ALUMINUM OXIDE (ALUMINA), ACTIVATED

1. Activated

The activated aluminas comprise a series of nonequilibrium forms of partially hydroxylated aluminum oxide [1344-28-1], Al_2O_3. The chemical composition can be represented by $Al_2O_{(3-x)}(OH)_{2x}$ where x ranges from about 0 to 0.8. They are porous solids made by thermal treatment ofaluminum hydroxide [21645-57-2] precursors and find application mainly as adsorbents, catalysts, and catalyst supports. Activated alumina, for purposes of this discussion, refers to thermal decomposition products (excluding α-alumina [12252-63-0]) of aluminum trihydroxides, oxide hydroxides, and nonstoichiometric gelatinous hydroxides. The term "activation" is used in this article to indicate a change in properties resulting from heating (calcining). Other names for these products are active alumina, gamma alumina, catalytic alumina, and transition alumina. Transition alumina is probably the most accurate because the various phases identified by x-ray diffraction are really stages in a continuous transition between the disordered structures immediately following decomposition of the hydrous precursors and the stable α-alumina which is the product of high temperature calcination.

2. Physical and Chemical Properties

In general, as a hydrous alumina precursor is heated, hydroxyl groups are driven off leaving a porous solid structure of activated alumina. The transformation is

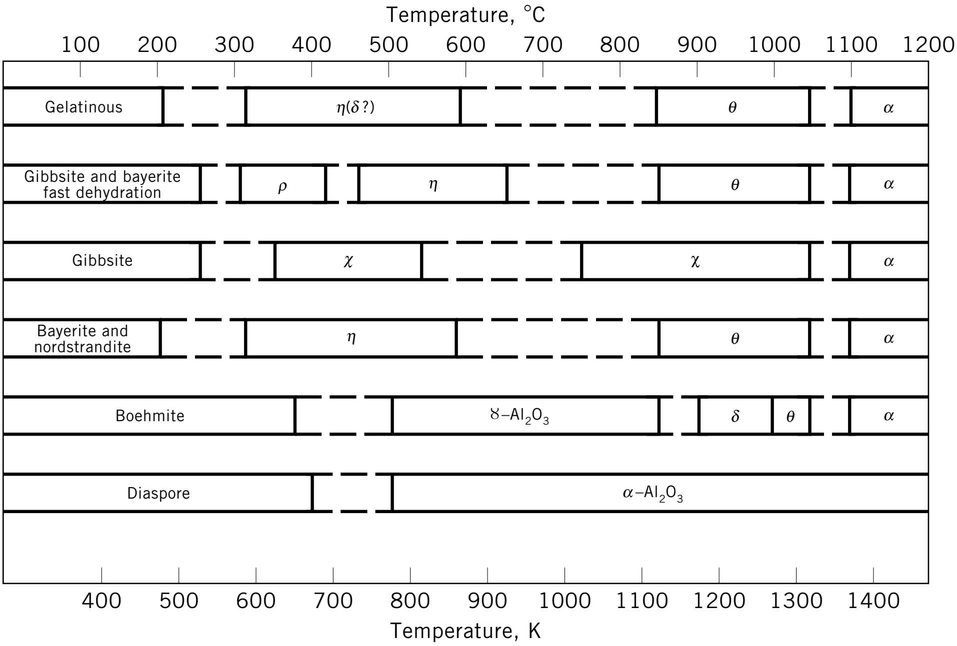

Fig. 1. Decomposition sequence of hydrous aluminas.

topotactic and little change in size or shape of the material is observed at low magnifications. At magnifications higher than about 10,000, changes in texture resulting from recrystallization can be seen. The physical properties of the material are set by the choice of precursor, the forming process, and the activation conditions.

Figure 1 shows the decomposition sequence for several hydrous precursors and indicates approximate temperatures at which the activated forms occur (1). As activation temperature is increased, the crystal structures become more ordered as can be seen by the x-ray diffraction patterns of Figure 2 (2). The similarity of these patterns combined with subtle effects of precursor crystal size, trace impurities, and details of sample preparation have led to some confusion in the literature (3). The crystal structures of the activated aluminas have, however, been well-documented by x-ray diffraction (4) and by nmr techniques (5).

2.1. Decomposition of Boehmite. Boehmite [1318-23-6], AlO(OH), can be synthesized having surface areas ranging from about 1 to over 800 m^2/g depending upon the method of preparation (6). The properties of activated boehmite products are strongly influenced by the crystallite size of the precursor material (7). When a crystallized, low surface area boehmite is heated, conversion to gamma alumina occurs at about 725 K yielding a low surface area product having a well-defined x-ray pattern. On further heating, the transition follows the gamma-delta-theta-alpha sequence shown in Figure 1. In contrast, a very high surface area boehmite (also referred to as pseudoboehmite or gelatinous boehmite) decomposes at 575–625 K yielding a high surface area product having

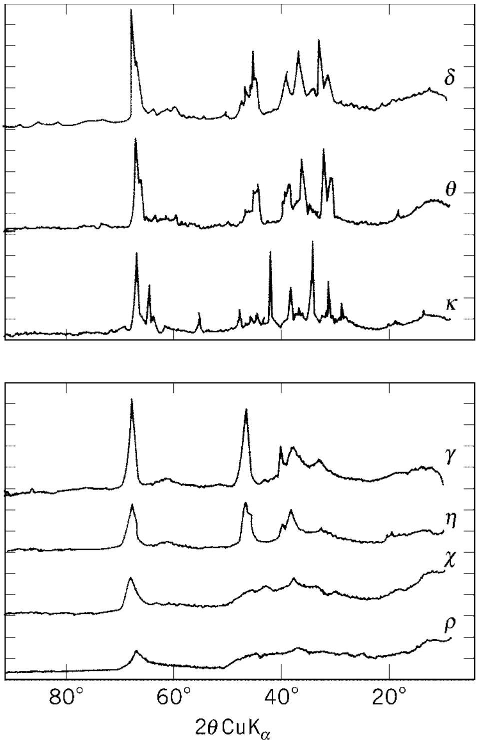

Fig. 2. X-ray diffraction patterns of transition aluminas.

a poorly defined gamma alumina x-ray pattern. On further heating, the transformation of this material to delta and theta phases may be retarded or may not occur at all, depending upon trace impurities (8). This sequence is also indicated in Figure 1. Figure 3 shows surface areas of three boehmite samples having different initial surface areas after heating for 16 hours at various temperatures. In

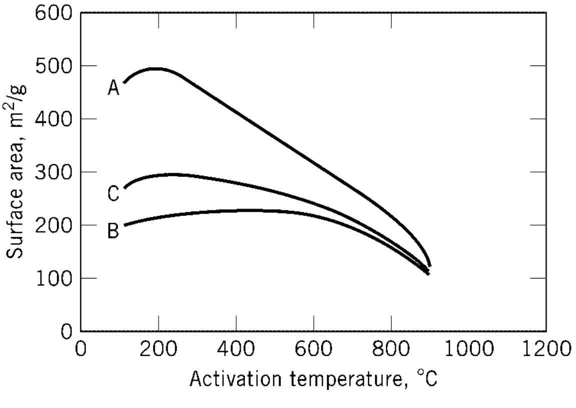

Fig. 3. Surface area of boehmite samples after activation, where A and B are experimental boehmite and C is Catapal SB.

the low temperature region, the highest surface area starting material maintains a higher surface area than the others, but in the range where delta-theta phases would be expected, the curves converge. This loss of surface area is a manifestation of coarsening of the activated alumina crystals and is also accompanied by a shift toward larger pore sizes on heating (9). In the high surface area materials, the surface area is mostly attributable to the external surface area of the precursor crystallites. Evolution of internal pore structure has been well-documented for activation of well-crystallized boehmite (10), but this is only a minor contribution to surface areas of the commercially important activated boehmites, typically in the 200–350 m^2/g range (11,12). Loss of surface area on heating is inevitable, but can be either retarded or accelerated by various additives (13).

2.2. Activation Products of Aluminum Hydroxide. Figure 4 shows the effect of temperature on some properties of activated alumina produced by heating gibbsite [14762-49-3], α-$Al(OH)_3$ (14). As the material is heated, surface area reaches a maximum of 300 m^2/g or more at about 650 K. As temperature is increased further, surface area decreases and the skeletal structure becomes more dense reflecting increased ordering of the crystalline structure during the progression from chi to kappa to alpha. At about 1450 K conversion to alpha alumina occurs with a major rearrangement of crystal structure and corresponding decrease in surface area to about 5 m^2/g. These trend vary somewhat according to precursor crystal size, purity, and the atmosphere of heating.

Unlike the case for boehmite, the surface area of activated gibbsite results mainly from internal porosity rather than external surface of the precursor. There is a minor effect of initial crystal size, however. Some (10% or less) boehmite tends to form when coarse crystals of gibbsite are activated because of hydrothermal conditions which are generated in the particles. On further activation, this boehmite decomposes, but contributes little to the overall surface area. For this reason, activation of fine gibbsite tends to give somewhat higher maximum surface areas than coarser material (15). High humidity in the activating gas can also promote boehmite formation and lower surface area.

Activation of bayerite [20257-20-9] and nordstrandite [13840-05-6] qualitatively follows the pattern shown in Figure 4, but the transition sequence is eta-theta-alpha. The structures of these transition phases are somewhat different from those obtained from gibbsite, reflecting the differences in crystal structure of the hydroxides (16).

If aluminum hydroxide is decomposed by heating at low temperature under vacuum or by rapidly heating at high temperature, a nearly amorphous (x-ray indifferent) phase known as rho alumina is produced which has the interesting property of recrystallizing (rehydrating) to boehmite or bayerite when mixed with water (17). This behavior is known as rehydration bonding and occurs to a significant degree in hot water at atmospheric pressure (18). Rho alumina can be formed from any of the aluminum hydroxides. The crystal structures are probably somewhat different depending upon which precursor (gibbsite or bayerite) is used, but this cannot be detected by x-ray and rehydration properties are similar. Some recrystallization to boehmite occurs in all of the activated aluminas under severe hydrothermal conditions and generally the degree to which this occurs decreases with increased crystalline order (higher activation

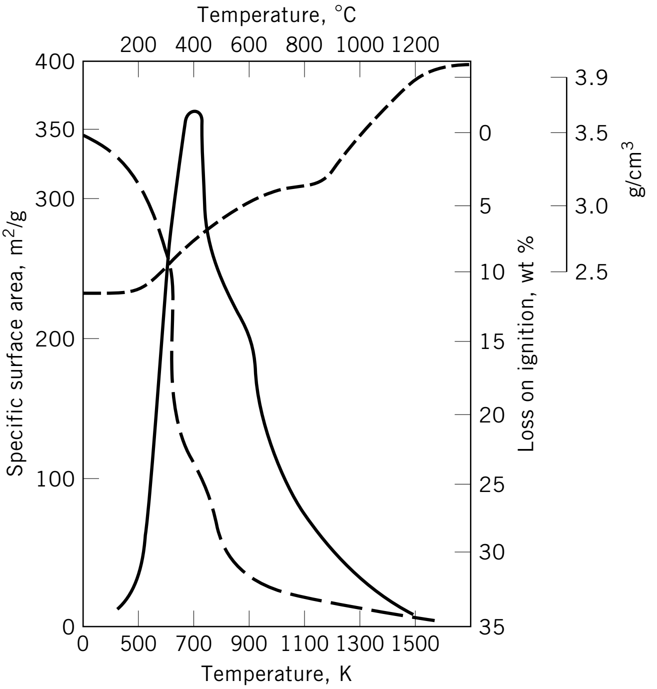

Fig. 4. Properties of activated gibbsite, where --- represents density, ——— is the loss of ignition, and — is specific surface area.

temperature). The ease with which rho alumina rehydrates sets it apart from the other activated alumina phases.

Except for rho alumina, the activated aluminas are quite stable when mixed with water in the pH range from about 4 to 10. Below pH 2 and above pH 12 they degrade rapidly.

The surface of activated alumina is a complex mixture of aluminum, oxygen, and hydroxyl ions which combine in specific ways to produce both acid and base sites. These sites are the cause of surface activity and so are important in adsorption, chromatographic, and catalytic applications. Models have been developed to help explain the evolution of these sites on activation (19). Other ions present on the surface can alter the surface chemistry and this approach is commonly used to manipulate properties for various applications.

3. Manufacturing Processes

The large majority of activated alumina products are derived from activation of aluminum hydroxide, rehydrated alumina, or pseudoboehmite gel. Other commercial methods to produce specialty activated aluminas are roasting of aluminum chloride [7446-70-0], $AlCl_3$, and calcination of precursors such as ammonium alum [7784-25-0], $AlH_7NO_8S_2$. Processing is tailored to optimize one or more of the product properties such as surface area, purity, pore size distribution, particle size, shape, or strength. A process for the production of β-alumina solid electrolyte without calcination has been reported (20).

3.1. Activated Aluminum Hydroxide. The principal precursor for this class of products is gibbsite derived from the Bayer process although a small amount of bayerite is also used for some specialty catalytic applications. Bayer process gibbsite is available in very large tonnages as an intermediate product in aluminum production. It is 99+% pure: the main impurity is sodium oxide [1313-59-3], Na_2O, at 0.2–0.3% on a $Al(OH)_3$ basis. Low cost, relatively high purity, and availability make gibbsite the raw material of choice for many activated alumina products.

Gibbsite is a free-flowing powder having a median particle diameter of about 100 micrometers. Traditionally, activated alumina powders were produced by passing this material through a rotary kiln at an appropriate temperature. However, the aluminum industry has developed fluid bed and flash heating technology for calcination of metallurgical alumina and Bayer alumina-based activated aluminas are often produced in this type of equipment (21,22). The particle sizes of the activated aluminas are essentially the same as that of the precursor powder and in the unground form are amenable to applications involving fluid bed handling. These powders can also be ground to sizes of 10 micrometers or less by standard techniques. Conventional activated aluminas are produced by slow heat treatment whereas “rehydratable” powders are activated within a few seconds.

Larger particle size products can be produced by direct activation of agglomerated gibbsite. The oldest product of this class is made from “scale” which forms on the walls of Bayer process precipitators and is periodically removed in massive chunks, then crushed and activated. This gives a granular product, having particle sizes up to 1 cm or more, which has good mechanical properties and is relatively inexpensive. A similar product is manufactured by high pressure compaction of gibbsite powder. Once the gibbsite agglomerates are formed they are converted to the desired activated alumina product by a combination of crushing, screening, and heat treatment.

Unrefined bauxite [1318-16-7] is also used as a precursor to low cost activated alumina because bauxites can contain as much as 90% gibbsite (dry basis). These materials represent the low cost, low performance end of the activated alumina spectrum of products, but they are still used in significant quantities.

A method for processing bauxite rich in alumina monohydrate had been reported. The monohydrate is more difficult to digest using the Bayer process and normally require higher temperatures (23).

3.2. Rehydration Bonded Alumina. Rehydration bonded aluminas are agglomerates of activated alumina, which derive their strength from the rehydration bonding mechanism. Because more processing steps are involved in the manufacture, they are generally more expensive than activated aluminum hydroxides. On the other hand, rehydration bonded aluminas can be produced in a wider range of particle shape, surface area, and pore size distribution.

The generic process used to manufacture these materials begins with an activated powder produced by rapid (flash) activation and grinding of Bayer process gibbsite. This powder is generally composed of a mixture of chi and rho forms, the proportion depending upon the specifics of the activation process. The powder is then mixed with water and formed into a shape by tumbling (24), extrusion (25), or dropping a slurry into an immiscible fluid (oil-drop) (26,27). During or after the forming process, the shapes are heated without drying from several minutes to several hours at temperatures between about 330 and 370 K. This allows partial rehydration to occur, rigidizing the structure. At this point, the alumina is a mixture of pseudoboehmite, bayerite, chi, and amorphous phases (18, 24). The shapes are then given a second heat treatment or calcination to establish the desired degree of activation. The surface area, which depends upon the micropores, is largely determined by the second heat treatment whereas the total pore volume is set by specifics of the forming operation. Activated aluminas prepared by this method are mixtures of phases reflecting the composition after rehydration.

3.3. Gel-Based Activated Aluminas. Alumina gels can be formed by wet chemical reaction of soluble aluminum compounds. An example is rapid mixing of aluminum sulfate [17927-65-0],$Al_2(SO_4)_3{\cdot}XH_2O$, and sodium aluminate [1302-42-7], $NaAlO_2$, solutions to form pseudoboehmite and a near neutral sodium sulfate [7757-82-6] solution (28). After extensive washing to remove the sodium sulfate, the resulting gel can be dried or partially dewatered and formed directly by extrusion or oil drop. If the gel is dried prior to forming, it can be ground to fine particle size, then mixed with water and tumbled or formed by the general processes described above (29). The shapes are then activated to produce relatively pure phase gamma alumina. Gels made from aluminates and aluminum salts must be carefully washed to remove undesirable anions and cations which would be detrimental to the final application.

Hydrolysis of aluminum alkoxides is also used commercially to produce precursor gels. This approach avoids the introduction of undesirable anions or cations so that the need for extensive washing is reduced. Although gels having surface area over 800 m^2/g can be produced by this approach, the commercial products are mostly pseudoboehmite powders in the 200–300 m^2/g range (30). The forming processes already described are used to convert these powders into activated alumina shapes.

"Oil-drop" covers an interesting group of processes to produce small activated alumina spheres or beads by dispersing an aqueous alumina sol or solution in an immiscible liquid. The surface tension effects cause the aqueous droplets to attain a spherical shape and, while in this condition, they gel. Gellation can be accomplished by neutralization with ammonia (28, 30), dehydration with alcohol, or through rehydration bonding (26). Beads of very uniform size have been generated by forming droplets of aluminum nitrate [13473-90-0], $Al(NO_3)_3$, solution using a specially designed nozzle, allowing them to be partially gelled with ammonia [7664-41-7] vapor then falling into an oil– ammonia solution for final gellation (31). A new process for producing aluminum oxide beads is discussed in Ref. 32.

The gel-based products have traditionally been the most expensive and highest performance activated alumina products. They have very good mechanical properties, high surface area, and their purity and gamma-alumina structure make them somewhat resistant to thermal degradation. On the other hand, they are the most difficult to manufacture; and disposal of by-product salts can present an environmental problem.

4. Economic Aspects

The least expensive products are those derived directly from Bayer-process gibbsite and powders are generally less expensive than formed products. The soda content (0.2–0.3% Na_2O) of Bayer gibbsite makes it unattractive for many catalytic applications. Gel-based products are normally used where low soda level is required. Soda content of gels prepared from inorganic salts or aluminate solutions is typically about 0.03% whereas soda in alkoxide-based gels is much lower. Specialty activated aluminas having purity as high as 99.99% are also available at a much higher price.

Shaped products used for adsorbent purposes are generally less sophisticated and therefore less expensive than catalytic products.

5. Safety and Handling

Activated alumina is a relatively innocuous material from a health and safety standpoint. It is nonflammable and nontoxic. Fine dusts can cause eye irritation and there is some record of lung damage because of inhalation of activated alumina dust mixed with silica [7631-86-9] and iron oxide [1317-61-9] (33). Normal precautions associated with handling of nuisance dusts should be taken. Activated alumina is normally shipped in moisture-proof containers (bags, drums, sling bins) because of its strong desiccating action.

U.S. and international exposure standards for aluminum oxide are given in Table 1 (35).

Table 1. **U.S. and International Exposure Limits for Aluminum Oxide**

U.S. OSHA PEL	TWA 15 mg/m^3, total dust TWA 5 mg/m^3, respirable fraction
U.S. NIOSH REL	No REL
U.S. ACGIH TLV	TWA 10 mg/m^3, A4
Australia	TWA 10 mg/m^3
Belgium	TWA 10 mg/m^3
Denmark	TWA 10 mg/m^3
France	TWA 10 mg/m^3
Germany (DFG MAK)	TWA mg/m^3 1.5, respirable fraction of the aerosol, (fume)
Ireland	TWA 10 mg/m^3, total inhalable dust TWA 5 mg/m^3, respirable dust
Japan (JSOH)	TWA 05 mg/m^3, respirable dust TWA 2 mg/m^3, total dust
The Netherlands	TWA 10 mg/m^3, inhalable
Poland	TWA 2 mg/m^3, STEL 16 mg/m^2
Russia	STEL 4 mg/m^3
Switzerland	TWA 6 mg/m^3

6. Uses

6.1. Catalytic Applications. Activated alumina is used commercially in catalytic processes as a catalyst, catalyst substrate, or as a modifying additive. Activated alumina serves as the catalyst in the Claus process for recovering sulfur from H_2S that originates from natural gas processing or petroleum refinery operations (34). The alumina is generally in the form of spheres, about 5 mm in diameter. This size has evolved as a good compromise between high activity and low pressure drop for fixed bed application (36). Promoting the alumina using a small amount of alkali has been claimed to enhance performance in certain Claus operations (37) (see SULFUR REMOVAL AND RECOVERY).

The largest application for activated alumina as a catalyst substrate is in hydrotreating of petroleum feedstocks (qv) (38). The purpose of hydrotreating is threefold: to increase the H/C ratio; to remove O, S, and N impurities; and to remove V, Ni, and other tramp contaminants, especially from residuum of heavier feedstocks. Specialized alumina-based catalysts typically promoted with compounds of Co, Mo, W, and Ni have been developed for these operations (34). The catalysts are usually in the form of extrudates having variously shaped cross sections (39) (circular, lobed, wagon-wheel) about one millimeter in diameter. Spherical catalysts 1 mm or less in diameter have also been described for hydrotreating (26). Much attention has been given to optimizing pore volume and pore size distribution of the activated alumina substrates used in these operations and the “optimum” properties vary with operating conditions and the petroleum feedstock being processed (40,41).

Another catalytic application for promoted alumina is in automotive exhaust catalysts which enhance oxidation of hydrocarbons, carbon monoxide, and nitrogen oxide in exhaust gas (see EXHAUST CONTROL, AUTOMOTIVE). There are two general configurations of catalyst currently in use: beads and monoliths. In both systems, the catalytic component is precious metal (platinum, palladium, and rhodium) on alumina (42). The bead system consists of a bed of 3-mm diameter alumina spheres which act as both catalyst substrate and mechanical support. In the monolith system, the mechanical support is provided by a porous ceramic (cordierite [12182-53-5]) multichannel “honeycomb” having a thin alumina coating (washcoat) as the catalytic substrate. Automotive exhaust catalyst was the largest volume application (about 18,000 t/yr) for promoted alumina in the mid-1970s and was a significant driving force in improving the technology of low density alumina sphere manufacture. Since that time, however, monoliths have become the system of choice for this application. Beads have maintained only a small share of the market and consequently the volume of activated alumina used in automotive catalysts has dropped significantly (43).

The largest tonnage single application for catalyst particles is in fluid cracking (FCC). These materials are typically made from zeolite having a clay or alumina–silica binder system to provide the necessary mechanical strength for fluid bed handling. Addition of alumina (aluminum hydroxide, pseudoboehmite, or rehydratable alumina) to these formulations has been reported to improve various properties (44,45). Any alumina powder would be activated under FCC processing conditions, thus the activated alumina is used as a modifying additive in the catalytic process. The actual usage of activated alumina in FCC catalysts is unclear because of the highly competitive and proprietary nature of this market.

A number of smaller but nevertheless important applications in which activated alumina is used as the catalyst substrate include: alcohol dehydration, olefin isomerization, hydrogenation, oxidation, and polymerization (46). A new method for synthesizing hydrocarbons using a silica–alumina support for group VIII metal has been reported (47).

6.2. Chromatographic Applications. Activated alumina has been used for many years in the separation of various organic compounds by normal phase chromatography (qv) because of its natural hydrophilic surface characteristics. More recently, stable surface coatings have been developed which impart hydrophobic properties to the particle surface (48). These coatings, coupled with improved technology to produce closely sized particles have allowed alumina to compete in reverse-phase chromatographic markets. Compared to silica, the dominant reverse-phase packing material, alumina has better chemical stability at moderately high pH levels, which gives it a natural advantage for separations in this pH range (49).

6.3. Membranes. Membranes comprised of activated alumina films less than 20 μm thick have been reported (50). These films are initially deposited via sol–gel technology (qv) from pseudoboehmite sols and are subsequently calcined to produce controlled pore sizes in the 2 to 10-nm range. Inorganic membrane systems based on this type of film and supported on solid porous substrates have been introduced commercially. They are said to have better mechanical and thermal stability than organic membranes (51). The activated alumina film comprises only a miniscule part of the total system (see MEMBRANE TECHNOLOGY).

6.4. Adsorbent Applications. One of the earliest uses for activated alumina was removal of water vapor from gases and this remains an important application. Under equilibrium conditions alumina adsorbs an increasing amount of water as the relative humidity of the contacting gas increases. At 50% rh, for example, a good quality activated alumina can adsorb water at levels of 15 to 20% or more of its own weight (52). By heating the activated alumina to about 525 K under low rh conditions, essentially all of the adsorbed water is removed and the alumina is returned to its original state. This adsorption–regeneration cycle can be repeated hundreds of times with little deterioration of the adsorbent. Industrial scale countercurrent drying systems use this cyclic approach, but generally are designed for lower water loadings because economic cycle times are too short for equilibrium to be established. Another consideration in the large beds of industrial dryers is the amount of heat generated which amounts to about 46 kJ/mol (11 kcal/mol) of water adsorbed. A common strategy is to operate the drying systems under pressure. This partially reduces the water content in the gas before it enters the bed by condensation and also increases the heat capacity of the gas, facilitating heat removal from the bed (53). Under proper operating conditions, moisture in the dried gas can be as low as 11 ppmv. The usual forms of activated alumina used in desiccant applications are granules or spheres about 3 to 12 mm in size. Besides air, a partial list of gases that can be dried includes Ar, He, H_2, CH_4, C_2H_6, C_3H_8, C_2H_2, C_2H_4, C_3H_6, Cl_2, HCl, SO_2, NH_3, and fluorochloroalkanes. Activated alumina is also used to remove water from organic liquids including gasoline, kerosene, oils, aromatic hydrocarbons, cyclohexane, butane and heavier alkanes, butylenes, and many chlorinated hydrocarbons.

Besides drying applications, activated alumina is used to selectively remove various species from gas and liquid systems. In refining and petrochemical operations, activated alumina is used to remove trace HCl from reformer hydrogen, fluorides from hydrocarbons produced by HF alkylation, and a variety of other stream cleanup applications (52). An important example in air pollution abatement is adsorption of fluoride vapors emanating from the aluminum smelting operation (see ALUMINUM AND ALUMINUM ALLOYS). Fluoride-contaminated air from the smelting cell area is countercurrently passed through fluid beds of alumina cell feed which adsorbs the fluorides for recycle. Fluorides are also effectively removed from potable water by activated alumina (54). Fluoride tends to adsorb on alumina at low pH levels and desorb (regenerate) when the pH is increased so in these systems, regeneration of the alumina adsorbent is accomplished by pH control rather than thermal treatment. Arsenic has also been effectively removed from potable water by activated alumina (55). Activated alumina is receiving renewed attention as an adsorbent and a wealth of information has been published on adsorption characteristics of many chemical species (56, 57). Decontamination of chemical warfare agents using activated aluminum oxide has been patented (58).

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Alan Pearson
Aluminum Company of America