



SYLOBEAD®

Adsorbents for Process Applications



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The GRACE Davison Silicas/Adsorbents Product Range

SYLOBEAD® Adsorbents for process applications
LUDOX® Colloidal silica
PHONOSORB® and PHONOSORB MTX® Adsorbents for insulating glass
SYLOSIV® Moisture scavengers for coatings and resins
SYLOBLOC® Anti-blocking agents for polymer films
SYLOID® Matting agents for coatings
SYLODENT®/SYLOBLANC® Toothpaste abrasive and thickening agents
SHIELDEX® Non-toxic anti-corrosion pigments
SYLOJET® Ink receptive coatings
TriSyl® Edible oil refining agents
DARACLAR® Beer stabilizer
VYDAC® Chromatographic media & columns
DAVISIL® Separations silica
SYLOWHITE® Titanium dioxide extender for paints and printing inks
ELFADENT® Thickener/mild abrasive for the toothpaste industry
PERKASIL® Reinforcing agent for the rubber industry & for multiple applications
DURAFILL® for the paper and pulp industry
DRI-PAX®, PROTEK-SORB® Packaged desiccants
TEL-TALE® Indicating silica gel

® = Registered Trademark of W.R. GRACE & Co., Columbia/MD

GRACE Davison Setting Standards in Performance. Worldwide

GRACE Davison is a core business of W.R. GRACE & Co., one of the world's largest specialty chemical companies, has as its focus silica and silica alumina products. These include silica gel, zeolites, colloidal silicas, precipitated silicas and precipitated silica aluminas among others.

This broad material portfolio has contributed to GRACE Davison's position as a leading global supplier of silica and zeolitic adsorbents and catalysts. These specialty chemicals can improve product performance or enhance manufacturing processes within a wide range of industrial applications.

We see our key strengths not only in the development of innovative technologies, which help improve product quality and performance but also in our manufacturing flexibility, global infrastructure and our commitment to close customer relationships. With manufacturing sites, research & development centers and sales offices worldwide, GRACE Davison can meet market requirements for speciality chemicals wherever that market may be.

To ensure product quality, all GRACE Davison sites are ISO certified and practice Total Quality Management.



Worms, Germany

Curtis Bay, USA



Safety First

Our first priority is to ensure the safety of everyone who works for us and comes into contact with our products.

At GRACE Davison, the environment is a major issue and we are proud to maintain an outstanding record of leadership and good corporate citizenship. Through the Commitment-to-Care® Program, every GRACE Davison facility worldwide fulfills high health and safety and environmental requirements. Indeed, for this program, we have earned recognition from government and civic

organisations in various states including the Environmental Protection Agency, the Audubon Society, the Environmental Defense Fund, the Centre of Environmental Technology in Hong Kong.

GRACE Davison's Commitment-to-Care® Program is accompanied by a strong commitment to work safety, which led to a substantial reduction in workplace injuries, close to the defined goal of zero accidents. This initiative has earned Grace several "Performance Improvement" awards from the American Chemistry Council.

Experience You can Buy. A Long History in Adsorption

GRACE Davison's interest in adsorption goes back over 85 years, initially with silica gels that were commercially developed in the first quarter of the last century.

In the 1960's, molecular sieves were added to the product portfolio. This long-standing experience presents a significant advantage in the design and optimisation of adsorbent units, well equipping us to face future challenges. With production sites for zeolites in the USA and Germany, and for silica gel in the USA, Germany, Brazil and Malaysia, we can offer excellent products, with a worldwide manufacturing, sales, marketing, R&D and technical service network.

Silica gel is still the material of choice in some processing areas. However, zeolites – or molecular sieves – have gained significant importance since first commercial availability. Over the years, GRACE Davison has become a

world leader in the development and production of commercial molecular sieves and silica gels, known as SYLOBEAD® grades. Our experience, combined with our commitment to research and development, has put us at the forefront of the adsorbents industry – not only for the process industry, but also for refrigerant drying, for the insulating glass industry, for packaging desiccants and desiccant powder. Our comprehensive product range is designed to meet your application needs, with state-of-the-art adsorbent technology, subject to continual evaluation and development.

GRACE Davison's involvement in the oil and gas, petrochemical and refining industries goes beyond adsorbents. GRACE Davison is the world's largest supplier of fluid cracking catalysts, and a major supplier of polyolefin catalysts, hydroprocessing catalysts, in addition to other catalysts and catalyst carriers.

An Insight to our Products. Molecular Sieves and Silica Gel

Structure and Composition of Molecular Sieves

Zeolite molecular sieves are crystalline, highly porous materials, which belong to the class of aluminosilicates. These crystals are characterised by a three-dimensional pore system, with identical pores of precisely defined diameter. This structure is formed by tetrahedras of (AlO_4) and (SiO_4) . These tetrahedras are the basic building blocks for various zeolite structures, such as zeolites A and X, the most common commercial adsorbents.

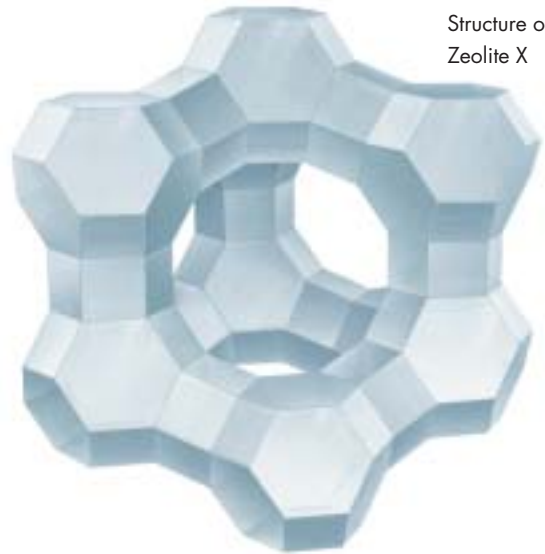
Structure of
Zeolite A



Due to the presence of alumina, zeolites exhibit a negatively charged framework, which is counter-balanced by positive cations resulting in a strong electrostatic field on the internal surface. These cations can be exchanged to fine-tune the pore size or the adsorption characteristics. For instance, the sodium form of zeolite A has a pore opening

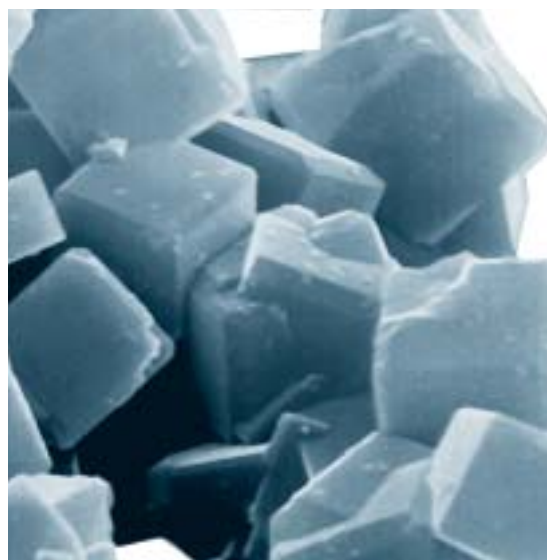
of approximately 4 Ångstrom (4×10^{-10} m), called 4A molecular sieve. If the sodium ion is exchanged with the larger potassium ion, the pore opening is reduced to approximately 3 Ångstrom (3A molecular sieve). On ion exchange with calcium, one calcium ion replaces two sodium

Structure of
Zeolite X



ions. Thus, the pore opening increases to approximately 5 Ångstrom (5A molecular sieve). Ion exchange with other cations is sometimes used for particular separation purposes. The pore opening of the sodium form of zeolite X (13X) is approximately 8 Ångstrom. The ability to adjust the pores to precisely determined uniform openings allows for molecules smaller than its pore diameter to be adsorbed whilst excluding larger molecules, hence the name "molecular sieve". The different pore sizes of synthetic zeolites open up a wide range of possibilities in terms of "sieving" molecules of different size or shape from gases and liquids.

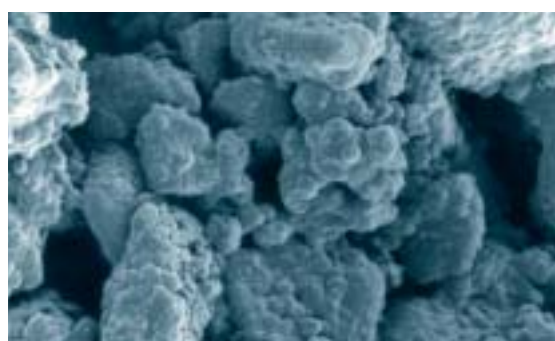
Each zeolite crystal is only a few microns across. To be of use in fixed bed adsorption processes, it is necessary to form particles in the range of 0.5 – 5 mm (4 – 36 mesh) in diameter. Within the above range, a wide selection of SYLOBEAD® particle sizes can be produced to suit the specific needs of the customer. The information gained from decades of research has shown that the usage of bead-shaped bodies for adsorber columns is the optimal solution due to the outstanding mechanical characteristics and excellent adsorption rate properties. GRACE Davison therefore concentrates exclusively on the production of beads.



Zeolite Crystals (Scanning Electron Microscope Picture)

Structure and Composition of Silica Gel

Silica Gel is a porous, amorphous form of silica (SiO_2). Although it has the same chemical composition as sand, silica gel is radically different to other SiO_2 -based materials, due to



Silica Gel Particle (Scanning Electron Microscope Picture)

its unique internal structure. It is composed of a vast network of interconnected microscopic pores. As opposed to zeolites, silica gels have larger pores with a wide range of diameters –

typically between 5 Å and 300 Å – and do not allow for the separation of molecules solely dependent on their size.

It is possible to adjust the pore size range in the manufacturing process. Silica gels synthesized with an average pore size of about 20 Å are known as narrow pore silica gels. Silica gels with an average pore size of about 110 Å are called wide pore silica gels.

As with zeolites, silica gels maintain their structure when activated. Activation frees the large internal surface area and pore volume, enabling physical adsorption and capillary condensation.

Based on the gelation process, silica gel can be manufactured in granulate or bead form, ranging from 0.5 – 6.0 mm (4 – 36 mesh).

A Comparison. Molecular Sieves and Silica Gel

The following overview shows the most important properties of GRACE Davison molecular sieves and silica gels in comparison:

Properties	Zeolite Molecular Sieves	Silica Gel
Chemical Composition	$Me^+_x [(AlO_2^-)_x (SiO_2)_y] \cdot y H_2O$ where Me can be cations like sodium, potassium, calcium, lithium	SiO_2 chemically inert, no metals or cations present
Chemical Stability	Chemically stable in basic, neutral and mild acidic environments	Chemically stable in acidic, neutral and mild basic environments
Mechanical Stability	Presence of liquid water may adversely affect the mechanical stability	Narrow pore gel is unstable against liquid water
Pore Size	Unique pore sizing Zeolite A: $\sim 3\text{\AA}$ or $\sim 4\text{\AA}$ or $\sim 5\text{\AA}$ Zeolite X: $\sim 8\text{\AA}$	Pore size range between 5\AA and 300\AA Average pore size narrow pore gel: 20\AA Average pore size wide pore gel: 110\AA
Surface Area	$800\text{ m}^2/\text{g}$	Narrow pore gels: $800\text{ m}^2/\text{g}$ Wide pore gels: $400\text{ m}^2/\text{g}$
Effective Pore Volume	$0.25 - 0.3\text{ cm}^3/\text{g}$	Narrow pore gels: $0.4\text{ cm}^3/\text{g}$ Wide pore gels: $1.2\text{ cm}^3/\text{g}$
Thermal Conductivity	Beads: $0.12\text{ W}/\text{m}/\text{K}$ $0.07\text{ BTU}/\text{ft}/^\circ\text{F}$	$0.14 - 0.2\text{ W}/\text{m}/\text{K}$ $0.08 - 1.2\text{ BTU}/\text{ft}/^\circ\text{F}$
Heat of Adsorption for Water	$4187\text{ kJ}/\text{kg H}_2\text{O}$ $1800\text{ BTU}/\text{lb H}_2\text{O}$	$2512\text{ kJ}/\text{kg H}_2\text{O}$ $1080\text{ BTU}/\text{lb H}_2\text{O}$
Heat Capacity	$0.96\text{ kJ}/\text{K}/\text{kg}$ $0.23\text{ BTU}/\text{lb}/^\circ\text{F}$	$0.92\text{ kJ}/\text{K}/\text{kg}$ $0.22\text{ BTU}/\text{lb}/^\circ\text{F}$

For Engineers Only. The Physics behind Adsorption

Many porous materials, when exposed to gases or liquids, exhibit a strong physical affinity for these fluids (physisorption). The molecules which make up the fluids, become 'trapped' on the internal surfaces of the materials' pores. These molecules are called 'adsorbates' and are 'adsorbed' in the process of 'adsorption'. Different porous solids exhibit different adsorption properties and different molecules are adsorbed

to different degrees. Adsorption is one of the major separation processes along with distillation, permeation, extraction, crystallisation and absorption.

For both, molecular sieves and silica gel, this adsorption process is completely reversible. Molecules, which have been adsorbed, can be released at high temperature and/or reduced pressures or concentrations.

Adsorption on Molecular Sieves

Zeolites function on the basis of physisorption. The main driving force for adsorption is the highly polar surface within the pores. This unique characteristic distinguishes zeolites from other commercially available adsorbents, enabling an extremely high adsorption capacity for water and other polar components even at very low concentrations.

In addition, the pore size plays a significant role, allowing or prohibiting the entrance of molecules to the pore system.

The adsorption on molecular sieves is therefore dependent on the following physical molecular properties:

- **Size and Shape**

Molecules larger than the pore opening of the molecular sieve can not be adsorbed, smaller molecules can (e.g. n-butane can be adsorbed on SYLOBEAD® 5A molecular sieve, i-butane can not). The Table „Size of Various Molecules“ shows critical diameters for various molecules. It is a useful but not infallible guide, since

molecular diameters, as shown in the literature, depend on the particular determination method. It is possible that molecules apparently larger than the relevant sieve pore can be adsorbed. If in doubt, an adsorption measurement should be made.

- **Molecular Polarity**

Molecules with large polarity or polarisability can be adsorbed preferentially under identical conditions. One example is the drying of methanol: water – with its dipole moment of 1.85 Debye – can be preferentially adsorbed over methanol with a dipole moment of only 1.70 Debye. For molecules with equal dipole moments, the molecule with the higher polarisability will be preferentially adsorbed. For example, carbon dioxide, nitrogen and oxygen have no dipole moment. However, carbon dioxide with a polarisability of $2.91 \cdot 10^{-24} \text{ cm}^3$ can be preferentially adsorbed over nitrogen ($1.74 \cdot 10^{-24} \text{ cm}^3$) and oxygen ($1.58 \cdot 10^{-24} \text{ cm}^3$). Therefore carbon dioxide can easily be removed from air.

Size of Various Molecules

Class	Molecule	Size [Å]
Rare Gases	Helium	2.0
	Argon	3.8
Inorganic Vapors and Gases	Water	2.6
	Ammonia	2.9
	Carbon Monoxide	3.7
	Carbon Dioxide	3.3
	Nitrogen	3.6
S-Compounds	Oxygen	3.5
	Hydrogen Sulphide	3.6
	Methyl Mercaptan	3.8
	Ethyl Mercaptan	3.8
Paraffins	Thiophene	5.3
	Methane	3.8
	Ethane	4.2
	Propane	4.3
	n - Butane and higher normal paraffins	4.3
	i - Butane and higher iso-paraffins	5.1
	neo-Pentane	6.2
Cyclohexane	6.0	
Unsaturates	Acetylene	3.3
	Ethylene	3.9
	Propylene	4.0
	Butene - 1	4.0
	Butene - 2 trans	5.1
	1,3 Butadiene	4.0
Aromatics	Benzene, Toluene	5.8
	p - Xylene	5.9
	o - Xylene	6.6
	m - Xylene	6.3
Amines	Triethylamine	7.8
Alcohols	Methanol	3.6 ⁽¹⁾
	Ethanol	4.2 ⁽²⁾
Cl-Compounds	Carbon Tetrachloride	5.9
	Chloroform	6.9

- Data have been taken from various sources. Data differ slightly for the different sources based on different calculation models. We have chosen those values which did fit best with our practical experiences regarding adsorption behaviour on molecular sieves.

⁽¹⁾ is adsorbed on 3 Å molecular sieve

⁽²⁾ is adsorbed on 4 Å molecular sieve

Adsorption on Silica Gel

Like zeolites, silica gel functions on the basis of physisorption. Adsorption occurs due to van der Waals interactions and capillary condensation at high humidities. The adsorption force is less than for zeolites, resulting in a lower adsorption capacity at low concentrations of adsorbates. At higher concentrations of adsorbates, the adsorption capacity is higher than for zeolites, due to the higher internal pore volume. Certain grades of silica can adsorb up to 1.2 times their own weight of water.

Adsorption Capacity of Grace SYLOBEAD® Molecular Sieves and Silica Gel

When molecular sieves or silica gels from GRACE Davison come into contact with an adsorbate (gases or liquids which can be adsorbed) under constant environmental conditions, the adsorbents remove increasing amounts of adsorbate until equilibrium is reached. The amount of adsorbate removed under these conditions is known as the equilibrium capacity.

The equilibrium capacity is mainly determined by the

- nature of the substance to be adsorbed and type of adsorbent
- adsorbate concentration
- pressure (if in gas phase)
- temperature

Other factors which effect equilibrium capacity are

- co-adsorption effects
- aging of the adsorbent

Adsorption Equilibrium Data for Water and Carbon Dioxide

Isotherms, such as shown in Figure 1, describe the dependency of the adsorption capacity upon the adsorbate partial pressure at a constant temperature.

Of note is the high capacity of SYLOBEAD® Molecular Sieve even at low water concentration, allowing to dry to very low water contents. Whereas, silica gel exhibits superior adsorption capacity at higher water concentrations.

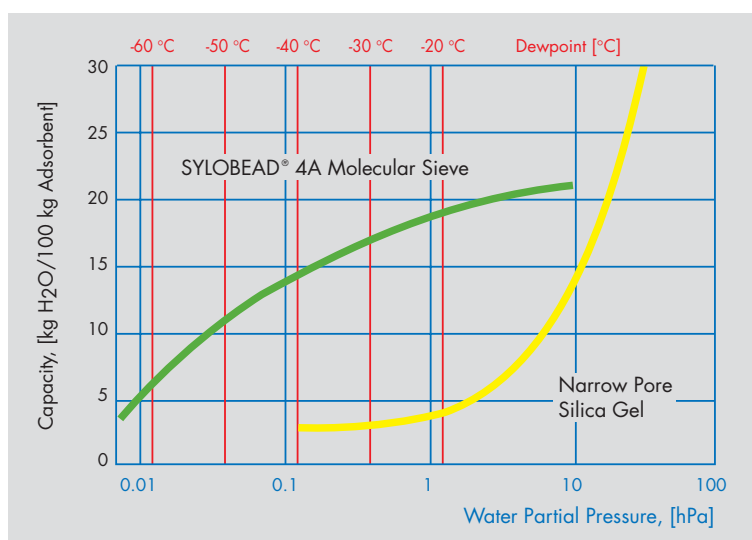


Figure 1 Isotherms for Water Adsorption Capacities of GRACE Davison Adsorbents (25 °C)

Isobars, as shown in Figure 2, describe the dependency of the adsorption capacity upon the adsorbent temperature at a constant adsorbate pressure.

The GRACE Davison SYLOBEAD® molecular sieve can retain high capacity at high temperature, which makes it the optimal material if drying needs to be carried out at comparatively high temperatures. Silica gel loses capacity with increasing temperature. Whilst this may have a negative effect on capacity, it is easier to regenerate.

The effects of temperature and concentration on adsorption as shown above, are qualitatively the same for all adsorbates.

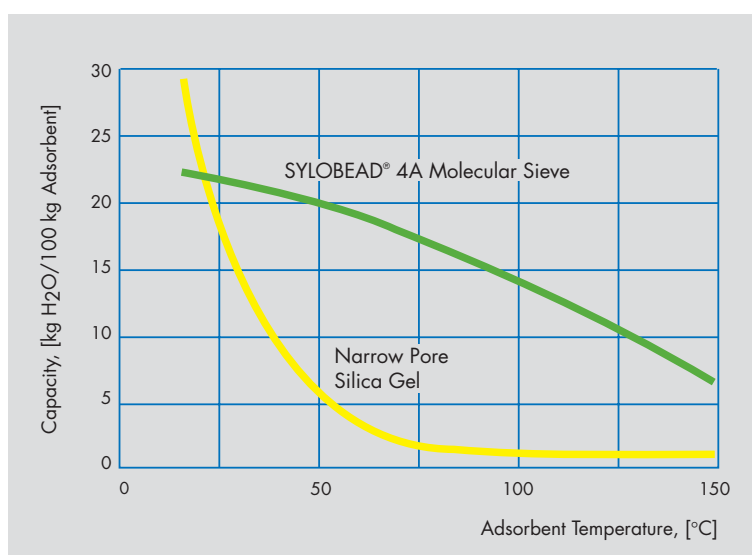


Figure 2 Isobars for Water Adsorption Capacities of GRACE Davison Adsorbents (10 hPa)

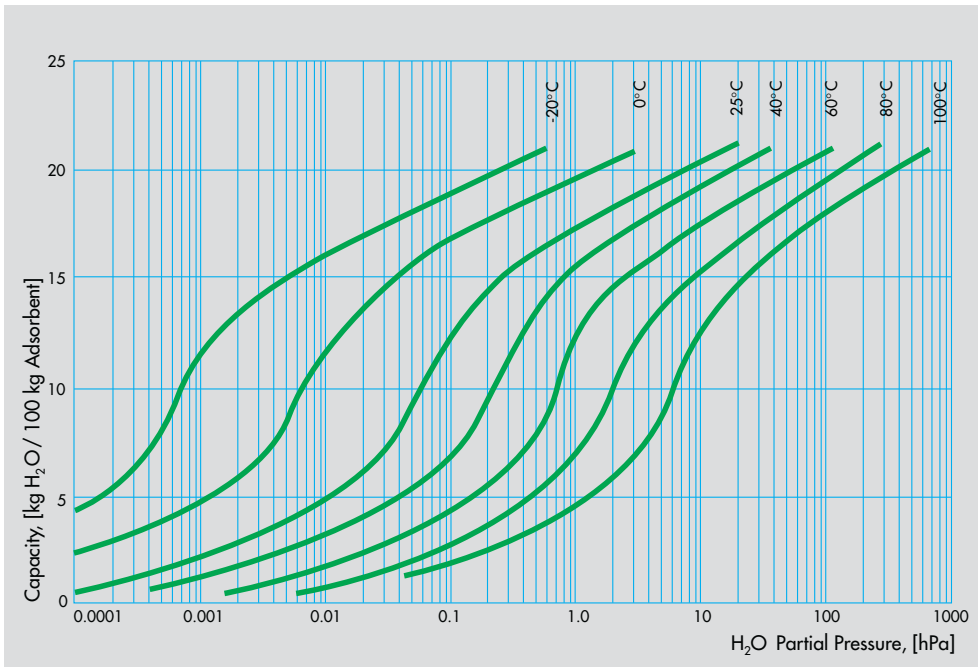


Figure 3: Isotherms for Water Adsorption Capacity of SYLOBEAD® 3A Molecular Sieve

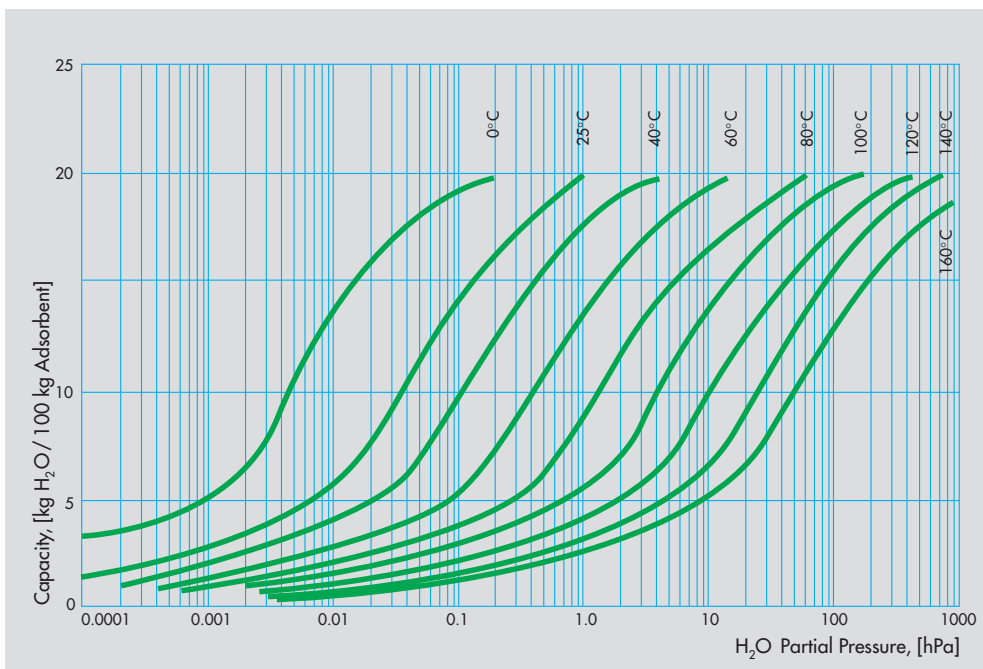


Figure 4: Isotherms for Water Adsorption Capacity of SYLOBEAD® 4A Molecular Sieve

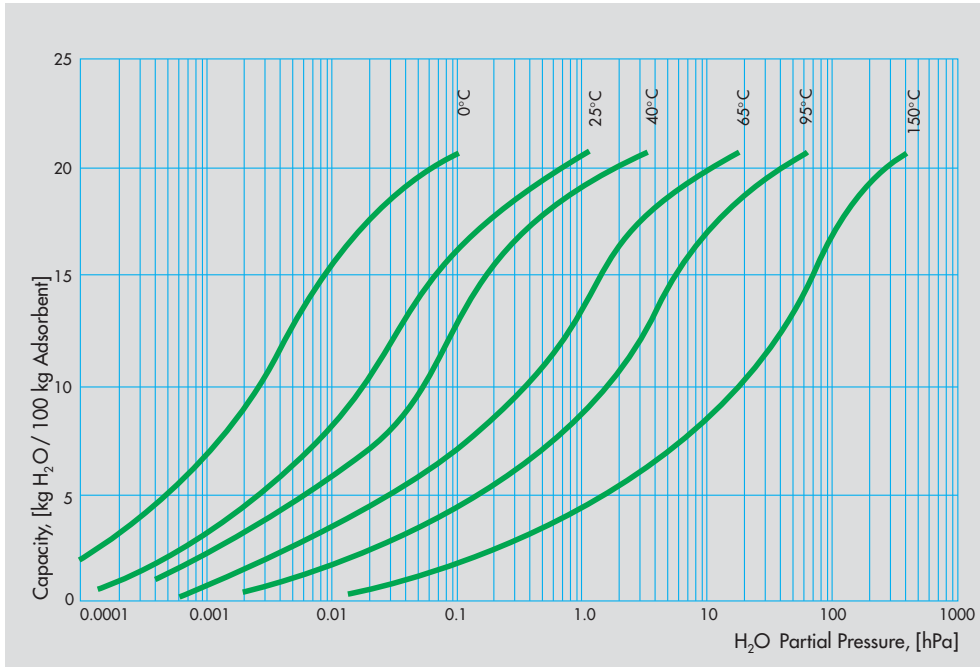


Figure 5: Isotherms for Water Adsorption Capacity of SYLOBEAD® 13X Molecular Sieve

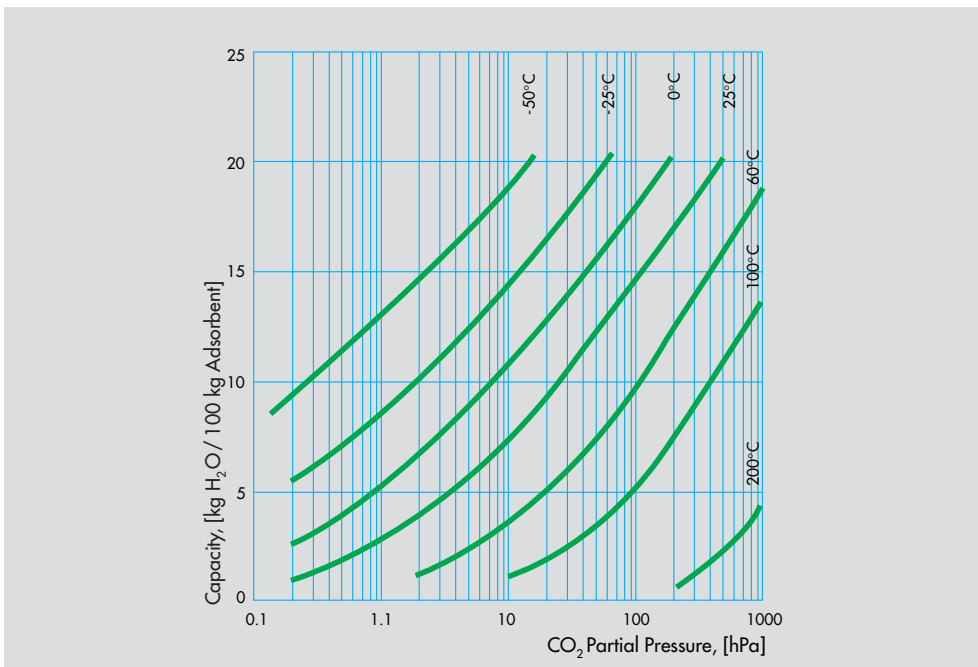


Figure 6: Isotherms for Carbon Dioxide Adsorption Capacity of SYLOBEAD® 13X Molecular Sieve

Industrial Applications. Using SYLOBEAD® Molecular Sieves and Silica Gels

GRACE Davison molecular sieves are successfully and universally used in many commercial drying and purification systems for gases and liquids. These applications can be broadly divided into the following sectors: natural gas, refining, petrochemical and industrial gas.

Additionally, GRACE Davison supplies narrow pore silica gel for natural gas and air drying, as well as for hydrocarbon dew point control. Wide pore buffer gel is commonly used to protect adsorbent beds against liquid carryover.

The following pages give a brief overview of the most common applications for molecular sieves or silica gels. For more specific information, a list of GRACE Davison references is available on request.

SYLOBEAD® for the Natural Gas Industry

The natural gas industry has strict specifications and limits on impurities in hydrocarbon gas and condensates. Water content has to be reduced to levels that prevent corrosion, hydrate formation, and freezing in cryogenic equipment.

When sulphur compounds and carbon dioxide levels are too high, they need to be removed to meet product specifications or avoid formation of solid CO₂. Special molecular sieves are available, which remove water and sulphur components while avoiding the formation of COS.

GRACE Davison adsorbents are used extensively to fulfil these requirements. Examples are:



Natural Gas
Drying Unit

SYLOBEAD®

- Natural gas drying prior to cryogenic processing with SYLOBEAD® molecular sieve
- Natural gas sweetening for removal of H₂S and mercaptans to meet pipeline specifications with molecular sieve
- Hydrocarbon dew point control and recovery. GRACE Davison supplies hydrocarbon grade silica gel for NGL extraction where hydrocarbon dew point specifications need to be met.

SYLOBEAD® for the Refining Industry

Molecular sieves are regularly used for the drying and purification of feedstocks for refinery applications such as isomerisation, alkylation and reforming, thereby improving operating performance and product quality. These processes often use catalysts, sensitive to impurities in the feed, which need to be protected by a feed purification step using SYLOBEAD® molecular sieve. In

addition, the removal of other impurities with molecular sieve can improve the quality of the final product.

Examples for the variety of uses in this area:

- Hydrocarbon gas drying with molecular sieves, prior to cryogenic extraction of LPG, to avoid ice and hydrate formation
- Alkylation and isomerisation feed drying: Removal of water for catalyst protection and corrosion prevention using molecular sieve
- Non-regenerative removal of chloride by molecular sieves to prevent contamination from isomerisation catalysts to avoid corrosion downstream the catalyst unit.
- Drying and purification of feed streams for MTBE and Butamer production
- LPG sweetening and butane destenching



Refinery

SYLOBEAD® for the Petrochemical Industry

The petrochemical industry uses molecular sieves for the purification of feedstocks and the drying and removal of impurities from products such as ethylene and propylene. This is necessary to both protect the catalyst in the downstream conversion unit and to avoid freezing of gases during a cryogenic process. Here are some of the most common applications where molecular sieves are used in the petrochemical industry:

- Cracked Gas drying: In order to prevent freezing, GRACE Davison has developed a special SYLOBEAD® 3A molecular sieve, which dries cracked gas without co-adsorption of olefins. Grace molecular sieve can also be used to dry hydrogen gas for acetylene converters.
- Drying and removal of CO₂ and oxygenates of ethylene and propylene

SYLOBEAD® for Industrial Gas and Pre-Purification

Where product quality needs to be improved, molecular sieves and silica gel can preferentially adsorb water vapour and impurities from industrial gases – for example, upstream of cryogenic separation processes, thus preventing freezing and hydrate formation. By removing various impurities, molecular sieves can also improve the quality of an industrial gas like synthesis gas.

Further applications in this industry are:

- The generation of oxygen from air by Pressure Swing Adsorption (PSA) or Vacuum Swing Adsorption (VSA) processes
- The removal of water and carbon dioxide from air, prior to cryogenic separation of nitrogen and oxygen
- PSA hydrogen gas upgrading. Refineries frequently require higher purity hydrogen for various hydrogenation processes. Molecular sieve is used on its own or with other adsorbents to upgrade refinery hydrogen to around 99% purity. Impurities commonly found in the feed include water, CO₂, CO, N₂, methane, and ethane.

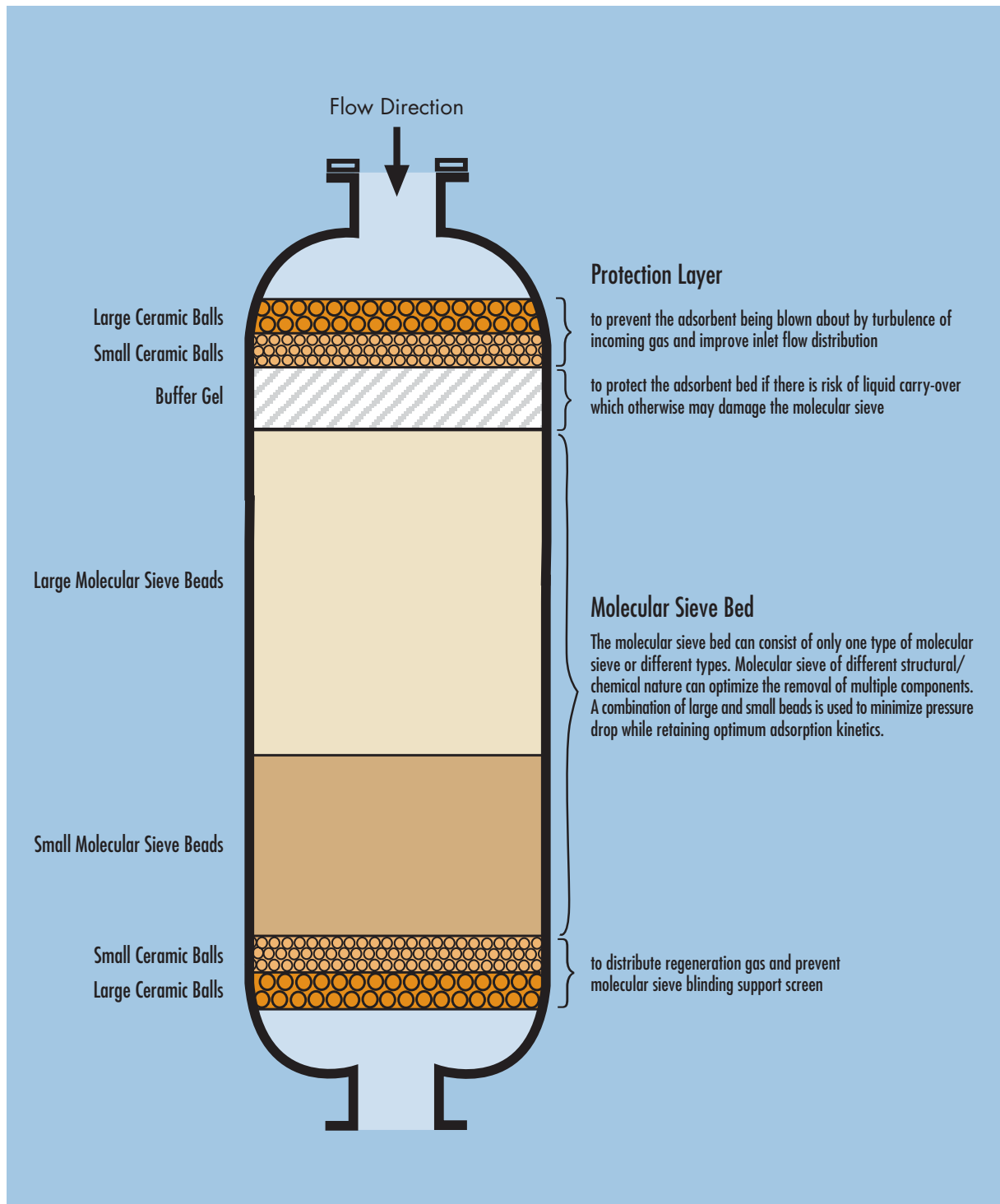
Further applications

Due to their unique adsorption properties, molecular sieves and silica gels can also be used in a variety of other drying and separation processes, for example:

- Liquid phase and gas phase ethanol dehydration: For fuel and beverage alcohol
- Separation of normal and iso-paraffins: For detergent and fuel production
- Air drying: For brake air, conveying air, compressed air, sterile environments and protection of sensitive equipment

For further technical information, please refer to our industry-related "Application Guides"

Look Inside.
A Typical Cross-Section of an Adsorber Bed



SYLOBEAD® Adsorbents. The Right Products for You



GRACE Davison offers a wide range of products to meet your specific purification/separation requirements. These include: SYLOBEAD® 3A molecular sieves, with improved aging resistance: These types are available in particle sizes of 1.6 – 2.5 mm (8 x 12 mesh) and 2.5 – 5 mm (4 x 8 mesh). GRACE also offers a special grade 3A molecular sieve with reduced catalytic activity and enhanced ethane/methane compatibility. SYLOBEAD® 4A and SYLOBEAD® 5A molecular sieves are available in bead sizes

of 1.6 – 2.5 mm (8 x 12 mesh) and 2.5 – 5 mm (4 x 8 mesh). The 4A grades are also available in bead sizes of 0,5 – 1 mm (20 x 36 mesh) and 1 – 2 mm (12 x 20 mesh). GRACE offers special 5A grade molecular sieves, ensuring minimum COS formation while retaining the high performance of the normal 5A grades.

SYLOBEAD® zeolite X, also known as 13X or 10A molecular sieve, is available in bead sizes of 1 – 2 mm (12 x 20 mesh), 1.6 – 2.5 mm

SYLOBEAD®

(8 x 12 mesh) and 2.5 – 5 mm (4 x 8 mesh). GRACE has both regular and high performance grades.

SYLOBEAD® Bead Gel is a spherical silica gel. It is available as a wide pore type for use as buffer gel, and a narrow pore type for the adsorption of water and hydrocarbons. Bead sizes range from 2 – 5 mm (4 – 12 mesh).

Granular Silica Gel from GRACE Davison is also available as wide and narrow pore types for use as buffer gel and for the adsorption of water and hydrocarbons, respectively. It can be obtained in various particle sizes.

For a better gas/fluid distribution and stabilisation of the adsorbent bed, GRACE Davison offers high purity Ceramic Balls in sizes between 1/8" and 1" .

Please contact our Technical Sales/Service support for advice in selecting the most suitable material for your application. Special Product Information Sheets for each grade are available on request.

GRACE Davison zeolites and silica gels are considered to be non-toxic. For detailed information, please refer to the appropriate Material Safety Data Sheet, available separately.

Properly Packaged

After production, our molecular sieves and silica gels are highly active with a high affinity for water. The proper packaging of the adsorbents is extremely important to maintain maximum performance.

GRACE Davison molecular sieves and silica gels are supplied in hermetically sealed steel drums, or in big bags with an inner lining. Whenever the logistics allow, GRACE Davison can deliver molecular sieves in silo trucks for rapid pneumatic loading of adsorption vessels.

See also "Storage Instructions for Grace Molecular Sieves", „Transport Instructions for Grace Molecular Sieves" and our Product Information Sheets.

Vessel Loading with GRACE Davison big bags



Delivery by Silotruck



We Make the Difference. Our Comprehensive Customer Service

GRACE Davison not only supplies superior adsorbents, but also provides comprehensive customer service to help you make the most of our products. Our highly trained technical support network is at your disposal. Worldwide. GRACE Davison is committed to a complete

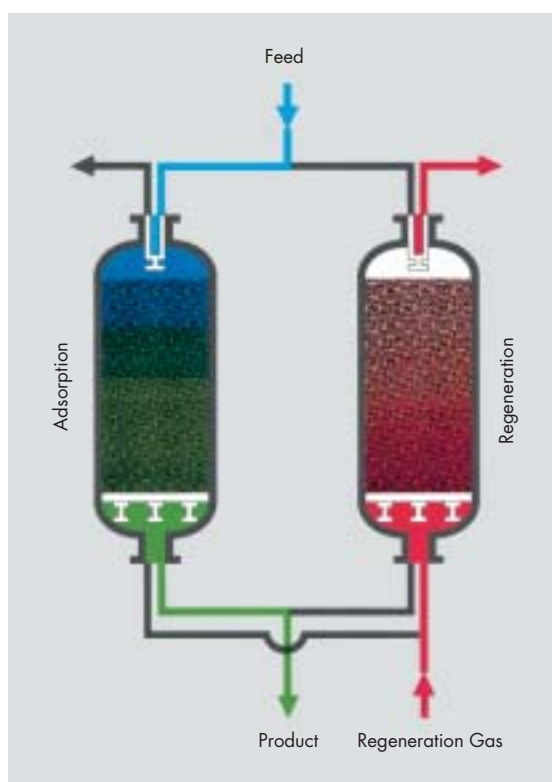
adsorbent lifecycle approach, from the conception of the unit to the replacement of the adsorbent at the end of its lifecycle. Each stage – conception of the unit, loading of the adsorbent, start-up and in-process control, unloading of the adsorbent – requires specialist know-how.

The following table gives an overview of the GRACE DAVISON adsorbent lifecycle services:

Stage	Service provided by GRACE Davison
Design and Engineering	Sizing and configuration of the adsorber unit Advice on flow schemes Advice on vessel configuration and sample points
Loading	Loading supervision Pneumatic loading service
Start-Up	Start-Up supervision Training for staff in the operation of adsorbers units
Operation	Adsorption performance measurements Pressure drop measurements Performance evaluation Optimisation of adsorber performance Operator training Adsorbent lifetime prediction Used adsorbent analysis
End of Life	Unloading advice

Experience in Performance. Design of the Adsorption Unit

GRACE Davison has a long history in designing adsorbers for the process industries. To achieve optimal results, GRACE draws on expertise gained from in-house design concepts, actual plant experience and concepts adapted from research institutes and universities.



Typical Arrangement of a Two Bed Adsorption System

The design of an adsorber is complex. It requires in-depth knowledge of adsorption theory, product characteristics and know-how regarding the

interaction of feed components and process conditions. An optimal design can maximise the value of your adsorption unit in terms of process efficiency and the overall unit lifecycle.

The following gives an outline of the main considerations to be made when designing an adsorption unit. The process design procedure is naturally more complex, but this should provide you with a general understanding for the criteria which determine the way we design adsorbers. For more specific advice, please contact your local GRACE Davison office.

Selecting the appropriate GRACE Davison Adsorbent

The first step towards the design of the adsorber is the selection of the types of adsorbent. The chosen SYLOBEAD® molecular sieve type needs to have a pore opening big enough to let in the molecules to be adsorbed, whilst at the same time excluding undesired molecules which might react during regeneration. For this reason, SYLOBEAD® 3A molecular sieve is usually chosen for drying olefin mixtures.

One must also take co-adsorption into account: When removing CO₂ from natural gas, a SYLOBEAD® 5A sieve is the usual choice. However, a SYLOBEAD® 4A sieve can be used if conditions indicate that hydrocarbon adsorption may inhibit CO₂ adsorption.

Calculating the Molecular Sieve Mass

Once the type of adsorbent has been selected, it is necessary to calculate the mass of adsorbent needed to remove the required amount of adsorbate according to customer specifications. This calculation is based on the adsorption capacity under given process conditions.

On page 9, we discussed factors which affect the equilibrium capacity of GRACE Davison molecular sieves. This capacity is NOT the capacity when operating the unit; while this capacity can be achieved in the inlet part of the adsorber bed, other sections will only be partially saturated or not saturated at all.

In an operating unit, the adsorbent can be regarded in three sections – see figure below:

- **Equilibrium Zone**

- The equilibrium zone is at the inlet end of the bed.
- The equilibrium zone can achieve the equilibrium capacity shown on isotherms, but discounted for any co-adsorption effects

and allowing for a drop in performance as the molecular sieve ages.

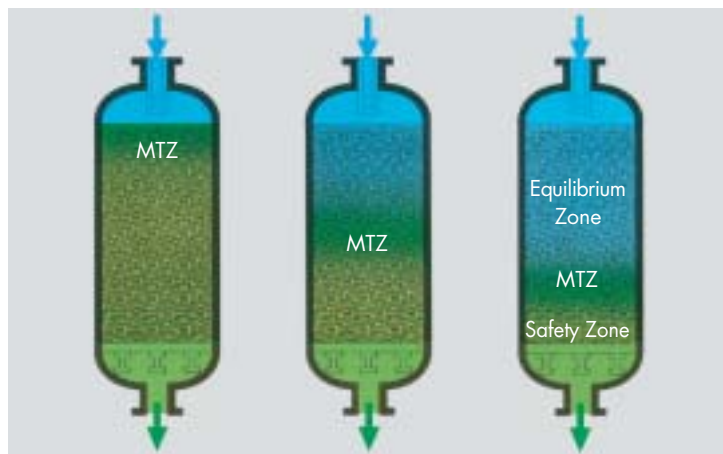
- The size of the equilibrium zone is directly related to the adsorption time.

- **Mass Transfer Zone**

As its name implies, the Mass Transfer Zone, (MTZ) is the part of the bed where the adsorption currently takes place. This is where the concentration of the adsorbate in the fluid is reduced from the inlet to the outlet level. The length of the mass transfer zone depends, in part, on the components to be adsorbed and the composition of the carrier stream. It is also dependent upon:

- Fluid velocity through the bed
- Inlet concentration
- Outlet concentration requirement
- Temperature
- Molecular sieve type
- Molecular sieve bead size

Mass transfer zones can range in length from a few centimetres to several metres.



Progress of Mass Transfer Zone (MTZ) through the Bed During the Adsorption Period (Beginning, middle and end of cycle)

- **Safety Zone**

Normally a system is designed with a detector to indicate when the mass transfer zone is likely to break through the end of the bed. A safety zone is installed to prevent actual breakthrough before the system has been able to change to the regenerated bed. Here, there is normally no adsorption.

The adsorbent mass required in each of these zones must be calculated to achieve the right unit design.

This calculation takes into consideration the special unit requirements of each customer: for example, the unit can be conceived with long cycle times giving long lifetimes at the expense of high capital costs. Or with short cycle times, which result in lower capital costs at the expense of a shorter lifetime.

Defining Bed Dimensions

Once the type and mass of adsorbent have been identified, the dimension of the adsorber bed can be calculated. We can help the customer to find an optimal mix between investment, operating costs and performance.

The following interrelationships should be considered:

- A narrow vessel is not as costly as a wide vessel. It has thinner walls, which implies a lower regeneration heat load.
- Narrower vessels result in higher velocities and thus higher pressure drops. These higher pressure drops may waste expensive compressor power. Additionally, if velocities are too high, movement of the bed may occur, which could damage the molecular sieve.

- A vessel which is too wide may demonstrate a poor mass transfer because the flowrate is too low to allow for good gas distribution.

Evaluating Regeneration Conditions

The next step is the evaluation of the regeneration conditions. Two main methods of regeneration are used commercially:

- **Heating (Thermal Swing Regeneration)**

This is the most common method, particularly where high purity products are required. Calculation methods to simulate thermal regeneration are very complex. In principle, the right amount of hot regeneration gas must be passed through the bed in order to remove the adsorbed material. The bed must then be cooled down close to adsorption temperature. Typical regeneration temperatures are 200 °C to 300 °C.

- **Pressure Swing Regeneration**

This is used for applications where moderate product purity is required, or where the molecular sieve shows a relatively weak attraction for the adsorbate. It is based on the principle that molecular sieve capacities are lower at lower pressures. The system is 'swung' from the high operating (adsorption) pressure to a low pressure and the adsorbed material is thus desorbed into a small flow of purge gas.

Our Technical Service Engineers will provide you with the optimal design for your unit, based on your feed gas, process conditions and costing requirements. Please ask for our "Data Inquiry Sheet" at your local GRACE Davison office.

The SYLOBEAD® Advantage. Pneumatic Loading

Typically, molecular sieve is loaded into vessels in big bags or drums. To improve the loading process, GRACE Davison offers pneumatic loading via silotruck, wherever logistics allow*. This greatly accelerates the loading process, reducing costly plant downtime. Where possible, pneumatic loading can be used to refill vessels without the need for shutdown. The pneumatic loading of adsorber vessels was implemented by GRACE Davison in the mid 1970s and, since then, thousands of tons of GRACE Davison molecular sieve have been loaded this way. The costs for manpower, equipment rental (such as cranes) and for packaging disposal can thereby be reduced or eliminated.

GRACE Davison produces highly attrition resistant molecular sieves in bead form. Only molecular

sieve beads have the mechanical properties and strength to withstand the forces employed in pneumatic loading. Molecular sieve cylindrical extrudates tend to break either when blasted through the pressurised hose, or during the freefall into the vessel when coming into contact with each other and the vessel walls. They are unable to withstand the loading pressures and flowrates, which present no problem to SYLOBEAD® molecular sieve beads.

The entire loading process is planned and supervised by highly trained, experienced GRACE Davison personnel, to ensure a safe, effective and environmentally friendly loading.

** For availability and further information on pneumatic loading, please contact your local Grace Davison Service Centre.*

Pneumatic
Loading



Our Service. Throughout the Lifecycle

GRACE Davison has a range of services available to ensure a smooth and cost-effective operation of the adsorption unit throughout its lifetime: Advice and support during start-up, on site measurements, process optimisation, troubleshooting and characterisation of used adsorbents. Some of these services are described in more detail below:



On-Site Breakthrough Measurement
by a GRACE Davison Engineer

On-Site Measurements

Adsorber performance can be checked during normal plant operation. Based on the results of such measurements, it is possible to optimise the operation of the adsorber, thus improving efficiency.

Examples of on-site measurements are:

- For drying units: Cycle optimisation, by determination of the breakthrough time of the adsorber, which is a measure of the overall water adsorption capacity and an indication of the ageing behavior of the molecular sieve
- For pre-purification units: Purification efficiency, by measuring the concentrations of impurities (CO₂, H₂S, etc.) in the raw and product gases
- Pressure drop measurements across the molecular sieve bed

Other special measurements can be carried out on request.

Analysis of Used Adsorbents

Used, but not necessarily exhausted adsorbent samples can be examined in the Technical Service laboratories. We can then advise our customers as to whether or when the bed needs to be changed out.

Adsorption Isotherm Measurement with McBain Microbalance



The examination includes

- the analysis of the mechanical integrity of the molecular sieve beads as well as
- the determination of the remaining capacity of the molecular sieve.

These analysis can also provide valuable information for the optimisation of the process conditions. Special measurements can also be conducted.

Troubleshooting

GRACE Davison Technical Service is committed to giving the maximum support to our customers to ensure satisfaction with our products. We help to investigate and identify causes for process problems and can conduct accompanying measurements. The resulting recommendation enables you to get back to effective business.



Sample Analysis at GRACE Davison using Scanning Electron Microscope

Unloading Adsorbents

At some time, the adsorbents will need to be replaced. This means that the vessel must be unloaded, and the disposal of the spent adsorbents has to be done at minimum cost and within strict environmental protection guidelines. GRACE Davison Technical service can advise you on unloading procedures and the disposal of the spent adsorbents.

More details are available in the Technical Information "Spherical Molecular Sieves for Process Applications – Unloading of Adsorbents" and "Handling of Spent Molecular Sieve"

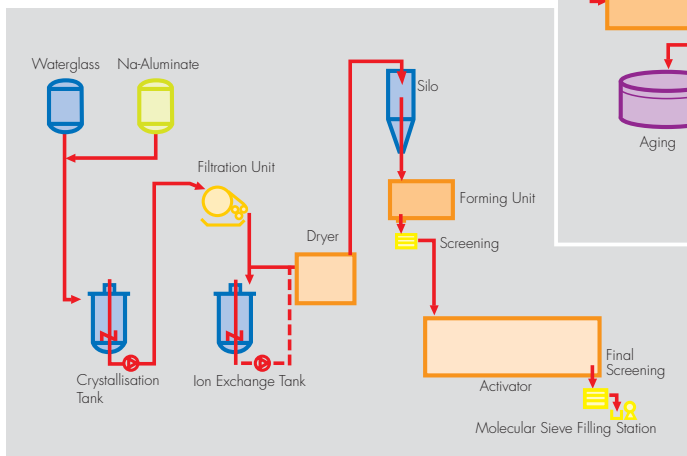
A Promise of Quality. GRACE Davison's Manufacturing

GRACE Davison manufactures adsorbents using state-of-the-art processes and technologies. For meeting highest customer's demands.

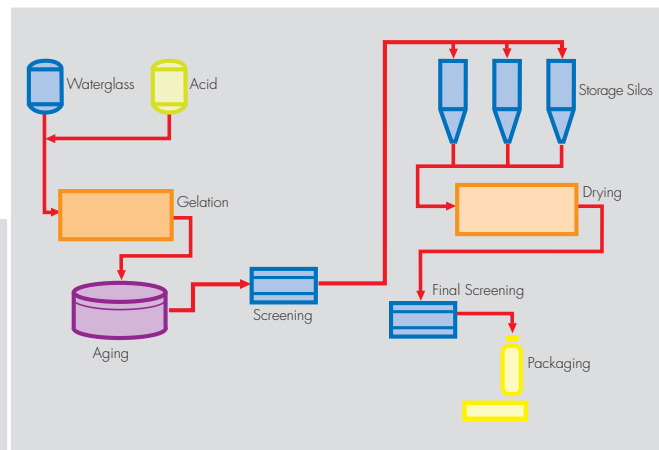
Molecular sieves are manufactured by crystallisation from aluminium hydroxide, sodium hydroxide and waterglass. Under carefully controlled conditions, the crystallisation process produces the required sodium aluminosilicate structure. The zeolite crystals can then be ion exchanged with either potassium or calcium. After drying the molecular sieve crystals, a small quantity of binder is added, forming the material into beads. These beads are dried, calcined and finally screened to the required particle size prior to packaging.

Silica gels are manufactured by reacting waterglass with sulphuric acid. The gel is then washed and aged. The precise control of the reaction conditions determines the pore structure of the resulting gel. To produce granular gel, the initial large gel granules are ground and screened into particles of the required size. The gel is then activated prior to packaging.

Beaded gel is produced by special equipment, resulting in spherical particles during the gelation process. The beads are then washed, activated and screened prior to packaging.



Molecular Sieve Production



Silica Gel Production

The Driving Forces. Research & Development, Quality Management and Six Sigma

Research & Development

GRACE Davison is a firm believer in innovation. Our researchers are continuously working to improve the quality of existing products and seeking ways to broaden our product range to fulfil the increasing requirements of our customers. With R&D centres in Columbia, USA, and Worms, Germany, GRACE can look back on more than 80 years of expertise in the development of adsorbents for the processing industries.

In our facilities we can conduct laboratory and pilot plant zeolite synthesis and forming, silica gelation, chemical/physical characterisation with state-of-the-art equipment, application testing such as breakthrough measurements, accelerated aging tests or adsorption isotherm measurements.

GRACE Davison Quality Management

Our Total Quality Management (TQM) Program is geared towards customer satisfaction and based upon GRACE Davison's philosophy of continuous improvement in every area of our organisation.

- All our sites are ISO 9001 or ISO 9002 certified and we implement internal and external audits to find ways to improve our services and processes.
- We employ the latest Statistical Process Control (SPC) to monitor and analyse production and related work processes.
- Our well-equipped Quality Control department works around the clock to ensure constant product quality.

- We continuously collect and assess customer information and feedback as an important factor within our TQM Program.

Our dedicated work force is our most important capital. Committed to full customer satisfaction.



Research & Development Test Facility for Breakthrough testing

The Six Sigma Advantage

We at GRACE Davison are not only committed to Total Quality Management but also to continuously improving our processes. To maintain GRACE Davison's high standards, we employ GRACE's Six Sigma tools. These were designed to investigate process parameters, quantify their effects and optimise these in order to achieve the best possible results. Our Six Sigma initiative aims at improving product consistency, production flexibility and capacity by using advanced statistical methods and evaluation procedures. Our customers benefit from products of the highest quality. Quality you can count on.

A world of difference from GRACE

Grace Davison's comprehensive sales and distribution network, combined with European, North/Latin American and Asian production facilities, means prompt delivery of our products around the world.

Our highly trained Sales Force has the experience to help you immediately and our worldwide Technical Customer Service Groups are staffed with experienced professionals who are ready to offer you innovative assistance. Our Sales Force and our Technical Customer Service Groups are supported by Grace research laboratories worldwide.

Grace Davison is a leading global silica and adsorbent materials company with technology expertise in separations, digital printing, coatings, CMP and electronic applications, pharmaceutical and personal care, food and beverage, plastics, packaging, precision investment casting, rubber, window and industrial process applications.

Your local contact

The information contained herein is based on our testing and experience and is offered for the user's consideration, investigation and verification. Since operating and use conditions vary and since we do not control such conditions, we must DISCLAIM ANY WARRANTY, EXPRESSED OR IMPLIED, with regard to results to be obtained from the use of this product. Test methods are available on request.

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