

Particle Size Reduction by Compressibility Process of the Alumina oxide.

Dr. Khalid Khazzal Hummadi *

Received on: 1 /6 / 2004

Accepted on: 21 / 11 / 2004

Abstract

The effect of the compaction applied pressure, compaction velocity and feed particle size on the size reduction of Alumina Oxide by using the compressibility process were investigated. The apparatus consist of two punches and large die which is a single punch pressing. The applied pressure varied over the range of (3.1 to 30.3) MPa and the compaction velocity adjusted from (0.5 to 100) cm/min. Feed sizes used are: 6000, 5000, 4500, 4000 and 3500 μm . The ground alumina oxide was screened by sieves analyses method. The increases in specific surface area and the percentages of the losses in input energy for each case were evaluated.

The results of this investigation indicated that the high size reduction occurred at a slower compaction velocity and a high applied pressure for all fed particle sizes. These results will help us in the design of mills in order to decreases the losses in input energy for size reduction of solid material.

الخلاصة:

في هذا البحث تم دراسة تأثير الضغط المسلط وسرعة الكبس والحجم الحبيبي الداخل على تصغير الحجم الحبيبي لأوكسيد الألمنيوم باستخدام قالب كبس كبير يتكون من مكبسين إحداهما ثابت والآخر متحرك واسطوانة سميكة وضغط مسلط متغير يتراوح بين (3.1 إلى 30.3) ميكا باسكال وسرعة كبس تتراوح بين (0.5 إلى 100) سم/ دقيقة. وكانت حجم الحبيبات المستخدمة 6000 و 5000 و 4500 و 4000 و 3500 مايكروميتر. تم تحليل الناتج المطحون باستخدام طريقة التحليل بالغريلة. ومن ثم حساب الزيادة في المساحة السطحية النوعية للناتج المطحون وحساب النسبة المئوية للطاقة المفقودة لكل حالة.

ومن خلال النتائج تم التوصل إلى أن زيادة الضغط المسلط وتقليل سرعة الكبس تعطي أعلى كفاءة في تكسير الحبيبات الصلبة باستخدام المكبس ولجميع أحجام التغذية وإن هذه النتائج تساعدنا في تصميم الطواحين لغرض التقليل من الهدر في الطاقة الداخلة لعملية تصغير الحجم الحبيبي للمواد الصلبة.

Nomenclature:-

- | | | | |
|--------------|---|-------------|--|
| a | The maximum displacement of the upper punch. | S_2 | The specific surface area after the compressibility process. |
| LE | The area under the hysteresis loops (A) (losses energy). | ΔS | Increase in specific surface area (cm^2/gm). |
| ΔQ_i | The weights fraction retained on sieve of size (i). | UPF | The applied force by upper punch. |
| S | The specific surface area (cm^2/gm). | UPW | The upper punch work or the gross input work. |
| S_1 | The specific surface area before the compressibility process. | $\bar{X}_i$ | The average particle size (μm). |

* Dept. of Biochemical Engineering, University of Baghdad 13

ρ_s The solid density of alumina oxide (=2.024 gm/cm³).

ψ The sphericity of particles
($\psi = \frac{\text{min. particle diameter}}{\text{max. particle diameter}}$).

1-Introduction:-

Size reduction is one of the oldest processes of solid particles that had been used and is respected to be the first operation in a chemical process or the last step before product packaging⁽¹⁾. Size reduction such as crushing and grinding is usually carried out to increase the specific surface area of catalysts or supports (such as Alumina Oxide) of the active components⁽²⁾ because the rates of mass transfer and reactions are directly proportional to the surface area of contact with a second phase⁽³⁾. One purpose of the size reduction operation is to prepare products in finer particle size ranges. Another purpose is to liberate the constituent minerals in ores as a preparation for subsequent separation processes.

A wide variety of equipment types are used differing in nature and intensity of forces applied and in geometry and velocity of breakage members^(4, 5). Compressibility is the ability of the material to reduce the volume under the pressure, this happens in the size reduction of the coarse particles inside the ball mill. Coarse particles of Alumina Oxide need more than 100KN to approach the compaction condition. While the maximum capacity of Instron machine that used in this work is 100KN. Since the compaction pressure more than any other factor largely controls the resulting mechanical properties of final product. Knowledge of the relationship between compaction applied pressure and the material properties is very important. As pressure is applied, re-arrangement of particles takes within die; so that large voids are filled and inter particles friction may be sufficient to cause fragmentation of the particles⁽⁶⁾. Further increase in pressure is believed to cause elastic and plastic deformation of the weaker particles being brought into close physical contact^(7,8). At the same time, air

entrapped inside the pores of the powder compacted was found a source of cracks in powder compacts is called laminations. This was attributed to the high compaction velocity⁽⁹⁾.

The dry and wet grinding operations have been discussed by many authors^(10, 11). Size reduction is usually considered as the most expensive energy consuming department. The largest problem lies in the fact that most of the energy input to the crushing or grinding machines is observed by the machine itself, and only a small fraction of the total energy is available for breaking the material. In the ball mill, it has been shown that less than 1% of the total energy input is available for actual size reduction, the bulk of energy being utilized in the production of heat, sound and other losses⁽¹²⁾. The total energy input during the compressibility process is used for particle rearrangement, interparticle friction, friction of the particles with the die-wall, elastic deformation, plastic deformation, fragmentation of bonds.

In the present work, the effect of applied pressure, compaction velocity and the feed particle size on the size reduction of Alumina Oxide (which is very important parameters in the design of size reduction equipments) by using the compressibility process were studied.

2-Experimental work:-

The experiments were performed in a hydraulic universal testing machine (model 1195, Instron Ltd., High Wycombe, U.K) fitted with 67.2 mm stainless steel die diameter as shown in fig (1). It is decided to use hardened steel (55%) for the punch and die cylinder to withstand the large applied pressure and abrasion.

The feed stoke employed was Alumina Oxide (from Alcoa international company) of fractions 6000, 5000, 4500, 4000 and 3500 μm . These fractions are the normal sizes of a

narrow range distribution as shown in table (1).

The size reduction of Alumina Oxide has been conducted in a cylinder die, upper punch and lower punch as shown in fig (2). The clearance between the die-wall and the punches is 0.2 mm in the majority of tests. The experiments were carried firstly at constant compaction velocity (0.5 cm/min) and the range of applied pressure is from 3.1 to 30.3 MPa in this work, secondly at constant applied pressure 25.2 MPa for the same size fractions but at different compaction velocity from 0.5 to 100cm/min.

The specific surface area of Alumina Oxide before and after compressibility process are calculated for each size fraction using equation (1) as represented by Allen(1981)⁽¹³⁾.

$$S = \frac{600}{\rho_s \psi} \sum_{i=1}^n \frac{\Delta Q_i}{X_i} \quad \dots\dots\dots (1)$$

The sphericity of each feed alumina oxide sizes were:

$$\Psi_{6000}=0.752, \Psi_{5000}=0.749, \Psi_{4500}=0.746, \Psi_{4000}=0.744 \text{ and } \Psi_{3500}=0.742.$$

For calculation the increase in specific surface area (ΔS), the following formula is used

$$\Delta S = S_2 - S_1 \quad \dots\dots\dots (2)$$

The energy used during the compressibility can be calculated from punch force and displacement curve (FD-curve). The plotting of the displacement of the upper punch on the axis of the abscise versus the force on the upper punch on the ordinate results in a force displacement curve. This curve is comprises the die-wall friction and inters particle friction works^(14, 15). During compaction, force displacement measurements are carried out according to a method which uses a planimeter to measure the area under the FD-curve, which is the Instron testing machine plotted it. The FD- curve of particles of alumina Oxide is shown in fig (3). Fig (3) consists of two hysteresis loops. The hysteresis loop (A) corresponds to the loss energy (LE) (die-wall friction work and interparticles friction work). The

hysteresis loop (B) represents the work carried back to the upper punch, and it's indicated by work of expansion⁽¹⁴⁾. This measure of elasticity of the compressed mass. The losses percentage in input energy (LE%) can be calculated from equation (3):

$$LE\% = LE/UPW \quad \dots\dots\dots (3)$$

UPW can be calculated from DeBley, et al. (1971)⁽¹⁶⁾ equation (4):

$$UPW = \int_0^a UPF \, da \quad \dots\dots\dots (4)$$

3-Results and Discussion:-

Fig (4) shows the increase in specific surface area (ΔS) of Alumina Oxide versus the applied pressure for various feed particles size. It can be shown that the increase in specific surface area (ΔS) increases with increasing the applied pressure for all feed particles size. This is a good explanation about the effect of applied pressure to the compressibility process. At small applied pressure, there are two important stages of compaction occurs, the filling of holes of the same order of size as the original particle and the filling of voids which are substantially smaller, by plastic flow or by fragmentation. At a high applied pressure, may be occurs the filling of the holes between primary particles and plastic deformation of the primary particles. Therefore when an increasing the applied pressures, the size reduction increases too. Fig (4) shows that the larger feed particle size gives a high increase in specific surface area greater than of smaller feed particle size due to the fact that the smaller particles are of greater hardness than the larger particles and at the same time, that as the feed particles size increase the production of fin particles increases too, therefore the increase in specific surface area (ΔS) increases with increasing the feed particle size as shown in fig (4).

Table (2) shows the results of the maximum displacement of the upper punch and the energy losses for different applied pressure for all feed particles size of Alumina Oxide.

The losses percentage in input energy (LE%) against the applied

pressure (P) for various feed particle size of Alumina Oxide was plotted in fig (5). Table (2) and fig (5) show that the energy losses and the losses percentage in input energy increases with increasing the applied pressure. This could be attributed that at high applied pressure, the amount of work, used to overcome friction of die-wall and the antiparticle friction, increase and relatively more than the work used for particles rearrangement. Fig (5) shows that the losses percentage in input energy increases with decreasing the feed particle size because the larger particles are softer than smaller particles as shown in table (2).

Fig (6) shows the increase in specific surface area (ΔS) of Alumina Oxide against the compaction velocity (V_{ps}) for various feed particle size. Fig (6) shows that the increase in specific surface area (ΔS) decreases with increasing the compaction velocity for all feed particles size. This could be due to that at high compaction velocity the air entrapped inside the pores of the compacted powder causes an increase in the total resistance of the powder during the compressibility process⁽⁹⁾. This resistance will lead to a small fragmentation of the coarse particles, because they reduce the total applied pressure to the single particles. Therefore the size reduction decreases with increasing the compaction velocity.

Table (3) shows the results of energy losses for different compaction velocity of Alumina Oxide for all particles size. The losses percentage in input energy (LE%) against the compaction velocity (V_{ps}) were plotted in fig (7). Table (3) shows that the energy losses decreases with increasing the compaction velocity also in fig (7) it can be shown that the percentage losses in input energy decreases with increasing the compaction velocity. This may be due to that the slower compaction velocity gives a lot of time to the air to escape from the pores of the particles during the compressibility process.

4-Conclusion:-

1-It was found that the maximum size reduction occurs at high pressure and low compaction velocity. Also the

larger particles of Alumina Oxide are softer than the smaller particles. This conclusion will help us for simulation and design of size reduction equipments.

2-The compressibility process gives a size reduction of a coarse particles with low energy losses approximately 21-25% as shown in figs (5 and 7) when comparison with that obtained by using the ball mill.

5-REFERENCES:

- 1- Heim A. and Pietrzykowski M., "Grinding in a Ball mill", Powder Handling and Processing, 5, 4 (1993)363-365.
- 2- Sivaraj ch., Reddy B.P., Rao B.R. and Rao P.K., "Preparation of catalytically active γ - Al_2O_3 from a basic Alumina succinate precursor precipitated from homogeneous solution", Applied Catalysis, 24(1986) 25-35.
- 3- Colson J.M. and Richardson J.F., "Chemical engineering", Vol.2, 3rd edition, Pergamon Press, New York (1978).
- 4- Erickson, H.W., "types of grinding mills and when to use them", Chem.Eng.Prog. Vol.49, No.2, (1953)63-68.
- 5- Kanada Y. et al., "Study of grinding rate constant for solid materials in a ball mill", Metal Powder Report, Vol.58, Iss. 6, June (2003)49-55.
- 6- Briscoe B.J. and Rough S.I., "The effect of wall friction on the ejection of pressed ceramic parts", Powder Technology, Vol.99, Iss3, 1 October (1998)228-233.
- 7- Ching K.H. and Glenn S.D., "Effect of cyclic pressure on the low temperature of several composite powder systems", Acta Materialia, Vol.45, Iss10, October (1997)4283-4296.
- 8- Denny P.J., "Compaction equation; a comparison of heckle and kawakita equations", Powder Technology, Vol.127, Iss. 2, 16 October (2002)162-172.

- 9- Al-Jewaree H.A.M. and Chandler H.W., "Air entrapment: a source of lamination during pressing" Br. Ceram. Trans. J., Vol.89 (1990) 207-210.
- 10- Kotake N., Suzuki K., Asahi S. and Kanda Y., "Experimental study on the grinding rate constant of solid materials in a ball mill", Powder Technology, Vol.122, Iss. 2-3, January (2002) 101-108.
- 11- Deniz V., "A study on the specific rate of breakage of cement materials in a laboratory ball mill" March (2003) 439-445.
- 12- Lynch A.J., "Mineral crushing and grinding circuits", Vol.1, 1st edition, Elsevier Scientific Publishing Company, New York (1977).
- 13- Allen T., "Particle size measurement", 3rd edition, London Press U.K. (1981).
- 14- Debale C.J. and Polderman J., "measurement of the work involved in tablet compression", pharm. weeklly Vol.80, (1971) 111-118.
- 15- Lammens R.F., Liem T.B., Polderman J. and De Baley C.T., "Evolution of force - displacement measurements during one-sided powder compaction in a Die", powder Technology, Vol. 26 (1980) 169-185.
- 16- Debale C.J. and Polderman J., and Von Ondthoorn C.B., "compression of pharmaceuticals. III. Sulfadimidine", pharm. Weeklly, Vol. 106 (1971) 589-599.

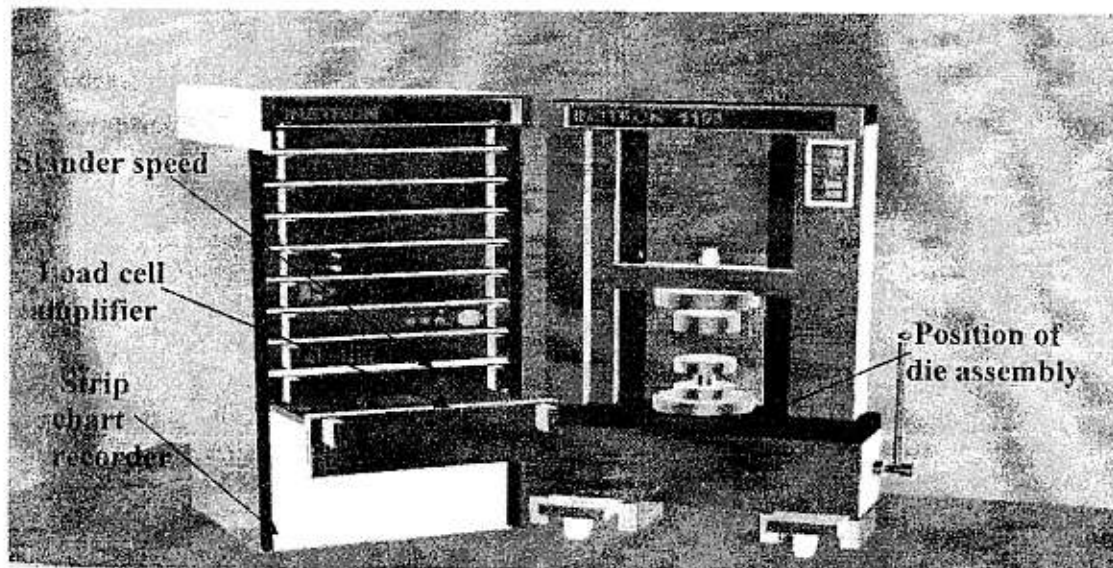


Fig.(1) Instron Universal Testing machine (model 1195, Instron Ltd)

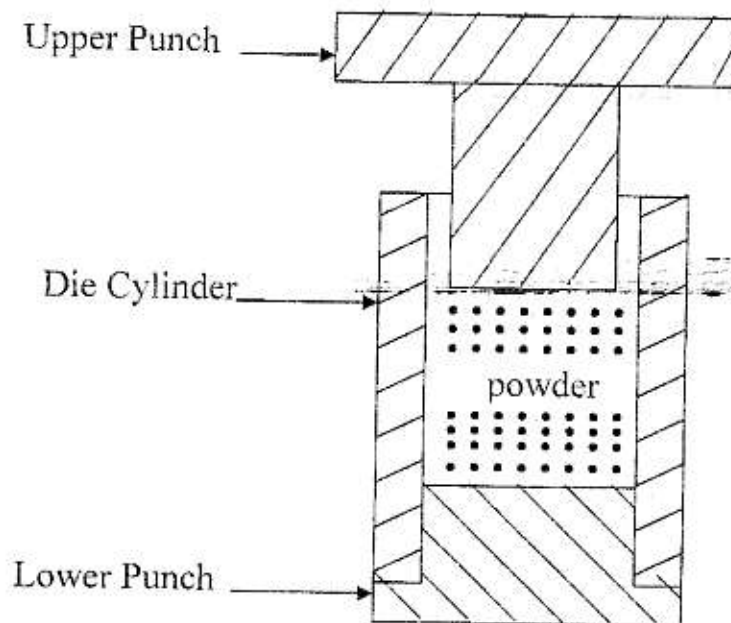


Fig. (2) Schematic diagram of the die assembly

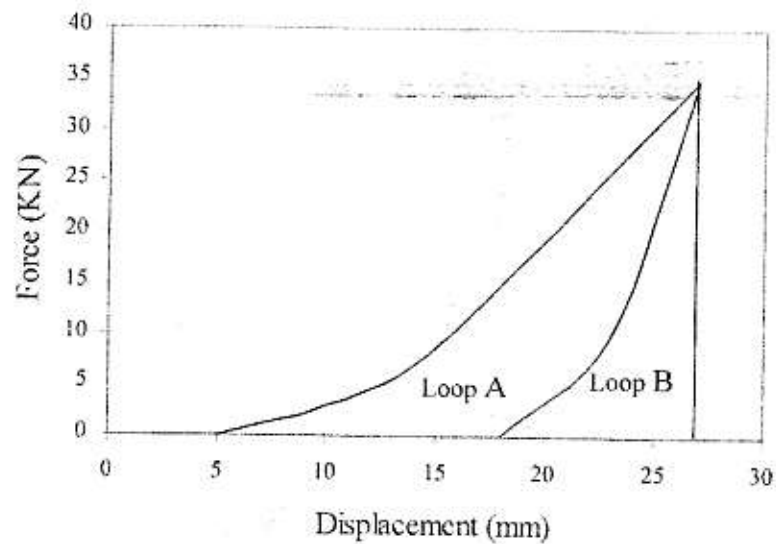


Fig. (3) Force displacement cycles of compressibility process

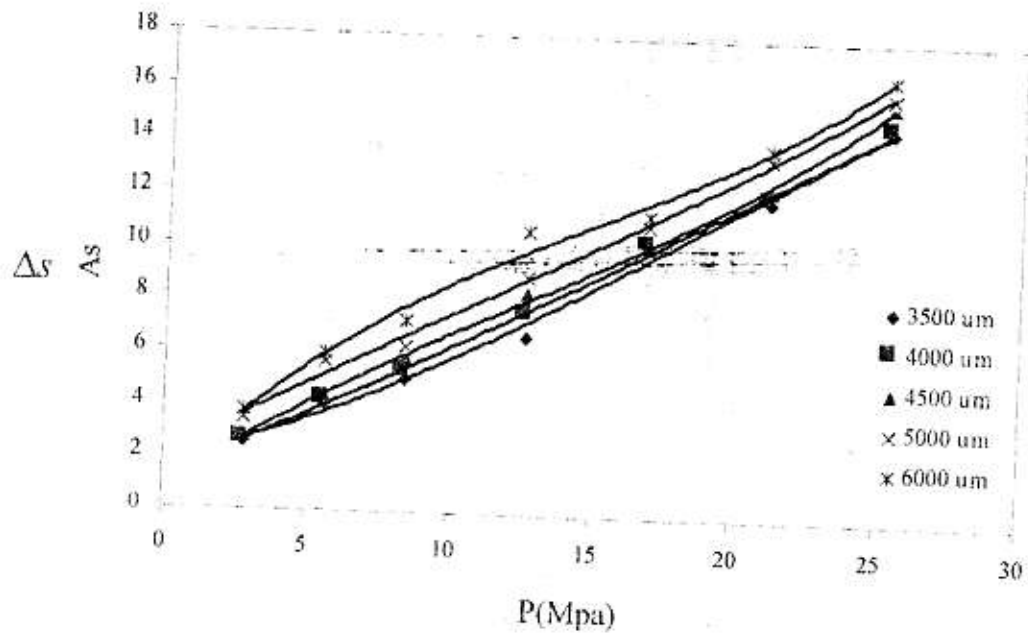


Fig. (4) Increase in specific surface area (Δs) versus the compaction applied pressure (p) for different feeds size of alumina oxide

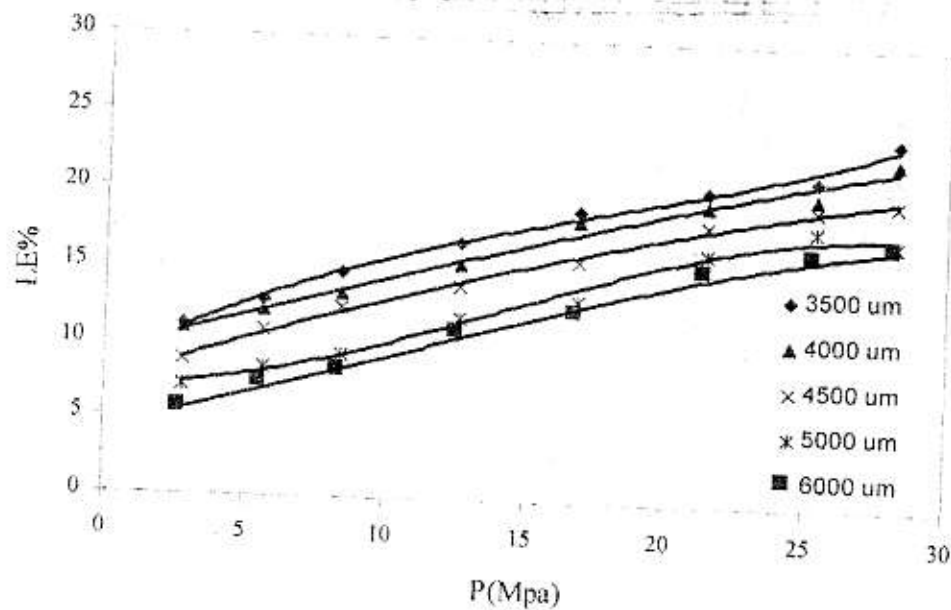


Fig. (5) The percentage of the losses in input energy (LE %) against the applied pressure (P) for different feeds size of alumina oxide

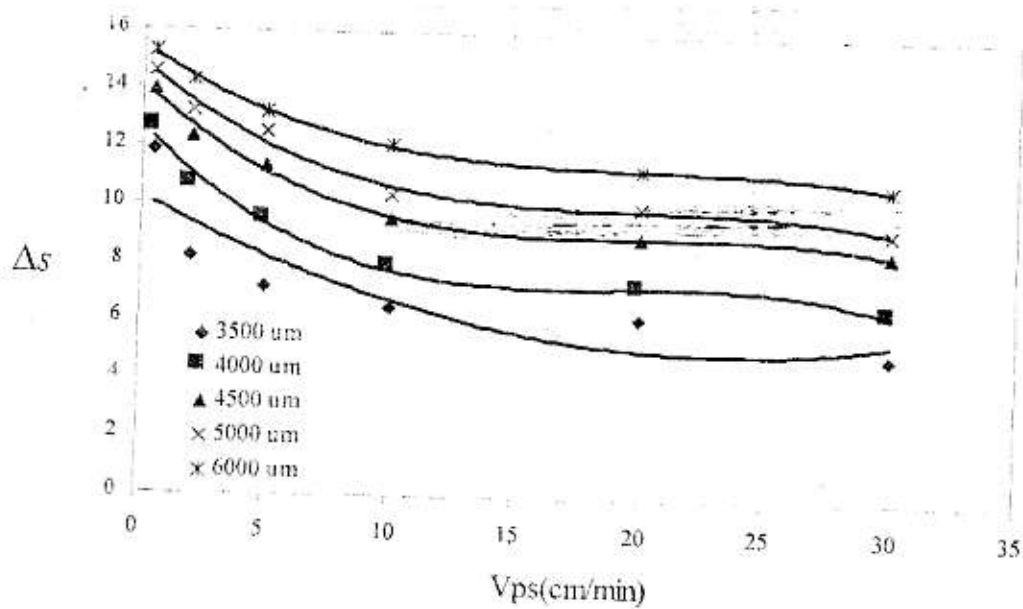


Fig. (6) Increase in specific surface area (Δs) versus the compaction velocity (Vps) for different feeds size of alumina oxide.

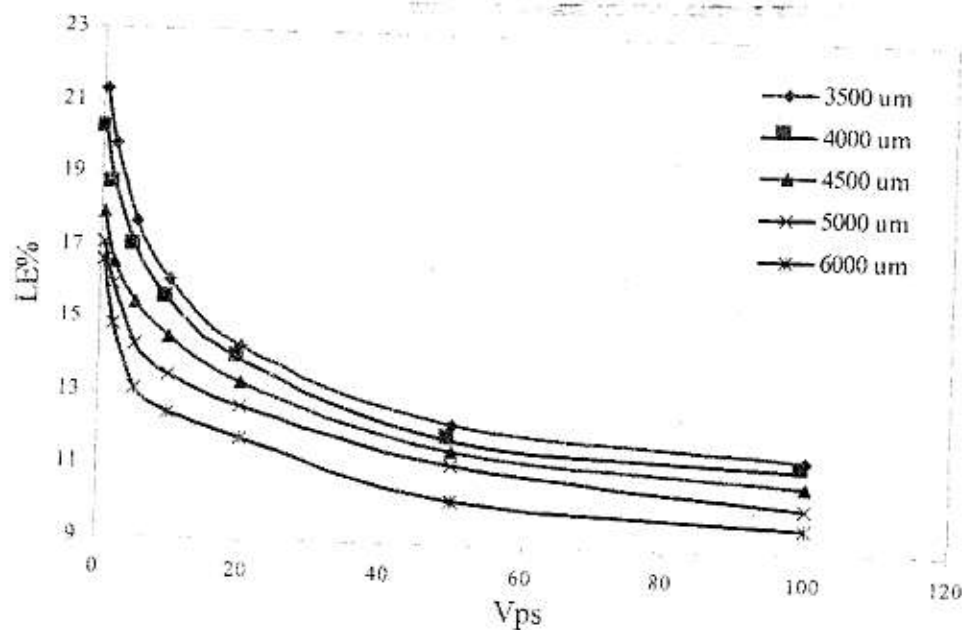


Fig. (7) The percentage of the losses in input energy (LE %) against the compaction velocity (Vps) for different feeds size of alumina oxide.