

Air and Gas Drying With AxSorb™ Activated Alumina



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Water is removed from air and other gases to avoid undesirable effects caused by liquid and ice formation, corrosion, and catalyst contamination. Water removal steps appear in a large number of applications, two examples of which are air conditioning and industrial gas drying. Activated alumina figures prominently among the ways to remove water from air or gas streams; it is particularly suited to drying compressed air and to drying gases having high relative humidities. Among the reasons for the widespread acceptance of alumina in these applications are its low cost, chemical and physical resistance, regenerability and high capacity for retaining water.

Commercial drying conditions are more demanding than ever, notably for air service where high gas velocities, pressures and regeneration frequencies are often specified by customers. The formulations of high quality aluminas we are offering to satisfy these needs are based on a half-century of know-how and experience in alumina development and manufacture.

Alumina and Axens

Our main alumina production facilities are in Salindres, southern France, a site whose history is strongly entwined with that of aluminum (Figure 1).



Figure 1. Axens Salindres Manufacturing Site

Built in the mid-nineteenth century, the Salindres plant was the first in the world to produce aluminum commercially from bauxite (after *les Baux*, the village in Provence where ore was discovered). A manufacturing plant there first began to produce alumina in 1953 using the "flash process" patented by Pechiney.

Since then, the site has been a major producer of alumina and catalysts with Procatalyse Catalysts eventually taking over the production, technology, development and commercial activities.

The production facilities became part of Axens on its establishment in 2001 with the merger of Procatalyse and the Industrial Division of IFP Energies nouvelles. Today, the Salindres plant develops and manufactures catalysts and aluminas.

Axens' Activated Alumina Know-How

The conventional raw material for obtaining activated alumina is bauxite. By reaction with soda, bauxite is transformed via the Bayer process to sodium aluminates. After washing and separation, the 'red mud' (with impurities such as iron oxide and silica) is removed and gibbsite is obtained (Figure 2).

In commercial practice, activated aluminas are manufactured by calcination at 400 to 600°C of gibbsite.

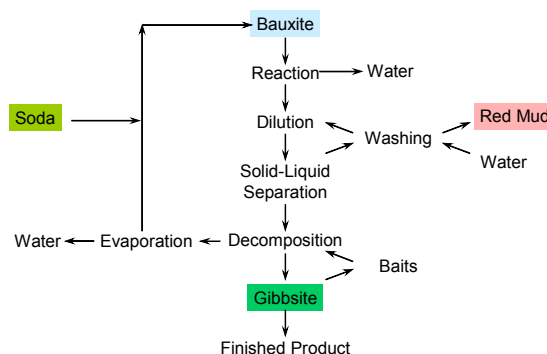


Figure 2. Bayer cycle for the bauxite transformation into gibbsite.

The relative amounts of two chemical species having different crystallographic forms, gibbsite and boehmite, in bauxite ore determine the final alumina quality. When coarse grains of gibbsite made by the above process undergo thermal dehydration without applying special process technology, the resulting product exhibits low specific surface area (only a few square meters per gram) and low pore volume due to the presence of inactive boehmite that attains levels as high as 40%. For that reason, a special process, called the "Flash process" was developed in Salindres in order to produce activated aluminas with high surface area, high specific pore volume and particularly high adsorptive properties.

We have established through extensive research and proprietary manufacturing technology the ratios needed for different alumina grades exhibiting the correct combination of specified adsorption capacity and desired physical characteristics such as crush strength and attrition resistance.

AxSorb Product Descriptions

We manufacture two grades of activated alumina in spheres of various diameters for air drying, which belong to the AxSorb product line:

AxSorb A. This is our standard product with optimized characteristics for use in purification or drying, in 3 sphere sizes:

1.5-3 mm or 1/16" (7 x 14 mesh)

2-5 mm or 1/8" (4 x 8 mesh)

4-8 mm (2.5 x 5 mesh)

AxSorb D. The product has been specifically developed for air drying. A smooth aspect of the bead has been designed for improved mechanical properties. Very high attrition resistance and crush strength are combined with high surface area and static adsorption capacity to give AxSorb D the

features required for the most demanding and safest drying operating conditions. This product is available in 3 spheres sizes.

This product enables longer life duration and thus provides direct savings by way of fewer adsorbent bed replacements. Transport, loading, disposal and administrative costs are reduced as well.

AxSorb alumina characteristics for air and gas drying are presented in Table 1.

For activated alumina, adsorption capacity remains the principal parameter for measuring adsorbent performance. Adsorption capacity depends on the surface site reactivity and is measured by the volume of adsorbed water per unit of surface area. Axens uses the terms $E_{0.1}$ and $E_{0.6}$ to express the static adsorbent capacity at 10% and 60% relative humidity, respectively.

Table 1. Typical Physical characteristics of AxSorb aluminas used for drying

	AxSorb A			AxSorb D			
	(Spheres)	1.5-3 mm	2-5 mm	4-8 mm	2-5 mm	2.5-5 mm	4-8 mm
Specific surface, m ² /g		325	335	330	335	335	330
Total pore volume, cm ³ /100g		44	44	44	44	44	44
Bulk Density, kg/m ³		840	820	785	820	820	790
Crush strength, daN		13	19	43	19	21	44
Attrition (AIF method), %		99,3	98,7	98	99,5	99,5	99,2
Na ₂ O content, ppm wt		3200	3200	3200	3200	3200	3200
$E_{0.1}$, %		7.5	7.5	7.5	7.5	7.5	7.5
$E_{0.6}$, %		21,5	21,5	21	21,5	21,5	21

Mechanical strength

Our activated aluminas have very high resistance to attrition and crushing. This is a result of the production process and the spherical shape of the beads. The attrition and crushing values for spheres are well adapted to their applications, the spherical form making possible homogeneous and compact loading with very little subsequent settling. Higher flow rates are enabled and taller drying vessels can be used.

Adsorption characteristics

Adsorption is a process in which a gas, liquid or solid adheres to the surface of a solid without intimate admixture with the atoms of the solid. A selective mass transfer occurs between these two phases.

Since adsorption is a surface phenomenon, good adsorbents are those having large surface areas per unit mass and high attractive forces for the compounds to be adsorbed, called the solute. The adsorption capacity (accumulation of the solute molecules at the surface of the solid) is directly proportional to the surface exposed and depends on

the solute partial pressure and the temperature. Although all molecules are adsorbed to a certain extent, those having high polarity are likely to be more strongly adsorbed than those having low polarity. Since water is a highly polar material, it is much more strongly adsorbed than hydrocarbons, for instance.

The optimized high porosity of activated alumina gives a high specific surface and a large adsorption capacity per unit weight. Even when saturated with water, activated alumina looks and feels dry. Moreover, activated alumina is chemically inert toward most liquids and gases. Its excellent resistance to fouling by olefin polymerization permits its wide use in the treatment of petrochemicals. The porosity of our activated alumina is distributed under carefully controlled procedures during manufacture to enable straightforward and mild operating conditions for both drying and regeneration.

Moisture adsorption and desorption isotherms

The water content of a gas is defined as the weight or volume of water vapor per unit weight or volume of gas (as ppm wt or ppm vol.). For any given temperature water content can also be expressed by its relative humidity, defined as the ratio of its partial pressure to its saturation pressure.

The water adsorption isotherm relates the equilibrium amount of water sorbed on a solid and the water content in a fluid at constant temperature and pressure. The amount of water trapped on a solid at a given relative humidity and temperature depends on its chemical affinity for the solid and the number of available sites for interaction. The capacity of a desiccant for water is expressed normally as the mass of water adsorbed per mass of desiccant.

An example of static adsorption of water on activated alumina is illustrated by adsorption isotherms in Figure 3.

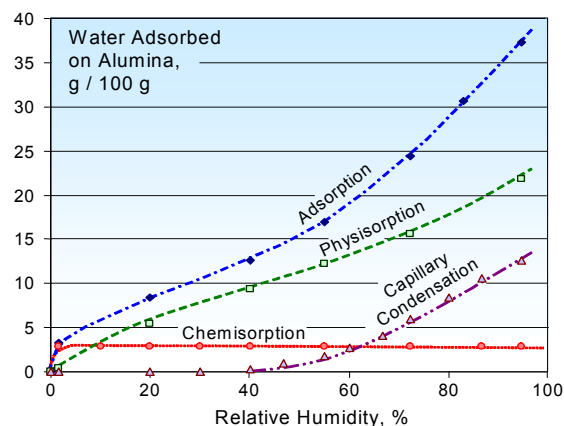
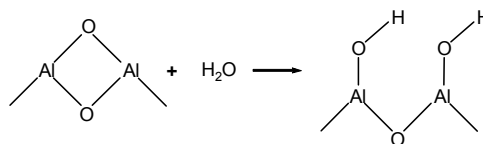


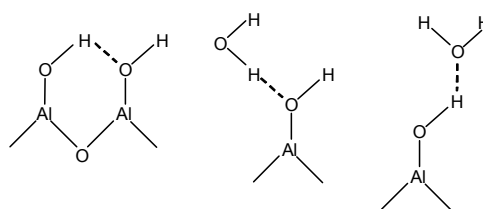
Figure 3. Relative participation of components making up the total water adsorption isotherm on activated alumina: chemisorption, physisorption and capillary condensation.

Water adsorption is the combined result of three phenomena, chemisorption, physisorption and capillary condensation:

- 1. Chemisorption:** dissociative addition of water molecules on aluminum oxide surface sites, forming the first layer at low partial pressures as illustrated by the following diagram.



- 2. Physisorption:** hydrogen bonding with some surface species (Van der Waals' forces), due to the formation of multiple layers by hydrogen bonding in the alumina pores



- 3. Capillary condensation:** growth of multilayers of water (Kelvin's law), where localized condensation takes place at temperatures above that of the bulk fluid's dew point

The activated alumina grades mentioned earlier all demonstrate these adsorption mechanisms, mainly due to their hydrophilic character, microporosity and surface area.

As the relative humidity increases from zero, the initial rapid increase in adsorbed water from zero to two or three per cent indicates that chemisorption is occurring and a monomolecular layer is forming on the surface of the adsorbent. In the case of water vapor, the net heat for the monolayer formation (difference between adsorption and liquefaction heats) is from 6 to 9 kilocalories per adsorbed mole, whereas additional layers (physisorption), this net heat is only about 0.5 kilocalories per mole.

The net heat of adsorption is very high on the most active sites making activated alumina a highly effective desiccant and capable of achieving low water dew points in this region. As the relative humidity increases, the less active sites adsorb water more gradually. The monolayer is essentially complete when the relative humidity reaches approximately 10%. Simultaneously, multilayers of water vapor form which indicates that the physisorption phenomenon is taking place. When the water vapor pressure is sufficiently high (relative humidity reaches 40%), the pores begin to fill by capillary condensation. When the relative humidity of the inlet gas attains 100% and the bed is in equilibrium, there is no further adsorption due

to the three mechanisms, and complete saturation occurs. The water adsorbed by the alumina is then at a maximum of about 40%.

In the case of porous products, a phenomenon of **hysteresis** (difference between the shape of isotherm in adsorption and desorption) can be observed which occurs via a mechanism of capillary condensation.

A typical example of the hysteresis effect is illustrated by the example in Figure 4 which represents static equilibrium conditions for a specific alumina at 25°C.

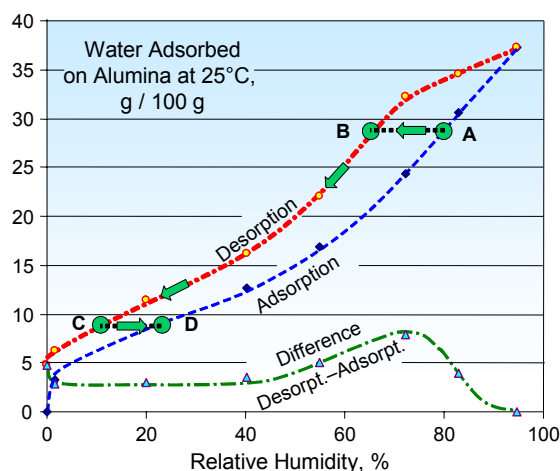


Figure 4. Water adsorption and desorption isotherms on an activated alumina AxSorb D, at 25°C, as a function of relative humidity.

Starting at point A at a relative humidity of 80% in the gas phase, there will be 29 grams of water adsorbed on the 100-gram alumina bed. If the relative humidity in the gas is reduced at constant temperature (desorption curve), no water desorbs until the relative humidity reaches about 65% (point B). With further reduction of the relative humidity to 10%, the amount of water adsorbed on the alumina bed follows the desorption curve, arriving at about 9 grams (point C). From there, when the relative humidity is increased, no adsorption will take place until the intersection of the adsorption curve at about 25% relative humidity (point D).

The general shape of the isotherm, the amount of moisture sorbed at low relative pressures in comparison with the specific surface area of the sample, the reversibility of moisture uptake, the presence of a hysteresis loop, and the shape of the hysteresis loop provide information on the manner of interaction of the solid with water.

A decided advantage of alumina compared with other desiccants is that neither capillary condensation nor free water destroys its structural properties. These phenomena are surface reactions which control the reaction rate. This is the reason why specific surface area is an important feature for

designing adsorbents in dehydration and other applications.

A high internal surface area through the presence of pores (capillaries) is necessary to create adsorption sites. Alumina is the most widely used adsorbent owing to the chemical properties of its surface and its ability to be formulated with well-defined pores defined as follows:

- Macropores (diameter > 500 Å) to enhance diffusion into the pore system.
- Mesopores (50 to 500 Å) to accommodate medium size molecules.
- Micropores (< 50 Å) to accommodate small molecules like water.

We have the capability of optimizing the pore size distribution for a specific adsorbent duty, while retaining high mechanical strength. Chemical species are sometimes used to alter acidity or basicity.

However, the most important parameter (discussed later in the text) is hydrothermal aging. We have developed adsorbents with good specific area and specific surface properties that are very stable over time. This compares favorably to other aluminas offered by the competition.

Aging

The design cycle length of a commercial unit generally corresponds to a capacity that is only a fraction of that available in a new adsorbent. The 'extra' capacity takes into account a gradual reduction in adsorbent capacity during the working life (aging). Multiple regenerations cause losses in effective surface area. In commercial units, hydrothermal deterioration and chemical contamination or fouling are the two main causes of aging.

• Hydrothermal Aging

Hydrothermal aging is the result of gradual irreversible changes in alumina structure, generally due to water exposure at regeneration temperatures. Aging is mainly dependent on the number of regenerations (i.e., adsorption/desorption cycles). This is why it pays to regenerate at longer intervals by using the available capacity to its fullest extent. This is done by adjusting the cycle length.

The main consequence is the surface area reduction is due to a "collapsed pores" effect (Figure 5).

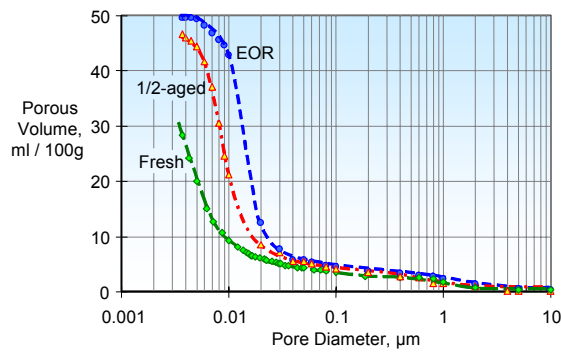


Figure 5. Influence of the hydrothermal aging on the pore diameter of AxSorb D.

Under normal working conditions, aging brings about a 50 % decrease in the initial properties of the alumina in a period of two to four years (Figure 6).

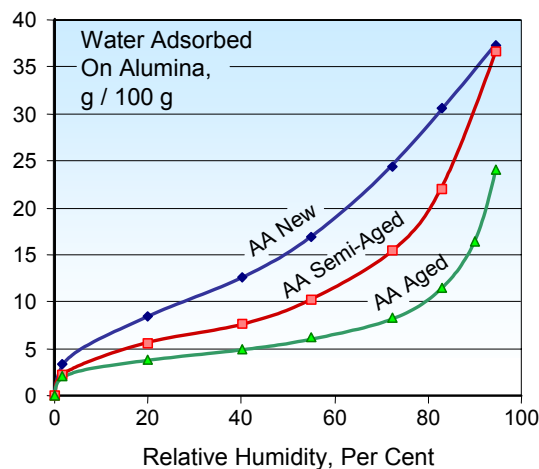


Figure 6. Influence of the hydrothermal aging on the capacity of AxSorb D

• Aging by Contamination or Fouling

Theoretically reversible, this form of aging occurs when the active surface of the alumina is coated or access to its active sites is blocked by either a direct deposit of oil, or by degradation, polymerization or oxidation of unstable compounds present in the fluid passing through the alumina. In practice, the phenomenon is not completely reversible and immobile carbon deposits increase at each regeneration, leading to a progressive decrease of the adsorption dynamic capacity.

Dynamic adsorption

Dynamic adsorption is the most widely used commercial adsorption technique. This is carried out in a vessel filled with beads of activated alumina through which passes the fluid to be dried in two consecutive zones: the equilibrium zone and the mass transfer zone (Figure 7):

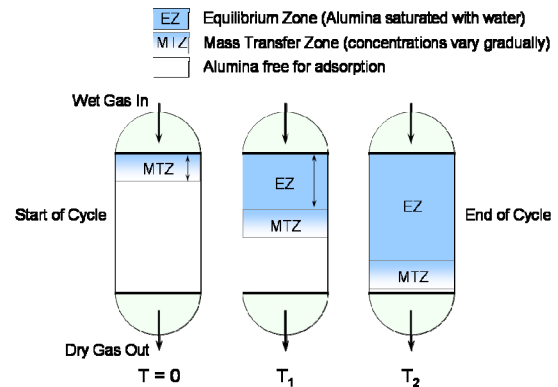


Figure 7. The progress of the Mass Transfer Zone (MTZ) and Equilibrium Zone (ZE) through the bed as a function of time from the start-of-adsorption cycle ($T=0$) to the end-of-cycle (T_2)

- The Equilibrium Zone (EZ) is the region in the bed where the adsorbate held by the alumina approaches the static equilibrium isotherm correlating the relative humidity of the adsorbate in the inlet gas stream with the weight of adsorbate in the bed at the operating temperature and pressure. In this part of the bed, the adsorbate loading can be considered as essentially “saturated”. The volume of the equilibrium zone increases with time, “pushing” the mass transfer zone ahead. After the equilibrium zone, the adsorbent is partially loaded with solute.
- The Mass Transfer Zone (MTZ) is downstream from the EZ where, although the adsorbate held on the bed is in dynamic equilibrium with the adsorbate in the gas at every point along the zone, the bed loading changes from saturation at the inlet to the zone to a small percentage at the zone outlet. The volume of the zone remains constant as the zone travels down the bed. Along the zone length, concentration of adsorbate in the gas can be represented by the “S”-shaped curve (Figure 4). A concentration gradient exists across the MTZ. If the leading edge of the MTZ reaches the end of the bed and the specified (water) concentration is exceeded, breakthrough is said to occur.

The MTZ length depends on many parameters but the main parameters concern transfer kinetics with the external diffusion constraints. These transfer limitations must be minimized if adsorbent efficiency is to be improved.

The following parameters affect transfer kinetics:

- Fluid composition (co-adsorption of other components / contaminants)
- Fluid properties (e.g., viscosity)
- Fluid velocity
- Bead sizing and porosity.

The EZ and MTZ are defined by:

- Quantity of water which has to be adsorbed
- Cycle length required
- Adsorption capacity
- Mass transfer efficiency.

To avoid a “breakthrough” of a component, that is, when its content in the effluent fails to meet a specification, bed design must provide for sufficient capacity to prevent arrival of the MTZ at the bed outlet, allowing time for a second bed to be regenerated. If the bed depicted in Figure 8 were allowed to remain on line much after T_2 , off-specification gas can be expected. At time T_3 , the bed no longer adsorbs any water.

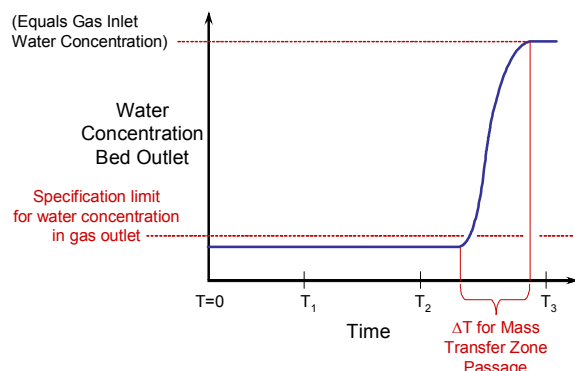


Figure 8. Bed outlet water concentration as a function of time. “Breakthrough” occurred after T_2 and by T_3 , the bed is completely saturated.

Main parameters for vessel sizing

The design of fixed bed adsorption systems is complex. The large number of interrelated parameters in the dehydration of gases and liquids and the regeneration of activated alumina desiccants complicates desiccant specification and system design.

For a given dew point, the breakthrough capacity determines drier performance which in turn depends on the activated alumina characteristics and the operating conditions, i.e., temperature, pressure, relative humidity, gas flow.

Two additional factors must be taken into account: the potential irreversible aging of alumina and, depending on regeneration conditions, the hysteresis phenomenon of alumina not fully recovering its initial properties.

• Relative humidity

The maximum quantity of water which can be fixed at the top of the vessel depends on the relative humidity of the inlet gas.

For example, referring to the isotherm in Figure 4, when for relative humidities of 100% and 25%, the adsorption values at equilibrium are 37 g and 9 g, respectively, of water per 100 g of alumina.

• Temperature

An increase in temperature reduces the adsorption capacity of activated alumina as the adsorption of water on alumina is exothermic. Therefore, it is best to operate an adsorption unit at as low a temperature as is practical.

• Pressure

When a gas is compressed, the partial pressure of the water present increases. At constant temperature, alumina’s adsorptive capacity for water increases with increasing water partial pressure (and relative humidity). Moreover, should the system pressure be increased to the point where the partial pressure of water in the gas exceeds its vapor pressure (the dew point), water will condense which, if it can be removed ahead of the dryers will reduce their duty.

However, operating a unit at a lower pressure than normal will have the reverse effect. This has happened during start-ups or other abnormal operations when the process dryers were operated at lower-than-normal pressures and thus failed to meet the effluent water specification.

• Linear velocity

The diameter of the drying vessel determines linear velocity of the gas, calculated on the empty vessel. The linear velocity affects the MTZ by increasing the kinetics of solute transfer from the fluid to the adsorbent surface.

To illustrate this point, experimental values for breakthrough capacities are given in Table 2 for drying saturated air at 20°C and atmospheric pressure to a dew point of - 40°C.

Table 2. Influence of contact time and linear velocity of the gas on dynamic capacity

	Adsorption Capacity, wt%			
	Contact Time 10 s	Contact Time 5 s	Contact Time 2 s	Contact Time 1 s
Linear velocity 10 cm/s	19	15	10	5
Linear velocity 20 cm/s	19.5	16	11	7.5
Linear velocity 40 cm/s	20	16.5	12	9

The volume of activated alumina to be used is determined by the amount of water to be removed and the process conditions. In practice it is convenient to specify dryer cycles between 8 and 24 hours and contact times from 5 to 30 seconds. The useful adsorptive capacity is on the order of 6 to 15%.

Nevertheless, it remains possible to obtain efficient drying with contact times of 2 seconds or less. This is the case of single-pass supersonic wind-tunnels where the air has to be dried for short periods but at extremely high flow rates; it is then necessary to place the alumina in thin beds to limit the pressure drop. With a 10 cm (0.33 ft) thick bed, a dew point below -40°C can still be attained by using AxSorb A in spheres of 2-5 mm.

• Vessel design and cycle length

Once the capacity has been estimated in accordance with the desired efficiency, the drying vessel dimensions depend on the following parameters:

- Cycle length
- Permissible pressure drop
- Fluidization (if dryers operated up-flow)
- Modified Reynolds number (flow type)
- Safety margin.

Pressure drop is an important economic factor in vessel sizing. For a constant desiccant volume, if the ratio of the height to the diameter of the vessel changes, the pressure drop varies inversely as the effective diameter. In practice, pressure drop must not exceed 0.4 meter of water per meter of bed.

Factors influencing pressure drop include:

- Shape: spheres are better than pellets or extrudates
- Particle size: pressure drop varies inversely with the effective particle diameter
- Porosity of the bed or void fraction. A small size distribution of particles is advised.

Our activated aluminas, available as spheres, feature particularly low pressure drops as compared with equal volumes of granules or extrudates. Their good loading qualities eliminate almost all risk of the channeling that is often observed with granular adsorbents. With these products, very tall vessels can be used because the pressure drop is small, and as these balls are very strong, there is no danger of breakage and attrition.

The pressure drop per unit length of bed $\Delta P/L$ can be calculated from the Ergun equation:

$$\frac{\Delta P}{L} = \frac{A \mu V_0 (1 - \varepsilon)^2}{\varepsilon^3 d_p^2} + \frac{B (1 - \varepsilon) \rho V_0^2}{\varepsilon^3 d_p}$$

ΔP = Pressure drop, Pa

L = Bed height, m

A, B = Shape factors

ε = bed void fraction

μ = dynamic viscosity, Pa • s

V = superficial fluid velocity, m/s

d_p = effective particle diameter, m

To perform the calculation, the effective diameter for AxSorb aluminas is taken as:

2 to 5 mm: 3.3×10^{-3}

4 to 8 mm: 6.0×10^{-3}

ε is between 0.35 and 0.4.

A & B shape factor are equal to 1 for spheres

ρ = fluid density, kg/m³

Choice of particle size

Pressure drop in the equilibrium zone guides the choice of the particle size. In the mass transfer zone, efficiency and pressure drop have to be considered simultaneously. In general, smaller particle sizes provide better mass transfer rates as diffusion is faster.

Configuration

Some dryer units, such as those in ethylene plant service, are required to achieve extremely low water contents (a few tenths of a part-per-million). The dryers play a critical role in that plant upsets due to moisture breakthroughs are avoided at all costs. The general practice today is to use three vessels, two of which operate in series while the third is being regenerated or on standby. The freshly regenerated bed is generally put on line in the second or "guard" position. The lead dryer can then be operated until the moisture breakthrough point allowing it to be fully loaded while the second (lag) dryer ensures the tight specification is met.

This method best protects the process and downstream equipment from upsets and the adsorptive capacity of the three vessels is maximized. It is also one of the most economical.

Drying liquids

Most fluids, including liquids, can be dried by our activated alumina. Liquids are generally dried in the up-flow mode to maximize distribution over the alumina bed. Regeneration can be performed either with a vaporized dry product or with a dry gas (for instance, methane).

In practice, contact times specified for drying liquids are much greater than those for drying gases because the moisture content is higher per unit of fluid volume. For some liquids, very efficient drying can be achieved with a contact time of one minute or less.

Regeneration

The most common configurations consist of two adsorbers, one operating in the adsorption mode while the other operates in the regeneration mode.

At some point, as the MTZ nears the bed outlet, most of the bed is saturated and the original adsorption capacity needs to be restored by desorption. So, once the adsorber columns have been sized, the next step is to determine how to regenerate the desiccant. For less-stringent applications like small dewpoint reductions, pressure reduction may be sufficient to regenerate the bed.

- **When changing relative pressure (PSA: pressure swing adsorption)**

This method, called "heatless drying" by pressure-swing, regenerate by expanding a portion (approx. 15% at 7 bar) of the dried air to atmospheric pressure. This "swing in pressure" causes the expanded air to become very dry as any water contained in the gas will then have a very low partial pressure. The dry purge air causes the moisture to desorb from the desiccant and then carries the desorbed water out of the dryer.

However, if low dewpoints are required, heat must be applied in addition to pressure reduction. If the partial pressure of the adsorbate in the gas is reduced only by depressurization, the adsorbate concentration on the solid phase will be higher than that predicted by the adsorption isotherm. This phenomenon, hysteresis, is due to the retention of strongly-held water molecules. These molecules are desorbed more completely by addition of heat using a hot dry gas at reduced pressure.

- **When changing the isotherm (TSA: temperature swing adsorption)**

Regeneration is performed by exposing the water loaded desiccant to a flow of hot gas. Temperatures of 160°C to 220°C are the most commonly used according to the composition of the regeneration

gas and its water content. Therefore, the regeneration gas may be:

- A dry gas under same or lower pressure than the wet gas being processed
- The same wet gas at the same or at reduced pressure.

Regeneration is generally carried out counter-current to the drying flow direction so that most of the water which is held at the inlet of the bed is not carried through the entire vessel. Thus, part of the vessel is not in contact with hot wet gases, and hydrothermal aging is reduced.

A temperature of 160°C in the bed is sufficient where moderate efficiencies are required (instrument air). When very high efficiency is needed, for instance with a water content of a fraction of ppm in the effluent (low temperature processing), a minimum regeneration temperature of 180°C has been found acceptable in industrial practice. Many inexpensive alternate sources of regeneration heat are thus possible. However, if the regeneration gas is wet, temperatures of 220°C are required. Subsequent cooling must of course be carried out without introducing water.

If the regeneration time is short, or if only a limited quantity of regeneration gas is available, the gas must be brought to a high temperature to provide a sufficient quantity of heat (sensible heat of desorption of the adsorbed water). A temperature up to 250°C is acceptable and causes a very small reduction in useful life.

It is necessary to cool the bed by passing cold gas through it, ensuring that water is not present in the cold gas which would reduce the adsorptive capacity of the bed before starting a new drying cycle.

If a dry gas is not available, it is essential to continue the closed-circuit cooling, as soon as the alumina temperature falls below 100°C. The quantity of water which is then inside the unit as a vapor can be neglected in comparison with the mass of desiccant. Care must be taken to avoid picking up water from the condenser or from unpurged parts of the unit. If cooling with wet gas cannot be avoided, it must be done co-currently with the drying flow direction.

Conclusion

Aluminas manufactured by Axens have earned wide recognition in comparison with standard market solutions. The high surface area and tailored pore distribution provide a high dynamic water adsorption capacity, coupled with excellent physical and mechanical properties. Their outstanding cyclic stability yields a long life time by achieving low water dew point, both with TSA and PSA processes.

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