

(12) STANDARD PATENT

(11) Application No. AU 2001237708 B2

(19) AUSTRALIAN PATENT OFFICE

(54) Title
A process for the manufacture of zeolite-A useful as a detergent builder

(51)⁷ International Patent Classification(s)
C01B 033/26 C11D 003/08

(21) Application No: **2001237708** (22) Date of Filing: **2000.12.19**

(87) WIPO No: **WO02/49962**

(43) Publication Date: **2002.07.01**
(43) Publication Journal Date: **2002.09.05**
(44) Accepted Journal Date: **2004.10.14**

(71) Applicant(s)
National Aluminium Company Limited; National Research Development Corporation

(72) Inventor(s)
Seth, M. M.; Gandhi, M. R.

(74) Agent / Attorney
Cullen & Co, 239 George Street, Brisbane, QLD, 4000

(56) Related Art
**JP 54-081200
DE 2722535**

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau



(43) International Publication Date
27 June 2002 (27.06.2002)

PCT

(10) International Publication Number
WO 02/49962 A1

(51) International Patent Classification⁷: C01B 33/26, C11D 3/08

(74) Agent: BANERJEE, Sudipta; L S Davar & Co., "Mon-alisa", Flats 1B & 1C, 17 Camac Street, Calcutta 700 017 (IN).

(21) International Application Number: PCT/IN00/00130

(22) International Filing Date: 19 December 2000 (19.12.2000)

(25) Filing Language: English

(26) Publication Language: English

(71) Applicants (for all designated States except US): NATIONAL ALUMINIUM COMPANY LIMITED [IN/IN]; * (IN). NATIONAL RESEARCH DEVELOPMENT CORPORATION [IN/IN]; 20-22 Zamroodpur, Community Centre, Kailash Colony Extension, New Delhi 110 048 (IN).

(72) Inventors; and

(75) Inventors/Applicants (for US only): GANDHI, M., R. [IN/IN]; * (IN). SETH, M., M. [IN/IN]; National Aluminium Co. Ltd. (IN).

(81) Designated States (national): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW.

(84) Designated States (regional): ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CII, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

Published:

— with international search report

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.



WO 02/49962 A1

(54) Title: A PROCESS FOR THE MANUFACTURE OF ZEOLITE-A USEFUL AS A DETERGENT BUILDER

(57) Abstract: A process for the manufacture of Zeolite-A useful as a detergent builder, which comprises: i) dilution of sodium aluminate liquor obtained as a pregnant liquor from Bayers process and having a concentration of at least 140 gpl of alumina therein and adjusting Al₂O₃-Na₂O molar ratio to 0.5±0.01, ii) thoroughly mixing alkali, water and sodium silicate to obtain a mixture with molar ratios SiO₂-Na₂O in the range of 1.25-1.66 and H₂O-Na₂O in the range of 35-45, iii) dosing hot sodium aluminate solution at a temperature in the range of 50-60 °C to the sodium silicate solution at ambient temperature with continuous mixing to obtain a synthesis mixture/crystallization bath having molar ratios: SiO₂-Al₂O₃ in the range of 1.6-2.0, Na₂O-SiO₂ in the range of 1.6-2.0, H₂O-Na₂O in the range of 30-40, iv) heating the crystallisation bath at a temperature in the range of ambient to 95 °C for a period in the range of 45 to 75 minutes under continuous stirring, v) recovering Zeolite-A by filtration and washing followed by drying and pulverising.

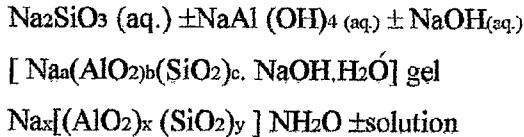
A process for the manufacture of Zeolite-A useful as a detergent builder.

FIELD OF INVENTION

5 This invention relates to a process for the manufacture of Zeolite-A useful as a detergent builder. In particular, this relates to a process for the manufacture of Zeolite-A powder useful as detergent builder and having a particle size distribution of 90% less than 4 μm .

BACKGROUND OF INVENTION

Synthesis of Zeolites depend on the starting materials such as reactive aluminosilicate gels prepared by mixing sodium silicate & sodium aluminate solutions; or amorphous solids such as metakaolin, siliceous earth, coal ash etc., pH, alkali concentration, hydrothermal conditions for crystallization with autogenous pressures and degree of supersaturation of the aluminosilicate gel leading to the nucleation of zeolite crystals. Using Na_2O - Al_2O_3 - SiO_2 - H_2O system, formation of gel and subsequently zeolite can be represented as under:



PRIOR ART

20 German patent Ger. Offen 2,517,218 describes a process to prepare Zeolite-A suitable as phosphate substitute in detergent formulations, by treating NaOH solution simultaneously with waterglass solution and sodium aluminate solution, gelled during 0.5 h, added to sodium aluminate solution at 80°C, and then crystallized during 3.5 h at 93°C. The zeolite obtained was having 93 wt % particles < 10 μm and 50 wt % < 6.5 μm .

In French patent no. 2,384,716, aqueous sodium aluminate solution is cooled to below ambient temperature and aqueous sodium silicate is mixed therewith to keep the resultant solution below ambient temperature. Such a mixture was heated to 60-100°C to form gel and maintained at this temperature for 12 min. to 3 h. As claimed, the process allowed 5 close control of particle size distribution of the product.

According to a German Patent, Ger. Offen 2,951,192 Zeolite-A having average grain size 0.9 μ m, calciumbinding capacity 300 mg CaCO₃/g and 134 mg MgCO₃/g was prepared by holding a solution containing H₂O 94.14, Na₂O 3.77, SiO₂ 1.77 and Al₂O₃ 0.52 mole at 60°C for 8h.

10 As described in Japanese patent JP NO. 89,05,905, zeolite having fine grain size is prepared from a mixture containing solidum aluminate and silicate, by hydrothermal reaction at the following conditions:

26-60 wt % zeolite concentration in reacting solution,
14-30 wt. % alkali (as Na₂O) concentration in mother liquor,
15 40°C at reactant solution mixing and 30-110°C for crystallization.

In a Japanese patent JP 04,342,416 a method is described to manufacture Zeolite-A, avoiding formation of hydroxysodalite and, wherein, an aqueous solution containing 0.1-0.3 mole per litre aluminate ions and 0.05-3 mole/l sodium ions is mixed with an aqueous solution containing 0.02-0.1 mole/silicate ions and 0.05-4 mole/l, Sodium ions, 20 at < 60°C not to form gel and the mixture is heated at 60-100°C to precipitate Zeolite-A.

A-type zeolite useful as detergent builder is effectively manufactured, without generation of other types of zeolites (HS), as described in Japanese patent JP 63,08,211. In such a patent, aqueous sodium silicate solution and aqueous sodium aluminate solution are mixed at < 60°C to give a slurry having alkali concentration (as Na₂O) > 12%, when the 5 zeolite crystallization rate becomes > 60% as determined by electron diffraction method (I) and < 80% by XRD method (II), the alkali concentration is lowered to 1-8 % and the resulting slurry is aged at 70-110°C to complete the crystallization process to give A-type zeolite having particle size 0.3 µm and Ca⁺ ion exchange capacity 160 mgCaO/g.

Another Japanese patent JP 63,265,808 describes a process for the manufacture of A-type 10 zeolite by (a) adding an aqueous solution of alkaline Al(OH)₃ [20-25 wt % Al(OH)₃] at 70-90°C and an aqueous solution of alkali silicate (20-29 wt % SiO₂) at < 40°C to water of < 40°C and by heating the mixture containing the resulting gel. Thus, an aqueous 15 solution containing 45.1 Kgs 49% NaOH, and 17.7 Kgs Al(OH)₃ of 80°C and 38.3 Kgs aqueous sodium silicate (SiO₂ 28-30 wt %) solution of 30°C were added to 59.9 Kgs water of 30°C, stirred 30 min. at 150 rpm, heated to 100°C for 2h, cooled to 40°C, filtered, washed and dried at 100-110°C for 3h to give Zeolite-A.

British Patent Application No. 2,005,653 describes a method to manufacture zeolite 20 useful for Ca⁺ exchange without amorphous or crystalline impurities by the hydrothermal crystallization of a completely dispersed aluminosilicate gel by combining solutions of silica, alumina and alkali. In an example of the aforesaid patent application, sodium aluminate solution containing 59.3% solids (Na₂O-Al₂O₃ molar ratio 1.2) is prepared by dissolving Al₂O₃ hydrate in hot NaOH, and sodium-silicate solution containing 47% solids (SiO₂:Na₂O::2.4:1) were heated to 72°C, diluted and mixed for 7 minutes in a homogenizer to produce a dispersed gel with no visible particles and viscosity 110 cps at

60°C. Zeolite-A seed material was added and the gel was heated for 80 minutes at 100°C. The crystalline Zeolite-A was separated, washed, dried for 5h at 105°C to give an uncontaminated material with Ca-exchange capacity 289 mg CaCO₃/g.

The drawbacks associated with the above mentioned known processes are:

5 i) Complex unit operations such as simultaneous addition of reacting solutions, maintaining the temperature of reactants and/or reaction mixture above or below ambient temperature, are involved in the preparation method.

ii) Gel formation at 60-110°C or below ambient temperature makes the preparation method energy consuming which adds to the production cost.

10 iii) Aging of gel, dispersing gel using homogenizer, prolonged crystallization time, hydrothermal conditions for crystallization etc. also contribute to make the process uneconomical.

iv) Precise pH control is required to obtain zeolite of uniform and small grain size.

20 v) Dilute solutions are used which is not favourable for large scale production of Zeolite-A

vi) Lowering the alkali concentration during the process and monitoring by sophisticated instruments (e.g. electron diffraction & XRD) is a tedious job. Even it requires much more time to obtain the product.

vii) Preparation of sodium-aluminate solution by dissolving Al_2O_3 hydrate in hot NaOH solution add to the production cost.

viii) High energy inputs are involved for the preparation a alumino silicate gel, since the raw material sodium aluminate powder has utilized more energy as compared to sodium aluminate liquor from which it was prepared. The gel of ambient temperature also requires more energy to achieve the crystallization temperature of more than 90°C.

French patent no. CA 1142501 describes a semi continuous method for production of Zeolite A. Though such a patent recognizes that the zeolite should have an average particle distribution size of 2 to 3 microns for use as a builder in detergents, the examples contained therein reflect that 98% of the average particle distribution size obtained by the process of the aforesaid patent is between 7 to 15 μm . Furthermore, the examples of the aforesaid patent reflect that a decomposed solution or Bayers liquor having low alumina content is employed as the starting material. Thus, such a patent employs a lean liquor after removal of alumina trihydroxide, namely a liquor which is unsaturated with respect to Al_2O_3 . A disadvantage in the use of a lean liquor is that it introduces complexity in the process. In order to obviate such a disadvantage, the patent teaches the additional step of adding more Al_2O_3 , which is then heated at a temperature of 100°C. Further, the aforesaid patent uses a liquor containing an excess of silica.

20 **OBJECTS OF THE INVENTION**

An object of the present invention is to propose an improved process for the manufacture of Zeolite-A useful as a detergent builder.

Another object of the present invention is to propose a process for the manufacture of zeolite-A having an average particle size distribution not exceeding 4 μm .

DESCRIPTION OF THE INVENTION

According to this invention there is provided a process for the manufacture of Zeolite-A useful as a detergent builder, which comprises:

- i) dilution of sodium aluminate liquor obtained as a pregnant liquor from Bayers process and having a concentration of at least 140 gpl of alumina therein and adjusting $\text{Al}_2\text{O}_3\text{-Na}_2\text{O}$ molar ratio to 0.5 ± 0.01 ,
- 5 ii) thoroughly mixing alkali, water and sodium silicate to obtain a mixture with molar ratios $\text{SiO}_2\text{-Na}_2\text{O}$ in the range of 1.25-1.66 and $\text{H}_2\text{O-Na}_2\text{O}$ in the range of 35-45.
- 10 iii) dosing hot sodium aluminate solution at a temperature in the range of 50-60°C to the sodium silicate solution at ambient temperature with continuous mixing to obtain a synthesis mixture/crystallization bath having molar ratios : $\text{SiO}_2\text{-Al}_2\text{O}_3$ in the range of 1.6-2.0, $\text{Na}_2\text{O-SiO}_2$ in the range of 1.6-2.0, $\text{H}_2\text{O-Na}_2\text{O}$ in the range of 30-40.
- 15 iv) heating the crystalisation bath at a temperature in the range of ambient to 95°C for a period in the range of 45 to 75 minutes under continuous stirring.
- v) recovering Zeolite-A by filtration and washing followed by drying & pulverising.

In the present invention, sodium aluminate liquor (Bauxite leachate) is used as received from the Bauxite leaching plant, to react with sodium silicate solution to form 20 aluminosilicate gel which is subjected to crystallization to obtain Zeolite-A (detergent

builder) powder with the composition $1.0 \pm 0.2 \text{ Na}_2\text{O} : \text{Al}_2\text{O}_3 : 1.8 \pm 0.2 \text{ SiO}_2 : \text{Y H}_2\text{O}$ (Y=upto 5) with 100 wt % particles below $20 \mu\text{m}$, 90 wt % particles in the range $1-5 \mu\text{m}$. Average particle size d_{50} (sedigraph) maximum $4 \mu\text{m}$ and calcium binding capacity of minimum 160 mg CaO/g absolute dry zeolite.

5 Specification of Sodium Aluminate liquor (Bauxite leachate) – an intermediate stream of alumina refinery used in the present invention is given in Table 1 below:

TABLE 1

Na ₂ O, (Total) gpl	165-175
Na ₂ O, (Caustic) gpl	146-152
10 Al ₂ O ₃ gpl	142-160
Carbonate [% of Na ₂ O (Total)]	12- 16
SiO ₂ , ppm	800-1000
Fe ₂ O ₃ (Total), ppm	8-12
Fe ₂ O ₃ (Soluble), ppm	3-6
15 Solid Content, ppm	20-40
Organic Carbon (% of Na ₂ O-Total)	2-2.5
Chloride (% of Na ₂ O-Total)	0.40-0.65

Specifically, the sodium aluminate liquor employed in the process of the present invention is a pregnant liquor obtained from the Bayers process and having a concentration of at least 140 gpl of alumina therein. Preferably, such a liquor has a concentration of 150 to 160 gpl of alumina.

Several distinct advantages are derived by the use of pregnant liquor in distinction to a

decomposed solution of Bayers liquor. One such advantage is a reduction in reaction time, eliminating aging of gel as well as heat energy during gel preparation and crystallization of Zeolite-A powder from superalkaline crystallization bath consisting of aluminosilicate gel. This method avoids the use of closed hydrothermal system and 5 homogenizer for dispersion of gel. The mother liquor/filtrate of the product contains mainly alkali, which can be recycled for Bauxite leaching or for the preparation of reactant solutions.

The sodium aluminate liquor may be diluted to adjust the $\text{Al}_2\text{O}_3\text{-Na}_2\text{O}$ molar ratio to 0.5 ± 0.01% by adding water and alkali hydroxide such as caustic soda, caustic lye. It has 10 been found that a deviation in such a ratio does have a detrimental effect on the properties of Zeolite A. The purpose of the present process is to provide crystalline zeolite without any grit. Thus, if such a ratio is 0.3, then Na_2O is present in excess and Al_2O_3 is less in the gel. In such an instance, free silica is present, which may trigger off the precipitation of silica aluminate. Thus, another critical factor is the $\text{Al}_2\text{O}_3\text{-SiO}_2$ ratio. If 15 the ratio of $\text{Al}_2\text{O}_3\text{-Na}_2\text{O}$ is higher than 0.5, an excess of alumina and less Na_2O and results in the formation of grits.

The sodium silicate solution may be prepared by mixing water, alkali such as caustic soda or caustic lye and sodium silicate.

The sodium silicate solution has a $\text{SiO}_2\text{-Na}_2\text{O}$ ratio 1.25 to 1.6. If the ratio is higher then 20 the silica content is higher.

The dosing time may be in the range of 60 to 120 minutes.

The Zeolite-A (detergent builder) may be crystallized by maintaining the temperature of crystallization bath from above 90°C to about 95°C.

The washing may be carried out till the pH of the filtrate is around 11.

The drying may be carried out at least for 6 h at 110°C in air dryer.

5 The process of the present invention is illustrated by the following examples which should not be construed to limit the scope of the present invention.

EXAMPLE 1

a) 300.7 L of sodium aluminate liquor ($\text{Na}_2\text{O}=168.18 \text{ gpl}$, $\text{Al}_2\text{O}_3=169.6 \text{ gpl}$), 14.74 Kgs of NaOH flakes and 199.3 L of water are mixed in a stirred tank, equipped with steam jacket/heating coil. The solution so prepared is heated to 50-60°C to simulate the conditions of temperature of sodium aluminate solution prepared using sodium aluminate liquor (Bauxite leachate) available in alumina refinery.

10 b) 155.25 Kgs sodium silicate (34.79 % SiO_2 , 14.24 % Na_2O , $\text{SiO}_2/\text{Na}_2\text{O}$ mole ratio 2.4), 19.48 Kgs of NaOH flakes and 355 of L water are mixed in a tank equipped with stirrer and stirred for 15 minutes.

15 c) Solution prepared in (a) is added to solution (b) within 1.5 h at ambient temperature with constant stirring to form aluminosilicate gel having molar ratios $\text{SiO}_2\text{-Al}_2\text{O}_3 1.8 \pm 0.2$, $\text{Na}_2\text{O}\text{-SiO}_2 1.6\text{-}2.0$, $\text{H}_2\text{O}\text{-Na}_2\text{O} 30\text{-}40$.

d) The aluminosilicate gel thus prepared is heated from ambient temperature to $93 \pm 2^\circ\text{C}$ in the same vessel as in ② and the same temperature is maintained for 1 h under continuous stirring.

5 e) The Zeolite-A powder produced in (d) is filtered using plate and frame type filter press.

f) 560 L filtrate obtained in (e) contains 8242 gpl Na_2O and less than 2 gpl SiO_2 & Al_2O_3 . This filtrate can be recycled in alumina refinery for Bauxite leaching process or can be partially used in preparation of reactant solutions.

10 g) The cake is washed with water till the pH of the wash water is around 11.

h) The product is dried in a cabinet type tray dryer at 110°C for about 6 h. The dried powder is pulverised and passed through 300 mesh BSS.

The product Zeolite-A (detergent builder) powder obtained was grit free having following specifications:

1.	Calcium binding capacity	171 mg CaO/g
15	2. Loss on ignition (800°C , 1h)	21.69%
3.	Particle size analysis: % finer than $4.5 \mu\text{m}$	90%
	Average particle size, d_{50} (sendigraph)	$2.9 \mu\text{m}$
20	4. Crystallinity, (XRD as compared to 4A BDH powder)	100%
	5. Whiteness index	97.3%

6. Chemical Composition:

	SiO ₂	33.10%
	Al ₂ O ₃	29.62%
	Na ₂ O	15.59%
5	H ₂ O	21.69%
7.	Mole composition	0.87 Na ₂ O: Al ₂ O ₃ : 1.90 SiO ₂ : 4.2 H ₂ O
8.	pH (5 % aq. Suspension)	11.0

EXAMPLE 2

a) 5.8 L of sodium aluminate liquor (Na₂O=182.0 gpl, Al₂O₃=176 gpl), 0.240 Kg NaOH flakes and 4.1 L water are mixed in a vessel to obtain 10 L of sodium aluminate solution having Na₂O- Al₂O₃ molar ratio 2.0. Heated to 60°C before adding to sodium silicate solution.

b) 3.075 Kgs sodium silicate (SiO₂ 35.12 % Na₂O=14.69 % & H₂O=50.19%) 0.367 Kgs NaOH flakes, and 7.1 L water are mixed in a vessel for 15 minutes.

15 c) Sodium aluminate solution as prepared in (a) is added to sodium silicate solution prepared as in (b) within 1.5 h at ambient temperature with constant stirring to form aluminosilicate gel having molar ratios SiO₂-Al₂O₃ 1.8 ± 0.2, Na₂O- SiO₂ 1.6-2.0, H₂O- Na₂O 30-40.

- d) The gel thus formed was heated upto $93\pm2^{\circ}\text{C}$ in the same vessel as prepared in © and the temperature maintained for 1 h under continuous stirring.
- e) The Zeolite-A powder product in (d) is obtained as described in example 1.
- f) 12.0 L filtrate obtained containing 85.63 gpl Na_2O and less than 2 gpl SiO_2 & 5 Al_2O_3 .

The product obtained according to example 2 was having following specifications.

1.	Calcium binding capacity	175 mg CaO/g
2.	Loss on ignition (800°C , 1h)	22.03%
3.	Particle size analysis	
15	% finer than $4.5\text{ }\mu\text{m}$	88%
	Average particle size, d_{50} (sendigraph)	$3.1\text{ }\mu\text{m}$
4.	Crystallinity, (XRD as compared to 4A BDH powder)	100%
5.	Whiteness index	Not Determined
20	6. Chemical Composition:	
	SiO_2	32.50%
	Al_2O_3	30.16%
	Na_2O	15.31%
	H_2O	22.03%
25	7. Mole composition	$0.84\text{ Na}_2\text{O} : \text{Al}_2\text{O}_3 :$ $1.83\text{ SiO}_2 : 4.14\text{ H}_2\text{O}$
	8. pH (5 % aq. Suspension)	11.0

EXAMPLE 3

a) 124.5 L sodium aluminate liquor ($\text{Na}_2\text{O}=187.4$ gpl & $\text{Al}_2\text{O}_3=174.15$ gpl), 3.9 Kgs NaOH flakes, 85.0 litres water are mixed to obtain 212.5 lits of sodium aluminate solution having 124 gpl Na_2O & 102 Al_2O_3 . The solution is heated to 5 60°C prior to mixing with sodium silicate solution.

b) 66.5 Kgs sodium silicate (34.53 % SiO_2 , 14.85 % Na_2O and 50.62 % H_2O), 7.664 Kgs NaOH flakes and 150.2 L water are mixed to obtain sodium silicate solution.

c) Solution prepared in (a) is added to solution prepared in (b) within 90 minutes at 10 ambient temperature so as to obtain a gel suitable to manufacture Zeolite-A (detergent builder).

d) The gel is heated to $93 \pm 2^\circ\text{C}$ and maintained at this retemperature for 1 h under continuous stirring.

e) The procedures followed thereafter are similar to those described in earlier 15 examples. Zeolite-A powder obtained according to this example was having following properties.

1.	Calcium binding capacity	173 mg CaO/g
2.	Loss on ignition (800°C , 1h)	21.70%
3.	Particle size analysis	
20	% finer than 4.5 μm	87%
	Average particle size, d_{50} (sendigraph)	3.2 μm
4.	Crystallinity, (XRD as compared to 4A BDH powder)	100%

5.	Whiteness index	96.8%
6.	Chemical Composition:	
	SiO ₂	33.39%
	Al ₂ O ₃	28.75%
5	Na ₂ O	16.16%
	H ₂ O	21.70%
7.	Mole composition	0.92 Na ₂ O: Al ₂ O ₃ : 1.97 SiO ₂ : 4.3 H ₂ O
8.	pH (5 % aq. Suspension)	11.1

Specifications of Zeolite-A (detergent builder) produced by the process of the present
10 invention are given in Table 2:

TABLE 2

1.	Calcium binding capacity	Min. 160 mg CaO/g
2.	Loss on ignition (800°C, 1h)	Min. 20%
3.	Particle size analysis:	
15	% finer than 4.5 μ m	90 \pm 10% ,
	Average particle size, d ₅₀ (sendigraph)	< 4 μ m
4.	Crystallinity, (as compared to 4A BDH powder by XRD)	Min. 95 %
5.	Whiteness index	Min. 95 %
20 6.	Chemical Composition:	
	SiO ₂	32-34%
	Al ₂ O ₃	28-30%
	Na ₂ O	14-16%
	H ₂ O	20-22%

7.	Mole composition	1.0 ± 0.2 Na ₂ O: Al ₂ O ₃ :
		1.8 ± 0.2 SiO ₂ : YH ₂ O
8.	pH (5 % aq. Suspension)	Y=Upto 5 10-12

Zeolite-A powder produced by the present invention is free of grit particles (i.e. particles > 45 μ m) having narrow particle size distribution (90% particles between 1-5 μ m),
5 average particle size d_{50} , sendigraph) < 4.0 μ m and has special properties required for the potential application as detergent builder to replace phosphates in detergent formulations.

The present invention has the following advantages.

1. An intermediate stream of alumina refinery as received from Bauxite leaching plant containing many impurities (see Table 2) has been used as source of alumina and alkali. This makes the final product cheaper as no extra processing is required for the preparation of sodium aluminate solution.
2. The unit operations involved in the process are so simple that it reduces the plant machinery cost and smooth production is possible.
- 15 3. The byproduct obtained in this process, i.e. filtrate, contains mainly alkali, which can be reutilised in Bauxite leaching or in the preparation of reactant solution. Thus making the process eco-friendly.
4. Even wash water which is slightly alkaline can be reused for the preparation of reactant solutions.
- 20 5. The process does not require any precise pH control or monitoring the crystallization by using sophisticated instruments. The only precise control required is that of temperature during crystallization.

WE CLAIM

1. A process for the manufacture of Zeolite-A useful as a detergent builder, which comprises:
 - i) dilution of sodium aluminate liquor obtained as a pregnant liquor from Bayers process and having a concentration of at least 140 gpl of alumina therein and adjusting $\text{Al}_2\text{O}_3\text{-Na}_2\text{O}$ molar ratio to 0.5 ± 0.01 ,
 - ii) thoroughly mixing alkali, water and sodium silicate to obtain a mixture with molar ratios $\text{SiO}_2\text{-Na}_2\text{O}$ in the range of 1.25-1.66 and $\text{H}_2\text{O-Na}_2\text{O}$ in the range of 35-45.
 - 10 iii) dosing hot sodium aluminate solution at a temperature in the range of 50-60°C to the sodium silicate solution at ambient temperature with continuous mixing to obtain a synthesis mixture/crystallization bath having molar ratios $\text{SiO}_2\text{-Al}_2\text{O}_3$ in the range of 1.6-2.0, $\text{Na}_2\text{O-SiO}_2$ in the range of 1.6-2.0, $\text{H}_2\text{O-Na}_2\text{O}$ in the range of 30-40.
 - 15 iv) heating the crystallisation bath at a temperature in the range of ambient to 95°C for a period in the range of 45 to 75 minutes under continuous stirring.
 - v) recovering Zeolite-A by filtration and washing followed by drying & pulverising.
2. A process as claimed in claim 1 wherein said sodium aluminate liquor used is an intermediate stream from Bauxite leaching plant of alumina refinery.

3. A process as claimed in claims 1 to 5 wherein the dosing time is in the range of 60 to 120 minutes.
4. A process as claimed in claims 1 to 6 wherein the Zeolite-A (detergent builder) is crystallized by maintaining the temperature of crystallization bath from above 90°C to about 95°C.
5. A process as claimed in claims 1 to 4 wherein the washing is carried out till the pH of the filtrate is around 11.
- 10 6. A process as claimed in claims 1 to 5 wherein the drying is carried out at least for 6 h at 110°C in air dryer.