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(54) METHOD OF PREPARING GRANULATED ACTIVATED ALUMINA

(71) We, INSTITUT KATALIZA SIBIRSKOGO OTDELENIA AKADEMII NAUK SSSR, Prospekt Nauki, 5 Novosibirsk, Union of Soviet Socialist Republics, a Corporation organised and existing under the laws of the Union of Soviet Socialist Republics, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to a method of preparing granulated activated alumina for use as an absorbing material, a catalyst, or a support for active components in the chemical and petrochemical industries.

The present invention provides a method of preparing granulated activated alumina, comprising decomposition of alumina trihydrate by heating, mixing of the obtained product of decomposition with water, its hydration, granulation, drying at a temperature of 20—150°C, and calcining at a temperature of 400—600°C, in which, according to the invention alumina trihydrate is decomposed by bringing it in contact with a bed of a solid heat carrier having a temperature of 350—600°C, for a period of time from 0.05 to 0.5 second, to obtain an amorphous aluminium hydroxide, which is mixed with water into a suspension having a concentration of 100—500 g/litre (as Al_2O_3), with subsequent hydration of this product of decomposition at a temperature of 20—100°C and a pH of 5—12 for 0.5—10 hours; before granulation of the hydrated product of decomposition, it is treated with an acid, that can form water-soluble basic salts of aluminium, at a temperature of 20—150°C.

Alumina trihydrate should preferably be decomposed in a fluidized bed of a solid heat carrier by bringing it in contact with the heated carrier bed for a period of time from 0.05 to 0.5 second.

In order to obtain a hydrated decomposition product containing not less than 50 per cent weight of pseudo-Boehmite, it is recommended to hydrate the decomposition product at a temperature of 20—80°C and a pH of 5—10 for 0.5—8 hours; for preparing a hydrated decomposition product containing not less than 80 per cent by weight of Bayerite aluminium hydroxide, the hydration process should be contacted at a temperature of 90—100°C and pH of 10.5 to 11.0 for 8—10 hours; for preparing a hydrated decomposition product containing not less than 80 per cent by weight of Nordstrandite aluminium hydroxide, the hydration process should be carried out at a temperature of 80—90° and pH of 11 to 12 for 8 to 10 hours in a medium of a 5 per cent solution of ethylenediamine for 8—10 hours respectively.

In order to obtain granulated activated alumina free from sodium admixtures, before the acid treatment, the hydrated product of decomposition is filtered, and washed with water to a residual sodium

content not exceeding 0.02 per cent by weight (as Na_2O).

The method according to the invention of preparing granulated activated alumina includes the step of decomposition of alumina trihydrate by bringing it in contact with a solid bed of a heat carrier, having a temperature of 350—600°C for a period of time from 0.05 to 0.5 second. Metallic, or ceramic particles of any configuration can be used as the solid heat carrier material. The result of the decomposition of alumina trihydrate is an X-ray amorphous product, having a loss on ignition of 10—16 per cent by weight, which is amorphous aluminium hydroxide close to alumina monohydrate with respect to its composition. The expression 'loss on ignition' is the standard loss in weight of a product heated at a temperature of 800°C for two hours. The sedimentation analysis of the amorphous aluminium hydroxide, which is the product of decomposition of alumina trihydrate, and of the starting alumina trihydrate, has shown that the distribution of their particles with respect to the size is the same. This means, that the particles of the starting alumina trihydrate are not destroyed in the process of their decomposition, but they are only cracked to release two molecules of water. The specific surface area of the decomposition product increases and sometimes attains a value of 400 sq.m/g.

The obtained product of decomposition of alumina trihydrate is mixed with water to obtain a suspension having a concentration of 100—500 g/litre (as Al_2O_3) and hydrated. Depending on the conditions of the hydration (temperature and pH of the medium), amorphous aluminium hydroxide, which is a product of decomposition of alumina trihydrate, can be converted into various modifications of aluminium hydroxide, viz. into pseudo-Boehmite, Bayerite, Nordstrandite or mixtures thereof. To prepare pseudo-Boehmite, the decomposition product is hydrated in an aqueous medium at a pH 5—10 and a temperature of 20—80°C. It should be noted that as amorphous aluminium hydroxide, which is the product of decomposition of alumina trihydrate, is mixed with water and hydrated, the pH of the medium increases to 11 on account of sodium ions that are liberated into water from the particles of the decomposed product. To adjust the pH below 11, from 1 to 8 g of an acid per gram of alumina are added to the mixture of the decomposition product in water.

In the hydration of the product of decomposition of alumina trihydrate to Bayerite or Nordstrandite, the acid correction of the pH is not required. In these cases amorphous aluminium hydroxide is hydrated at a temperature of 90—100°C and the pH 10.5—11, for preparing Bayerite, and at a temperature of 80—90°C and the pH 11—12 (in 5 per cent ethylenediamine) for preparing Nordstrandite.

To obtain activated alumina free from sodium the hydrated product of decomposition of alumina trihydrate, before the acid treatment, should be filtered and washed with water. After filtration, before the wash, the residual content of sodium in the precipitate is 0.05—0.17 per cent by weight (as Na_2O). To obtain higher purity, the hydrated product of alumina trihydrate decomposition is washed with distilled water, in an amount of 7—9 litres per kg of alumina in the precipitate.

Further, the hydrated products of decomposition of alumina trihydrate are processed with acid that can form water-soluble basic salts of aluminium, e.g. with nitric acid, to prepare plastic aluminium hydroxide that can be granulated by known methods.

The granules are dried at a temperature of 20—150°C and calcined at a temperature of 400—600°C preferably in a stream of dry air or inert gas, their consumption preferably being at least 1000 $hour^{-1}$.

The resultant granulated activated alumina has a specific surface area of 200—400 sq.m/g, a total pore capacity of 0.45—0.90 cc/g, and various pore structures.

The advantages of the method according to the invention compared with the known method of preparing granulated activated alumina, are as follows.

Carrying out the process of decomposition of alumina trihydrate by bringing it in contact with a fluidized bed of a solid heat carrier intensifies the heat and mass exchange in the decomposition zone, which in turn makes it possible to decrease the temperature of decomposition with an appreciable reduction of the time during which the particles of alumina trihydrate are present in the high temperature zone.

It is not obligatory to carry out the process of decomposition of alumina trihydrate to the formation of transition alumina of the type χ-Al_2O_3 and ρ-Al_2O_3, having a loss on ignition of 5—10 per cent by weight. The method of the invention can be used to obtain an amorphous (to X-rays) product having a loss on ignition of 10—16 per cent by weight, i.e. an amorphous aluminium hydroxide close, with respect to its composition, to alumina monohydrate. The properties of this product of decomposition are practically the same as those of alumina of the type χ-Al_2O_3 and ρ-Al_2O_3. Decomposing alumina trihydrate by the method of the invention makes it possible to preserve one molecule of water

in the structure of the decomposition product, and this considerably decreases the consumption of heat in the decomposition process, since the heat required to remove the last molecule of water (35.5 kcal/mole) is practically the same as the total heat required to remove the first two molecules of water (36.3 kcal/mole).

Moreover, changing the hydration conditions (temperature, pH of the medium, and duration) of the product of decomposition of alumina trihydrate in water in the form of a suspension having the concentration of 100—500 g/litre (as Al_2O_3- or changing the acid treatment conditions of the hydrated product of decomposition makes it possible to modify within a wide range the pore structure of the end product, granulated activated alumina.

The acid treatment of the hydrated products of decomposition makes it possible to obtain plastic hydroxides of aluminium that can be granulated by the known methods.

The invention will be further described with reference to the following illustrative Examples.

Example 1

A mixture consisting of alumina trihydrate, having particles not greater than 200 μ, and air, is passed through a reactor filled with fluidized bed of a solid heat carrier in the form of 4-mm long cylindrical granules of aluminium having a diameter of 4 mm. The fluidized bed of the solid heat carrier has a temperature of 500°C. Its height in the stationary state is 60 mm. The mean time of contact of alumina trihydrate particles with the hot solid heat carrier is 0.1 sec. During this time alumina trihydrate is decomposed to amorphous aluminium hydroxide, having a specific surface area of 250 sq.m/g; the loss on ignition is 14 per cent by weight. X-ray analysis shows that the obtained decomposition product is amorphous.

Now 1 kg of the obtained decomposition product is mixed with 3 litres of distilled water to prepare a suspension having a concentration of 215 g/litre (as Al_2O_3). In order to adjust the pH of the medium to 8, 4.3 g of nitric acid are added to the suspension. The product of decomposition is hydrated at a temperature of 70°C for 6 hours with constant stirring. The resultant product is the hydrated decomposition product having a residual moisture content of 60 per cent. The specific surface area of the hydrated product dried at a temperature of 110°C is 240 sq.m/g. X-ray analysis of the product shows that its composition is as follows: 70 per cent by weight of pseudo-Boehmite, 30 per cent by weight of Bayerite.

After hydration, the hydrated product of decomposition is filtered and washed with water to remove sodium admixtures. The water requirements are 7 litres per kg of Al_2O_3 in the precipitate. The residual sodium content in the precipitate is 0.02 per cent by weight (as Na_2O).

2.1 kg of the hydrated product are mixed with 2.2 litres of distilled water to prepare a suspension having a concentration of 250 g/litre of (Al_2O_3) and treated with nitric acid taken in a quantity of 0.15 mole per mole of Al_2O_3. The acid treatment is carried out at a temperature of 130°C for four hours with constant stirring to prepare a plastic mass of aluminium hydroxide that does not delaminate.

The obtained mass is granulated into spheres, that are dried at a temperature of 105°C and calcined in a furnace at a temperature of 550°C for four hours, with an air consumption of 1000 $hour^{-1}$.

The end product is granulated activated alumina in the form of spherical granules having a diameter of 2.5±0.5 mm, and characterized by the following properties: specific surface area, 350 sq.m/g; total pore capacity, 0.71 cc/g; prevalent radius of pores, 60Å; compressive strength, 95 kg/sq.cm.

Example 2

A mixture consisting of alumina trihydrate, having particles not greater than 200μ, and air, is passed through a reactor filled with a fluidized bed of a solid carrier in the form of ceramic balls, 2—3 mm in diameter. The temperature of the fluidized bed is 350°C, and its height (stationary) is 100 mm. The mean time of contact of the alumina trihydrate particles with the hot solid heat carrier is 0.2 second, during which alumina trihydrate is decomposed to an amorphous aluminium hydroxide having a specific surface area of 200 sq.m/g; the loss on ignition is 16 per cent by weight. X-ray analysis shows that the product of decomposition is amorphous, with traces of residual alumina trihydrate.

Now 1 kg of the obtained product is mixed with 1.4 litre of distilled water to prepare a suspension having a concenttration of 350 g/litre (as Al_2O_3). To adjust the pH of the medium to 9.5, 2.5 g of nitric acid are added to the suspension. The product is hydrated at a temperature of 80°C for 5 hours with constant stirring. The obtained hydrated product has a residual moisture content of 48 per cent by weight. The specific surface area of the hydrated product, dried at a temperature of 110°C, is 220 sq.m/g. X-ray analysis shows the following composition: 60 per cent by

weight of pseudo-Boehmite and 40 per cent by weight of Bayerite.

A suspension of the hydrated product having a concentration of 350 g/litre (Al_2O_3) is treated with hydrochloric acid taken in a quantity of 0.20 mole per mole of Al_2O_3. The acid treatment is effected at a temperature of 150°C for six hours with constant stirring. The resultant plastic mass is shaped into cylindrical granules, dried at a temperature of 20°C for 24 hours, and then at a temperature of 150°C for an hour, and finally calcined in a furnace at a temperature of 400°C for six hours in a stream of air.

The end product is granulated activated alumina, in the form of 4—6 mm long cylindrical granules, 4 mm in diameter, having the following characteristics: specific surface area, 300 sq.m/g; total volume of pores, 0.45 cc/g; prevalent radius of pores, 50 Å; compressive strength, 140 kg/sq.cm (pressure applied to the end).

Example 3

A mixture consisting of alumina trihydrate, having particles not greater than 200μ, and air, is passed through a reactor filled with a fluidized bed of a solid heat carrier in the form of copper cylinders, 2 mm long and 2 mm in diameter. The fluidized bed temperature is 600°C and its height (in the stationary state) is 40 mm. The mean contact time is 0.05 second, during which alumina trihydrate is decomposed to an amorphous (to X-rays) product having a specific surface area of 380 sq.m/g; loss on ignition is 15 per cent by weight.

Now 1 kg of the product of decomposition of alumina trihydrate is mixed with 7.5 litres of distilled water to prepare a suspension having a concentration of 100 g/litre (as Al_2O_3). To adjust the pH of the medium to 10, 0.85 g of nitric acid is added to the suspension. The decomposition product is hydrated at a temperature of 50°C for 8 hours with constant stirring, to obtain a hydrated product of decomposition having a residual moisture content of 55 per cent by weight. The specific area of the product, dried at a temperature of 110°C, is 300 sq.m/g. X-ray analysis shows the following composition of the product: 60 per cent by weight of pseudo-Boehmite and 40 per cent by weight of Bayerite.

The suspension of the hydrated product of decomposition, having a concentration of 100 g/litre (Al_2O_3) is treated with nitric acid taken in the quantity of 0.1 mole per mole of Al_2O_3. The acid treatment is effected at a temperature of 110°C for 6 hours with constant stirring. The obtained mass of aluminium hydroxide is shaped into granules ('fine sphere'), dried at a temperature of 100°C and calcined at a temperature of 400°C for 4 hours in a stream of air.

The obtained granulated activated alumina is spherical granules, from 0.1 to 2 mm in diameter, characterized by the following properties: specific surface area, 380 sq.m/g; total pore capacity, 0.65 cc/g; prevalent radius of pore, 150 Å and 1000 Å.

Example 4

A mixture consisting of alumina trihydrate, having particles not greater than 200μ, and air, is passed through a reactor filled with a fluidized bed of a heat carrier, which is bed of regularly placed rings having an inner diameter of 1 mm and an outer diameter of 3 mm. The bed of the solid heat carrier has a temperature of 450°C and the height of 300 mm. The time during which the particles of alumina trihydrate are in contact with the hot bed of the heat carrier is 0.5 sec, during which alumina trihydrate is decomposed to an amorphous (to X-ray) product having a specific surface area of 400 sq.m/g; the loss of ignition is 10 per cent by weight.

Now 1 kg of the product of decomposition is mixed with 0.8 litre of distilled water to obtain a suspension having a concentration of 500 g/litre (as Al_2O_3). To adjust the pH of the medium to 8, 4.5 g of nitric acid are added to the suspension. The hydration is carried out at a temperature of 60°C for 8 hours with constant stirring. The residual moisture content of the thus obtained product is 43 per cent by weight. The specific surface area of the hydrated product, dried at a temperature of 110°C, is 230 sq.m/g. The composition of the product, as revealed by X-ray analysis, is: 80 per cent by weight of pseudo-Boehmite, 20 per cent by weight of Bayerite.

After the hydration, the suspension is filtered, and the precipitate washed to remove sodium admixtures. The consumption of water is 7 litres per kg of Al_2O_3 in the precitate. The residual sodium content is 0.015 per cent by weight (as N_2O).

1.6 kg of the washed precipitate are mixed with 2 litres of distilled water to prepare a suspension, having a concentration of 350 g/litre (as Al_2O_3), and treated with acid as in Example 2. The obtained mass of aluminium hydroxide is shaped into ring-shaped granules, dried at a temperature of 100°C and calcined at a temperature of 550°C for 4 hours, in a stream of air, delivered at a rate of 1000 $hour^{-1}$.

The obtained granulated activated alumina is ring-shaped granules, having an inner diameter of 2 mm and an outer diameter of 4 mm, and 4—8 mm long. The properties of the product are as follows:

specific surface area, 270 sq.m/g; total capacity of pores, 0.5 cc/g; prevalent radius of pores, 100 Å; compressive strength, 40 kg/sq.cm.

Example 5

A plastic mass of aluminium hydroxide prepared by a procedure described in Example 3, after acid treatment, is shaped into microspherical granules, dried at a temperature of 100°C, and calcined at a temperature of 400°C for 4 hours in a current of air.

The obtained granulated activated alumina is spherical granules, 20—100μ in diameter, characterized by the following properties: specific surface area, 330 sq.m/g; total capacity of pores, 0.6 cc/g; prevalent pore radius, 100 Å and 700—1000 Å.

Example 6

A suspension of hydrated product of decomposition, obtained as described in Example 2, is treated with nitric acid taken in a quantity of 0.05 mole per mole of Al_2O_3. The acid treatment is effected at a temperature of 70°C for three hours with constant stirring. The obtained mass of aluminium hydroxide is shaped into 3—5 mm long cylindrical granules, 1 mm in diameter, dried at a temperature of 100°C, and calcined at a temperature of 500°C for 5 hours in a stream of air, delivered at a rate of 1000 $hour^{-1}$.

The obtained granulated activated alumina has the following characteristics: specific surface area, 300 sq.m/g; total pore capacity, 0.55 cc/g; prevalent pore radius, 50 A, 1000 A, and 10,000A; compressive strength (pressure applied to the granule end), 90 kg/sq.cm.

Example 7

2.1 kg of washed hydrated product of decomposition, obtained as described in Example 1, are mixed with 0.3 litre of distilled water and treated with nitric acid taken in a quantity of 0.08 mole per mole of Al_2O_3. The acid treatment is effected at as temperature of 20°C for 3.5 hours with constant stirring. The obtained plastic mass is shaped into 6—10 mm long cylinders having a diameter of 5 mm, dried at a temperature of 100°C, and calcined at a temperature of 550°C for 5 hours in a stream of air, delivered at a rate of 1000 $hour^{-1}$.

The obtained granulated activated alumina has the following characteristics: specific surface area, 250 sq.m/g; the total pore capacity, 0.75 cc/g; prevalent pore radius, 100 Å and 10,000 Å; compressive strength (pressure applied to the cylinder end) 80 kg/sq.cm.

Example 8

2.1 kg of washed hydrated product of decomposition, obtained as described in Example 1, are mixed with 0.9 litre of distilled water and treated with nitric acid taken in a quantity of 0.02 mole per mole of Al_2O_3. The acid treatment is effected at a temperature of 90°C for an hour with constant stirring. The obtained plastic mass is shaped into 4—8 mm-long cylinders, having a diameter of 3 mm. The cylinders are dried at a temperature of 100°C and calcined at a temperature of 550°C for 5 hours in a stream of air delivered at a rate of 1000 $hour^{-1}$.

The obtained activated granulated alumina has the following characteristics: specific surface area, 340 sq.m/g; total pore capacity, 0.5 cc/g; prevalent pore radius, 50 Å and 500 Å; compressive strength (pressure applied to the end of the cylinder), 180 kg/sq.cm.

Example 9

1 kg of the product of decomposition of alumina trihydrate, obtained as described in Example 1, is mixed with 7.6 litres of distilled water, and hydrated at a temperature of 90°C for 10 hours with constant stirring. The pH of the medium is 11 which is due to liberation of sodium ions from the products of decomposition. The residual moisture content of thus obtained product is 45 per cent by weight. The specific surface area of the hydrated product dried at a temperature of 110°C is 60 sq.m/g. X-ray analysis proves the following composition: 90 per cent by weight of Bayerite, and 10 per cent by weight of pseudo-Boehmite.

The hydrated product is filtered and washed to remove sodium admixtures. the consumption of washing water is 9 litres per kg of Al_2O_3 in the precipitate. The residual sodium content of the precipitate is 0.02 per cent by weight (as Na_2O).

1.6 kg of the washed hydrated product of decomposition are mixed with 1.4 litre of distilled water and treated with nitric acid taken in a quantity of 0.05 mole per mole of Al_2O_3. The acid treatment is carried out at a temperature of 90°C for 4 hours with constant stirring. The obtained plastic mass of aluminium hydroxide is shaped into 4—5 mm-long cylinders having a diameter of 5μ, dried at a temperature of 100°C, and calcined at a temperature of 450°C for 5 hours in a stream of air delivered at a rate of 1000 $hour^{-1}$.

The obtained granulated activated alumina has the following characteristics; specific surface area, 380 sq.m/g; total pore capacity, 0.90 cc/g; prevalent pore radius, 40 and 300 Å; compressive strength

(pressure applied to the cylinder end), 50 kg/sq.cm.

Example 10

1 kg of a product of decomposition of alumina trihydrate, obtained as described in Example 3, is mixed with 7.5 litres of distilled water and hydrated at a temperature of 90°C for 8 hours with constant stirring. The pH of the medium, at which the decomposition product is hydrated, is 11 due to sodium ions liberated into water from particles of the decomposed product. The residual moisture content of thus obtained product is 50 per cent by weight. The specific surfaca area of the hydrated product, dried at a temperature of 110°C, is 80 sq.m/g. X-ray analysis proves the following composition: 80 per cent by weight of Bayerite, and 20 per cent of by weight of pseudo-Boehmite.

The further treatment of the hydrated product of decomposition (filtration, washing, acid treatment, granulation, drying, and calcining) is effected as described in Example 9.

The end product is granulated activated alumina, which is 4—5 mm-long cylinders having a diameter of 3 mm, and characterized by the following properties: surface area, 350 sq.m/g total pore capacity, 0.85 cc/g; prevalent pore radius, 40 and 300 Å; compressive strength (pressure applied to the cylinder end), 60 kg/sq.cm.

Example 11

1 kg of the product of decomposition of alumina trihydrate, obtained as described in Example 4, is mixed with 8 litres of distilled water and hydrated at a temperature of 100°C and a pH of 10.5 for ten hours with constant stirring. The pH of 10.5 accounts for the liberation in water of sodium ions from the products of decomposition. The residual moisture content of the hydrated product is 40 per cent by weight. The specific surface area of the hydrated product, dried at a temperature of 110°C, is 90 sq.m/g. X-ray analysis proves the following composition: 70 per cent by weight of Bayerite, 20 per cent by weight of Nordstrandite, and 10 per cent by weight of pseudo-Boehmite.

The hydrated product of decomposition is washed to remove sodium as described in Example 9.

1.5 kg of the washed hydrated product of decomposition of alumina trihydrate is mixed with 1.2 litres of distilled water and treated with nitric acid taken in a quantity of 0.1 mole per mole of Al_2O_3. The acid treatment is effected at a temperature of 110°C for 6 hours with constant stirring. The further processing of the acid-treated plastic mass is effected as described in Example 9.

The resultant granulated activated alumina has the following characteristics: specific surface area, 390 sq.m/g total pore capacity, 0.8 cc/g; prevalent pore radius, 50 and 10,000 Å; compressive strength (pressure applied to the end), 50 kg/sq.cm.

Example 12

1 kg of the product of decomposition of alumina trihydrate, obtained by a procedure described in Example 3, is mixed with 7.5 litres of 5% aqueous solution of ethylenediamine. The pH of the medium is 12. The hydration is carried out at a temperature of 90°C for 8 hours with constant stirring. The residual moisture content of the obtained hydrated product is 45 per cent by weight. The specific surface area of the hydrated product, dried at a temperature of 110°C, is 60 sq.m/g. X-ray analysis proves the following composition. 90 per cent by weight of Nordstrandite, and 10 per cent by weight of pseudo-Boehmite.

The further treatment of the obtained hydrated product of decomposition of alumina trihydrate is effected as described in Example 9. The obtained granulated activated alumina has the following characteristics: specific surface area, 350 sq.m/g; total pore capacity, 0.75 cc/g; prevalent pore radius 50 and 5,000 Å; compressive strength (pressure applied to the end), 60 kg/sq.cm.

Example 13

Granulated activated alumina is prepared by a procedure similar to that described in Example 12 except that the hydration process is carried out at a temperature of 80°C for 10 hours with constant stirring. The residual moisture content of the hydrated product is 50 per cent by weight; its specific surface area is 80 sq.m/g; the composition is 80 per cent by weight of Nordstrandite and 20 per cent by weight of pseudo-Boehmite.

Example 14

A mixture consisting of alumina trihydrate, having particles not larger than 200μ, and air, is passed through a reactor filled with a fluidized bed of a solid heat carrier, which comprises ring-shaped granules having an inner diameter of 2 mm and an outer diameter of 4 mm. The bed of the solid heat carrier has a temperature of 550°C and a height of 200 mm. The time during which the alumina trihydrate particles are present in the bed of the hot heat carrier is 0.35 sec; during this time alumina trihydrate is decomposed to an amorphous (to X-ray analysis) product. Its specific surface area is 300 sq.m/g; the loss on ignition is 12 per cent by weight.

1 kg of the product of decomposition of alumina trihydrate is mixed with 7.8 litres of distilled water to prepare a suspension having a concentration of 100 g/litre (as Al_2O_3). To adjust the pH to 5,7 of nitric acid are added to the suspension. The hydration of the product of decomposition is effected at a temperature of 20°C for 8 hours with constant stirring. The residual moisture content of the hydrated product is 55 per cent by weight. The specific surface area of the hydrated product dried at a temperature of 110°C, is 210 sq.m/g X-ray analysis proves the following composition: 70 per cent by weight of pseudo-Boehmite, and 30 per cent by weight of Bayerite.

The further treatment of the hydrated product and the properties of the end product are the same as in Example 3.

Example 15

A mixture consisting of alumina trihydrate, having particles not exceeding 200μ in size, and air, is passed through a reactor filled with a fluidized bed of a solid heat carrier in the form of 3-mm long cylinders of aluminium having a diameter of 2 mm. The fluidized bed of the heat carrier has a temperature of 450°C; its (stationary) height is 50 mm. The mean time during which the alumina trihydrate particles are brought in contact with the hot heat carrier is 0.08 second. During this time alumina trihydrate is decomposed to an amorphous aluminium hydroxide having a specific surface area of 210 sq.m/g; its loss on ignition is 16 per cent by weight.

1 kg of the amorphous alumina hydroxide, which is the product of decomposition of alumina trihydrate, is mixed with 2 litres of distilled water and 5 g of nitric acid. The hydration is carried out at a temperature of 80°C and a pH of 7 for 6 hours with constant stirring. The residual moisture content of the hydrated product is 62 per cent by weight. X-ray analysis proves the following composition of the resultant product: 75 per cent by weight of pseudo-Boehmite, 25 per cent by weight of Bayerite.

The further treatment of the hydrated product of decomposition of alumina trihydrate is carried out as described in Example 1. The end product, actjvated alumina, is spherical granules having a diameter of 2.5±0.5 mm, characterized by the following properties: specific surface area 300 sq.m/g; total pore capacity, 0.65 cc/g; prevalent pore radius, 100 and 1000 Å; compressive strength, 70 kg/sq.cm.

Example 16

1 kg of the decomposition product obtained as described in Example 1 is mixed with 2.5 l of distilled water. The result is a suspension having a concentration of 240 g/l (for Al_2O_3). For establishing the pH of the medium equal to 5.8, 6.5 g of nitric acid are introduced into the suspension. Hydration of the decomposition product is carried out at a temperature of 20°C for 0.5 hr under continuous intensive stirring. The residual moisture content of the hydrated product is 60 per cent. The specific surface area of the hydrated decomposition product dried at 110°C is 320 sq.m/g. X-ray analysis proves the following composition of the resultant product: 70 per cent by weight of pseudo-Boehmite, and 30 per cent by weight of amorphous aluminium hydroxide (product of decomposition of alumina trihydrate).

After the hydration the hydrated product of decomposition is filtered and washed to remove sodium, the water consumption for washing being 75 l per kg of Al_2O_3 in the precipitate, the residual content of sodium in the precipitate ultimately being 0.02 per cent by weight (for Na_2O).

2.1 kg of the hydrated product of decomposition are mixed with 2 l of distilled water to prepare a suspension having a concentration of 260 g/l for Al_2O_3, and treated with nitric acid in an amount of 0.1 mole NHO_3 per mole Al_3O_3. The treatment is carried out at a temperature of 130°C for 4 hours under constant stirring. The resulting product is a plastic mass of aluminium hydroxide.

This mass is then shaped into spherical granules, which are subjected to drying at a temperature of 100°C and calcining at 500°C for 4 hours, the air consumption being 1000 hr^{-1}.

The end product, granulated activated alumina, comprising spherical granules having a diameter of 2.5±0.5 mm and the following characteristics: specific surface area, 320 sq.m/g; total pore capacity, 0.55 cc/g; prevalent pore radius, 50 Å, compressive strength, 100 kg/sq.cm.

Example 17

1 kg of the decomposition product prepared as described in Example 14 is mixed with 2.5 l of distilled water to prepare a suspension having a concentration of 250 g/l for Al_2O_3.

For adjusting the pH of the medium to be equal to 6, 6.3 g of nitric acid are introduced into the suspension. Hydration of the decomposition product is carried out at a temperature of 20°C for 3 hours under constant stirring. The residual moisture content of the hydrated product is 55 per cent. The specific surface area of the hydrated decomposition product dried at 110°C is 370 sq.m/g. X-ray analysis proves the following composition of the resultant product: 60 per cent by weight of pseudo-Boehmite, and 40 per cent by weight of

amorphous aluminium hydroxide (decomposition product).

After the hydration the hydrated decomposition product is filtered and washed to remove sodium, the water consumption for washing being 7.5 l per kg of Al_2O_3 in the precipitate, the residual content of sodium in the precipitate ultimately being 0.02 per cent by weight (for Na_2O).

1.96 kg of the hydrated decomposition product are mixed with 1.6 l of distilled water to prepare a suspension having a concentration of 280 g/l for Al_2O_3, and treated with nitric acid in an amount of 0.1 mole HNO_3 per mole Al_2O_3. The treatment is carried out at a temperature of 130°C for 5 hours under constant stirring. The resulting product is a plastic mass of aluminium hydroxide.

This mass is shaped into spherical granules, which are then dried at a temperature of 100°C and calcined at 550°C for 4 hours, the air consumption being 1000 hr^{-1}.

The end product, granulated activated alumina, comprises spherical granules having a diameter of 2.5±0.5 mm and the following characteristics: specific surface area, 360 sq.m/g; total pore capacity, 0.5 cc/g; prevalent pore radius, 40 Å; compressive strength, 120 kg/sq.cm.

WHAT WE CLAIM IS:—

1. A method of preparing granulated activated alumina which comprises decomposing alumina trihydrate by bringing it in contact with a bed of a solid heat carrier having a temperature of 350 to 600°C, the time of contact being from 0.05 to 0.5 second, to form a decomposition product of amorphous aluminium hydroxide; mixing the decomposition product with water to form a suspension having a concentration of 100 to 500 g/litre calculated with reference to aluminium oxide; hydrating the product by heating the suspension at a temperature of 20 to 100°C and pH of 5 to 12 for 0.5 to 10 hours; treating the hydrated decomposition product with an acid so as to form water-soluble basic salts of aluminium at a temperature of 20 to 150°C; followed by granulating; drying at a temperature of 20 to 150°C; and calcining at a temperature of 400 to 600°C.

2. A method as claimed in Claim 1, in which alumina trihydrate is decomposed in a fluidized bed of the solid heat carrier.

3. A method as claimed in Claim 1 or 2, in which the product of decomposition of alumina trihydrate is hydrated at a temperature of 20—80°C and a pH 5 to 10 for 0.5 to 8 hours, to obtain a hydrated decomposition product containing not less than 50 per cent by weight of pseudo-Boehmite.

4. A method as claimed in Claim 1 or 2, in which the product of decomposition of alumina trihydrate is hydrated at a temperature of 90—100°C and a pH of 10.5 to 11 for 8 to 10 hours, to obtain a hydrated decomposition product containing not less than 80 per cent by weight of Bayerite.

5. A method as claimed in Claim 1 or 2, in which the product of decomposition of alumina trihydrate is hydrated at a temperature of 80 to 90°C and a pH of 11 to 12 in a 5 per cent solution of ethylenediamine for 8 to 10 hours, to obtain a hydrated decomposition product containing not less than 80 per cent by weight of Nordstrandite.

6. A method as claimed in any preceding claim, in which after hydration and before the acid treatment, the hydrated product of decomposition of alumina trihydrate is washed with water until residual quantities of sodium therein do not exceed 0.02 per cent by weight, calculated with reference to Na_2O.

7. A method of preparing granulated activated alumina substantially as herein disclosed in any of the foregoing Examples.

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