PREPARATION OF ZEOLITE 4A AND STUDYING THE OPERATING CONDITIONS OF THE AGGLOMERATION IN THE INCLINED ROTARY DISK

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Abstract

Zeolite type 4A (molecular sieve) was prepared by using the Iraqi kaolin. The prepared zeolite was blended with kaolin (as a binder) in a ratio of zeolite to kaolin 5:1 (by weight). Agglomeration (into spherical particles) was achieved in a rotary inclined disk (laboratory scale).

During agglomeration process, various water content (35,40,45,and 50 wt%) was used with different rotational speed as 30,40,50,and 60 rpm (the disk angle was fixed at 45°). For 20% water content, the disk angle was varied as 30°,40°,45°,and 55° with the same rotational speeds .

The particle mean diameter was calculated by Carl Zeiss apparatus.It was found that the particle mean diameter (of the agglomerated blended zeolite) increased with the increase in the water content and disk rotational speed. The increase in the disk angle will be responded by a decrease in the particle mean diameter.

Introduction

Synthetic zeolites are widely used in industry as adsorbent of various gases and vapors, and as a catalyst for many petroleum industries. Structurally the zeolite are "frame work" alumineosilicates which are based on infinitely extended three dimensional network of AlO_4 and SiO_4 tetrahedral linked to each other by sharing all oxygen's[2,3]. The properties, which are structure related, include [2]:

1- High degree of hydration.
2- Low density and large void volume when dehydrated.
3- Stability of crystal.
4- Cation exchange properties.
5- Uniform molecular sized channels in the dehydrated crystals.
6- Adsorption of gas and vapor. Zeolite is a high capacity and selective adsorbent.
7- Catalytic properties.

Kaolin clay is used as a binder. The blended clay zeolite mixture is agglomerated into the required shape (spherical, cylindrical…etc). After drying the particles, calcination was carried out at a high temperation (550 °C) in order to convert the clay into a amorphous binder of considerable strength (2). The objectives of agglomeration are (12).

a- Reduce dust losses , b-Reduce hazards , c- the agglomeration material is suitable to store, d- prevent caking and lump formation , e- create uniform blends.

Aim and scope

The aim of this research work was to prepare zeolite 4A from Iraqi kaolin. The prepared zeolite was mixed with keolin binder to produce a blended zeolite of 20 wt% keolin. Agglomeration was carried out in the inclined rotary disk.

For 45° disk angle, different rotational speeds (30, 40, 50, and 60 rpm) were used with various water content of the agglomerated particle (35, 40, 45, and 50 weight percent).

For 45 (wt%) water content, disk angle of 30°, 40°, 45°, and 55° were used for different disk rotational speed.

Particle mean diameter (of the calcined agglomerated zeolite) was measured by the particle size analyzer.

Theory

Kapur, et al [8], studied the mechanism of agglomeration as:

A- Nucleation:

Initially water is held in a pendular state, as distinct lens shaped rings to the points of contact particles. Here the cohesive forces are small and the granules has little surface plasticity and an irregular in shape. As granulation proceeds, the particles are kneaded closer together and eventually the rings coalesce to give continuous network interspersed with air (funicular state) and finally the capillary state is reached when all the pores spaces are completely filled with water [15]. (Fig.(6) shows the mechanism).

B- Transition:

The nucleus grows by two possible mechanism: single particle addition by pendular bridging, or combination of two or more nuclei followed by reshaping.

C- Ball growth:

Large granules were formed by the coalescence of two or more granules regardless on their relative sizes[9]. Metakaolin is formed during calcination [2]

At 550-600 °C: $2Al_2Si_2O_5(OH)_4 \rightarrow 2Al_2Si_2O_7 + 4H_2O$
Kaolin Metakaolin

Metakaolin is not a simple mixture of amorphous silica and alumina but retains some order, which is associated with the hexagonal layers. Zeolite 4A is formed according to[2]:

$$6Al_2Si_2O_7 + 12NaOH \xrightarrow{100\,^\circ C} Na_{12}[(AlO_2)_{12}(SiO_2)_{12}].27H_2O$$
Metakaolin Zeolite 4A

Caps and Danckwerts[6] considered that the torque produced by tumbling motion probably determined an upper limit size of granules which could coalesce. They concluded that the coalescence occurred in an early stage of growth but the subsequent granule growth resulting from crushing and layering.

Ouchiyama, et.al [11], suggested that for two granules to coalesce, it is necessary that a collision occurs first in the former, and that a loading occurs first in the latter. In each case, the cohesion exerted in the deformed portion must be strong enough to endure the successive separating forces, thus loading means an application of force through the neighbors to the contact point between the two granules which are in contact with each other.

Caps, C. E., [5], and Butensky, M., et al [4], found that the particle mean diameter increases with increasing the liquid content.
Fuerstenau, et al [8], proved that the particle mean diameter increased as the speed of rotation increase.

Experimental work

1- Kaolin powder was sieved (Retsch equipment) in order to get particles of less than 45 microns.
2- Calcination of this powder was carried out in a programmable electrical furnace maintained at 550 °C for 3 hours, followed by slow cooling [2].
3- Zeolite 4A was produced by adding (8 wt%) NaOH solution (with stirring) to the calcined kaolin. The reaction was adapted in a beaker (maintained at 100 °C) for 3 hours. The zeolite was separated from the solution by filtration and washing with distilled water until reaching pH of 10 [7].
4- The zeolite produced was dried overnight at 110 °C.
5- The dried zeolite was crushed in the rotary ball mill (Retsch). Then was sieved to get powder of less than 45 microns.
6- Zeolite produced is mixed with kaolin (20 wt% kaolin) in a rotating cylinder (for 5 minutes) to produce homogeneous mixture.
7- Agglomeration was carried out in the inclined rotary disk (40 cm diameter and 10 cm rim height). Feeding rate was kept constant for all experimented as 0.1 Kg per minute, and the feeding was continued for 10 minutes. During this period tumbling occurs and the measured quantity of water was sprayed over the particles. Spherical particles of different sizes were obtained.
8- The produced particles were dried overnight at 110 °C.
9- The dried spherical particles were calcined again using the same conditions mentioned earlier.
10- Particle mean diameter was calculated by [10]:

$$D = \frac{\sum N_I D_I}{\sum N_I}$$

where:

D: is the particle mean diameter

D_I: is the mid point of the I^{th} size interval.

N_I: is the frequency of the I^{th} size interval.

Carl Zeiss particle analyzer [1] was used to determine the size distribution of the spherical particles. The particles (of a given batch) were photocopied, then a direct comparison was made between the projected area of a particle and the area of a reference circle. The reference circle is a pot of light adjustable in size of a particle. When the spot of light had been made equal in area to the projected area of the particle, it is recorded in a preset size category by the depression of a foot switch.
The experimental results were recorded in Tables 1 and 2.

Table (1) Experimental results of the particle mean diameter (D) for 45 disk angle

Disk	Water Content			
Speed	35 wt%	40 wt%	45 wt%	50 wt%
rpm	$D \times 10^3$ (m)	$D \times 10^3$ (m)	$D \times 10^3$ (m)	$D \times 10^3$ (m)
30	1.2	1.4	1.8	2.0
40	4.2	4.5	5.0	5.5
50	6.3	7.0	7.4	8.0
60	7.0	7.3	7.9	8.4

Table (2) Experimental results of the particle mean diameter for 45 wt% water content

Disk	Disk Angle			
Speed	30 °	40 °	45 °	55 °
rpm	$D \times 10^3$ (m)	$D \times 10^3$ (m)	$D \times 10^3$ (m)	$D \times 10^3$ (m)
30	2.5	2.3	1.8	1.2
40	6.5	6.0	5.0	3.3
50	8.3	7.8	7.4	6.6
60	8.8	8.3	7.9	7.3

Results and Discussions

1- For a fixed 45° disk angle:

A. Effect of water content on the particle mean diameter:

Figure (1) shows the effect of water content (of the agglomerated zeolite) on the particle mean diameter for different rotational speed. It is shown that the increase in the water content will be responded by an increase in the particle mean diameter. This is in a good agreement with Caps, C. E. [5]. This effect can be explained that the increase in the quantity of water (between the grains of a particle) means that water will surround the grains and fill the pores, thus capillary forces (and bridging effects) bring the grains to coalesce and binding effect will be greater.

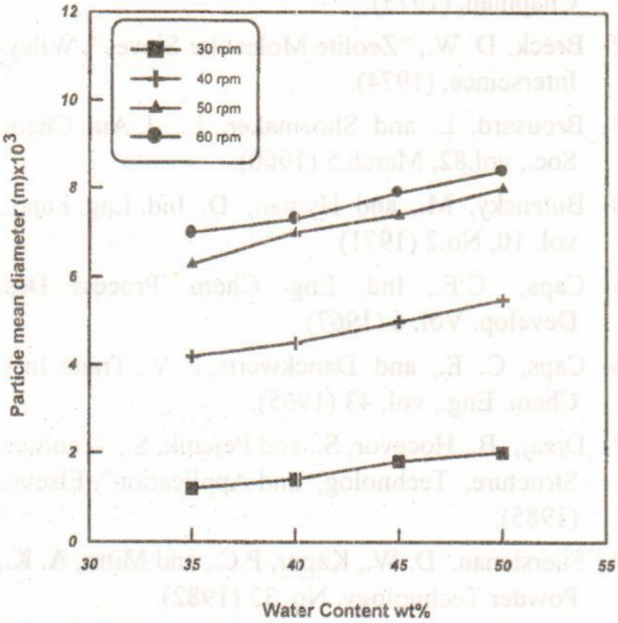

Fig.(1) The effect of water content on particle mean diameter for 45° degree disk angle

Thoroughly studying this figure, it can be seen that the change in water content cause a small variation in the particle mean diameter, i.e., the water content cause small effect to obtain larger particles.

B. Effect of disk speed on the particle mean diameter:

Particle mean diameter against rotational disk speed (for different water content) was plotted in Fig.(2). From this plot, it can be seen that the particle mean diameter will increase as the disk speed increases. This agrees with Fuerstenau, D.W. [8]. The explanation is that as the speed increases, more chances for the small particles to tumbling, i.e., the coalescence of smaller particle increases to become larger particle, also layering will increase.

Carefully noting this plot, it shows a high rate of increase in the particle mean diameter as varying the disk speed.

2- For a fixed 45 wt% water content:

A- Effect of disk angle on the particle mean diameter:

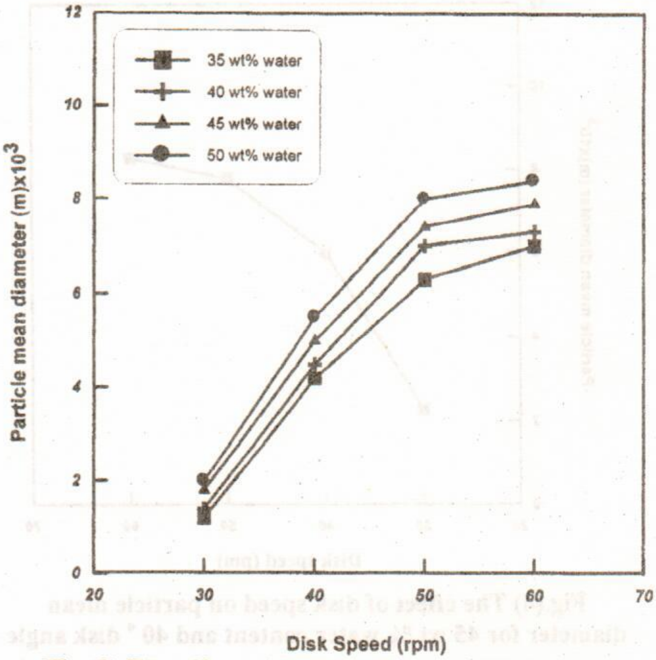

Fig.(2) The effect of disk speed on particle mean diameter for 45° degree disk angle

It can be expected that as the disk angle increases, the particle mean diameter will decrease, this is shown in Fig.(3). The increasing in the inclination of rotating disk will lower the coalescence rate, i.e., less time for particles to tumble over each other, because the particles will leave the disk quickly.

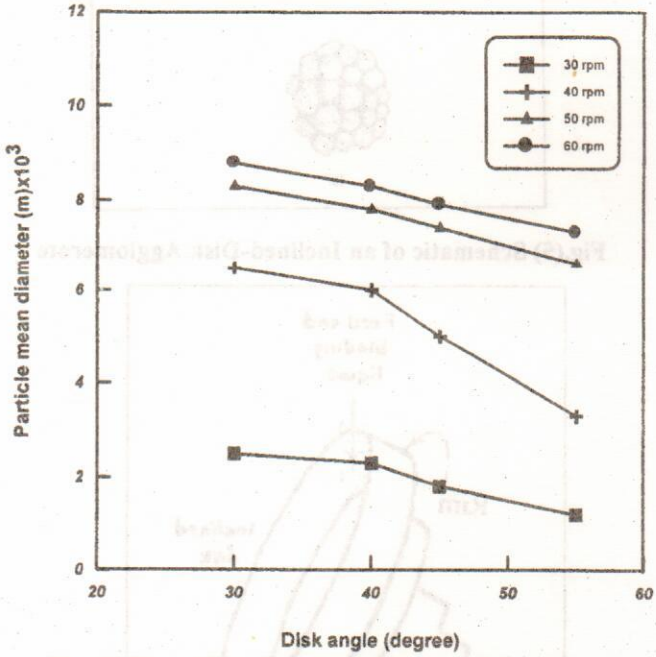

Fig.(3) The effect of disk angle on particle mean diameter for 45 wt % water content

Another indication from Fig.(3), that there will be a small change of particle mean diameter with the variation of disk angle.

B- Effect of disk speed on the particle mean diameter:

As the disk speed increases, the response will be an increase in the particle mean diameter, this is shown in Fig.(4).

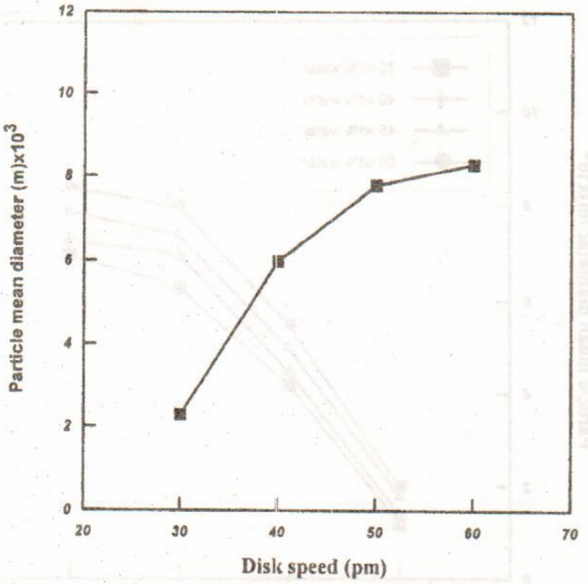

Fig.(4) The effect of disk speed on particle mean diameter for 45 wt % water content and 40 ° disk angle

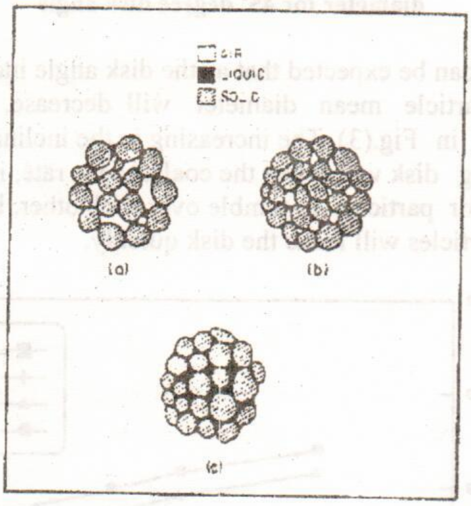

Fig.(5) Schematic of an Inclined-Disk Agglomerate

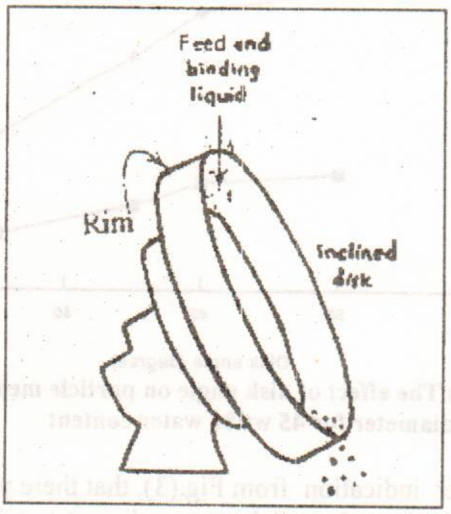

Fig.(6) : Structure of a Porous Agglomerate with Reference to the Relative Distribution of Air-Water-Solid Phases. (a) Pendular (Liquid Trapped), (b) Funicular (Air Trapped), (c) Capillary (Air Expelled-Liquid Saturated).

Conclusions

1- Using the inclined rotary disk, the particle mean diameter will increase with the increase of water content of the (blended) zeolite.

2- The increase in the disk rotational speed will be responded by an increase in the particle mean diameter.

3- If the disk angle increases, the particle mean diameter will decrease.

4- To produce larger particles, the disk speed is more significant factor than the water content.

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