



## Studying the Effect of Nitric Acid on the Physical Chemistry Properties of the Prepared Extrudates Catalyst Support from Boehmite Powder of Nephleensinite Ore Mines

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**Abstract:** In this research, for the first time, the extrudates gamma alumina prepared from Boehmite powder of the Nephleensinite ore mine. In order to enhance the physical properties of the catalyst support, the prepared extrudates were treated in high concentration nitric acid at different times, and the effect of treatment times on the basic properties as the Surface Area (S.A.) and Average Pore Diameter (A.P.D.) were studied. These effects were studied by using X-ray diffraction (XRD), N<sub>2</sub>-adsorption/desorption (BET) and Scanning Electron Microscope (SEM) techniques. The sample 3 with 72 hr aging time was selected as the optimized sample.

**Keywords:** acidic aging time, extrudates gamma alumina, acidic treatment, catalyst support

### I. INTRODUCTION

The production of alumina is done under two general methods of using Bauxite and extraction from Nephleinsinite ore throughout the world, Nephleinsinite is a type of igneous ore that contains useful compounds of K<sub>2</sub>O, Na<sub>2</sub>O, Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub> which become diverse commercial products in the course of various processes. Appropriately, Alumina production from bauxite is the most commonly used aluminum production methods. However, the economics of making alumina from Nephleinsinite are more cost effective because of the supply of cement, sodium carbonate, potassium carbonate, potassium sulfate and silicate. Since the cost of using Nephleinsinite is 15 to 20 percent cheaper than Bauxite, the technology of alumina production from Nephleinsinite is perfectly considered and expanding in most of the countries over the globe. So that, in order to improve the physical properties of the catalyst support, the extrudates were stored in a concentrated nitric acid solution at different times. The effects of nitric acid on the catalyst support were studied by using XRD, BET and SEM techniques.

### II. EXPERIMENTAL SECTION

In this research, Boehmite powder was produced from Nephleinsinite Azar Shahr ore mine as a support powder and all the other raw materials, including nitric acid and hydroxyethyl cellulose, were used with laboratory purity (Merck Germany). Therefore, the PW1800 was utilized to perform the XRD test. The Belsorp mini II device was used to perform the BET tests.

#### a) General Method for preparing the Gamma Alumina Catalyst support

In order to prepare the extruded catalyst support, firstly; Boehmite powder is mixed with 5% by weight of hydroxyethyl cellulose powder (HEC) and then spray enough water on it. Afterwards, the mixture is well blended to obtain a homogeneous paste. The paste was passed through the extruder and the extrudates were dried for 2 hours. Then they kept at 120 ° C for 1 night in an oven. Later, they were calcined in the furnace with a temperature of 100 ° C / hr to 600 ° C.

#### b) Studying aging time

After preparation of the extrudates catalyst support, 65% nitric acid solution was prepared in four containers so as to evaluate the effect of aging time. Alternatively, extrudates were stored in each of these containers for 72, 48, 24 and 120 hours, and then extrudates were kept in oven for 1 night at 120 ° C.

### III. RESULTS AND DISCUSSION

#### a) The effects of aging times on the extrudates properties

The effect of aging time was investigated on catalyst support by nitric acid concentration for five times. The results are shown in Table 1. All prepared samples except sample 4 had a high mechanical strength. Because sample 4 had a fragile texture, the BET test was not done at it.

The maximum Pore Volume (P.V.) and A.P.D. of the catalyst support are resulted in 65% nitric acid with 72 hr aging time. Besides, longer aging time will lead to a



reduction in the mechanical strength of the catalyst support. Therefore, sample 3 was selected as an optimized sample.

Table 1. The BET and mechanical results of untreated catalyst support and treated samples

Sample Name	Aging time(hrs)	S.A (m <sup>2</sup> /gr)	P.V.(cc/gr)	A.P.D.
Untreated	0	231	0.54	9.06
Number 1	24	221	0.6	10.88
Number 2	48	216	0.67	12.39
Number 3	72	226	0.76	13.49
- Number 4	120	-	-	-

#### b) XRD Analysis

The crystalline structure of the untreated and the optimized sample (sample No.3) was investigated using XRD analysis. It can be observed that both samples have  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> structure. Furthermore, aging in nitric acid did not affect its crystalline phase in spite of its effect on the properties of the tissue of the sample. In Table 2, the XRD analysis of these two samples is presented properly.

Table 2 - XRD analysis results of two untreated and an optimized sample

Optimized sample(No 3)		Untreated sample	
JCPDS Card no.	d spacing (observed)	JCPDS Card no.	d spacing (observed)
004-0858		004-0858	
2.75	2.7806	2.750	2.734
2.43	2.4095	2.430	2.453
2.300	2.2746	2.300	2.289
2.000	1.9746	2.000	2.014
1.400	1.3984	1.400	1.410

#### c) Scanning Electron Microscopy Studies

Figure 1 shows the SEM images for both of the untreated (left) and the optimized sample (right), the SEM results show that with nitric acid treatment no agglomeration was occurred.

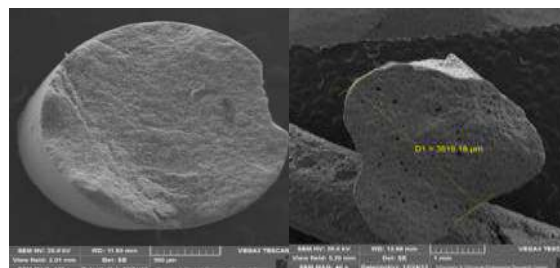


Figure 1. SEM images of untreated and optimized sample

#### IV. CONCLUSION

- Aging the extrudates in nitric acid cause to pilling effect. By increasing the aging times in nitric acid, the P.V. and A.P.D. of extrudates are increased.
- Due to the corrosive nature of nitric acid, the mechanical strength properties of the extrudates which are aged more than 72 hr are reduced.
- Aging in nitric acid does not change the crystalline phase of catalyst support; the the gamma alumina crystalline phase remains appropriately.
- The extrudates catalyst support obtained from the aging in nitric acid has optimized S.A. properties. They can be used as a catalyst support in the desulfurization processes of heavy oil fractions containing large sulfur molecules.

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