# Process for Preparing Pseudo-Boehmite from Aluminium Trihydrate

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## Abstract

Pseudo-Boehmite is an essential raw material, used for making catalyst substrate for various applications in petroleum and polymer industries. It is having property of self-binding and easily forms into extruded parts and pellets. Extruded pseudo-Boehmite material can be activated into transition phases, having high surface area, pore volume and excellent thermal stability. The unstable transitional alumina (pseudo-Boehmite) has the property of forming gel with acids such as Hydrochloric acid, acetic acid or formic acid. Gelling property of pseudo-Boehmite makes it highly desirable for use in the manufacture of FCC (fluid catalytic cracking) catalysts as FCC of petroleum products requires gelling catalysts for efficient cracking of the petroleum products.

Pseudo-Boehmite is generally manufactured commercially by processes involving neutralization of aluminium salts by alkalis, such as acidification of aluminate salts with acids and hydrolysis of aluminum alkoxide. In above processes, there is generation of large amounts of hazardous wastes due to large scale precipitation and washing, resulting in environmental pollution.

This paper elaborates on new and improved processes for preparing pseudo-Boehmite from aluminium trihydrate, which produce pseudo-Boehmite in high yield and high purity. This process is simple and easy to carry out, economical, reduce wastage, and it is environmental friendly. It also reduces energy requirements

**Keywords:** Pseudo-Boehmite, FCC (fluid catalytic cracking), Aluminium trihydrate, Calcination, Digestion.

## 1. Introduction

The term, Boehmite, is used in the chemical industry to describe alumina hydrates which exhibit X-Ray Diffraction (XRD) patterns close to that of the Aluminum oxide-hydroxide [AlO(OH)], naturally occurring Boehmite or diaspore. Boehmite, usually broadly describes a wide range of alumina hydrates which contain different amounts of water of hydration, different surface areas, pore volumes, specific densities and also exhibit different thermal characteristics while undergoing thermal treatments. XRD patterns of alumina hydrates, although exhibit the characteristic of Boehmite [AlO(OH)] peaks, usually vary in their widths and can also shift in their location. The sharpness of the XRD peaks and their location are indicative of degree of crystallinity, crystal size and amount of imperfections in the crystal structures [1].

Broadly, Boehmite aluminas can be classified into two categories; the first category contains Boehmites which have been synthesized and/or aged at temperatures close to 100 °C and most of the time under ambient atmospheric pressures. This type of Boehmite is poorly crystallised form of Boehmite and is generally referred to as quasi-crystalline Boehmites or pseudo-Boehmites.

The second category of Boehmite consists of so-called micro-crystalline Boehmites. Micro crystalline Boehmites are Boehmites with high degree of crystallinity, relatively large crystal size and low surface area. XRD pattern of pseudo-Boehmite differs from crystalline Boehmite in broadening of the reflections and higher ‘d’spacing values. Pseudo-Boehmite is widely used for making catalyst substrate for various applications, majorly in petroleum and polymer industries. Pseudo-Boehmite is having property of self-binding and easily forms into extrudes and pellets.

Extruded pseudo-Boehmite material can be activated into transition phases having high surface area, pore volume and excellent thermal stability. The unstable transition alumina (pseudo-Boehmite) is having property of formation of gel with acids like hydrochloric acid, acetic acid or formic acid. Gelling property of pseudo-Boehmite makes it highly desirable for use in the manufacture of Fluid Catalytic Cracking (FCC) catalysts. FCC of petroleum products requires gelling catalysts for efficient cracking of the petroleum products.

Pseudo-Boehmite is generally manufactured in commercial scale by processes involving neutralisation of aluminium salts by alkalis, acidification of aluminate salts and hydrolysis of aluminium alkoxide [2] In the said processes, there is a generation of large amounts of hazardous wastes due to large scale precipitation and washing, thereby leading to environmental pollution.

Thus, there is need and scope for a new and improved process for preparing pseudo-Boehmite from aluminum trihydrate, which can produce pseudo-Boehmite in high yield and purity, which is simple and easy to carry out, economical and reduce wastage, environmental friendly and reduce energy requirements [3].

## 2. Experimental Details

The present study describes about a process for preparing pseudo-Boehmite crystals from aluminum trihydrate. The process provides pseudo-Boehmite crystals with high specific surface area and may provide enhanced functionality. The process for preparing pseudo-Boehmite crystals comprises of calcinating aluminum trihydrate to form alumina. The aluminium trihydrate is calcinated at a temperature in the range of 300 °C to 500 °C.

The calcination may be carried out in any known equipment including but not limited to a calciner, an electric furnace, a vertical shaft kiln or a rotary kiln. In a preferred embodiment, the aluminium trihydrate is calcinated in a calciner. In accordance with an embodiment, the alumina formed by calcining the aluminium trihydrate has a specific surface area in the range of 150 to 300 $m^2/g$. Preferably, the alumina has a specific surface area of 200 to 300 $m^2/g$.

The alumina was milled to obtain alumina particles having a particle size distribution in the range of 10 to 70 µm. Milled alumina enables efficient conversion to pseudo-Boehmite and requires lower energy. The calcined alumina was milled in a grinding media such as a ball mill made of alumina. Preferably, the calcined alumina is milled with 18 to 25 % of grinding media made of alumina having greater than 90 %.

A slurry of the alumina particles is formed in an aqueous solution of acetic acid. The aqueous solution of acetic acid for forming the slurry of alumina had a volume/volume ratio of water and acid in the range of 1:2. The slurry of the alumina was prepared with solid content in the range of 15 to 25 %. The slurry of the alumina particles in the aqueous solution of acetic acid was maintained at a pH in the range of 2 to 4.

This process further comprised of heating the slurry under pressure. The slurry of the alumina particles was heated at a temperature in the range of 170 ºC to 220 ºC. The slurry of the alumina was heated at pressure in the range of 2000 kPa to 3000 kPa. The slurry of alumina particle was heated under pressure in an autoclave. The slurry was flashed from autoclave by withdrawing the pressure to form pseudo-Boehmite.

The pseudo-Boehmite crystals thus obtained were separated from the slurry through filtration. The separated pseudo-Boehmite crystals were dried in the tray drier in the range of temperature between 80 – 90 °C. The obtained products were analysed in the XRD equipment; PAN Analytical Xpert Pro, Co-lamp (Wavelength = 1.78 A°), 45 kV.

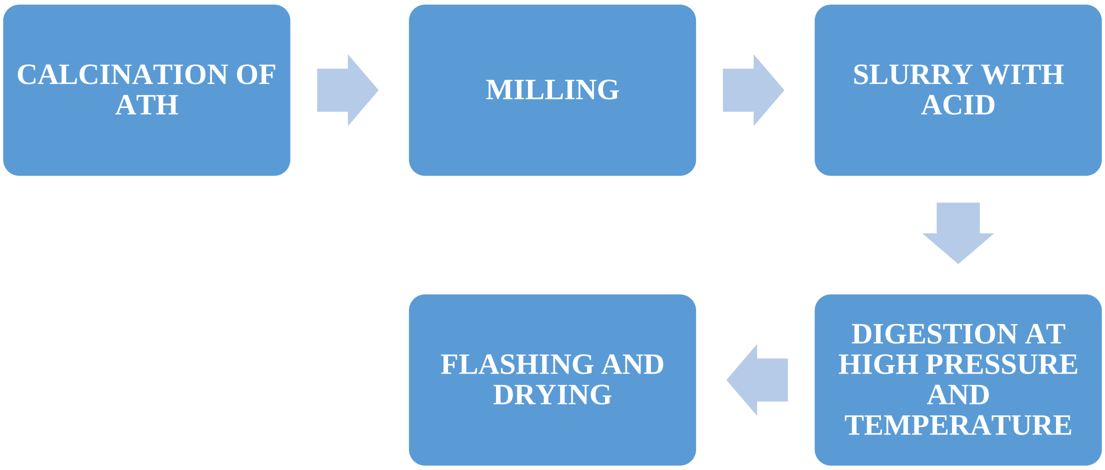

**Figure 1. Process flow diagram for pseudo-boehmite**

## 3. Results

### 3.1 Effect of heating time with coarse alumina feed

Aluminum trihydrate was calcined in a calciner at 300 °C – 500 °C in a continuous mode at feed rate of 20 to 22 kg/hour. Calcined alumina (390 g) was transferred into an autoclave and water (2210 g) was added to the calcined alumina to form 15 % solid slurry having a pH 9. The slurry was heated to 220 °C for different periods of time at a pressure of 3000 kPa. The slurry was flashed by withdrawing the pressure abruptly. The product was filtered out and dried overnight in a tray drier at 80 °C. Phase analysis of the product by XRD (X-Ray Diffraction) was carried out. XRD phase and size of the product are as shown in the following Table 1.

**Table 1. Properties of Boehmite, obtained with different heating time.**

| $d_{50}$ (µm) | Heating time (minutes) | Specific Surface Area ($m^2/g$) | XRD patterns |
|---|---|---|---|
| 70 | 120 | 40.7 | Boehmite |
| 70 | 60 | 113.1 | Boehmite |
| 70 | 5 | 182 | Boehmite |

Table 1 shows that the product formed was Boehmite crystals. Specific surface area (SSA) of the product decreases with increasing heating time. Fig 2 of the accompanying drawing represents XRD pattern of the product for different heating period.

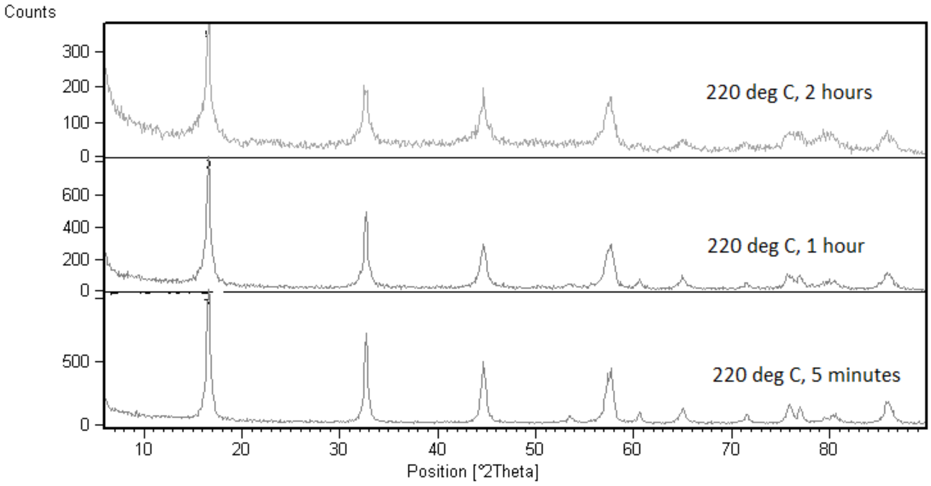

**Figure 2. XRD patterns of Boehmite formed with different heating time.**

### 3.2 Effect of heating temperature with fine feed alumina

Aluminum trihydrate was calcined in a calciner at 300 – 500°C in a continuous mode at feed rate of 20 to 22 kg/hour. Calcined alumina (390 g) was milled in a jar bill to an average alumina particle size distribution of 5 – 10 µm. Alumina particles were transferred into an autoclave and water (2210 g) was added to the alumina particles to form a 15 % solid slurry, having a pH 9. The slurry was heated to varying temperatures for 5 minutes at a pressure of 3000 kPa atmospheric pressure. The slurry was flashed out by withdrawing the pressure abruptly. The product was filtered and dried overnight in a tray drier at 80 °C. XRD patterns and size of the product are shown in the following Table 2.

**Table 2. Properties of Boehmite, prepared with Fine alumina feed.**

| $d_{50}$ (µm) | Heating temperature (°C) | Specific Surface Area ($m^2/g$) | XRD patterns |
|---|---|---|---|
| 10 | 200 | 79.9 | Boehmite |
| 10 | 160 | 212 | Boehmite |

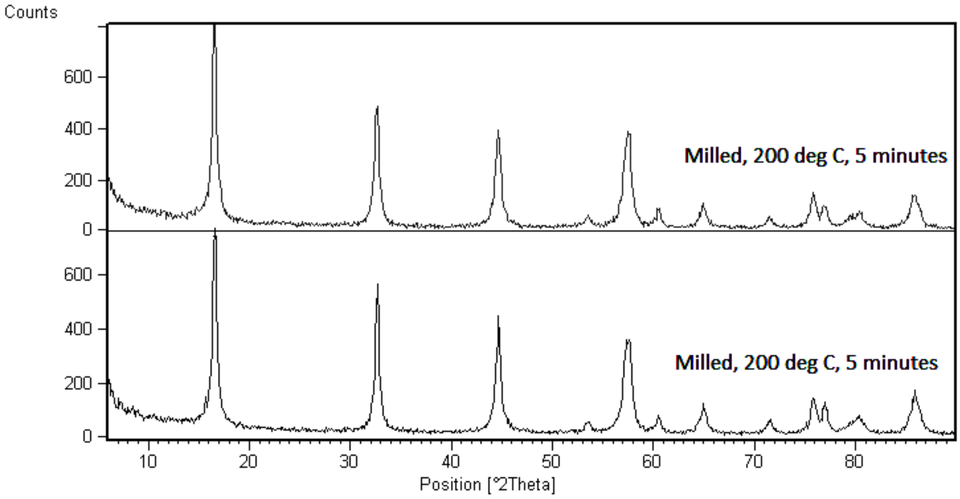

**Figure 3. XRD pattern of Boehmite, formed at 200oC for 5 minutes**

The XRD patterns of the product obtained, indicates that only Boehmite is obtained when the slurry of Alumina particles is formed in water alone. Milled alumina heated to 200 °C for 5 mins and having SSA of 79.9 is as shown in Figure 3.

### 3.3 Effect of heating time with fine feed alumina

Aluminum trihydrate was calcined in a calciner at 350 – 500 °C in a continuous manner at a feed rate of 20 to 22 kg/hour. Calcined alumina particles were transferred into an autoclave and water (2210 g) and HCl (100 ml) in 1:1 v/v ratio were added to the calcined alumina to form a 15 % solid slurry having an acid pH between 2 and 4. The slurry was heated to 200 °C at different periods of time at a pressure of 3000 kPa The slurry was flashed out by withdrawing the pressure abruptly. The product was filtered and dried overnight in a tray drier at 80 °C. XRD patterns and sizes of the products are shown in the following Table 3:

**Table 3: Properties of Boehmite, prepared with fine alumina feed**

| $d_{50}$ (µm) | Heating Time (Minutes) | Specific Surface Area ($m^2/g$) | XRD patterns |
|---|---|---|---|
| 10 | 120 | 189.5 | Boehmite |
| 10 | 30 | 255.5 | Boehmite |

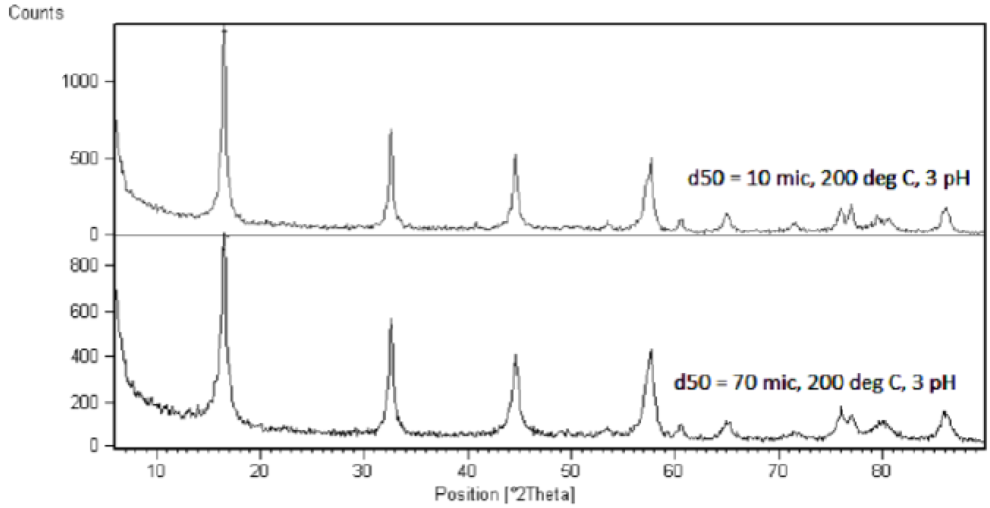

**Figure 4. XRD pattern of Boehmite formed at 200 ºC with different heating time.**

Figure 4 illustrates the XRD pattern of the product. The XRD patterns of the product obtained, indicated that only Boehmite is obtained when the slurry of Alumina particles is formed in HCl.

### 3.4 Effect of pH with feed alumina

Aluminum trihydrate was calcined at 300 – 500 °C in a continuous mode at a feed rate of 20 to 22 kg/hour. Calcined alumina particles were transferred into an autoclave and water (2210 g) and acetic acid (100 ml) in 1:1 v/v ratio were added to the calcined alumina to form a 15% solid slurry having an acid pH between 2 and 4. The slurry was heated to 120 °C for 120 minutes at a pressure of 3200 kPa. The slurry was flashed out by withdrawing the pressure abruptly. The product was filtered and dried overnight in a tray drier at 80 °C. XRD phase and sizes of the product were as shown in the following Table 4.

**Table 4. Properties of Pseudo-Boehmite, formed with acetic acid slurry**

| $d_{50}$ (µm) | Heating Time (Minutes) | Specific Surface Area ($m^2/g$) | XRD patterns |
|---|---|---|---|
| 10 | 120 | 150 | Pseudo-Boehmite |
| 70 | 120 | 260 | Pseudo-Boehmite |

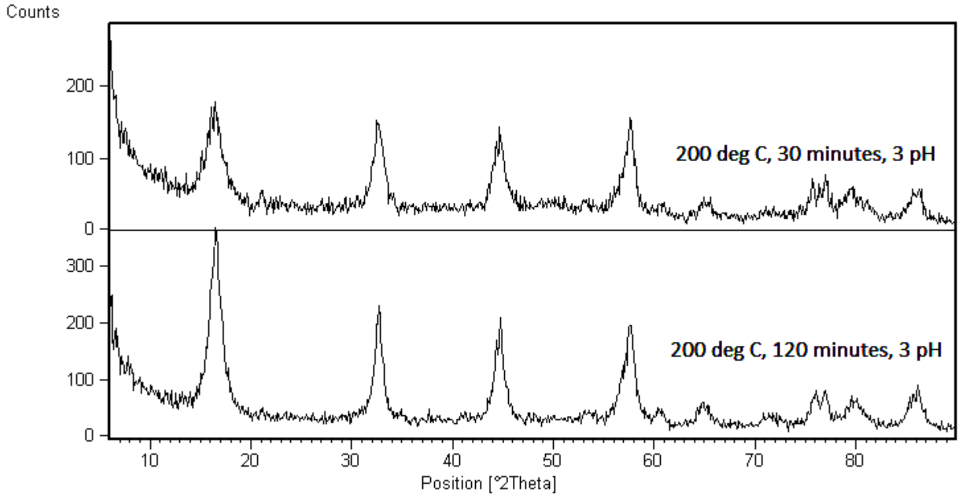

**Figure 5. XRD pattern of Pseudo-Boehmite prepared with acetic acid slurry.**

Figure 5 of the accompanying drawing shows XRD pattern of product obtained. The XRD patterns of the product obtained, indicated that only Pseudo-Boehmite is obtained when the slurry of Alumina particles is formed in water and acetic acid.

## 4. Conclusions

A process was developed for preparing pseudo-Boehmite crystals comprising from aluminum tri-hydrate. In this process, aluminium tri-hydrate is to be calcined in flash calciner to form amorphous alumina with specific surface area in the range of 200 to 300 m$^2$/g. The aqueous solution of acetic acid has a volume/volume ratio of water and acetic acid in the range of 1:2 and heated at a pressure in the range of 20 bar to 30 bar.

The slurry of the alumina particles has a solid content of 15 to 25 % and heated at a temperature range of 120 to 220 $^o$C. The pseudo-Boehmite crystals are dried at a temperature ranging from 80 to 90 °C having the surface area in the range of around 250 m$^2$/g.

Maintaining the pH of the slurry of the alumina particles between 2 to 4 suppresses the crystalline phase of the Boehmite, during heating of the slurry. As a result, the pseudo Boehmite crystal remains semi amorphous Boehmite, thus avoiding the formation of gibbsite. The pseudo Boehmite crystals obtained by this process have a specific surface area in the range of 90 to 260 m$^2$/g. The pseudo-Boehmite crystals obtained, having a specific surface area in the said range are ideal for use as refinery catalysts for fluid catalyst cracking of hydrocarbons.

The disclosed process enables a process for preparation of pseudo Boehmite crystals with high specific surface area that are ideal and well suited for use as refinery catalysts for fluid catalyst cracking of hydrocarbons. Specifically, the above process to obtain pseudo Boehmite crystals is simple, easy to perform, economical, reduces the process time and energy required, and significantly improves properties of the desired product. High yield, simple to perform process steps and reduced process time makes the process very efficient

## 5. References

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