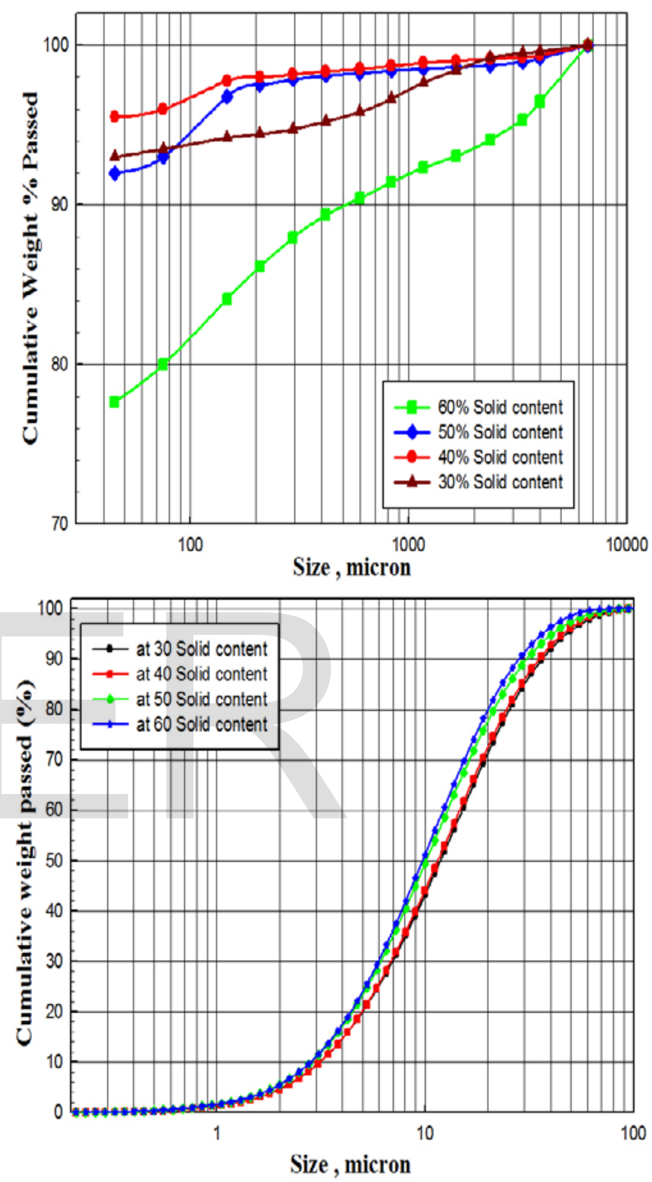

Figure 6: Size distribution at different solid content in slurry percentage by volume (a) Ground productFraction less than 4

3.3.4. Wet regrinding in attritor mill

To obtain fraction < 10 micron, the fines (-45 µm) has been reground in a series of experiments to study the effect of slurry solid content and ratio of media to talc volume at 460 r.p.m and different grinding times.

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3.3.4.1. Effect of slurry solid content

The effect of slurry solid content on the grinding performance has been studied at constant media 30% from total mill volume, stirrer speed 460, and different times. The feed d90 and d50 were 35 µm and 11 µm.

Figures 7 (a, b) show the product size analysis at different slurry solid content at 30 min and 90 min, respectively. It has been shown that; at different solid percent and 30 min, the grinding rate was almost similar, where d50 approximately 9.8 micron, figure (7a). By increasing the grinding time above 30 min, the grinding rate increased by decreasing the slurry solid content, and d90 and d50 decreased from 25.5 and 9.2 µm at 40% solid to 20.3 and 7.5 µm at 20 % solid at 90 min, respectively figure (7b).

Figure 8 (a, b) represents the 3D relationship between the slurry solid content, grinding time and d50, d90, depicting an optimum size reduction at 20% solid content. It has been shown that grinding at high solid content (40%) exhibits less effect on particle size reduction. Furthermore, decreasing the solid content from 25 to 10% had no effect on grinding performance. It has been shown that the best fitting of curve in figures followed Paraboloid model with mathematical model equations 1 and 2.

$$d_{90} = 34.0008 - 0.3017x - 0.1582y + 0.0075x^2 + 0.0006y^2 \quad (1)$$

$$d_{50} = 10.8345 - 0.0288x - 0.0382y + 0.0012x^2 + 6.6667E-005y^2 \quad (2)$$

Where x is slurry solid content and y is the grinding time (min) and R2 for d90 and d50 were 0.9243, and 0.8800, respectively.

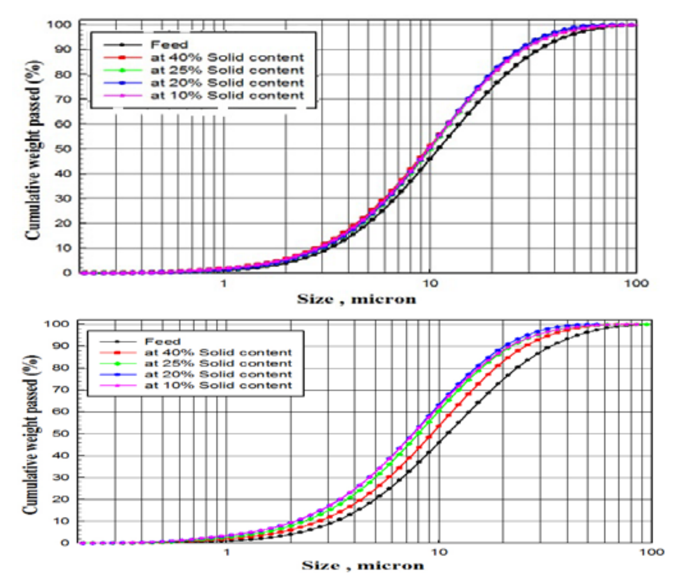

Fig. 7: Size analysis of products at different slurry solid content
(a) during 30 min grinding (b) during 90 min grinding

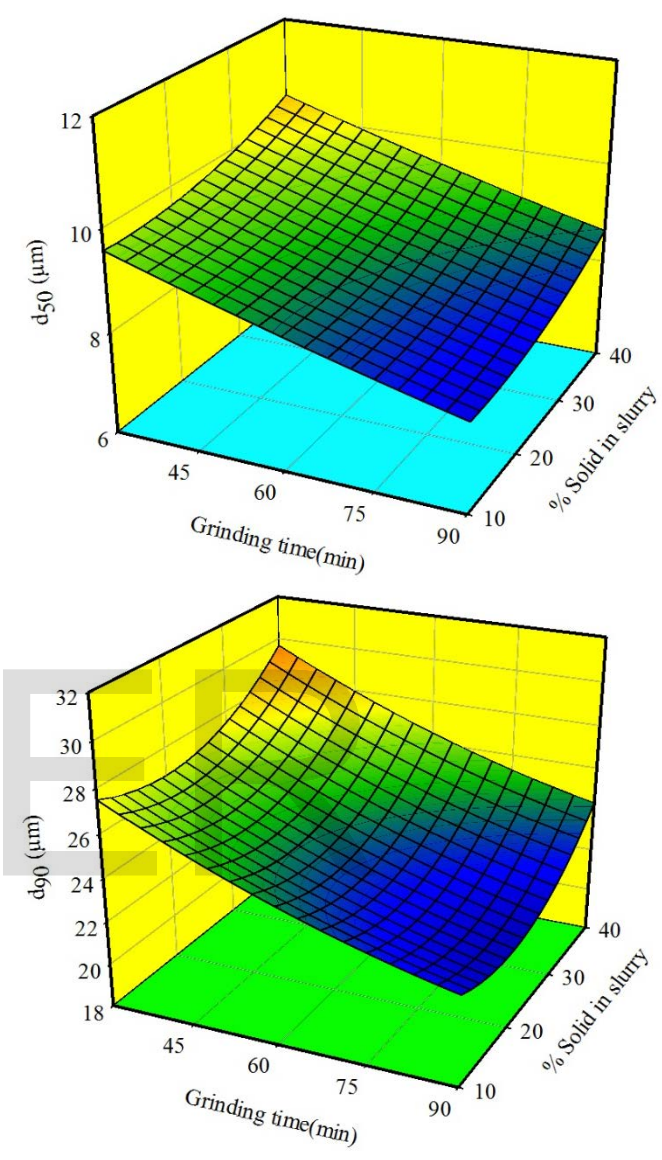

Fig. 8: The relationship between solid in slurry (%) and grinding time
(a) On d50 (b) On d90

3.3.4.2. Effect of ratio of media to talc

The effect of media percentage on size reduction has been investigated at 20% solid and 460 rpm.

Figure 9 (a, b) shows size analysis of ground product at different grinding times, 20% slurry solid content and different media percentage, where the grinding rate has been found to rise sharply by increasing the media %. Increasing the grinding time up to 240 min at 83.5% media, raised the fines formed, and d90 and d50 reached their maxima (15.3 and 5.3 µm); whereas, after 240 min, the fines were nearly the same and the

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size enlargement took place, figure (9a). Meanwhile, increasing the media % to 90% resulted in a faster agglomeration rate due to Inter-particle forces, such as van der Waals forces [14, 15].

Figure 10 (a, b) shows the interaction between media percentage and grinding time on d50, d90. A glance at the figure reveals that the grinding rate has increased by four folds while increasing the media % from 83% to 90%. Also shown is that the minimum d90 and d50 were 12 µm and 3.8 µm at 88% media %. Therefore, the 88% media is the optimum percentage in talc wet grinding. This may be due to by increasing the ball percentage above the above mentioned value, the total mass of the system increases, hence the dissipated energy also increased so the grinding performance decreased. The fitting of curves followed Paraboloid model with mathematical model equations.3 and 4.

$$d_{90} = 1155.7548 - 25.1941x - 0.2585y + 0.1407x^2 + 0.0011y^2 \quad (3)$$

$$d_{50} = 196.8576 - 3.9007x - 0.0869y + 0.0202x^2 + 0.0003y^2 \quad (4)$$

Where x is media percentage (%) and y is the grinding time (min) R2 for d90 and d50 were 0.9251, and 0.9825 respectively.

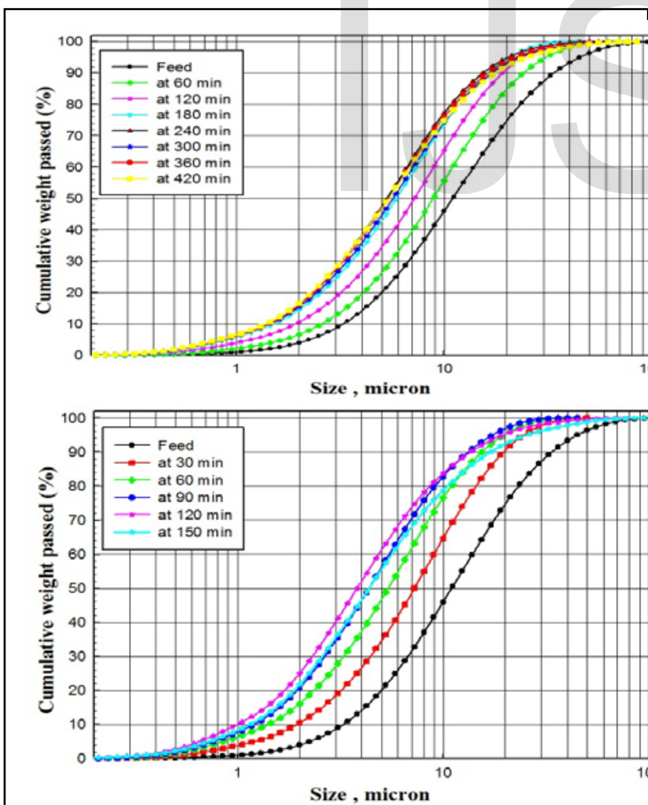


Fig.9: Size analysis of products ground with different media percentage and different grinding time (a) at 83.5 % media(5:1 media/talc ratio) (b) at 90 % media(9:1 media/talc ratio)

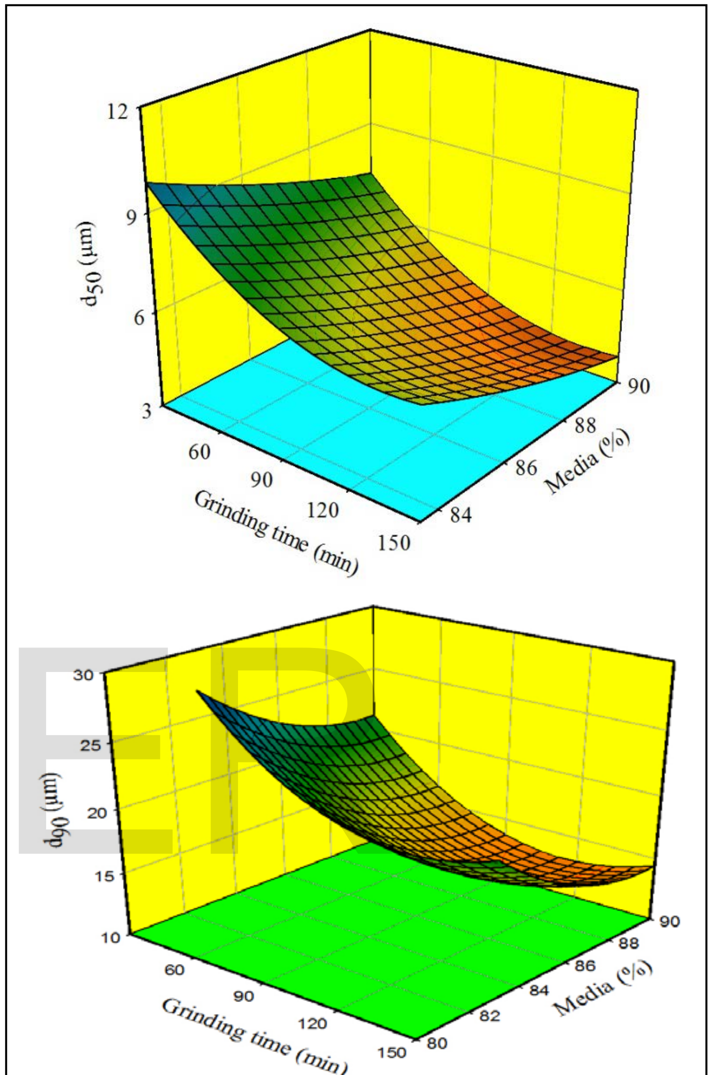


Fig.10: Relationship between media percentage and grinding time.

(a) On d50 (b) On d90

3.3.4.3. Effect of Wet Grinding on Talc Morphology and Crystal Structure

Talc is very sensitive to grinding. The layers are bonded together only by weak van der Waals forces, resulting in a low Moh's hardness Aglietti [16]

Figures 11 and 12 show XRD and SEM analyses for the first and second (regrinding) steps, It has been shown that the intensity of peak decreases with increasing the time of grinding and distortion of platy structure occurred after grinding and regrinding of talc more than 2 hours, figure 12. Table 2 also shows the values of the crystal sizes (on the basis of the Scherrer formula) and its corresponding (d spacing) during grinding and regrinding processes. It has been shown that crystal size decreases with increasing the grinding time. It decreased from 143 in the feed to 59 after four hours at (2 □) 28o.

In order to keep the platy structure and crystal size of talc to be used as filler in different industrial applications such as paints and plastics, it is recommended not to grind talc above 3 hours.

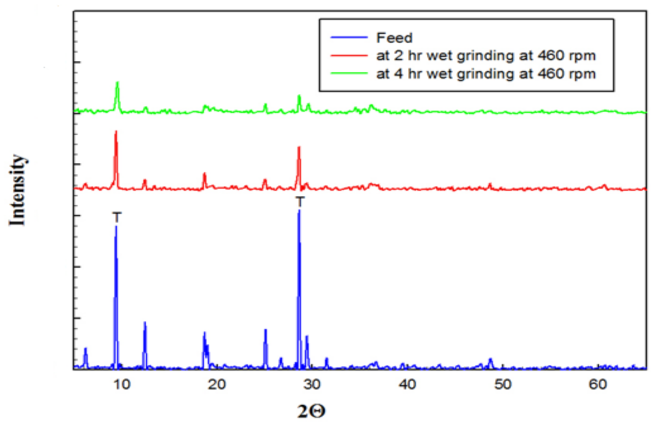
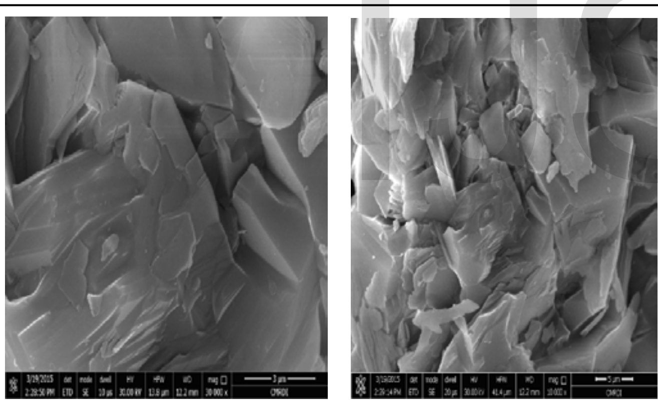
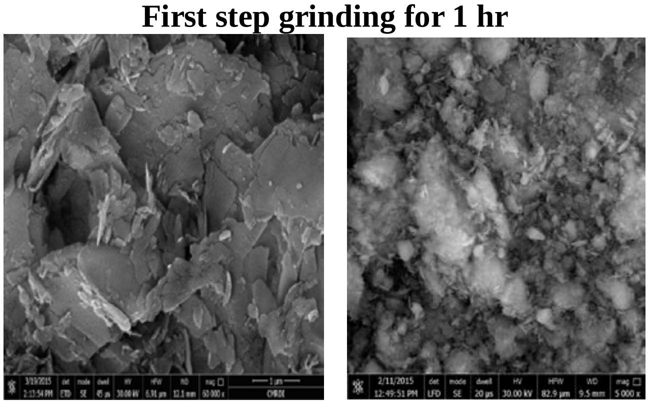


Figure 11: XRD of talc wet grinding for different time at 460 rpm



First step grinding for 1 hr



Second step (regrinding) for 2 hr Second step (regrinding) for 4 hr

TABLE 2

Change in crystal size with time during wet grinding

Total grinding time	Angle (2θ)	d value (A)	Crystal size (nm)
0	9.46	9.33	118.7
	28.6	3.11	143
3 hr	9.40	9.39	59.4
	28.59	3.11	85.1
5 hr	9.53	9.26	58.9
	28.7	3.107	59

4.CONCLUSIONS

1- In wet grinding of coarse feed size (less than 6630µm), higher grinding rate was proven to be obtained with the larger media size, the higher impeller rotational speed. Increasing solid content above 50% has a great effect on decreasing the grinding rate and efficiency due to viscosity increase. About 96% by weight with d90 and d50 33 and 11 µm was obtained at 385 r.p.m, 40% solid in slurry, and 60 min grinding time by using 10 mm balls as grinding media.

2- In wet regrinding, the particle size sharply decreased by decreasing solid content in the slurry up to 20 % and increasing the ratio of media to talc up to 88%. The maximum size reduction obtained at 460 r.p.m, 20 % solid in slurry by volume, 7.5:1 media to talc volume (88 % media), and 120 min grinding with 4 mm balls, where d90 and d50 are 12 µm, 3.8 µm compared with 33 and 11 µm of feed.

3- In long wet grinding time and higher impeller speed, the distortion of talc platy structure and crystallinity decreased sharply by grinding over three hours at 460 rpm. Therefore, in order to keep the platy structure of talc to be used as filler material in different industrial applications such as paints and plastics, it is recommended not to grind talc more than the mentioned time.

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