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(71) Applicant (for all designated States except US):
ADITYA BIRLA SCIENCE & TECHNOLOGY CO. LIMITED [IN/IN]; Aditya Birla Centre, 2nd Floor, C Wing, S. K. Ahire Marg, Worli, Mumbai 400 025 (IN).

(72) Inventors; and

(75) Inventors/Applicants (for US only): **MOHAPATRA, Sanjaya, Ranjana** [IN/IN]; Aditya Birla Science & Technology Co. Ltd., Aditya Birla Centre, 2nd Floor, C Wing, S. K. Ahire Marg, Mumbai 400 025, Maharashtra (IN). **JADHAV, Rahul** [TH/TH]; Aditya Birla Science & Technology Co. Ltd., Aditya Birla Centre, 2nd Floor, C Wing, S. K. Ahire Marg, Mumbai 400 025, Maharashtra (IN). **PATIL, Narendra, Singh** [IN/IN]; Aditya Birla Science & Technology Co. Ltd., Aditya Birla Centre, 2nd Floor, C Wing, S. K. Ahire Marg, Mumbai 400 025, Maharashtra (IN). **KULKARNI, Sadguru** [IN/IN]; Aditya Birla Science & Technology Co. Ltd., Aditya Birla Centre, 2nd Floor, C Wing, S. K. Ahire Marg, Mumbai 400 025, Maharashtra (IN). **PURI, Prashant, Mickey** [IN/IN]; Aditya Birla Science & Technology Co. Ltd., Aditya Birla Centre, 2nd Floor, C Wing, S. K. Ahire Marg, Mumbai 400 025, Maharashtra (IN).

(74) Agent: **DEWAN, Mohan**; R.K. Dewan & Company, Podar Chambers, S.A. Brelvi Road, Fort, Mumbai 400 001, Maharashtra (IN).

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(54) Title: A PROCESS FOR MANUFACTURING OF ALUMINIUM FLUORIDE

(57) Abstract: There is provided a process for manufacturing aluminium fluoride having larger particle size wherein a by product of the fertilizer industry predominantly containing hydrofluorosilicic acid is used as the starting material along with aluminium hydroxide. Solid aluminium fluoride as obtained by the process of the present invention has particle size in the range of 80 microns to 90 microns and moisture content less than 1%.



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A PROCESS FOR MANUFACTURING OF ALUMINIUM FLUORIDE

Field of the invention

This invention relates to a process of manufacturing aluminium fluoride.

Background of the Invention

Aluminum fluoride (AlF_3) is an inorganic solid well known in the art of heterogeneous catalysts preparation. It is used as such and as a support for metal salts having catalytic activity due to its strong acidity according to Lewis and/or its thermal and chemical inertia. It is widely used, for example, in the fluorination of chlorinated organic compounds to obtain the corresponding fluorinated compounds. Aluminium fluoride is also used as a raw material for ceramics such as an optical glass. Aluminium fluoride (AlF_3) is also used as a fluxing agent for the electrolysis of aluminium. The anhydrous aluminium fluoride improves the production yield of aluminum smelters, and lowers its electrolytic cost.

The preparation on an industrial scale of the aluminum fluoride is carried out by fluorination of the aluminum oxide (alumina) with anhydrous hydrofluoric acid (HF).

There are two known methods for manufacturing aluminium fluoride namely a dry method and a wet method. The dry process comprises fluorinating aluminum hydroxide with a hydrogen fluoride gas and the wet process comprises adding aluminum hydroxide to hydrofluoric acid to produce a super-saturated solution of aluminum fluoride and crystallizing aluminum fluoride from this solution.

These abovementioned conventional methods mainly employ Hydrofluoric acid (HF) as one of the starting material. Since the use of hydrofluoric acid is expensive, these methods are industrially and economically are not viable methods.

Existing Knowledge

US Patent 6080210 discloses a process for producing aluminum fluoride particles, comprising the steps of adding aluminum fluoride seed crystals to a super saturated solution of aluminum fluoride; allowing aluminum fluoride particles to be precipitated under heating and agitation; collecting the aluminum fluoride particles by filtration; and drying them. However, the process results into an aluminium fluoride having smaller particle size and greater silica content.

US Patent 5707406 discloses a method of manufacturing aluminum fluoride anhydride by means of a wet method. This method is featured in that a seed crystal of $\text{AlF}_3 \cdot 3\text{H}_2\text{O}$ containing not more than 5% of fine particle 40 micrometer or less in diameter is added into the super saturated solution of aluminum fluoride in such a ratio that the total surface area of the seed crystal is in the range of 40-100m² per 1 kg of $\text{AlF}_3 \cdot 3\text{H}_2\text{O}$ to be precipitated until an initial concentration of super saturated solution is reduced to 1.6%. The initial concentration of super saturated solution is adjusted to 8-15%. Resultant slurry is heated under agitation, thereby precipitating in batch wise large $\text{AlF}_3 \cdot 3\text{H}_2\text{O}$ particles, which are then separated, dried and dehydrated. In this process, large amount of AlF_3 seed

are required to prepare large particles of AlF_3 , which makes the process uneconomical.

There is thus felt a need for a process of manufacturing aluminium fluoride having low silica content and larger particle size in a cost-effective manner.

Object of the Invention

It is an object of the present invention is to provide a process for the preparation of aluminum fluoride anhydride having low silica content.

Another object of the present invention is to provide a process for preparation of aluminum fluoride anhydride having large particle size.

Yet another object of the present invention is to provide a process for preparation of aluminum fluoride anhydride in high purity and high yield.

Yet another object of the present invention is to provide a cost-effective and economically feasible process for preparation of aluminum fluoride anhydride.

Still another object of the present invention is to provide a process for preparation of aluminum fluoride anhydride which is carried out at faster rate.

Summary of the Invention

In accordance with the present invention there is provided a process for manufacturing aluminium fluoride having large particle size comprising the following steps:

1. A process for manufacturing aluminium fluoride anhydride having large particle size comprising the following steps:
 - a. reacting aluminium compound having moisture content less than 2% with a preheated Hydrofluorosilicic acid solution (40 to 105⁰C) in a reactor to obtain a first slurry containing silica and aluminium fluoride;
 - b. filtering the first slurry immediately in a continuous solid liquid separator to separate silica cake and to yield a first filtrate containing aluminium fluoride;
 - c. subjecting the first filtrate to crystallization by seeding the first filtrate with aluminium fluoride trihydrate to obtain a second slurry containing crystals of aluminium fluoride;
 - d. filtering the second slurry to yield a wet cake of aluminium fluoride crystals; and
 - e. calcining said wet cake to obtain aluminium fluoride anhydride having moisture content less than 1%.

Preferably, the aluminium compound is selected from a group consisting of aluminium hydroxide and aluminium oxide.

Typically, the mole ratio of aluminium compound to Hydrofluorosilicic acid is in the range of 1.7 to 1.99.

Preferably, the aluminium compound is aluminium hydroxide and the mole ratio of aluminium hydroxide to Hydrofluorosilicic acid is 1.97:1.

Preferably, the aluminium compound is aluminium hydroxide having moisture content less than 0.5 % in process step (a).

Preferably, the aluminium hydroxide is heated at a temperature in the range of about 90 -110⁰C.

Typically, the hydrofluorosilicic acid in process step (a) is a byproduct of the fertilizer industry.

Typically, the hydrofluorosilicic acid solution is heated at a temperature in the range of about 40 °C to 105 °C in process step (a).

Preferably, the concentration of the hydrofluorosilicic acid solution is in the range of 5 % to 25 % in process step (a).

Preferably, the process step (a) is carried out at a temperature in the range of about 40 °C to 110 °C for 1 to 20 minutes.

Typically, the process step (a) is continued until the hydrofluorosilicic acid left in the reactor is less than 0.5% by mass.

Typically, the silica in process step (a) is obtained as a silica cake having 30% moisture content.

Typically, the first slurry is filtered within 15 minutes from its formation in process step (b).

Preferably, the first slurry is filtered using continuous filter centrifuge in process step (b).

Typically, the crystallizer is maintained at a temperature in the range of about 70 -90°C in process step (c).

Typically, the filtrate containing aluminium fluoride is fed to the crystallizer in batches in process step (c).

Typically, the aluminium fluoride trihydrate in process step (c) is in the form of wet powder.

Typically, the aluminium fluoride trihydrate is added in the amount of 15 % by mass of the total weight of the aluminium fluoride into the crystallizer in the process step (c).

Preferably, several similar batches are introduced to the crystallizer at regular time interval such that the soluble aluminium fluoride concentration is not less than 9% by mass in the process step (c).

Preferably, crystallization is carried out for about 4 hours.

Typically, the aluminium fluoride wet cake crystals are separated out by continuous filtration centrifuge in the process step (d).

Typically, the aluminium fluoride cake has moisture content less than about 20 % by mass.

In accordance with this invention, the process step of calcining comprises

- (i) heating the wet cake of aluminium fluoride at a temperature of about 160⁰C to remove the surface moisture; and
- (ii) heating the resultant aluminium fluoride at a temperature of about 500-600⁰C in an oven to obtain aluminium fluoride powder having moisture content less than 1%.

In accordance with this invention the particle size of the aluminium fluoride anhydride is in the range of 80 microns to 90 microns.

Brief Description of the accompanying Drawings

The invention will now be described with reference to accompanying drawings, in which

Figure 1 illustrates the X-ray powder diffractogram of aluminum hydroxide;

Figure 2 illustrate the X-ray powder diffractogram of aluminum fluoride trihydrate;

Figure 3 illustrates the X-ray powder diffractogram of aluminum fluoride anhydride

Figure 4 shows a flowchart of the process for preparation of aluminium fluoride in accordance with the invention.

Figure 5 shows a graph wherein product yield of AlF_3 (On Y-Axis) is plotted against the different initial concentrations of AlF_3 in the crystallizer on X-axis;

Figure 6 shows a graphical representation shows the effect of reaction time on silica content in aluminium fluoride on Y-axis along with the time on X-axis;

Figure 7 shows a graph wherein the silica content of AlF_3 (On Y-axis) is plotted against the different mole ratios of aluminium hydroxide to hydrofluorosilicic acid.

Detailed Description of the Invention

Aluminium fluoride is the inorganic compound with the formula AlF_3 . Aluminium fluoride trihydrate is rarely found in nature as mineral rosenbergite. Aluminium fluoride is an important additive during electrolyte aluminium production because it lowers the melting point of the alumina feed and increases the electrolyte's conductivity. The conventional method for manufacturing of aluminium fluoride mainly uses hydrofluoric acid as one of the raw material. This makes the overall process expensive and thus industrially and economically not feasible methods. Accordingly, the present invention provides an effective process for manufacturing of aluminium fluoride having larger particle size. A process in accordance with the present invention is a cost-effective process used for manufacturing of aluminium fluoride in high yield and high purity.

Hydrofluorosilicic acid, which is a raw material useful for producing potassium fluoride, is a by-product produced in large amount in the manufacture of well known phosphate fertilizers. Rock phosphate is a raw material used in the manufacture of most commercial phosphate fertilizers. Rock phosphate containing fluorides such as calcium fluoride and silica as impurity and mineral phosphates having 5% to 10% calcium fluoride is treated with previously diluted sulphuric acid. This reaction converts the tertiary calcium phosphate into primary phosphate rendering it soluble in water and liberates the fluorine of the calcium fluoride, forming hydrofluoric acid. This acid in turn combines with the silica forming silicon tetrafluoride, a gas, escapes into the air and is detrimental to surroundings. In order to prevent the escape of this gas, the gas is then sprayed with water in condensing towers into a series of scrubbers and dissolved in water and this decomposes the silicon tetrafluoride into Hydrofluorosilicic acid and silicic acid. This is the crude form of Hydrofluorosilicic acid. The purified form is obtained by distillation of the crude acid. The hydrofluorosilicic acid can also be prepared by the reaction of hexafluorosilicate, apatite and/or fluorite (fluorspar) with sulphuric acid. Until now, Hydrofluorosilicic acid has been barely utilized, and mostly discarded as a toxic waste. However, in recent times increasing attention has been paid to the utilization of hydrofluorosilicic acid in view of saving natural resources, and reducing environmental pollution.

The present invention envisages the use of Hydrofluorosilicic acid, a byproduct of the fertilizer industry and aluminium hydroxide for the preparation of aluminium fluoride.

Thus, in accordance with this invention, a process for manufacturing aluminium fluoride having larger particle size comprises the following steps:

A by-product from the fertilizer industry predominantly containing hydrofluorosilicic acid which is preheated at a temperature in the range of about 40⁰C to 105⁰C (having concentration in the range of 5% to 25% is reacted with aluminium compound. This reaction is carried out at a temperature in the range of about 40⁰ to 110⁰C for 1-20 minutes to obtain a first slurry containing silica and aluminium fluoride. Typically, the reaction is continued until the hydrofluorosilicic acid left in the reactor is less than 0.5% by mass.

In accordance with process of the present invention the aluminium compound is selected from a group consisting of aluminium hydroxide and aluminium oxide. The mole ratio of aluminium compound to hydrofluorosilicic acid is in the range of 1.7 to 1.99. More particularly, the aluminium compound is aluminium hydroxide and the mole ratio of aluminium hydroxide to hydrofluorosilicic acid is 1.97:1.

In accordance with the process of this invention the aluminium hydroxide with 98.1% purity is heated to 90-110⁰C to reduce the moisture content less than 0.2%.

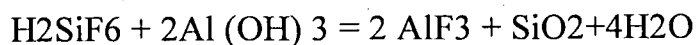
As the resultant aluminium fluoride solution is metastable and the trihydrate begins to crystallize out quickly at temperature of about 90⁰C, the first slurry from the reactor is filtered immediately in a continuous filter centrifuge to separate silica cake and to yield a first filtrate aluminium fluoride. Preferably, the silica cake having moisture content 30% by weight and aluminium fluoride content 10% by mass is obtained. To avoid the aluminium fluoride being contaminated with silica, the first slurry is filtered. The filtrate predominantly containing aluminium fluoride is subjected to the crystallization by seeding the first filtrate with aluminium fluoride trihydrate. The crystallizer is maintained at a temperature in the range of

about 70-90⁰C and the crystallization is carried out for about 4 hrs to obtain a second slurry containing crystals of aluminium fluoride. The stirring speed is such that it is just sufficient to keep all the aluminium particles in motion. Typically, a wet powder of aluminium fluoride trihydrate is added in the amount of 15% by mass of the total weight of the aluminium fluoride into the crystallizer. The crystallization process is continued till the soluble aluminium fluoride content falls below 3% by mass.

In accordance with the process of the invention, several similar batches are prepared and introduced to the crystallizer in 20 minutes time interval such that the soluble aluminium fluoride concentration is not less than 9% by mass to get larger particle size of aluminium fluoride crystals. Then the second slurry is filtered by using continuous filtration centrifuge to yield a wet cake of aluminium fluoride crystals.

The separated aluminium fluoride wet cake is subjected to heating at a temperature of about 160⁰C to remove the surface moisture and then subjected to calcination at a temperature in the range of about 500-600⁰C in an oven to obtain aluminium fluoride anhydride powder having moisture content less than 1% and particle size in the range of 80 microns to 90 microns.

In accordance with this invention the aluminium fluoride is produced by the following reaction



The invention will now be described with respect to the following examples which do not limit the invention in any way and only exemplify the invention.

Example-1

375Kg of aluminum hydroxide (98.1% purity) was heated to 110⁰C for having moisture content less than 0.2% and added to preheated (86⁰C) 1725Kg of 20% Hydrofluorosilicic acid solution. The reaction was carried out at 98⁰C for 10 minute. In this reaction mole ratio of aluminum hydroxide to Hydrofluorosilicic acid was 1.97:1.

After this, silica (236.1 Kg wet cake having moisture content 30% by wt and aluminum fluoride content 10 % by wt) was filtered out within 5 minutes by continuous filter centrifuge and filtrate was transferred to the crystallizer maintained at 86⁰C to get one reactor batch. 300 kg of water was used for washing.

100 Kg of seed (aluminum fluoride trihydrate solid wet powder) was added to it. After 20 minute interval, another four similar reaction batches were transferred to the crystallizer one after another. The aluminum fluoride concentration in crystallizer was above 13 % till last batch added to crystallizer. After addition of the last batch was completed, the crystallization continues for another 4 hrs. The soluble aluminum fluoride amount in crystallizer was found to be 1.9 %.

Then aluminum fluoride wet cake was filtered (having moisture content 12% by wt) and calcined at 600⁰C to give 339.2 Kg aluminum fluoride anhydride per reaction batch after subtracting the amount of seed added. The silica content of aluminum fluoride anhydride was found to be 0.12% by wt. The average particle diameter of aluminum fluoride anhydride was 90 micron. The moisture content was less than 0.4% by wt and purity was 98% by wt. The isolated product yield was 85.6 %. The filtrate can be re used for making Hydrofluorosilicic acid.

Example 2: (Particle Size distribution)

The particle size of aluminum fluoride anhydride was analyzed by sieve. The following table shows the particle size distribution in terms of percentage by mass.

Aluminum fluoride anhydride particles	% by mass
+105 micron	44
+75 to +105 micron	50
+45 to +75 micron	4.9
-45 micron	1.1

Example 3: (Effect of moisture content of aluminum hydroxide on reaction time)

The reaction was carried out as per Example-1 by adding aluminum hydroxide powder having different moisture content and the reaction was monitored by fluoro-meter by analyzing free fluoride content in solution.

Amount of aluminum hydroxide added (Kg) and its purity	% Moisture content of aluminum hydroxide	Time taken (in min) to complete the reaction	% Un reacted Hydrofluorosilicic acid calculated from free fluorine content by fluoro meter.
376.2(97.8% purity)	0.5	12	0.25
380.8 (96.6% purity)	1.7	18	0.25
388.9 (94.6% purity)	3.7	28	0.25

This showed that, during the reaction not only the preheated hydrofluorosilicic acid but also the moisture content of aluminum hydroxide powder added to a pre heated Hydrofluorosilicic acid, plays an important role for faster completion of the reaction. The higher the moisture content, longer is the time taken for reaction completion.

Example 4 (Silica cake separation by centrifuge)

The reaction was carried out as per Example-1 and the silica cake was filtered out with conventional belt filter. It was found that the centrifuge has advantages over belt filter.

The table given below shows the comparison between belt filter and basket type continuous centrifuge filter (used in example-1).

Equipment used	Moisture content of silica cake	% aluminum fluoride in wet silica cake	Total weight of silica cake obtained
Vertical shaft basket centrifuge Filtration medium : polypropylene filter bag 25 micron size Speed: 1440 rpm, Centrifugal acceleration: 210'G' (as example-1)	30%	10%	236.1
Belt filter having polypropylene filter cloth of 25 micron size	40%	15%	314.8

The results mentioned in the above table shows the advantages of continuous centrifuge filtration over the belt filter.

Example 5: (Reaction time vs. silica content in aluminum fluoride anhydride) (fig. 6)

The reaction was carried out as per Example-1, with different reaction time for aluminum hydroxide and Hydrofluorosilicic acid, the silica content in the final aluminum fluoride anhydride was found to be varied. The results were given below.

Thus it was found that from the figures given in the table, the extended reaction time results into the reduced silica content of the product.

Reaction time for aluminum hydroxide and Hydrofluorosilicic acid (in min)	% Silica content in the final aluminum fluoride anhydride
5	0.74
7	0.41
9	0.22
12	0.12
18	0.01

Example 6: (Mole ratio of aluminum hydroxide to Hydrofluorosilicic acid vs. silica content in aluminum fluoride anhydride)

When the reaction was carried out as per Example-1 with different amount of aluminum hydroxide and Hydrofluorosilicic acid, the silica content in the final aluminum fluoride anhydride was found to be varied. The result was given below.

Aluminum hydroxide amount (Kg).	Mole ratio of aluminum hydroxide to Hydrofluorosilicic acid	% Silica content in the final aluminum fluoride anhydride
333.14	1.75	3.5
342.66	1.8	2.4
371.2	1.95	0.2
375	1.97	0.12

The observations obtained from the above table showed that for low silica content the mole ratio of reactants (aluminum hydroxide or oxide to fluosilicic acid) was preferably in the range of about 1.93 to 1.99.

Example 7: (Aluminum fluoride concentration in crystallizer and aluminum fluoride trihydrate yield) (fig 5)

When the reaction was carried out as per Example-1 and the filtrate goes to crystallization vessel, where aluminum fluoride trihydrate crystallized out. Before crystallization starts the aluminum fluoride concentration in crystallizer was adjusted with water as mentioned in table given below, and 20 Kg of seed (aluminum fluoride anhydride) was added. No further reaction batches were added to the crystallizer. The crystallization was carried out similar to example-1. After 4 hrs of crystallization, the soluble aluminum fluoride amount in crystallizer was found to be 2% for all experiments. Then aluminum fluoride wet cake was filtered (having moisture content 12% by wt) and calcined at 600 deg C to give aluminum fluoride anhydride. For each concentration the amount of aluminum fluoride produced by calcining aluminum fluoride trihydrate was measured. The %

aluminum fluoride remains in solution (which did not precipitate) after saturation level reached from its super saturation level is also measured.

% Aluminum fluoride in solution for crystallization	% Aluminum fluoride loss as it did not precipitate out compared to total aluminum fluoride present in crystallizer.	% Aluminum fluoride crystallized out compared to total aluminum fluoride present in crystallizer.
17.5	9.4	90.7
15	11.2	89
13.5	12.2	87.8
12	13.2	86.4
9	18.3	81.5
6	27.5	72.3

The observations obtained from the above table showed that for better isolated AlF_3 yield, the concentration of AlF_3 in crystallizer was found to be more than 13%.

Example 8: (Seed amount vs. particle size)

The reaction was carried out as Example-1. 10 Kg of seed (aluminum fluoride anhydride) added to it. No further reaction batches were added to the crystallizer. The crystallization was carried out similar to example-1 by adding different amounts of aluminum fluoride trihydrate as a seed. After 4 hrs of crystallization, the soluble AlF_3 amount in crystallizer was found to be 2%. Then aluminum fluoride wet cake was filtered (having moisture

content 12% by wt) and calcined at 600 deg C to give 355.5 Kg aluminum fluoride anhydride. The particle size was analyzed and is as below:

Amount of seeds(as AlF ₃ ·3H ₂ O) added.(in kg)	% (by Wt) fines (below 63 micron)	% (by Wt) particles (above 90 micron)
49.6	14.33	44
424.1	53.97	45
1598.3	12.26	84
1884	3.4	93
2511.4	0.06	99.9

The result shows for less than 5% fines, one need to 1884 Kg AlF₃ powder in the conventional manner. However, in the present invention (as shown in Example-1) the same has been achieved by adding only 100Kg AlF₃.

Technical Advancement

The process as disclosed in the present invention offers several advancement over processes disclosed in the prior art in terms of particle size, yield, purity, faster reaction rate and cost-effectiveness. Furthermore, the process for the preparation of aluminium fluoride anhydride gives cheaper process by adding small amount of seed to get larger aluminium fluoride particles. The process offers faster reaction rate by maintaining the temperature during addition of aluminium hydroxide to preheated Hydrofluorosilicic acid. The process uses a continuous filtration centrifuge technique which lowers the filtration time and moisture content of the silica cake thereby increasing both the productivity and product yield of the aluminium fluoride anhydride.

While considerable emphasis has been placed herein on the specific steps of the preferred process, it will be appreciated that many steps can be made and that many changes can be made in the preferred steps without departing from the principles of the invention. These and other changes in the preferred steps of the invention will be apparent to those skilled in the art from the disclosure herein, whereby it is to be distinctly understood that the foregoing descriptive matter is to be interpreted merely as illustrative of the invention and not as a limitation.

Claims:

1. A process for manufacturing aluminium fluoride anhydride having large particle size comprising the following steps:
 - a. reacting aluminium compound having moisture content less than 2% with a preheated Hydrofluorosilicic acid solution (40 to 105°C) in a reactor to obtain a first slurry containing silica and aluminium fluoride;
 - b. filtering the first slurry immediately in a continuous solid liquid separator to separate silica cake and to yield a first filtrate containing aluminium fluoride;
 - c. subjecting the first filtrate to crystallization by seeding the first filtrate with aluminium fluoride trihydrate to obtain a second slurry containing crystals of aluminium fluoride;
 - d. filtering the second slurry to yield a wet cake of aluminium fluoride crystals; and
 - e. calcining said wet cake to obtain aluminium fluoride anhydride having moisture content less than 1%.
2. A process as claimed in claim 1, wherein the aluminium compound is selected from a group consisting of aluminium hydroxide and aluminium oxide.
3. A process as claimed in claim 1, wherein the mole ratio of aluminium compound to Hydrofluorosilicic acid is in the range of 1.7 to 1.99.

4. A process as claimed in claim 1, wherein the aluminium compound is aluminium hydroxide and the mole ratio of aluminium hydroxide to Hydrofluorosilicic acid is 1.97:1.
5. A process as claimed in claim 1, wherein the aluminium compound is aluminium hydroxide having moisture content less than 0.5 % in process step (a).
6. A process as claimed in claim 5, wherein the aluminium hydroxide is heated at a temperature in the range of about 90 -110⁰C.
7. A process as claimed in claim 1, wherein the hydrofluorosilicic acid in process step (a) is a byproduct of the fertilizer industry.
8. A process as claimed in claim 1, wherein the hydrofluorosilicic acid solution is heated at a temperature in the range of about 40 °C to 105 °C in process step (a).
9. A process as claimed in claim 1, wherein the concentration of the hydrofluorosilicic acid solution is in the range of 5 % to 25 % in process step (a).
10. A process as claimed in claim 1, wherein the process step (a) is carried out at a temperature in the range of about 40 °C to 110 °C for 1 to 20 minutes.

- 11.A process as claimed in claim 1, wherein the process step (a) is continued until the hydrofluorosilicic acid left in the reactor is less than 0.5% by mass.
- 12.A process as claimed in claim 1, wherein the silica in process step (a) is obtained as a silica cake having 30% moisture content.
- 13.A process as claimed in claim 1, wherein the first slurry is filtered within 15 minutes from its formation in process step (b).
- 14.A process as claimed in claim 13, wherein the first slurry is filtered using continuous filter centrifuge in process step (b).
- 15.A process as claimed in claim 1, wherein the crystallizer is maintained at a temperature in the range of about 70 -90°C in process step (c).
- 16.A process as claimed in claim 1, wherein the filtrate containing aluminium fluoride is fed to the crystallizer in batches in process step (c).
- 17.A process as claimed in claim 1, wherein the aluminium fluoride trihydrate in process step (c) is in the form of wet powder.
- 18.A process as claimed in claim 1, wherein the aluminium fluoride trihydrate is added in the amount of 15 % by mass of the total weight of the aluminium fluoride into the crystallizer in the process step (c).

- 19.A process as claimed in claim 1, wherein several similar batches are introduced to the crystallizer at regular time interval such that the soluble aluminium fluoride concentration is not less than 9% by mass in the process step (c).
- 20.A process as claimed in claim 1, wherein crystallization is carried out for about 4 hours.
- 21.A process as claimed in claim 1, wherein the aluminium fluoride wet cake crystals is separated out by continuous filtration centrifuge in the process step (d).
- 22.A process as claimed in claim 18, wherein the aluminium fluoride cake has moisture content less than about 20 % by mass.
- 23.A process as claimed in claim 1, wherein the process step of calcining comprises (i) heating the wet cake of aluminium fluoride at a temperature of about 160⁰C to remove the surface moisture and (ii) heating the resultant aluminium fluoride at a temperature of about 500-600⁰C in an oven to obtain aluminium fluoride powder having moisture content less than 1%.
- 24.A process as claimed in claim 1, wherein the particle size of the aluminium fluoride anhydride is in the range of 80 microns to 90 microns.

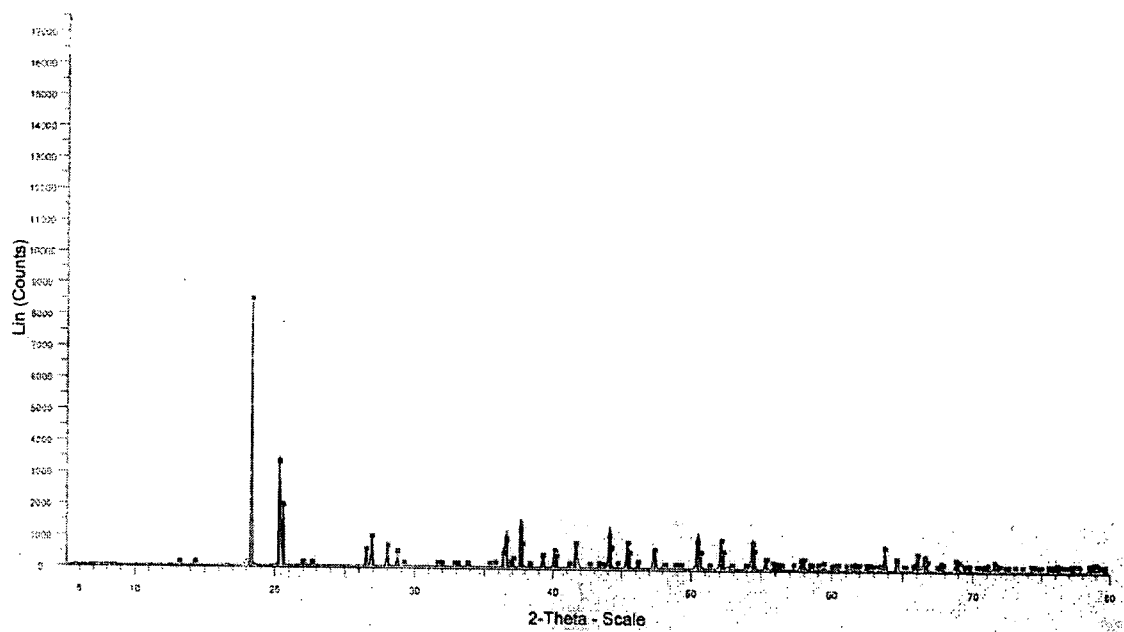


Figure 1

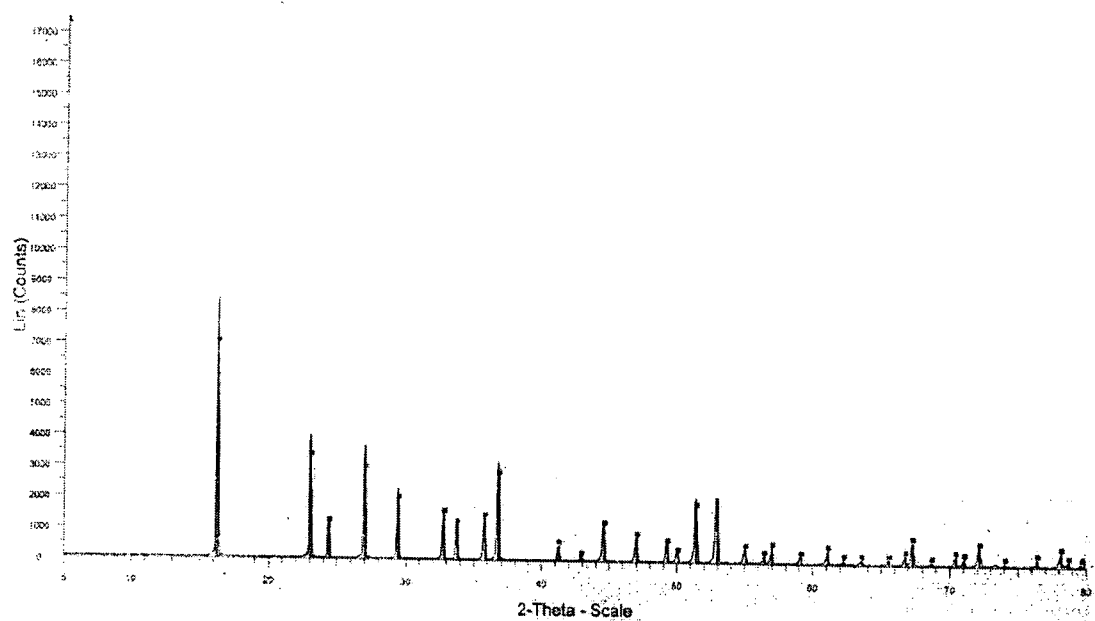


Figure 2

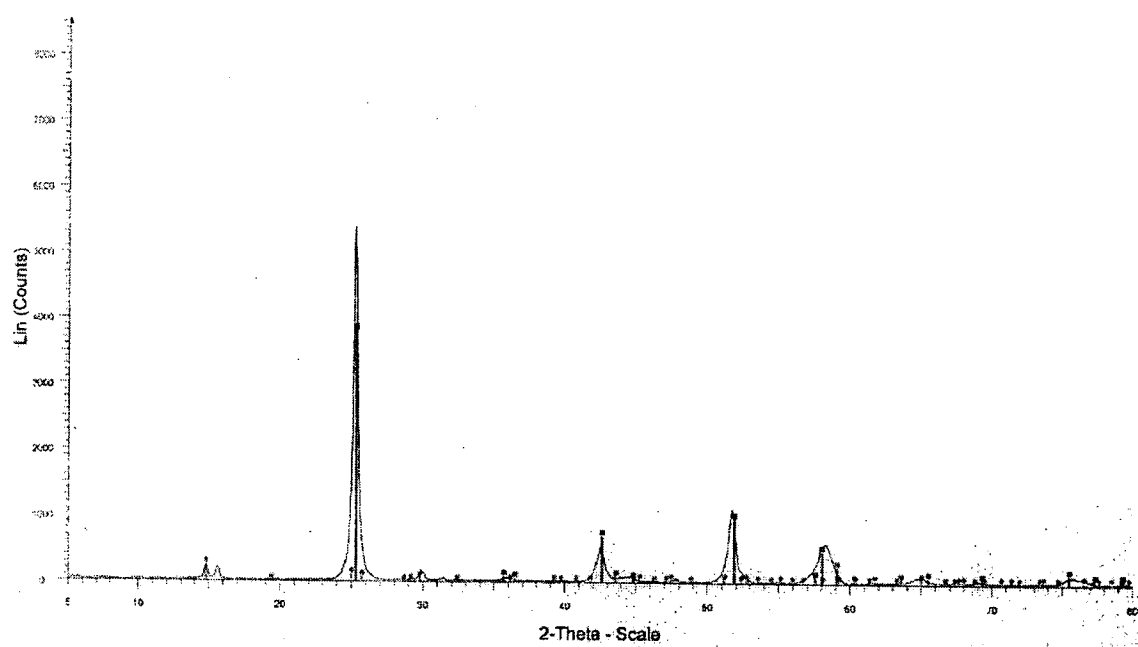


Figure 3

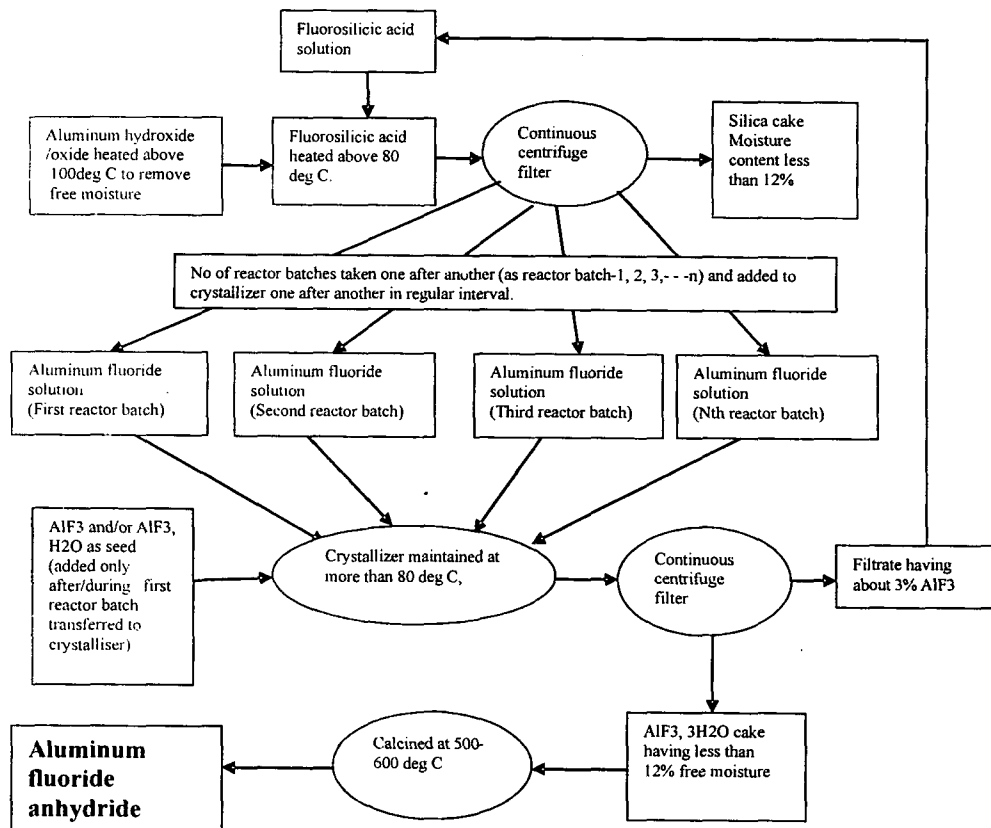


Figure 7

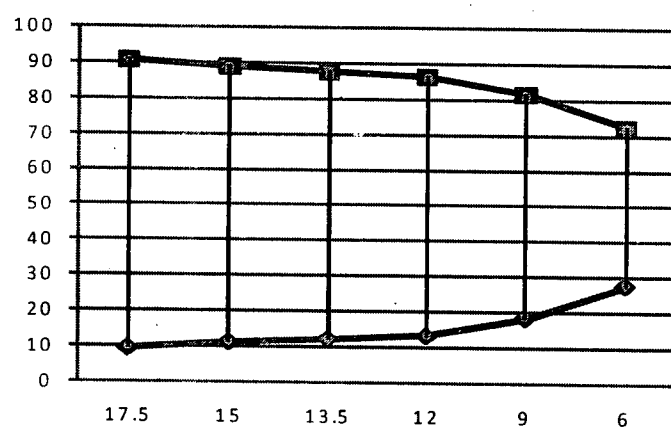


Figure 5

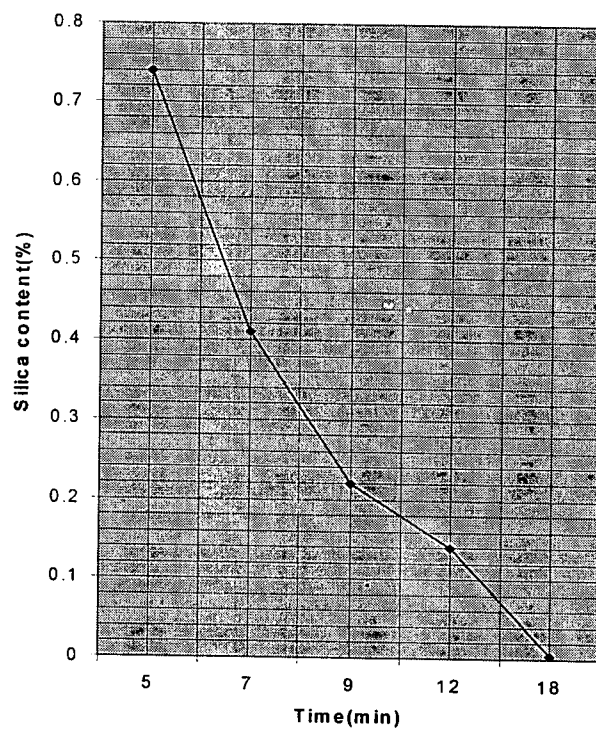


Figure 6

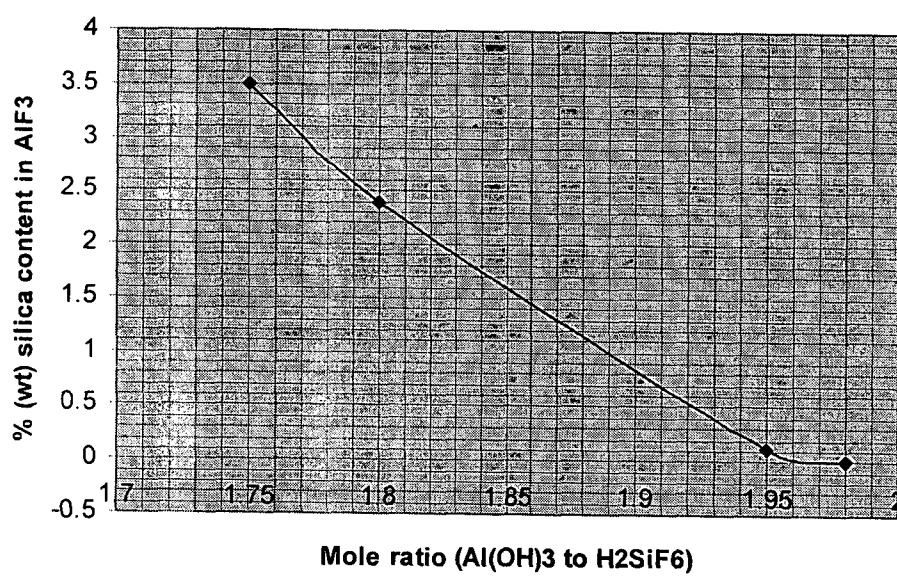


Figure 7