# STUDYING THE FACTORS EFFECTS ON THE FLOWABIITY OF ZnO-CuO/γAL<sub>2</sub>O<sub>3</sub> CATALYST THROUGH HOPPER BEFORE TABLETING PROCESS

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# ABSTRACT

One of the most important problems in tablet process is to control the flow of the catalyst through the hopper; Controlling the flow can be done either by changing the size of particles or adding the lubricant (stearic acid, starch, graphite) with different percentages (0, 2, 5, 8, and 11 %). The study showed that for most cases, starch (lubricant) had the major effect on flowability, then the stearic acid come in the second order of effect, graphite showed small effect (and some times negative effect) on the flowability.

## INTRODUCTION

The catalyst used in this research was (ZnO– CuO/ $\gamma$ Al2O3) which was in exyrogel phase, green color, non cohesive powder. This catalyst is calcined after tableting process to become finished catalyst Zinc–Copper over Gama Alumina (Zn – Cu / $\gamma$ Al2O3) in gray color.

Adding lubricants to catalyst is important to improve the flowing of the catalyst through the hopper and facilitating the tableting process in the tablet machine<sup>[1]</sup>. The lubricants affect the flow; compaction; and ejection behavior. Some of lubricants will decrease the flow and decrease ejection; others such as graphite will improve all the flow and ejection <sup>[2]</sup>.

The angle of repose is defined as the angle to the horizontal assumed by forming a pile of bulk solids<sup>[3]</sup>. The angle of repose is one of the utmost factors to indicate the flowability of powder, the angle of repose is a common test of inter particle friction <sup>[4]</sup>; therefore, the angle of repose is a direct indication of the potential flowability of material<sup>[5]</sup>, which can be measured by four methods (Fixed – Height Cone, Fixed – Base Cone, Tilting Box, and Rotating Cylinder). Each powder has flow properties different from another one<sup>[6]</sup>, typical method of formation of the heap is to pour powder on the top of a growing conical mound (fixed – height cone)<sup>[7]</sup>.

Lubricants typically used in tableting operation are graphite, starch, talc, stearic acid and others<sup>[8]</sup>. Figure (1)<sup>[9]</sup> illustrates the blending of the particles with different lubricants which

are very important in flow behavior of the catalyst, (1.A) shows the small particles with lubricants which have same particles size, (1.B) shows the medium particle sizes with lubricants without change in size, (1.C) shows the large particles with lubricant.



Fig. (1) Particles - Lubricants Mixture<sup>[9]</sup>

The aim of the study is to show the effect of flow (ZnO-CuO/ $\gamma$ Al2O3) catalyst by using different types of lubricants (stearic acid, starch, graphite) and blending of them, to show their effects on the angle of repose and on the mass flow of the catalyst through the hopper. Lubricant content of (0, 2, 5, 8, and 11 %) were used.

## EXPERIMENTAL WORK

#### Angle of Repose

The angle of repose was measured using The fixed – height cone method by a standard hopper

(Fig. 2), for pouring the powder, a platform (Fig. 3) was used for the heap of powder, that allows excess material to slip away, the shape of plate is rectangular and it was divided into concentric circles to measure the diameter of the cone which was formed with powder (Fig. 4).



Fig. (2) Standard Hopper

The drained angle of repose is measured by allowing a deep container to empty through a standard hopper in its base; the heap of powder remaining on a platform suspended in the container then measuring the height of the heap and the base diameter of the cone formed.



The height of the heap (H) and the base diameter of the cone formed (D), (as shown in Fig. 3) were measured:

Angle of Response 
$$(\alpha) = tan^{-1}(H/r)$$
 (1)

Where r = the base radius = D / 2. Three measurements were made for the powder and the mean value was taken as the true angle of repose.



Fig. (4) Measure of Angle of Repose

### **Catalyst Flow**

In this research a special hopper was used to measure the flow of catalyst (which has the same dimensions of the hopper in the company which is used in the tablet machine) as shown in Fig. (5)



Fig. (5) Special Hopper

The mass flow rate of powder (g/s) was measured using the flow procedure:

- 1. Weighing the catalyst (W).
- Allowing the catalyst to pour through the hopper, and calculate the time (t) needed for the catalyst to drain.

Mass flow rate = 
$$\frac{W}{t}$$
 (2)

Lubricants were used as additives to the catalyst. Five different percentages of lubricant material (0, 2, 5, 8, and 11%), with three different types of lubricants (stearic acid, starch, and graphite) were prepared. The procedure was as follows:

- 1. Weigh a sample of cut used (W).
- 2. Calculate weigh of lubricant content percentage used (WL) which mixed it with the catalyst, for example, when used (2%) of lubricant:

$$W_L = \left[\frac{W}{(1-0.02)}\right] - W \tag{3}$$

- 3. Mixing the catalyst with lubricant and weigh them (WM).
- Allowing the mixture to pour through the hopper, and calculate the time (t) for the mixture to drain.

Mass flow rate = 
$$\frac{W_M}{t}$$
 (4)

Three measurements were made for the catalyst and the mean value is taken as the true mass flow rate.

# RESULTS AND DISCUSSION

# Effect of Particles Size on the Flow

Figure (6) shows the mass flow rate with the average diameter of catalyst. The mass flow rate (kg/s) increased with the increase of size of particles, because, the small particles will cause high friction with themselves. The attraction and compaction are high for small particles; therefore, the flow is slow. As a result, the large particles have good flow because the surface area of particles is large (the compaction and attraction are low), therefore, the friction is low.



Fig. (6) Effect the Particles Sizes on the Flow (Without Lubricants)

As shown in the figure, when the average diameter is greater than (0.72 mm), the mass flow rate decreases with the increase in the average diameter because the diameter of hole of the hopper which is (2 cm) is fixed and the size of particles became larger (0.72 - 1.875) mm, therefore, the particles were crowded and do not able to flow through hopper and the resistance of flow will be high.

# Effect of Particle Sizes on the Angle of Repose

Figure (7) shows that the angle of repose decreases with the increase in the average diameter of particles through flow of particles on free surface. The small size particles have high angle of repose and the large sizes of particles have low angle of repose. This can be explained that large particles will fall and flow on the free surface, while small size particles like to accumulate on the free surface.





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#### Effect the Lubricants on the Flow

#### The Cut [(0.075 – 0.125) & (0.125 – 0.15)]

The effect of lubricant content on the mass flow rate of catalyst is shown in Fig. (8). It is clear to note that there was no effect of lubricant on the mass flow rate in this cut. The main reason for this can be explained that the particle size of lubricants (0.06 - 0.09 mm) is close to the particle size of catalyst, thus lubricants can not be able to surround the particle or form any film over catalyst surface.



Fig. (8) Influence the Lubricants on the Flow Cut [(0.075 - 0.125) & (0.125 - 0.15)]

#### The Cut [(0.15–0.2) & (0.2–0.3) & (0.3–0.4)]

When the size of catalyst particles increased the effect of lubricants in the flow becomes clear than the cut of small size, because the adhesion increase increases with contact area Figure (9) shows (10).the increasing of stearic acid (lubricant) will content improve the flowability until 5 % becomes where the flow relatively This is attributed to the small constant. lubricant available quantities of to surface area of catalyst cover the high particles.

Using up to 2 % starch or graphite lubricants, the value of flow is relatively constant, but over 2 % the flow will decrease. In this cut adding more starch or the graphite (lubricant) make catalyst particles crowded with the lubricant particles where of close they are dimensions.

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Fig. (9) Influence the Lubricants on the Flow Cut [0.15 - 0.2) & (0.2 - 0.3) & (0.3 - 0.4)]

#### The Cut [(0.4 - 0.6) & (0.6 - 0.84)]

Figure (10) shows that there is an increase in flow rate when using starch. This effect shows the important of using starch to control the flowability of powder. That is because starch has the property of non cohesiveness. Stearic acid gives small significant effect on the flow rate of catalyst. Graphite shows insignificant effect.



Fig. (10) Influence the Lubricants on the Flow Cut [(0.4 - 0.6) & (0.6 - 0.84)]

#### The Cut [(0.84 - 1.25) & (1.25 - 2.5)]

Figure (11) shows the same behavior of lubricants on mass flow rate of powder. Also the starch has the significant effect on the mass flow rate. In comparing with Fig. (10), the flow rate here is lower, this is because of large particles crowded on the hole of the hopper and there is not enough space available for them to flow.





# Effect the Lubricants on the Angle of Repose

Figures (12 - 15), illustrated the angle of repose in different cuts of catalyst when using three types of lubricants with (11 % weight content). These results agree with the fact that low angle of repose will give high mass flow rate.



Fig. (12) Angles of Repose for Cuts [(0.075-0.125) & (0.125-0.15)] (11 % wt. lubricant)



Fig. (13) Angles of Repose for Cuts [(0.15-0.2) & (0.2-0.3) & (0.3-0.4)] (11 % wt. lubricant)



Fig. (14) Angles of Repose for Cuts [(0.4-0.6) & (0.6-0.84)] (11 % wt. lubricant)



Fig. (15) Angle of Repose for Cuts [(0.84-1.25) & (1.25-2.5)] (11 % wt. lubricant)

# CONCLUSIONS

- 1- The flow of catalyst (with or without lubricants) through the hopper depends upon the particles size, the flow increases with particles size until (0.72) mm and then decrease.
- 2- The angle of repose (which gives an indication for the flow of powder), which depend also on the particles size. Decreases with increasing of particles size.
- 3- The lubricants (stearic acid, starch, and graphite) show no effect on flow when particles size lower than 0.15 mm.
- 4- Stearic acid and starch have good lubricant properties when particles size more than 0.4 mm.
- 5- Graphite gives no contribution of lubrication to flow for particles size (0.15 - 0.4) mm, but have significant effect for particles size between (0.6 - 2.5) mm.

#### NOMENCLATURE

D	Diameter of heap	m
H	Height of heap	m
t	The time of drain catalyst	S
W	Weight of catalyst	kg
WL	Weight of lubricant percentage	kg
W <sub>M</sub>	Weight of catalyst – lubricant mixture	kg
α	Angle of repose	Degree

# REFERENCES

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- 1- Little, A. and Mitchell, K. A., "Tablet Making", 1963.
- 2- Komarek, K. R., Chemical Engineering, Vol. 74, December 4, P. 154 – 155, 1967.
- 3- Peschl, I., "The Accutron Vibratory Feed System for Bulk Solids", Us Patent No. 3 973 703, 1976.

- 4- "Powder Metallurgy", 2000, by internet.
- 5- Saenz, I., Papazoglou, E. and Lee, R., "Quantitative Evaluation of Feeding Different Physical Forms of Stabilizers", by internet.
- 6- Brown, R. L. and Richards, J. C., "Principles of Powder Mechanics", 1970.
- 7- Peschl, I., "The Accutron Vibratory Feed System for Bulk Solids", Us Patent No. 3 973 703, 1976.
- Browning, J. E., Chemical Engineering, Vol. 74, December 4, P. 151 – 153, 1967.
- 9- Hardo, P. J., "Manufacturing by Selective Laser Sintering", by internet.
- Lieberman, A., Chemical Engineering, April 10, P. 209 – 218, 1967.