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Effect of the Forming Conditions in the Production of Gamma Alumina Catalyst Support on the Crushing Strength Property

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Abstract

An investigation was conducted for the determination of the effects of the forming conditions in the production of Gamma Alumina catalyst support on the crushing strength property. Eight variables were studied , they are ;binder content which is the sodium silicate , Solvent content which is the water, speed of mixing , time of mixing, drying temperature , drying time , calcinations temperature and the calcinations time

Design of the experiments was made by using the response Surface method in Minitab 15 software which supply us 90 experiments .

The results of this investigation show that the crushing strength for the dried Gamma alumina extrudate was affected by the drying temperature and the drying time only and there is no interaction effect between the variables studied.

Furthermore, the results show that, the crushing strength for the calcined extrudate was affected by the speed of mixing only and the optimum speed is 900rpm.

The maximum crushing strength of 38.38 after calcinations and 11.865 Kg/mm after drying were obtained

Keywords: Gamma Alumina, Catalyst Support, Crushing strength.

Introduction

The physical characteristics of the naphtha reforming catalysts are determined primarily by the material which serves as a support for the metal or bimetallic function. Alumina is the support for nearly all reforming catalysts .The strength of the macroscopic catalyst particles is an important property. For most fixed- bed operations, if a catalyst can survive the handling during manufacturing and loading, it has adequate strength [1].

The physical strength parameters of the catalysts, such as crushing strength and resistance to abrasion, are of significant importance. In industrial reactors, a catalyst with greater abrasion and crumbling tendency can cause severe pressure drop in reactors and pipe lines. Changes in the environment inside the reformer caused by physical degradation of the catalyst adversely affect the the selectivity, activity, and ultimately the life of the catalyst [1].

The crush strength is the resistance of a solid to compression, a property of paramount importance not only for industrial catalysts, but also for ceramics, pharmaceutical tablets and many other solid materials. In The case of catalysts, it is noteworthy that many plant shutdowns occur due to mechanical failure of the catalyst (and not due to its loss of activity) [2].

As far as catalysts are concerned, the ASTM standard methods D 4179 and D6175 are recommended. D 4179 refer to tableted pellets which can be measured in either axial or radial way. As the axial crush strength is much higher, it is not representative of the catalyst behavior in the reactors, therefore the radial crush strength must be measured for industrial purposes (the type of the crush strength measured must always be specified).D 6175-98 procedure refers to extrudates (the radial measurements can be only performed in this case).The radial crush strength is usually expressed in Kg/mm [2].

The crush strength of a catalyst is not appreciably dependent on reactor loading , but mainly on reactor operation .When the catalyst does not require activation , the crush test should be conducted on fresh dried samples, when the catalyst has to be activated in the reactor , only measurements on activated samples are significant [2].

Single pellet radial or, for irregularly shaped particles, bulk crush strength must be measured for any catalyst used in fixed –bed and trickle-bed reactors. A minimum value of the force required to break the pellets is usually specified for single –pellet crush strength (such values should not refer to axial measurements) [2].

For bulk crush strength, either the minimum pressure giving 1% fines

(ASTM D 7804-04) or the maximum percentage of fines formed under the pressure of 23 bar are specified [2].

The crushing strength of the Alumina spheres measured by single –pellet radial is 7 Kg minimum. Without knowledge of the mechanical properties of the materials used, it is impossible to utilize high pressure, temperatures and rates which characterize the present day development of all industries [3].

Contact masses experience different loads when operating under the conditions of fixed and supensed beds. Under the conditions of a fixed bed, the grains are under the pressure of the overlying beds, i.e., they" work" for compression under the conditions of different temperatures and media .Under the conditions of suspension , friction forces and to a certain extent impact Forces act on the catalyst .Taking account of the difference in the load, contact masses are tested likewise by different methods [3].

The purpose of this research is to show which of the variables participating in the forming of gamma alumina support has an effect on the mechanical characteristic of the support by measuring the radial crush strength of the extrudate.

Experimental work

The variables that were studied in this work and which is participating in the steps of forming the gamma alumina supports from the gamma alumina powder are as shown in table 1 with the upper and the lower limit for each of them (based on gamma alumina powder) .

Table 1 Details of the variables and its lower and upper limits

No.	Variable	Lower limit	upper limit
1	Binder: Sodium silicate (50% soln.) %	20%	50%
2	Solvent: Water %	70%	90%
3	Speed of mixing ,rpm	300	1500
4	Time of mixing,min	10	45
5	Drying temperature,C^o	100	200
6	Drying time,min	30	120
7	Calcination temperature, C^o	300	700
8	Calcination time,hr.	2	6

Raw Materials

Gamma alumina powder supplied from Suzhou Yuguang Lighting Materials Co. Ltd. From China was used with the properties shown in table 2 .The XRD chart for it is shown in fig. 1.

Table 2 Properties of the Gamma alumina powder

No.	Specification	Value
1	Name	High purity Alumina powder
2	Type	Gamma-Al_2O_3
3	Purity%	99.99
4	Particle size,µm	0.2
5	Loose density, g/cm^3	0.16
6	BET,m^2/g	120
7	K,ppm	30
8	Na,ppm	18
9	Fe,ppm	10
10	Si,ppm	20
11	Mg,ppm	10
12	Mn,ppm	9
13	Ti,ppm	10
14	Cr,ppm	10
15	Cr,ppm	10
16	Zn,ppm	10

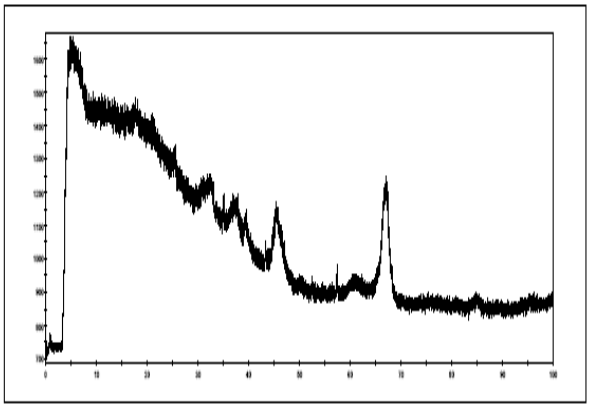

Figure 1 XRD chart for the Gamma alumina powder used

Commercial Sodium silicate solution was used as a binder, its specific gravity is 1.500 and its concentration is 44%

Gamma Alumina Extrudate manufacturing steps

Figure 2 show the steps used in making the alumina paste and forming the extrudate for the support [4].

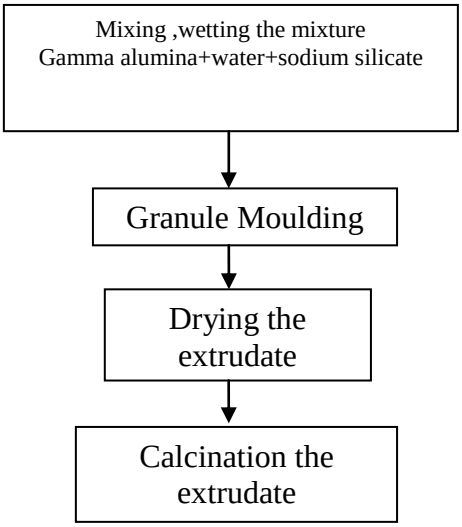

Figure 2 Block diagram of the steps used in producing the Gamma alumina Extrudate (Support)

Method of Moulding

There are many methods to mould the catalyst supports [3], In this work the gamma alumina paste was rubbed into the aperture of perforated steel plate. The size of granules produced is determined by the thickness of the plate and the diameter of the aperture, this is the same as mentioned in reference [3].

Design of Experiments

The response surface method from the Minitab 15 software was used to get the table of design for the 8 variables mentioned above, 90 experiments were got from the Minitab software [5,6] .

From each experiment two samples was tested for crush strength , one after drying and other after calcination, so from the 90 experiments 180 samples were gotten and the crush strength was repeated 20 times for each sample, i.e. , 3600 single pellets were tested for the crushing strength according the standard method, ASTM D-6175-03 [7].Each pellet length over diameter ratio was measured before test , to assure that the L/D is equal or greater than 1.1 as mentioned in the ASTM method above.

Results and Discussion

From the analysis of variance for the dried and calcined crush strength made by the Minitab Software, we get that there is a linear regression for the dried crush strength,also it appeared that there is no any interaction effects between the variables affect the crush strength for both the dried and calcined extrudate.

The estimated regression coefficients for the dried crush strength show that drying temperature and the drying time were the only variables that affect the crush strength in dried state. Figure 3 show the interaction effect for the two above variables on the dried crush strength. From figure 3 , it can be seen that the crush strength increased with increasing the drying time for each drying temperature .Also we can see that the best drying time is between 90-120 minutes.The best drying temperature is between 150-200 C^{o}.

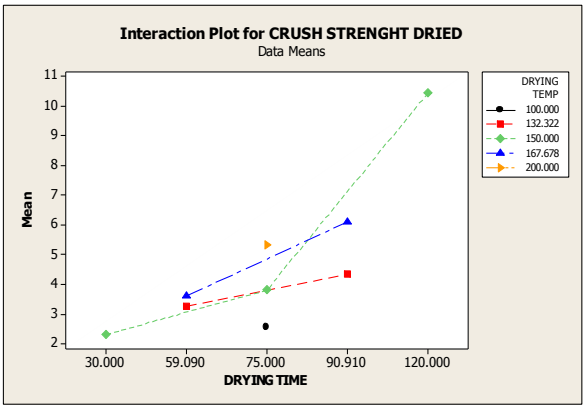

Figure 3 The interaction plot of the drying temperature and drying time on the crush strength in dried extrudate

figures 4 and 5 show that only the drying time and the drying temperature has clear affect on the mean crush strength.

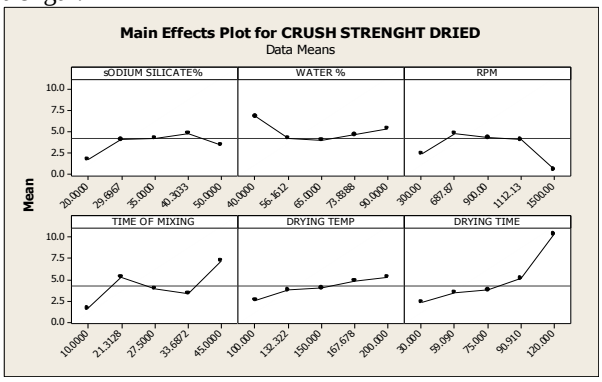

Figure 4 the main effects plot for the crush strength dried

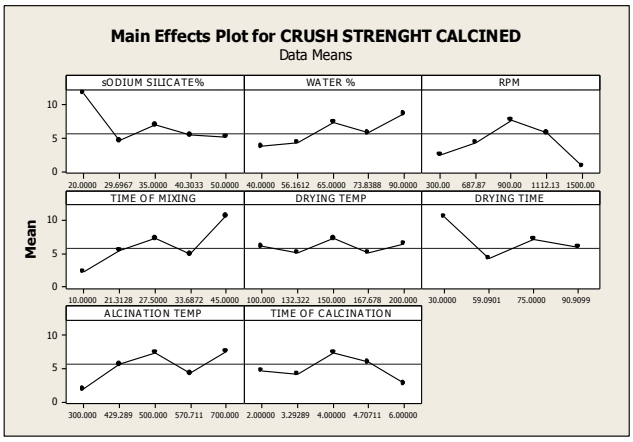

Figure 5 the main effect plot for the crush strength calcined.

The estimated regression coefficients for the calcined crush strength show that speed of mixing is the only one variable that has a little affect on the crush strength in calcined state. .

High crush strength values which is agree with the required minimum values of the alumina support (7Kg/mm) obtained in the experiments shown in table 3.The detailed formulation conditions of the above experiments is shown in table 4.

Table 3 details of the experiments of high crush strength in the dried and claimed conditions.

No	experiment	state	Crush value,KG
1	12	Calcined	10.545
2	13	Calcined	11.585
3	14	Calcined	13.555
4	15	Calcined	38.36
5	16	Calcined	19.689
6	18	Dried	11.865
7	24	Calcined	13.875
8	31	Dried	10.776
9	31	Calcined	12.07
10	34	Calcined	7.665
11	37	Calcined	10.7042

Table 4 details of the formulation conditions for the high crush value experiments

Experiment	Sodium Silicate%	WATER %	RPM	TIME OF MIXING,min	DRYING TEMP,C°	DRYING TIME,min	CALCINATION TEMP,C°	TIME OF CALCINATION,hr,
12	35	65	900	27.5	150	30	500	4.00
13	20	65	900	27.5	150	75	500	4.00
14	29.69	73	687.8	33.6	132.	90.	570	4.70
15	35	65	900	27.5	150	75	500	4.00
16	40.30	73	1112	21.31	132	59.	429	4.70
18	29.696	56	687	21.31	167	90.	570	4.70
24	29.69	73	1112	33.6	132	90.	429	4.70
31	40.30	73	1112	21.31	167	90.	429	4.70
34	29.69	73	1112	33.68	167	90.	429	3.29
37	35	65	900	45	150	75	500	4.00

Figure 6, shows that the best concentration of sodium silicate which give high values of crush strength was 40.30%.

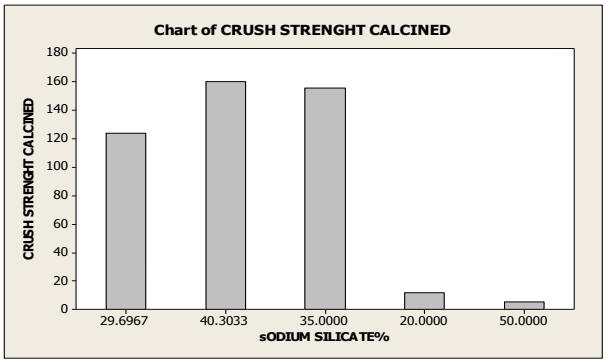

Figure 6 Sodium silicate effect on calcined Crush strength

Figure 7, shows that the best water content which give high crush strength is 65 and 73%, it is compatible with the results of table 3 and 4.

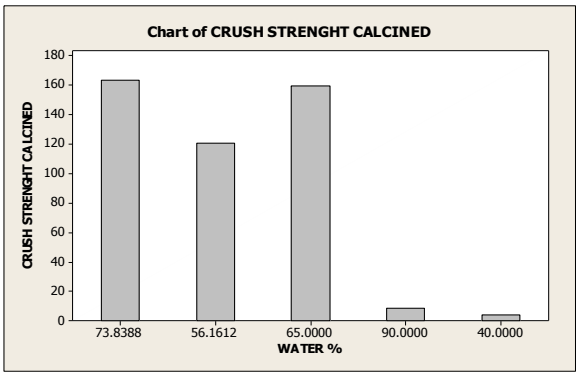

Figure 7 Water content effect on calcined crush strength

Figure 8 show that the best speed of mixing which gives high crush strength is 900 rpm, it is compatible with the results of table 3 and 4.

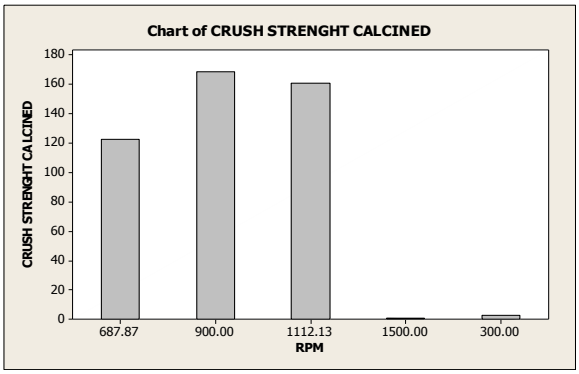

Figure 8 speed of mixing effect on calcined crush strength

Figure 9 show that the time of speed which gives high crush strength is between 21-33 minutes, it is compatible with the results of table 3 and 4.

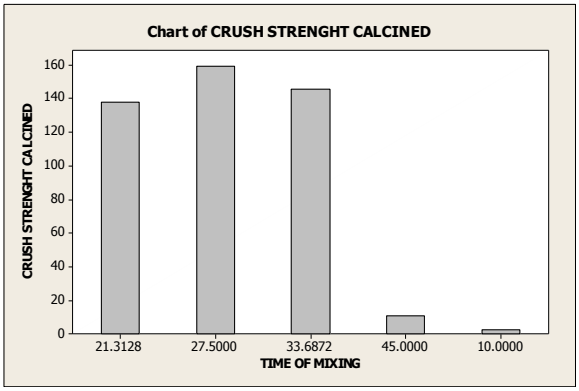

Figure 9 main effect of the time of mixing on calcined crush strength.

Figure 10. shows that the range of the drying temperature which gives high calcined crush strength is 132-150 C^{o}.

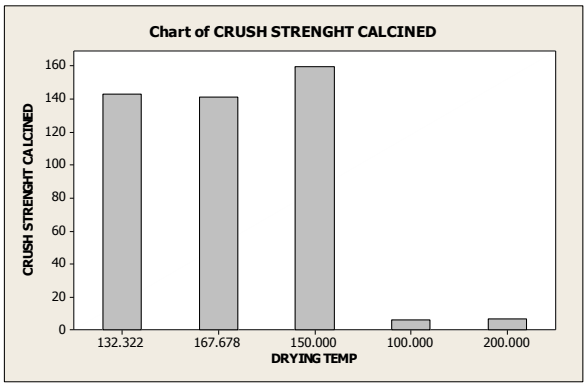

Figure 10 main effect of drying temperature on calcined Crush strength

Figure 11 shows that the drying time which give high calcined crush strength is between 75-90 minutes, it is compatible with the results of table 3 and 4.

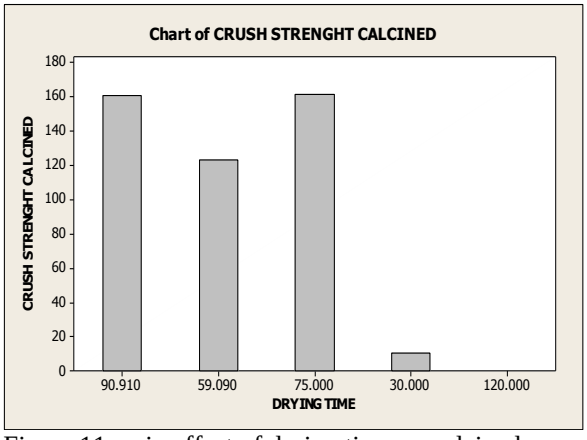

Figure 11 main effect of drying time on calcined Crush strength.

Figure 12 shows that the calcined time which give high calcined crush strength is 4 hours , it is compatible with the results of table 3 and 4.

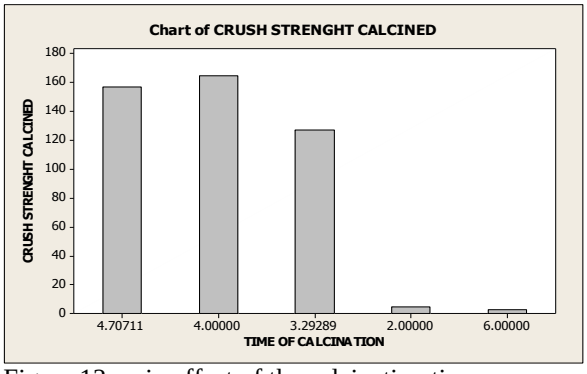

Figure 12 main effect of the calcination time on

Crush strength

Figure 13 shows that the calcination temperature which give high calcined crush strength is 500 C^{O} .

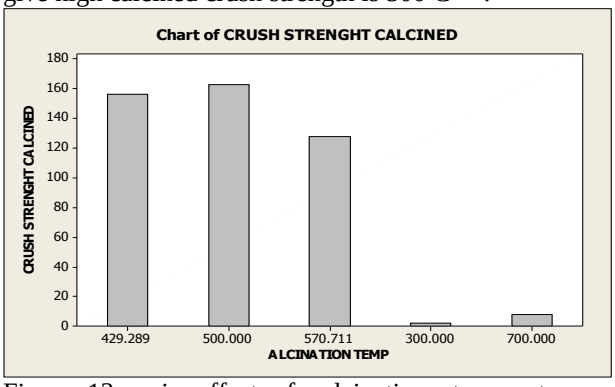

Figure 13 main effect of calcinations temperature on Crushgt strength

For dried crush strength the best results was obtained at the following operating conditions:

a. Sodium silicate solution concentration 40.30%
b. Water content 73%
c. Speed of mixing: 900 rpm.
d. time of mixing 21 minutes
e. Drying temperature 167 C^{o}.
f. Drying time 90 minutes.

Conclusions:

1. The drying time and the drying temperature are very important variables on the crush strength values of the gamma alumina extrudates while forming them.
2. The best drying temperature is between 150-167 C^{o},whileThe best drying time is 1.5hours.
3. There is no interaction effect between the variables studied in forming the gamma alumina extrudate.
4. There is linear regression between the dried crush strength and the drying time and drying temperature.
5. Calcination in general raise the crush strength of the dried extrudate to the required acceptable value.
6. The best calcination temperature and time of calcination are is 500 C^{o} and 4h respectively.
7. The best mixing speed in the step of making the alumina paste and the time of mixing are is 900 rpm and 27 minutes respectively.

References:

1. Antos,George J., Aitani, Abdullah M.,Parea,Jose M., " Catalytic Naphtha Reforming Science and Technology", Marcel Dekker Inc.,USA,1995.
2. www.materialstechnologies.com
3. Mukhlyonov,I.P.,Dobkina, E.I,Deryuzhkina,V.I,Soroko,V.E.,"Catalyst Technology" ,MIR Publishers,Moscow, 1976.
4. US, Patent 4579728- April,1 1986,wide pore Alumina Supports,.
5. Statistics using SPSS, Ray Pub., Syria,2007.
6. Montgometry,"Design and Analysis of Experiments", 1997.
7. ASTM method D-6175-03.