ALUMINUM OXIDE (ALUMINA), HYDRATED

1. Introduction

The term alumina hydrates or hydrated aluminas is used in industry and commerce to designate aluminum hydroxides. These compounds are true hydroxides and do not contain water of hydration. Several forms are known; a general classification is shown in Figure 1. The most well-defined *crystalline forms* are the trihydroxides, $Al(OH)_3$: gibbsite [14762-49-3], bayerite [20257-20-9], and nordstrandite [13840-05-6]. In addition, two aluminum oxide–hydroxides, AlO(OH), boelimite [1318-23-6] and diaspore [14457-84-2], have been clearly defined. The

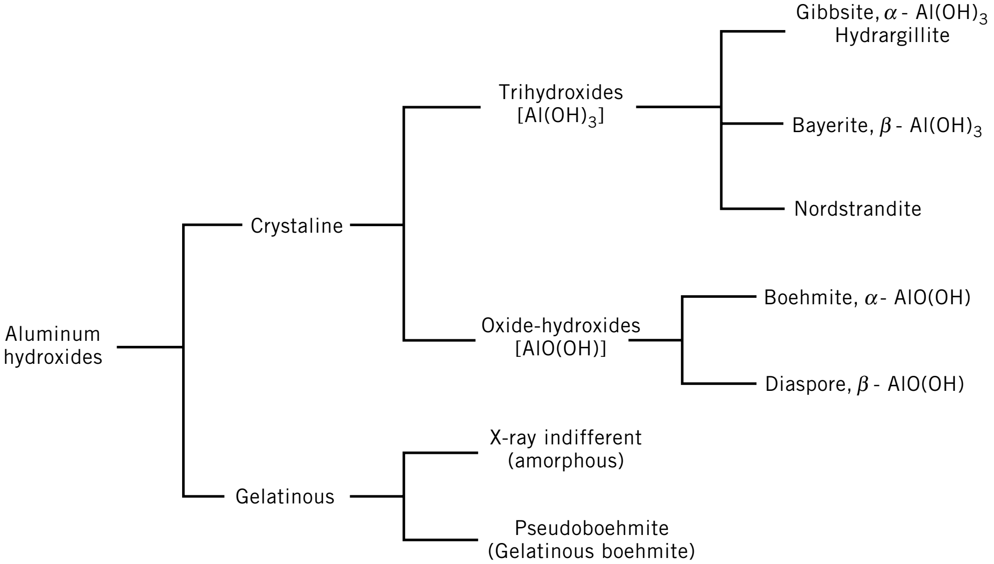

Fig. 1. Classification of aluminum hydroxides. Courtesy of Aluminum Company of America.

existence of several other forms of aluminum hydroxides have been claimed. However, there is controversy as to whether they are truly new phases or structures having distorted lattices containing adsorbed or interlamellar water and impurities.

The terms gelatinous alumina or alumina gel cover a range of products in which colloidal hydrated alumina is the predominant solid phase. Structural order varies from x-ray indifferent (amorphous) to some degree of crystallinity. The latter product has been named pseudoboehmite or gelatinous boehmite. Its x-ray diffraction pattern shows broad bands that coincide with the strong reflections of the well-crystallized boehmite.

2. Crystalline Alumina Hydrates

The mineralogical, structural, physical, and thermodynamic properties of the various crystalline alumina hydrates are listed in Tables 1, 2, and 3, respectively. X-ray diffraction methods are commonly used to differentiate between materials. Density, refractive index, tga, and dta measurements may also be used.

2.1. Gibbsite (α-Aluminum Trihydroxide, Hydrargillite). Gibbsite, commonly associated with bauxite [1318-16-7] deposits of tropical regions, is the most important aluminum compound. The gibbsite lattice consists of double layers of hydroxide ions, and aluminum occupies two-thirds of the interstices within the layers. The hydroxyls of adjacent layers are situated directly opposite each other. The layers are somewhat displaced in the direction of the a-axis and the hexagonal symmetry (brucite type) is lowered to monoclinic. The particle size of gibbsite varies from 0.5 to nearly 200 μm depending on the method of prepara-

Table 1. **Mineralogical Properties of Aluminum Hydroxides**

Material	Index of refraction[a] α	β	γ	Cleavage	Brittleness	Mohs' hardness	Luster
gibbsite	1.568	1.568	1.587	(001) perfect	tough	$2\frac{1}{2}-3\frac{1}{2}$	pearly vitreous
boehmite	1.649	1.659	1.665	(010)		$3\frac{1}{2}-4$	
diaspore	1.702	1.722	1.750	(010) perfect	brittle	$6\frac{1}{2}-7$	brilliant pearly

[a] The average index of refraction for bayerite is 1.583.

Table 2. **Structural Properties of Aluminum Hydroxides**

Material	Crystal system[a]	Space group	Unit axis length, nm a	b	c	Angle	Density, g/cm³
Al(OH)$_3$							
gibbsite	monoclinic[b]	C_{2h}^5	0.8684	0.5078	0.9136	94°34′	2.42
bayerite	monoclinic	C_{2h}^5	0.5062	0.8671	0.4713	90°27′	2.53
nordstrandite	triclinic	C_1^1	0.5114	0.5082	0.5127	70°16′ 74° 0′ 58°28′	
AlO(OH)							
boehmite	orthorhombic	D_{2h}^{17}	0.2868	0.1223	0.3692		3.01
diaspore	orthorhombic	D_{2h}^{16}	0.4396	0.9426	0.2844		3.44

[a] Unit cell contains two molecules unless otherwise indicated.

[b] Unit cell contains four molecules.

tion. The smaller crystals are composed of plates and prisms whereas the larger particles appear as agglomerates of tabular and prismatic crystals. The basic crystal habit is pseudo-hexagonal tabular.

The usual commercial method of preparation of gibbsite is by crystallization from a supersaturated caustic aluminate, $NaAlO_2$, solution. Seed gibbsite crystals are used.

$$NaAlO_2 + 2\,H_2O \longrightarrow Al(OH)_3 + NaOH$$

Table 3. **Thermodynamic Data for Crystalline Aluminum Hydroxides at 298.15 K and 0.1 MPa**[a]

Substance	Molecular weight	Molar vol, cm³/mol	ΔH°_f, kJ/mol[b]	ΔG°_f, kJ/mol[b]	S°, J/(mol·K)[b]	C_p, J/(mol·K)[b]
gibbsite	78.004	31.956	−1293.2	−1155.0	68.44	91.7
bayerite	78.004		−1288.2	−1153.0		
boehmite	59.989	19.55	−990.4	−915.9	48.43	65.6
diaspore	59.989	17.76	−999.8	−921.0	35.33	53.3

[a] To convert MPa to psi, multiply by 145.

[b] To convert J to cal, divide by 4.184.

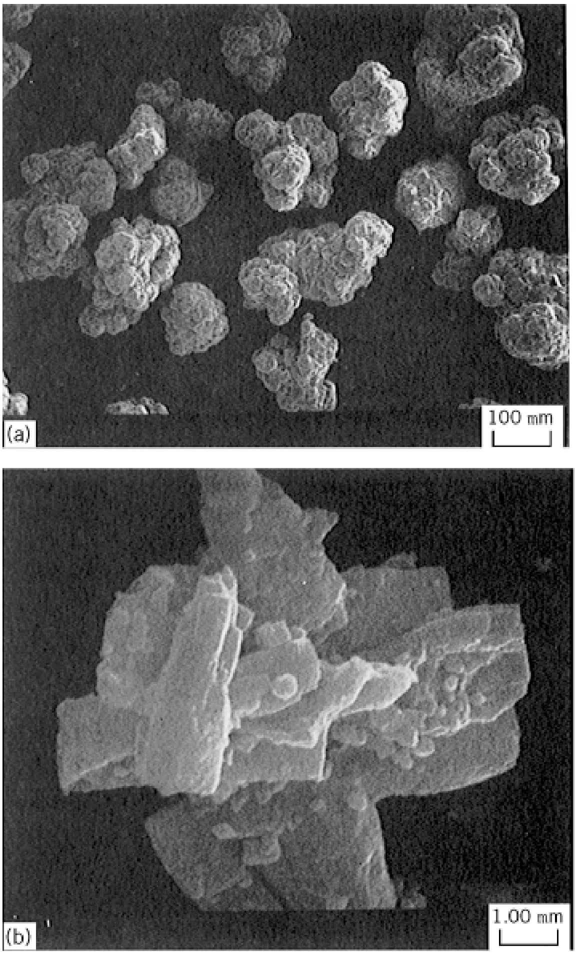

Fig. 2. Aluminum trihydroxides: (**a**) coarse gibbsite from the Bayer process, ×100; (**b**) Schmäh bayerite, ×10,000. Courtesy of Aluminum Company of America.

Alternatively, neutralization of sodium aluminate [1302-42-7] by CO_2 can be employed.

$$2\,NaAlO_2 + CO_2 + 3\,H_2O \longrightarrow 2\,Al(OH)_3 + Na_2CO_3$$

Crystallization at temperatures of about 40°C results in heavy nucleation and a fine product. At temperatures above 75°C, only crystal growth occurs, giving rise to large, well-crystallized aggregates composed of hexagonal rods and prisms (Fig. 2**a**).

Gibbsite usually contains several tenths of a percent of alkali metal ions; the technical product, precipitated from a sodium aluminate solution, contains up to 0.3% Na_2O which cannot be washed out even using dilute HCl. Several authors (1,2) suggest that these alkali ions are an essential component of gibbsite structure.

Gibbsite is an important technical product and world production, predominantly by the Bayer process. Most (90%) is calcined to alumina [1344-28-1], Al_2O_3, to be used for aluminum production. The remainder is used by the chemical industry as filler for paper, plastics, rubber, and as the starting material for the preparation of various aluminum compounds, alumina ceramics, refractories, polishing products, catalysts, and catalyst supports.

2.2. Bayerite (β-Aluminum Trihydroxide). Bayerite is rarely found in nature. It has been synthesized by several methods: A pure product is prepared by the Schmäh method (3) in which amalgamated aluminum reacts with water at room temperature. Other methods include rapid precipitation from sodium aluminate solution by CO_2 gassing, aging of gels produced by neutralization of aluminum salts with NH_4OH, and rehydration of transition rho alumina.

Unlike gibbsite, pure bayerite can be prepared without any alkali ions and there is evidence that bayerite converts irreversibly to gibbsite in the presence of alkali (Na and K) metal ions. Bayerite does not form well-defined single crystals for proper structural analysis. The most commonly observed growth forms are spindle or hourglass shapes formed by stacking of $Al(OH)_3$ layers in a direction perpendicular to the basal plane (Fig. 2**b**). The bayerite lattice is also composed of double layers of OH, but hydroxyl groups of one layer lie in the depressions between the OH positions of the second. This approximately hexagonal close packing results in the higher density of bayerite compared to that of gibbsite.

Bayerite is a commercially available technical product that is produced in small quantities mainly for alumina catalyst manufacture. High purity aluminum [7429-90-5] metal has been converted to bayerite to produce very high purity aluminum oxides.

2.3. Nordstrandite. The x-ray diffraction pattern of an aluminum trihydroxide which differed from the patterns of gibbsite and bayerite was published (4) prior to the material, named nordstrandite, being found in nature. The nordstrandite structure is also assumed to consist of double layers of hydroxyl ions and aluminum occupies two-thirds of the octahedral interstices. Two double layers are stacked with gibbsite sequence followed by two double layers in bayerite sequence.

Pure nordstrandite has been prepared (5) by reaction of aluminum, aluminum hydroxide gel, or hydrolyzable aluminum compounds with aqueous ethylenediamine [107-15-3]. However, no commercial production or uses have been reported.

2.4. Boehmite (α-Aluminum Oxide-Hydroxide). Boehmite, the main constituent of bauxite deposits in Europe, is also found associated with gibbsite in tropical bauxites in Africa, Asia, and Australia. Hydrothermal transformation of gibbsite at temperatures above 150°C is a common method for the synthesis of well-crystallized boehmite. Higher temperatures and the presence of alkali increase the rate of transformation. Boehmite crystals of 5–10 μm size (Fig. 3) are produced by this method. Fibrous (acicular) boehmite is obtained under acidic hydrothermal conditions (6). Excess water, about 1% to 2% higher than the stoichiometric 15%, is usually found in hydrothermally produced boehmite.

The structure of boehmite consists of double layers in which the oxygen ions exhibit cubic packing. Hydroxyl ions of one double layer are located over the depression between OH ions in the adjacent layer such that the double layers

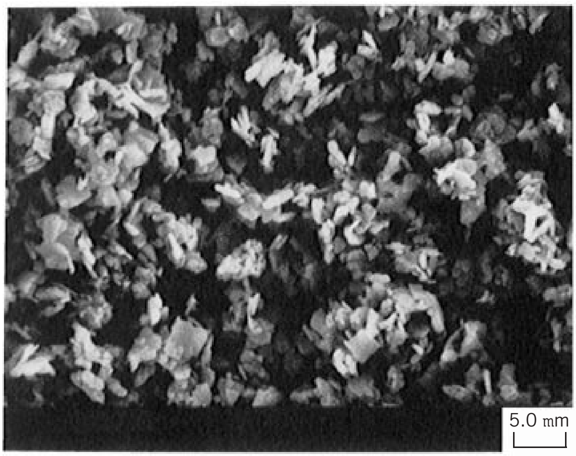

Fig. 3. Aluminum oxide–hydroxide: hydrothermally prepared boehmite, ×2,000. Courtesy of Aluminum Company of America.

are linked by hydrogen bonds between hydroxyls in neighboring planes. There is some technical production and use of synthetically produced boehmite.

2.5. Diaspore (β-Aluminum Oxide Hydroxide). Diaspore, found in bauxites of Greece, China, and the former USSR, can also be obtained by hydrothermal transformation of gibbsite and boehmite. Higher (>200°) temperatures and pressure (>15 MPa–150bar) are needed for synthesis and the presence of diaspore seed crystals helps to avoid boehmite formation.

In the diaspore structure, the oxygen ions are nearly equivalent, each being joined to another by way of a hydrogen ion and arranged in hexagonal close packing. This arrangement accounts for the higher density of diaspore as compared to boehmite. Although diaspore-containing bauxites and clays have been used for the production of high alumina refractories, no commercial use or large-scale synthesis of diaspore has been reported.

3. Gelatinous Aluminum Hydroxides

Apart from the crystalline forms, aluminum hydroxide often forms a gel. Fresh gels are usually amorphous, but crystallize on aging and gel composition and properties depend largely on the method of preparation. Gel products have considerable technical use.

The amphoteric behavior of aluminum hydroxide, which dissolves readily in strong acids and bases, is shown in Figure 4. In the pH range of 4 to 9, a small change in pH towards the neutral value causes rapid and voluminous precipitation of colloidal hydroxide which readily forms a gel. Gels are also formed by the hydrolysis of organoaluminum compounds such as aluminum alkoxides (see ALKOXIDES, METAL).

Aluminum hydroxide gels contain considerable excess water and variable amounts of anions. Even after prolonged drying at 100–110°C, the water content

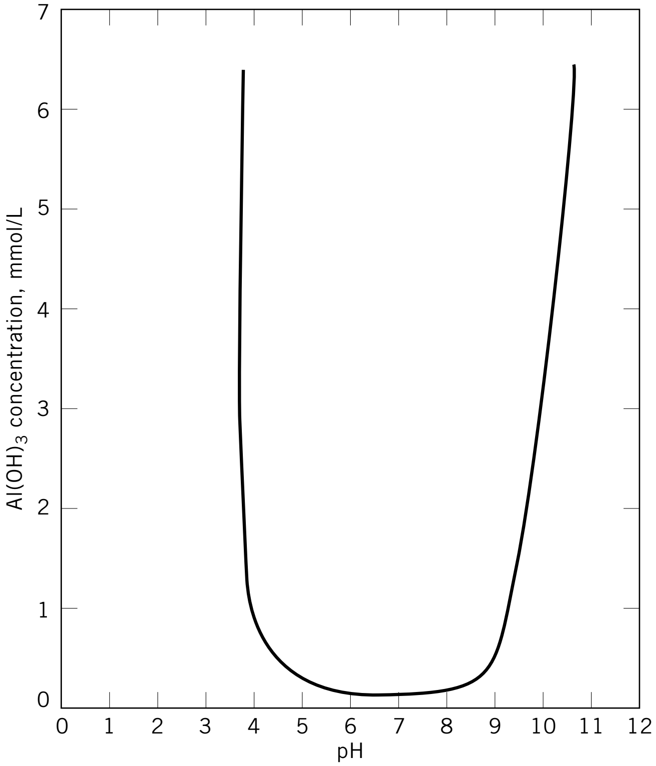

Fig. 4. Amphoteric behavior of $Al(OH)_3$. Courtesy of Aluminum Company of America.

can be as high as 5 mols H_2O/mol Al_2O_3. The initial precipitated product is usually amorphous (x-ray indifferent), except for material prepared at a pH above 7 or at high temperatures. Gradual transformation to crystalline hydroxide (aging) occurs; the rate is dependent on the OH ion concentration and temperature. Gelatinous boehmite is the first x-ray identifiable crystalline phase and the diffraction pattern shows broad bands that coincide with strong reflections of well-crystallized boehmite (7). In contact with a mother liquor having a pH greater than 7, gelatinous boehmite transforms into crystalline trihydroxides; the rate of transformation increases with pH and temperature. The transformation sequence may be represented by (8)

$$Al^{3+} + 3\,OH^- \xrightarrow{\text{pH}<8} \text{basic } Al^{3+} \text{ salt}^{\text{ pH 8}} \text{ xray amorphouse gel}^{\text{ pH 9}}$$

$$\text{gelantinous boehmite} \xrightarrow{\text{pH 10}} \text{bayerite (or nordstrandite)} \xrightarrow{+Na^+ \text{ or } K^+} \text{gibbsite}$$

Gelatinous boehmite, called alumina gel in commercial use, is used in the preparation of adsorbents, desiccants (qv), catalysts, and catalyst supports (see CATALYSTS, SUPPORTED). A significant amount is used in pharmaceutical preparations.

4. Phase Relations in the Al_2O_3–H_2O System

Under equilibrium vapor pressure of water, the crystalline trihydroxides, $Al(OH)_3$ convert to oxide–hydroxides at above 100°C (9,10). Below 280°–300°C, boehmite is the prevailing phase, unless diaspore seed is present. Although spontaneous nucleation of diaspore requires temperatures in excess of 300°C and 20 MPa (200 bar) pressure, growth on seed crystals occurs at temperatures as low as 180°C. For this reason it has been suggested that boehmite is the metastable phase although its formation is kinetically favored at lower temperatures and pressures. The ultimate conversion of the hydroxides to corundum [1302-74-5],Al_2O_3, the final oxide form, occurs above 360°C and 20 MPa.

Several nonequilibrium forms of aluminum oxides have been observed (11,12) in hydrothermal experiments at low water vapor pressures in the temperature region of 300–500°C. The KI–Al_2O_3 form, also known as tohdite [12043-15-1], $Al_2O_3 \cdot 1/5H_2O$, is characterized by a distinct x-ray diffraction pattern.

5. Production

Aluminum hydroxides are technically the most widely used members of the alumina chemicals family. The most important source of aluminum hydroxides is the bauxite refining plant for alumina production. A small amount of somewhat purer aluminum hydroxide is produced by the Sinter process.

The operating conditions and practices of alumina refining plants are generally geared to the production of a single grade of metallurgical alumina and the hydroxide has rather limited chemical uses in the form produced for this conversion. Variations in particle size, purity, and other properties are generally needed before the hydroxide can be utilized for chemical applications. Thus a part of the aluminate liquor is diverted to special purpose crystallization vessels. The crystallized product is filtered, washed thoroughly using clean hot water to remove adhering caustic liquor, and dried. Care has to be exercised in the drying operation to avoid exposure to high (>150°C) temperatures which could cause dehydration and thus affect such properties as surface activity and dissolution rate in acids and alkali. Several commercial grades of aluminum hydroxide are produced. The properties of some grades are given in Table 4.

Hydroxide grades can be surface-treated to modify dispersion behavior and rheological properties. The most widely used surface coating agents are stearic acid [57-11-4] and stearates. Additionally, compounds from the silane group have been used as coupling agents to give improved adhesion to polymers when the hydroxide is used as a filler.

5.1. Normal Coarse Grade Bayer Hydrate.

The normal coarse grade Bayer hydrate has the lowest cost. It corresponds to the usual Bayer plant product produced for conversion to metallurgical alumina. Traditionally, the American Bayer product is coarser than the European product; chemical compositions are nearly identical. The hydroxide is approximately 99.5% pure. Principal impurities are soda, Fe_2O_3, SiO_2, and small amounts of TiO_2. Organic impurities originating from bauxite impart an off-white or yellowish color to the hydroxide. This low cost commodity grade material is widely used for the manufacture of alum and other aluminum chemicals.

Table 4. **Properties of Commercial Grade Aluminum Hydroxides**

Property	Normal coarse grade[a]	Normal white grade[b]	Ground[c]	Fine precipitated[d]
Al_2O_3, wt %	65.0	65.0	65.0	64.7
SiO_2, wt %	0.012	0.01	0.02	0.04
Fe_2O_3, wt %	0.015	0.004	0.03	0.01
Na_2O (total), wt %	0.40	0.15	0.30	0.45
Na_2O (soluble), wt %	0.05	0.05	0.05	0.1–0.25
LOI at 1200°C, wt %[e]	34.5	34.5	34.5	34.5
moisture at 100°C, wt %	0.1	0.1	0.4	0.3–1.0
specific gravity	2.42	2.42	2.42	2.42
bulk density (loose), g/cm^3	1.2–1.4	1.0–1.1	0.7–1.25	0.13–0.22
surface area, m^2/g	0.1	0.15	2–4	6–8
color	off-white	white	off-white	white
refractive index	1.57	1.57	1.57	1.57
Mohs' hardness	2.5–3.5	2.5–3.5	2.5–3.5	2.5–3.5
Particle size, cumulative wt %				
retained 100 mesh = 149 μm	5–20	0–1		
retained 200 mesh = 74 μm	65–90	5–15		
retained 325 mesh = 44 μm	90–98	30–65	1–2	0.1–0.2
passing 325 mesh = 44 μm	2–10	35–70	98–99	99.8
median particle size, μm			6.5–9.5	0.6

[a] Alcoa C-30.

[b] Alcoa C-31.

[c] Alcoa C-330.

[d] Alcoa Aydral 710.

[e] Loss on ignition.

5.2. White Hydroxide. The soda sinter process applied to bauxite or bauxite residue produces a hydroxide that is completely free from organic coloring matter and is very white. A value of more than 95% is obtained on the GE brightness scale relative to TiO_2 as followed in the paper (qv) industry. This compares to about 70% on the same scale for the normal Bayer product. The white hydroxide is preferred in the paper, toothpaste, and artificial marble industries.

5.3. Ground Hydroxides. Many applications of aluminum hydroxides require smaller particle sizes obtained mostly by grinding the dry hydroxide from the refining plant, followed, if necessary, by size classification. Types of grinding equipment include: ball and jar mills (sometimes ceramic lined and using ceramic balls to avoid iron contamination), vibrating mills, disc mills, and air or steam jet pulverizers. Aluminum hydroxide is relatively easy to grind; consistency of particle size distribution is desirable in the production and applications of ground hydroxides. Direct precipitation of a fine hydroxide is possible, but is not widely practiced because filtration, washing, and drying of fine products are relatively more difficult. Ground hydroxides are used extensively as fire retardant fillers in plastics (see FLAME RETARDANTS).

5.4. Low Soda Hydroxide. The Na_2O content of normal Bayer hydroxide is around 0.2–0.4%, 0.1% of which can be removed by thorough washing. The remaining soda is trapped within the hydroxide crystal. Experience shows that the occluded soda content is reduced when crystallization is carried out

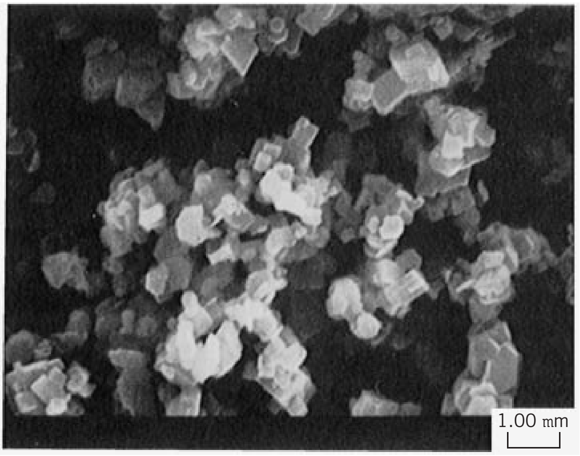

Fig. 5. Fine precipitated aluminum hydroxide, ×10,000. Courtesy of Aluminum Company of America.

under low alumina-supersaturation conditions and at relatively higher temperatures (80–95°C). Soda contents as low as 0.05% Na_2O can be obtained by this procedure. However, these conditions also reduce hydroxide yield and thus increase the production cost. Low soda aluminum hydroxide is generally employed in the production of aluminas for the ceramics industries.

5.5. Extra-Fine Precipitated Hydroxide. Very fine (<1 μm-diameter) particle size hydroxide is produced by precipitation under carefully controlled conditions using specially prepared hydroxide seed. Precipitation is usually carried out at low (30–40°C) temperatures causing massive nucleation of fine, uniform hydroxide particles (Fig. 5). Tray or tunnel type dryers are used to dry the thoroughly washed filter cake to a granular product which is easily pulverized to obtain the fine hydroxide. Alternatively, the washed product is spray dried. Precipitation from an organic-free aluminate liquor, such as that obtained from the soda–sinter process, yields a very white product. The fine precipitated hydroxide is used by the paper and plastic industries as fillers.

5.6. Production of Other Aluminum Hydroxides. Commercial production of gelatinous aluminum hydroxide involves the neutralization of aluminum sulfate [10043-01-3] or ammonium alum [7784-25-0] by ammonia or sodium hydroxide. Neutralization of a sodium aluminate solution by acids, $NaHCO_3$, or CO_2 has also been used. The gelatinous product is difficult to filter and wash, and the product is often aged to improve washability. The filter cake is either dried and pulverized, or directly extruded and cut to the desired shape and size, and then dried. Spray drying has also been used. The dried powder may be agglomerated to yield a spherical product or pressed into pellets. Drying and dehydration in a hot oil bath also has been used to produce spherical particles. Rate, temperature, pH variation, agitation during neutralization, and the subsequent aging all play significant roles in the development of reproducible properties of the final product. Gelatinous aluminum hydroxides are extensively used in the preparation of adsorbent and catalytic aluminas. They are also used in the pharmaceutical industry and in the manufacture of printing inks.

The Ziegler process involves the formation of aluminum alkoxides at an intermediate stage (see ALCOHOLS, HIGHER ALIPHATIC). Hydrolysis of the alkoxides produces aluminum hydroxide having pseudoboehmite structure. The hydroxide product is further processed to remove residual alcohols and then dried. The chemical purity is generally high; the most significant contaminant is sometimes TiO_2. Catalytic applications have been the principal market for this product because of its higher purity.

Commercial production of bayerite is relatively small and employs CO_2 neutralization of caustic aluminate liquor obtained from either Bayer or sinter processes. The product obtained is about 90% crystalline bayerite having small amounts of gibbsite, pseudoboehmite, and amorphous aluminum hydroxides.

6. Shipping and Analysis

Shipping of aluminum hydroxide powders is usually in paper bags of 10 to 25 kg size. Bulk shipment by road or rail wagons is also common. Aluminum hydroxides are not hygroscopic, but could be dusty and precautions against dust inhalation should be taken during handling.

Many of the procedures used for technical analysis of aluminum hydroxides are readily available from the major producers of aluminum hydroxides.

Phase Composition. Weight loss on ignition (110°–1200°C) can differentiate between pure (34.5% $Al(OH)_3$) trihydroxides and oxide–hydroxides (15% $Al(OH)_3$). However, distinction between individual trihydroxides and oxide–hydroxides is not possible and the method is not useful when several phases are present together. X-ray powder diffraction is the most useful method for identifying and roughly quantifying the phase composition of hydroxide products.

Thermal Analyses. Thermal analysis often complements x-ray data in providing information on phase composition. The thermal behavior of aluminum hydroxides is particularly important in filler type applications.

Particle Size and Shape. Particle size information is important for many aluminum hydroxide applications. Sieve analysis, both dry and wet, is commonly used in the 20 μm (using micro-sieves) to 200 μm size range. Sizes in the 2–200 μm range have been analyzed by light microscopy, sedimentation (eg, Andreasen pipette, sedimentation balances, Sedigraph instrument), Coulter Counter, and MicroTrac instruments. Electron microscopy is the only reliable procedure for characterizing particles below 2-μm size. Scanning electron microscopy has proved useful in obtaining information on particle shape and the relation to behavior in many applications.

Alkalinity (Soluble Soda) Determination. The surface alkalinity or soluble or leachable soda is determined by making a fixed weight percent slurry in water and determining the alkalinity of the solution by pH measurement or acid titration. Sodium ion-sensitive electrodes have been investigated.

Whiteness and Brightness. Photometric instruments, originally developed by the paper industry, are used for these measurements. Values are compared against standard white pigments such as $BaSO_4$, TiO_2, or MgO.

Chemical Analysis. Chemical impurities commonly analyzed include Na_2O, Fe_2O_3, and SiO_2. The hydroxide is first dissolved in boiling concentrated HCl. Atomic absorption methods have replaced older colorimetric procedures.

Other Measurements. Other tests include free moisture content, rate of dissolution and undissolved residue in acids and alkali, resin and plasticizer absorption, suspension viscosity, and specific surface area. Test procedures for these properties are developed to satisfy application-related specifications.

7. Economic Aspects

U.S aluminum hydroxide production capacity was nearly 940×10^3t in 1997 (13). About 90% of world production came from the Bayer process; the remaining came from Sinter, Ziegler, and gel processes. Although many alumina plants possess the capability to make the normal Bayer-grade hydroxide, specialty grade aluminum hydroxides for the chemical industry are generally produced in alumina refining plants dedicated to nonmetallurgical alumina products.

In 2000, 1×10^3t of aluminum hydroxide were consumed in the U.S.; 960×10^3t were consumed in Western Europe, and 430×10^3t were used in Japan (14).

8. Health and Safety Factors

Aluminum hydroxides are minimally absorbed by the body and LD_{50} values for ingestion are unavailable. Death upon ingestion occurs from intestinal blockage rather than systemic aluminum toxicity (15). It is only as a fine particulate suspended in air that aluminum hydroxides may gain entry (via the lungs) into the body in amounts of physiological significance. Evidence collected among workers in the alumina refining industry has failed to show any effect of aluminum hydroxide dust on the lungs. However, in recognition of the possible adverse effects of long term exposure to alumina dusts, threshold limit values have been established by the ACGIH (16) as follows: 10 mg/m^3 TLV–TWA and 20 mg/m^3 TLV–STEL. Aluminum hydroxide [21645-51-2] and aluminum hydroxide oxide [24623-77-6] are reported in EPA TSCA inventory.

9. Uses

In 2000, aluminum hydrate was used in flame retardants, reinforcement fillers in plastics, elastomers, and adhesives, filler pigments, coatings in papermaking, precursors for the production of activated alumina and other specialty aluminas, and as a raw material for the production of aluminum (14).

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