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THE SPECIFIC SURFACE AREA (S) OF COARSE PARTICLES DURING THE COMPRESSIBILITY PROCESS

H.A.M. Al-Jewaree, A.F. Srrat and E.A. Basha

Department of Mechanical Eng.,Institute of Polytechnic – Al-Zaweyah
Tripoli - Libya, P.O. Box 13750
Email: drhaaljewary@yahoo.com

ABSTRACT

The compaction apparatus is used in this study to measure the specific surface area of coarse particle during the compressibility process for approaching to the real grinding of the particles in the ball mill in order to be used for design purposes. The apparatus consist of two punches and a large die which a single punch pressing. The increments of specific surface area (ΔS) and the size reduction ($\overline{\Delta X}$) are measured because it gives an indication to the grinding product of the coarse particles of white cement clinker. The effect of the compaction mass, particles size, and compaction applied pressure and compaction velocity are studied. Results indicated that the low compaction speed, high applied pressure, decrease the weight of a compacted material and those larger particles give a high specific area of coarse particle during the compressibility process.

1. INTRODUCTION

Specific surface area (S) is one of the most commonly used properties to describe the general product in a ball mill.

Compressibility is the ability- of the material to reduce the volume under the pressure; this nearly what happens in the crushing and grinding of the coarse particle inside the ball mill. Therefore, it is used a coarse particles of white cement clinker and these particles need more than 100 kN to approach the compaction condition. While maximum capacity is 100 kN for our Instron machine.

The compression of powdered or granular material into cohesive mass during the formation of a compact is widely used. As pressure is applied, re-arrangement of powder particles takes place, within die, so that large void are filled and interparticles friction may be sufficient to cause fragmentation of the weaker particles. Further increase in pressure is believed to cause elastic and plastic deformation of the particles which also cause a fragmentation of the some primary particles. Therefore considered that if particles of known size are compressed and then allowed to disintegrate, evidence of fragmentation or interparticles bonding might be obtained.

Size reduction is an important step in many processes by which raw materials are converted into final products. It may be the first operation in a chemical process or the last step before product packaging. Examples may be taken as; the preliminary grinding of phosphate rock in phosphoric acid-production, and the final grinding of clinker in the manufacture of cement. In chemical industry, size reduction is usually carried out to increase the surface area because in most reactions involving solid particles, the rate is directly proportional to the area of contact with the second phase.

2. MATERIALS AND METHODS

Using a laboratory sieving machine (Retsch Ltd, UK) white cement clinker manufactured by Al-Khomes* cement industry from the following raw materials, 10 % sand, 10% clay and 80% limestone is classified into (-1100+900 µm), (-2000+1800 µm), (-2500+2100 µm), (-2800+2500 µm), (-3550+3350 µm) and (-4500+4000 µm). These fractions are the normal sizes of narrow range as follow; 1000, 1900, 2300, 2650, 3450 and 4250 µm. Compression is produced using a hydraulic universal testing machine (Model 1190, Instron Ltd, High Wycombe, U.K) fitted with 67.2mm stainless steel die diameter. It is decided to use hardened steel (55) for the punch and die cylinder to withstand the large applied-pressure and abrasion. By honing the die-wall with a fine stone, the groves in the surface had a depth of less than 0.5µm and 0.6µm, at the end of the experimentation. After every compaction measurement the die is demounted, and if damage to the die wall is perceptible to the naked eye, the die is honed, honing oil removed by rinsing with methyl ethyl ketone (MEK) followed by ultrasonic cleaned in ethanol and acetone in turn. The clearance between the die-wall and the punches is 0.2mm in the majority of tests.

3. RESULTS AND DISCUSSION

It is possible to examine the grinding of the product of white cement clinker by compression process, if the size reduction ($\overline{\Delta X}$) during compressibility process was measured. This size reduction ($\overline{\Delta X}$) in the coarse particles is calculated from the following formula:

$$\overline{\Delta X} = \overline{X2} - \overline{X1} \tag{1}$$

Where:

$\overline{X1}$: The mean particles size diameter after compression process.

$\overline{X2}$: The mean particles size diameter before compression process.

Both mean particles diameters ($\overline{X1}$ and $\overline{X2}$) are calculated from the moment of the sum of all the elementary areas of thickness (dx) about ordinate equals the sum of all the moments:

$$\overline{X} = \frac{\sum Xi \cdot \Delta Q}{\sum \Delta Q} \tag{2}$$

Where:

Xi is the average size and ΔQ is the weight percentage in range (Alien, 1981) or the cumulative weight percentage.

When compaction pressure is applied to the top punch a resisting pressure builds up in the powder or other materials, especially in the zone where the face of the moving top punch meets the wall of the die. The total resistance (Tr) has been found an important factor to cause a size reduction and the increment of the specific surface area. The total resistance of a compacted material is consisted of:

a) Structure of the compacted material resistance (Sr).

b) Friction resistance (Fr), this include the interparticles and particle-die wall frictions.

c) ir- entrapped resistance (Ar).

The best term explains the specific area(S) of coarse particles, which is calculated from the following formula (Heywood(1947) and Allen (1981):

$$\mathbf{S} = \frac{600}{\rho s \, \psi} \sum \frac{\Delta Qi}{Xi} \tag{3}$$

Where:

S is the specific surface area (cm^2/gm),

ρs is the solid density of cement clinker (3 gm/cm^3),

ψ is the sphericity of particles which was defined by Allen (1981)

$$\psi = \frac{MinimumParticleDiameter}{MaximumParticleDiameter} \tag{4}$$

ΔQ is the weight fraction retained on sieve of size i

Xi is the average particle size (μm).

For calculation of the increment of specific surface area (ΔS), the following formula is used:

$$\Delta S = S_2 - S_1 \tag{5}$$

Where

S_1 is the specific surface area before the compression process, and

S_2 is the specific surface area after the compression process (cm^2/gm).

Various compaction parameters are examined to find the success of this process to causing the size reduction and to approach the total resistance which effects the grinding of the coarse particles in the ball mill. These compaction parameters are studies separately and as shown respectively:

3.1 Weight of a Compacted Material

The compression of white cement clinker, feed size fraction is 2650μm and 4250μm, have been conducted in a large die (see Fig. 1) under constant applied pressure (25.375 MPa) and constant compaction speed (0.5 cm/min.). The chosen weight in this study comprises a single bed [0.25 layers (L), 0.5L, 0.75L and 1L] and packed bed (2L, 3L and 4L). The weights are multiplied by the number of the layers.

The cumulative weight percentage (Δ Q) are calculated for each weight of fraction size and plotted versus the particle size for each fraction using the highly recommended method suggested by Hukki (1975), Abdul-Wahab (1990) and Limwong (2004). This method includes the plotting of the overall cumulative weight versus the size distribution of a cumulated product on log-log scale and thus producing smooth curves. The curves are shown in Figs. 2--5. These figures show the distribution curve for the compaction product of feed fraction 2650 μm for various weight of a compacted material and compaction applied force (P) from 30kN to 90kN, these curves are the same for another size particle.

In Figs.2 & 3, it are shown that the distribution curve of 0.5L and one layer (1L) single bed is a smooth convex curve, which is due to absence of the effect of interparticles friction force and the die-wall friction force. But, Figs 4,5 and 6 illustrate the distribution curves of many (2L,3L and 4L) layers packed bed, while the particles friction began to effect on the compaction process. Therefore, a slightly scattering on the convex curve happened. In the compression of cement clinker as layers, the distribution curve are largely scattered on the smooth convex curve as shown in Figs. (4-6). Thus scattering, because, of the friction forces began to influence strongly the compaction process.

But in figure (7) represents how the increment of specific surface area (ΔS) of coarse particles varies from a single bed to a packed bed for feed sizes (4000-4500 μm). However, the (ΔS) increase with the decreasing the weight of compacted material and increase with applied forces (P) as concluded in table 1 and shown as Fig. (7). Actually, at the single bed (one layer) the particles fragmentation easily because of the total pressure applied will disturbed to the all particles equally in the die and all friction resistance with air-entrapped resistance are small than packed (1-4 layers), so, this will leads a high fragmentation to the particles and lead to high specific surface area (S2) as shown in figure (8). The results for sizes (4000-4500 μm) of this section are tabulated in table l.

Table 1: The result of different weight of size (4000-4500 μm) to the increment of specific surface area(ΔS cm^2/gm) during the compressibility process. (S_1 =6.7 cm^2/gm)

Weights (gm)	*ΔS (P=30 kN)*	*ΔS (P=60 kN)*	*ΔS (P=90 kN)*
3.5 (0.25L)	51.54	62.99	87.83
7 (0.5L)	63.68	53.017	65.265
10.5 (0.75L)	21.75	33.81	38.847
14 (1L)	13.03	23.785	28.814
28 (2L)	9.572	15.517	19.796
42 (3L)	9.185	14.622	16.884
56 (4L)	**5.995**	**10.753**	**13.774**

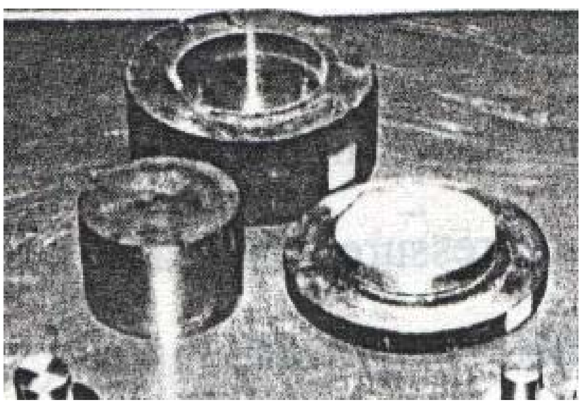

Fig 1: Die assemble

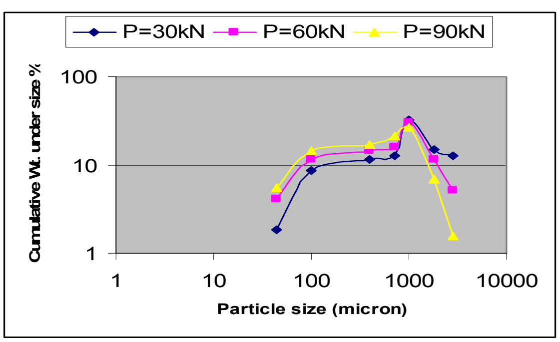

Fig 2: Size distribution of cement clinker for different applied force (P) and feed size (-2800+2500 μm) of 0.5L (single bed)

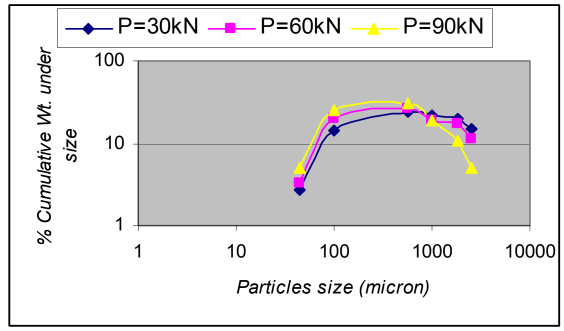

Fig 3: Size distribution of cement clinker for different compaction applied force (P) to the feed size (-2800+2500 μm) of 1 Layer single bed.

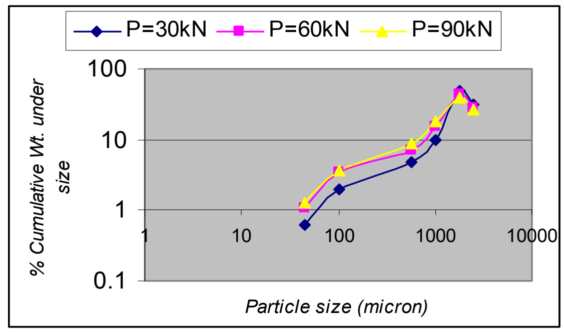

Fig 4: Size distribution of cement clinker for different compaction applied force (P) to the feed size (-2800+2500 μm) of 2 Layers packed bed.

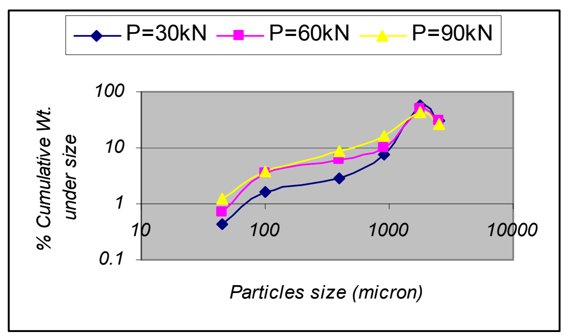

Fig 5: Size distribution of cement clinker for different compaction applied force (P) to the feed size (-2800+2500 μm) of 3 Layers packed bed.

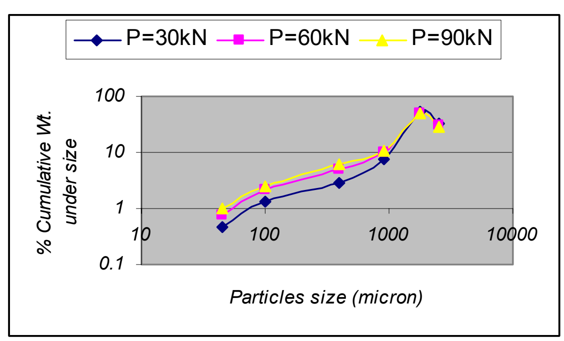

Fig 6: Size distribution of cement clinker for different compaction applied force (P) to the feed size (-2800+2500 μm) of 4 Layers packed bed.

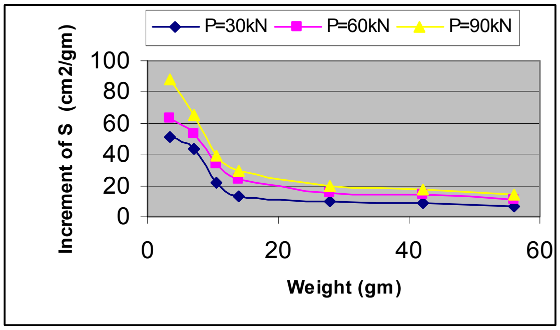

Figure7: Effect the weight of compressibility process on specific surface area (ΔS) of coarse particles at different applied forces (P), for feed size (-4500+4000 µm).

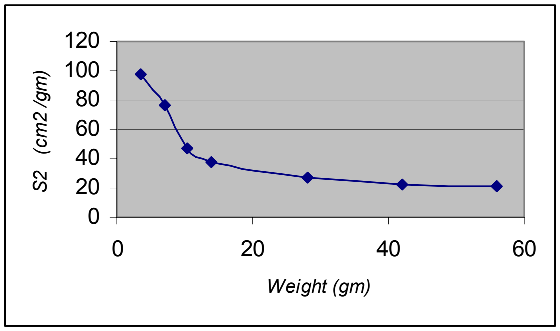

Fig 8: The variation of specific surface area (S_2) of compressibility process of different weight of feed size (-4500+4000 µm) of cement clinker at constant force (P=95kN).

3.2 Particle Size

In the present study it is intended to examine the effect of particle variation on the size reduction of compact formed from white cement clinkers. Furthermore, since changes in particle size distribution may well change the predominant compaction mechanism, the percentage of size reduction is employed to investigate this effect.

Five size fraction of a sample of white cement classified as follows:-1120 to +900µm, -2000 to +1800µm, -2800 to +2500µm, -3550 to +3350 µm and -4500 to +4000µm are used. These have a mean diameter ($\overline{X}_2$) of 1000, 1900, 2650, 3450 and 4250µm before compaction. These are passed through a suitable size to break down any agglomerates.

The results are presented at a constant weight of (50 gm) and at the same die and punch and a constant compression applied pressure (28.195 MPa) and constant compaction velocity (0.5 cm/min). It is found the increment of specific surface area (ΔS) increase with the increasing of the particle size as marked in Fig. (9). However, the specific surface before compressed the coarse particles (S_1) has an inverse relation with mean particle size as shown in Fig.(10). It is deduced from the above figure (Fig. 9). Those larger particles (4000-4500µm) produce more fragmentation than the smaller particles during the same compression process condition. The cleavage of large particles will cause more small particles.

The smaller fragmented particles tend to occupy the interparticles voids and thus increasing the density of a compacted material and increase the friction resistance. The interparticles friction force will increase and lead to more fragmentation in the weaker particles and the original compacted particles. Results of this work are summarized in table (2).

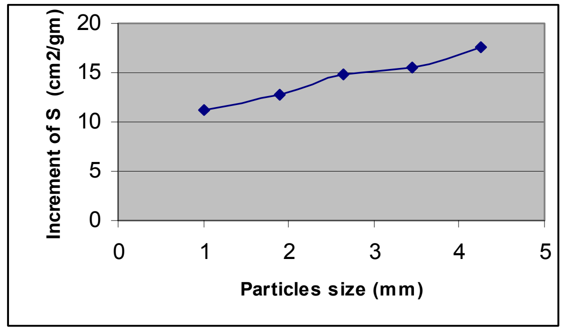

Fig 9: Effect the mean particles size of compressibility process on increment of specific surface area (ΔS).

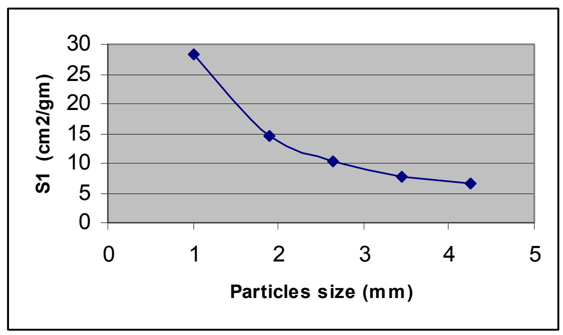

Fig 10: Represent the specific surface area before compression (S1) for different mean particle diameter of cement clinker ($\overline{X}_2$).

Table 2: Size analysis of coarse particles for weight 50gm of different particles size ($\overline{x}_2$) at constant pressure (28 MPa) and constant velocity (0.5 cm/min).

Feed size (µm)	$\overline{X}_2$ (mm)	$\overline{X}_1$ (mm)	ΔS (cm^2/gm)	S_1 (cm^2/gm)
-1100+900	1	0.82	11.16	28.2
-2000+1800	1.9	1.394	12.75	14.6
-2800+2500	2.65	1.8125	14.78	10.2
-3550+3350	3.45	2.342	15.46	7.8
-4500+4000	4.25	2.72	17.67	6.7

3.3 Compaction Applied Pressure

Since the compaction applied pressure (P) more than any other factor largely controls the density and the resulting mechanical properties of final products. Knowledge of the relationship between compaction applied pressure and the material properties is very important.

To estimate the increase of size reduction for a particle size distribution, a better description should be given for fragmentation process. Fragmentation has been defined as the formation of smaller, discrete particles from initial material. This implies that the characterization method should give direct information about the number of cleavages in particle after submission to compaction pressure.

Various sieve fraction of white cement clinkers are used, which had a size range 900-1120,1800-2000, 2500-2800, 3350-3550 and 4000 - 4500µm with weight represent as the fourth layers. The compaction velocity is constant (0.5 cm/min) and the compaction applied pressure has a range from 2-25 MPa in this study. The increment of specific surface area (ΔS) increased with increasing the applied pressure (Pc) as seen in Fig.(11). It can be conclude from this figure, the l

inear relation occur due to the high of structure resistance (which represent the total elasticity of the wholly compact) and the friction resistance. in Fig. (11). Actually the interparticles friction and particle-die wall friction forces are responsible for increasing the fragmentation of coarse particles and this lead to increase the specific surface area.

Table 3 summarizes the result of surface area of coarse particles at different applied pressure

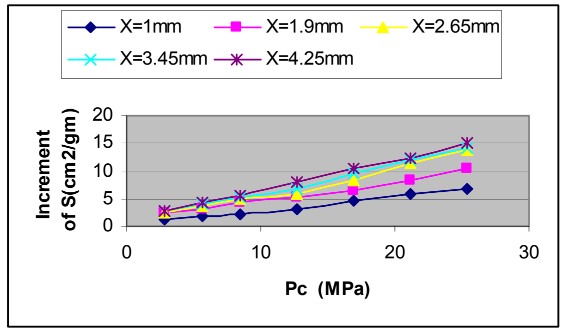

Fig 11: Effect of the applied pressure (Pc) of the compressibility process to increment (ΔS) of the specific surface area to different size of coarse particles**.**

Table 3: The inc increment of specific surface area (ΔS cm^2/gm) at different applied pressure (Pc) and different coarse particles size during the compressibility process.

Pc (MPa)	*ΔS at (X=1mm)*	*ΔS at (X=1.9mm)*	*ΔS at (X=2.65mm)*	*ΔS at (X=3.45mm)*	*ΔS at (X=4.25mm)*
2.82	1.13	2.494	2.453	2.54	2.794
5.64	1.74	3.127	3.733	4.037	4.353
8.46	2.23	4.168	4.873	5.16	5.665
12.68	3.11	5.307	5.888	6.67	8.078
16.92	4.47	6.316	8.418	10.236	10.313
21.14	5.73	8.356	11.415	11.93	12.163
25.375	6.83	10.46	13.951	14.676	15.103

3.4 Compaction Velocity

Air entrapped inside the pores of the powder compact was found bigger than atmospheric pressure [Long and Alderton 1960, James and Newton 1983, and At-Jewaree and Chandler (1990)]. This was attributed to the high compaction velocity, which was absent in the last work. In this work, the increment of specific surface area (ΔS) is examined at different compaction velocities and the effect of air entrapped to the fragmentation of the coarse particles as shown in figure (12). However the entrapping of air inside the pores of the particles is found to increase the total resistance of the powder during the compressibility process. This resistance will leads to a small fragmentation of the coarse particles, because they reduce the total applied pressure which reached to the single particles. Supporting the above evidence, it is found the size reduction ($\overline{\Delta X}$) decreasing with the increasing of the compaction velocity as illustrated in Fig.(13). The test are done at constant applied pressure up to 25.375 MPa and a constant weight equal to 44 gm as four layers to a size range from 2100 to 2500µm, but at different speed ranging from 0.5 to 50 cm/min. Table (4) summarize the effect of compression velocity (Vc) on increment of surface area(S).

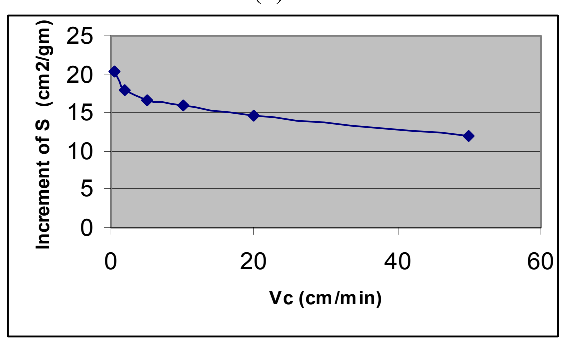

Fig 12: Effect the compression velocities (Vc) on increments of surface area (ΔS) of coarse particles.

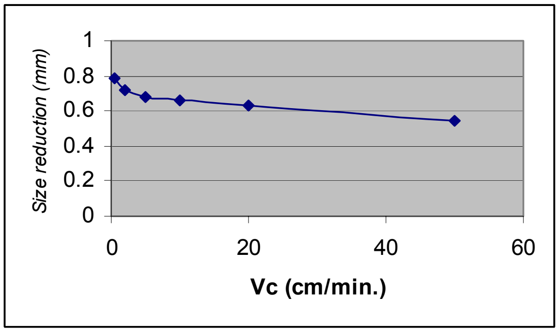

Fig 13: Represent the relationship between the compression velocity (Vc) and the size reduction $\overline{\Delta X}$ for feed size (-2500+2100 µm) of coarse cement particles

Table 4: The increment of specific surface area at different compression velocity (cm/min), where S_1 = 12.3 cm^2/gm for 2.3mm mean particles size.

Compression Velocity (cm/min.)	*ΔS (cm^2/gm)*
0.5	20.42
2	18.09
5	16.58
10	16.08
20	14.734
50	12.188

4. CONCLUSIONS

The following conclusions are drawn out from the present study:

1) The increment at the size reduction ($\overline{\Delta X}$) and the specific surface area (ΔS) mean decreasing in the total resistance of the compressed coarse materials.
2) It is found that the high increment of specific surface area (ΔS) of coarse particles at higher applied pressure, slower compaction velocities, larger coarse particle and at reducing the weight of compressed material (see Figs.7,9,11&12)
3) It is obvious that for a certain particle size, the higher the compaction load, the more fragmentation will occur (see Fig. 10). On the other hand, one should forget that fragmentation can not take place when porosity drowns near zero. Thus considering fragmentation to be a function of particle size and porosity. It is clear from Figs.(9&10), that the large particles have more fragment than the smaller particle of white cement clinker and these increase the specific surface area during the compressibility process.
4) **It** may be concluded that compaction apparatus is good enough to cause a size reduction in the coarse particles and for approach to the real grinding of the materials inside the ball mill.

5. REFERENCES

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*Al-Khomes is a city in east of Libya.