

D. DE MEIS

Dipartimento Fusione e Tecnologie per la Sicurezza Nucleare
Divisione Fisica della Fusione
Laboratorio Sorgenti, Antenne e Diagnostica
Centro Ricerche Frascati, Roma

OVERVIEW ON POROUS INORGANIC MEMBRANES FOR GAS SEPARATION

RT/2017/5/ENEA



ITALIAN NATIONAL AGENCY FOR NEW TECHNOLOGIES,
ENERGY AND SUSTAINABLE ECONOMIC DEVELOPMENT

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OVERVIEW ON POROUS INORGANIC MEMBRANES FOR GAS SEPARATION

D. De Meis

Riassunto

Negli ultimi 20 anni, la tecnologia di separazione dei gas con le membrane è notevolmente avanzata e può essere considerata come un metodo industriale competitivo. È stato dimostrato essere una tecnologia tecnicamente ed economicamente superiore alle altre. Questa superiorità è dovuta ad alcuni vantaggi che le membrane hanno rispetto alle altre, tra cui il basso investimento di capitale, il peso ridotto, il basso impatto ambientale, i requisiti di spazio e l'alta flessibilità di processo.

La tecnologia di separazione dei gas con le membrane si è sviluppata fino a raggiungere i 150 milioni di dollari/l'anno di fatturato.

La prima applicazione industriale moderna di membrane porose inorganiche è stata la separazione degli isotopi dell'Uranio U_{238} e U_{235} in forma di UF_6 negli anni 1940 e 1950.

Oltre ad avere ben note proprietà di stabilità termica e chimica, le membrane porose inorganiche di solito hanno flussi di gas molto più alti rispetto a quelle polimeriche.

Il potenziale delle membrane inorganiche non era stato ancora ampiamente riconosciuto fino a che non furono prodotte membrane ceramiche porose di alta qualità per le applicazioni industriali.

L'attenzione dei ricercatori si è concentrata su materiali che hanno proprietà di setaccio molecolare, come silice, zeoliti e carbonio che sembrano essere promettenti nella separazione dei gas.

Questo documento è una panoramica preliminare sulle membrane porose inorganiche per la separazione del gas.

Parole chiave: separazione gassosa, membrane inorganiche, membrane ceramiche, membrane porose

Abstract

In the past 20 years, membrane gas separation technology has advanced greatly and can now be regarded as a competitive industrial method for gas separation. It has been proven to be technically and economically superior to other technologies. This superiority is due to certain advantages which membrane technology benefits from, including low capital investment, low weight, environmentally friendly, space requirement and high process flexibility.

Membrane gas separation technology has developed into a \$150million/year business.

The first modern industrial application of inorganic porous membranes was separation of U_{238} and U_{235} isotopes in form of UF_6 in the 1940 and 1950.

Besides having well known thermal and chemical stabilities, inorganic porous membranes usually have much higher gas fluxes as compared to polymeric membranes.

The potential of inorganic membranes was not widely recognized until high quality porous ceramic membranes were produced for industrial applications.

The attention of the researchers has focused on materials that exhibit molecular sieving properties, such as silica, zeolites and carbon which appear to be promising in separation of gas.

This paper is a preliminary overview on inorganic porous membranes for gas separation.

Keywords: gas separation, inorganic membranes, ceramic membranes, porous membranes

INDEX

1. Introduction	7
2. Definition of membrane	8
3. Inorganic membranes	10
3.1 Commercialized inorganic membranes	13
4. Gas membrane application	14
5. Ceramic membranes	15
6. Mechanisms for gas permeation across porous membranes	19
Acknowledgments	21
References	21

1. Introduction

Due to high energy prices, energy efficiency and energy saving have become important components of government policy. As energy costs rise, the development of membranes is considered an important task in the introduction of new energy saving. Membrane technology is therefore currently one of the most innovative and rapidly growing fields.

In the last 20 years, membrane gas separation technology has developed into a \$150million/year business (Baker, 2002). Membrane separation processes are attractive as they are considered environmentally friendly, simple, versatile and have a low energy consumption. Conventional technologies such as cryogenic distillation, adsorption and absorption require a gas-to-liquid phase change in the gas mixture that has to be separated. This phase change adds significant energy costs to the separation cost (Pengilley 2015). An overall comparison of the four relevant gas separation technologies is given in Table 1.

Gas Separation Technologies		
Technology	Advantages	Disadvantages
Adsorption	High purity	Lower recovery Low efficiency Requires adsorbent regeneration
Absorption	High recovery	Complex process Many stages of operation
Cryogenics	High purity	Complex operations Energy intensive
Membrane	Good for bulk separation Low capital cost Simple process Energy efficient Low maintenance	Membrane design is complex Moderate product purity

Tab. 1 Gas separation technologies (Pengilley 2015)

2. Definition of membrane

A membrane can be described as a selective barrier between two phases that has the ability to separate gases or liquids with the aid of a driving force. This driving force could result from differences in total pressure, partial pressure, concentration or electrical potential.

The overall principle in any membrane separation is sketched in figure 1.

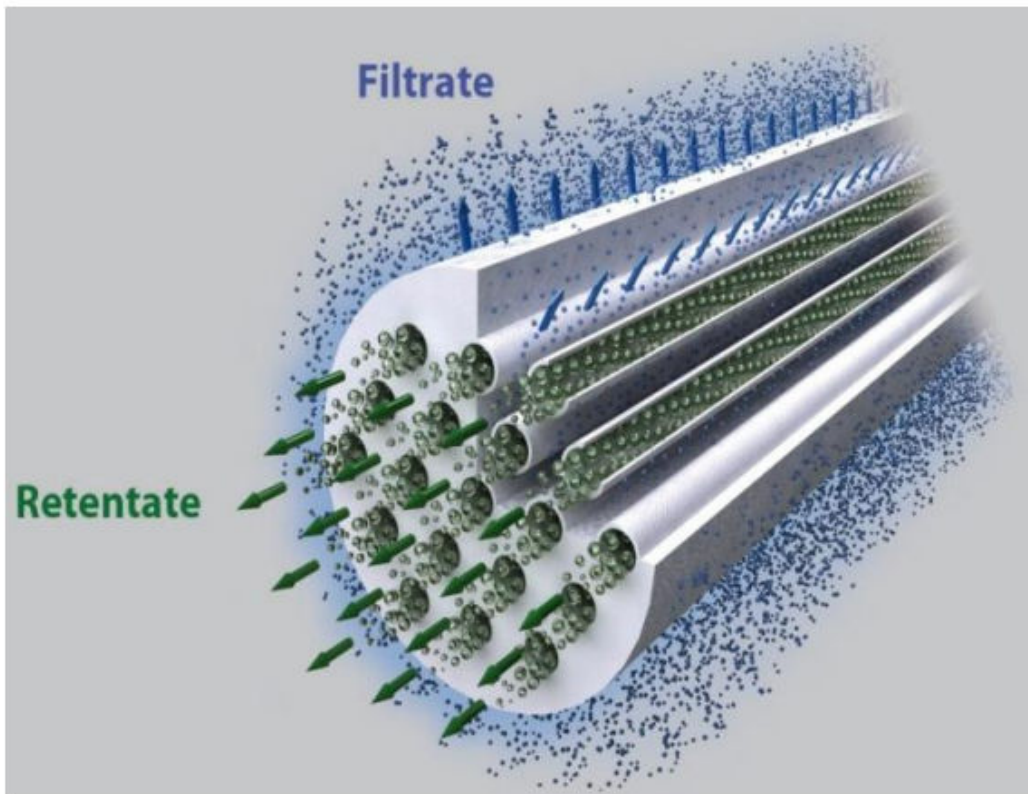


Fig. 1 The principle in membrane separation (Manohar 2012)

Membranes can also be classified by their morphology or structure, since membrane structure determines the separation mechanism, and hence the application (Mulder, 1996).

A simple sketch of gas separation by membranes is provided in fig. 2 (Sridhar 2014) and fig. 3 (Zhang 2011).

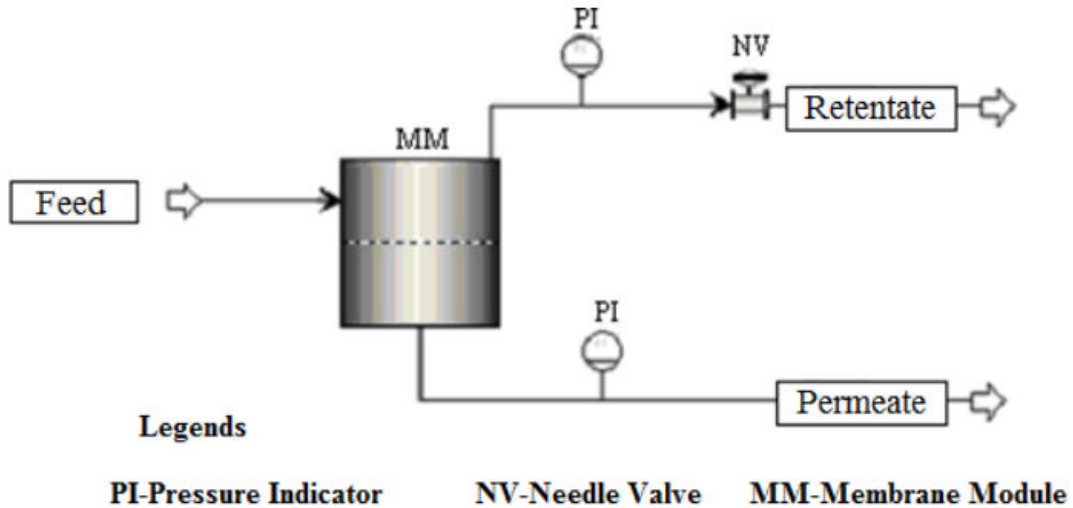


Fig. 2 Schematic of Membrane separation process

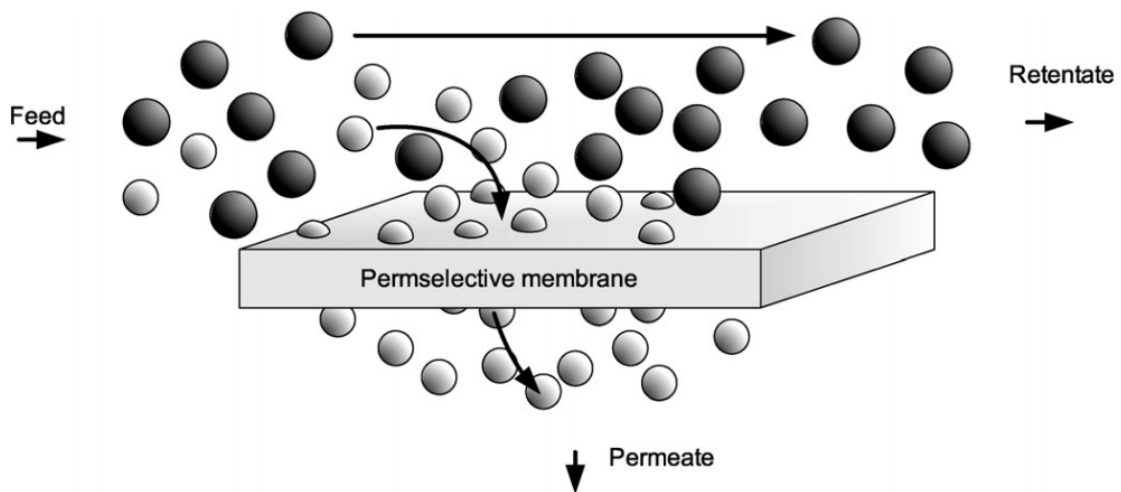


Fig. 3 Schematic of Membrane separation

As a first classification membranes can be divided into biological and artificial membranes. Biological membranes are beyond the scope of this work. Synthetic membranes can be modified natural materials or synthetic (man-made). Synthetic membranes can be further divided into organic and inorganic membranes. Generally polymeric membranes are fabricated from organic polymers. Polymers commonly used are cellulose acetate, fluorocarbon polymers and aromatic polyamides. Unfortunately the efficiency of polymeric membranes decreases with time due fouling, compaction, chemical degradation and thermal instability (Pingelley 2015).

3. Inorganic membranes

Inorganic membranes consist of oxides, or metal, and can be present in multi-layer supporting structures, or as a self-supporting structures (Ismail 2001). Furthermore, the layers can be dense (defect-free) or porous thus involving different mass transfer mechanisms of the gas through the membrane.

The development of porous inorganic membranes dates from before 1945, long before the development of synthetic organic membranes. Not much publicity was given to the early development of inorganic membranes because the first porous inorganic membranes were developed in the Manhattan Project to enrich uranium by separating uranium isotopes in form of gas molecules UF₆ (Keizer 1996, Pandey 2001). This was the first large-scale membrane gas separation process.

Also in the 1940s Corning Glass developed a homogeneous porous glass (Vycor) with 20-40 Å pores.

Before gaining importance in gas separation, the inorganic membranes were developed in the 1960-1980 period for the treatment of liquids and, especially, for water purification. In particular, these membranes have become important apparatus for beverage production, water purification and the separation of dairy products (Ismail 2001, Keizer 1996).

Non-nuclear applications of inorganic membranes for gas separation started at the beginning of the 1980s with Membralox produced by Ceraver (now SCT), Carbosep produced by SFEC (now TECHSEP) and Ceraflo produced by Norton (now by SCT) (Soria 1995). Some useful information on materials, pore diameter and geometry are reported in tab. 2 of paragraph 3.1.

Inorganic membranes are increasingly being explored to separate gas mixtures. Besides having well known thermal and chemical stabilities, inorganic membranes usually have much higher gas fluxes as compared to polymeric membranes (Pandey 2001).

The potential of inorganic membranes was not widely recognized until high quality porous ceramic membranes were produced for industrial usage on a large scale (Hsieh 1990).

Nowadays, inorganic membranes are used primarily for civilian energy-related applications such as (Abedini 2010, Venkataraman 1992):

- the removal of Hydrogen, sulfide, sulfur dioxide, carbon dioxide, and nitrogen existing in oil reservoir before transportation to the pipeline to avoid gas flaring;
- contaminant removal to protect process components and to control emissions from advanced coal-based power systems;

Numerous European, American and Japanese companies are now competing to produce inorganic membranes (Soria 1995) for the gas separation processes of industrial sector.

Hsieh has provided a technical overview of inorganic membranes in his paper (Hsieh 1990). He divided the inorganic membranes into two major categories based on its structure: porous inorganic membranes and dense (non-porous) inorganic membranes as shown in Fig. 4.

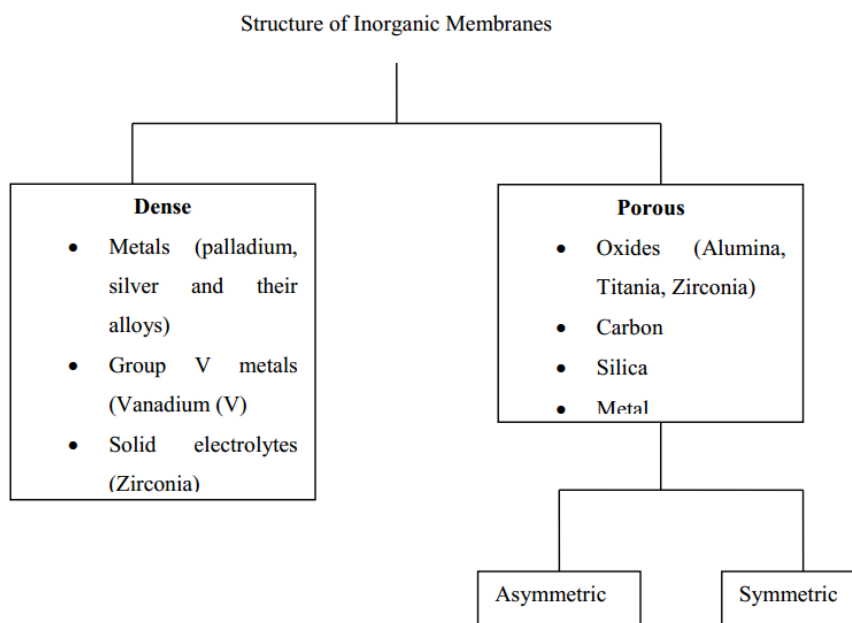


Fig. 4 Structures of inorganic membranes (Ismail 2001)

Porous inorganic membranes with pores more than 0.3 nm usually work as sieves for large molecules and particles.

Glass, metal, alumina, zirconia, zeolite and carbon membranes are commercially used as porous inorganic membranes. Other inorganic materials, such as cordierite, silicon carbide, silicon nitride, titania, mullite, tin oxide and mica also have been used to produce porous inorganic membranes. These membranes vary greatly in pore size, support material and configuration.

On the other hand, dense membranes made of palladium and its alloys, silver, nickel and stabilized zirconia have been used or evaluated mostly for separating gaseous components. These membranes are characterized by low permeabilities, but by high selectivities (Pandey 2001). Application of dense membranes is primarily for highly selective separation of hydrogen and oxygen. In these dense membranes only atoms or ions of selected gases can pass through the dense membrane layers.

However, the dense membranes have limited industrial application due to their low permeability and high cost (it is the case of the Pd-alloys) compared to porous inorganic membranes. Therefore, today's commercial inorganic membranes market is dominated by porous membrane (Keizer 1996, Hsieh 1990).

Although inorganic membranes are more expensive than organic polymeric membranes, they possess advantages of: temperature and wear resistance, well-defined stable pore structure, and chemically inertness. Moreover, compared to organic membranes, inorganic membranes can work at higher pressures (up to 10 MPa) and can be cleaned with steam, which is impossible with organic membranes (Zhang 2011). These advantageous characteristics encouraged many researchers in the early 1980s to investigate the gas separation properties of these membranes.

Furthermore, many studies regarding applications of inorganic membrane reactors have been carried out (Keizer 1996).

At present, interest in the development of porous inorganic membranes providing better selectivity, thermal and chemical stability than polymeric membranes has grown. The attention has focused on materials that exhibit molecular sieving properties, such as silica, zeolites and carbon (Fuentes 1998), which appear to be promising in separation of gas. Silica-based inorganic membranes selectively separate hydrogen from other gases but permselectivity between similar-sized molecules, such as oxygen and nitrogen is not sufficient (Ismail 2001). Zeolites exhibit a high selectivity and can separate isomers, however it is difficult to produce large, crack-free zeolite membranes. On the other hand, carbon molecular sieve membranes are easily produced (Ismail 2001).

Membranes can also be classified as symmetric or asymmetric according to their the pore structure. Symmetric membranes, also called “isotropic”, have uniform structure and character. In asymmetric membranes, also called “anisotropic”, chemical and physical features vary along their thickness (Zhang 2011). In fact in most cases, membranes consist of several layers with a different pore size in each layer. Generally, there are three layers: a macroporous support that provides mechanical strength to the system; an intermediate layers bridge the gap between the large pores of the support and the small pores of the top layer. The top layer provides the separating capability. In some cases, the top layer can be a dense layer: these membranes are called composite membranes (Soria 1995).

The membranes vary greatly in pore size, support material and configuration and can further be classified according to the IUPAC classification of (Zhang 2011):

- macroporous with $d > 50$ nm
Used as supporting structures and particulate filtration applications
- mesoporous with $2 < d < 50$ nm
Used as supporting structures or in water purification
- microporous with $0,5 < d < 2$ nm
Highly selective for CO₂, H₂O and hydrocarbon

Where d is the pore diameter.

Microporous membranes are usually prepared as thin films on porous inorganic supports of larger pore size that provide the mechanical strength.

Compared to polymeric membranes, inorganic membranes can be more selective and permeable, and, especially, can withstand more extreme conditions.

Examples of important applications of inorganic membranes are (Verweij 2012):

- Separation of H₂ from coal-derived gas
- Separation of CO₂ from natural gas and coal plant flue gas
- Separation of O₂ from air for efficient combustion, and (petro-) chemical applications.
- Separation of H₂O from chemical reaction mixtures
- Removal of dissolved salts and other contaminants from water

The main process applications are: separation, recovery, enrichment, removal of undesired components, drying, purification, recycling and reuse of specific substances. In particular, inorganic membranes can operate under harsh conditions and be highly permeable. Further, when properly designed these membranes can achieve also high selectivity for specific molecules.

3.1 Commercialized inorganic membranes

Inorganic membranes commercially available today are dominated by porous membranes, and particularly by porous ceramic membranes.

Today, twenty firms are manufacturing inorganic membranes, most of them were introduced to the market over the last five or ten years (Table 2).

The most prevalent membrane materials are metallic oxides and more precisely alumina and zirconia, but other materials, such as titania, carbon or glass, are available (Soria 1995).

Commercialized inorganic membranes exist in three configurations: disks, tubes and multichannels/honeycombs.

The use of disks has been limited to medical and laboratory applications whereas tubes and monoliths are used in various industrial applications.

The multichannel/honeycomb configuration represents a technical evolution from single-tube or tube bundle geometry as it potentially offers high mechanical strength and higher membrane packing density.

Manufacturer	Trade name	Material	Pore ϕ	Geometry
USF/SCT	MEMBRALOX®	ZrO ₂ /Al ₂ O ₃	20–100 nm	Monolith
		Al ₂ O ₃ /Al ₂ O ₃	5 nm–12 μ m	
TECH-SEP	CERAFLO®	Al ₂ O ₃ /Al ₂ O ₃	0.2–1.0 μ m	Monolith
	CARBOSEP®	ZrO ₂ /C	10–300 kD	Tube
		TiO ₂ /C	0.14 μ m	
	KERASEP®	TiO ₂ /Al ₂ O ₃ + TiO ₂	0.1–0.45 μ m	
Le Carbone Lorraine		ZrO ₂ /Al ₂ O ₃ + TiO ₂	15–300 kD	Monolith
CERASIV		C/C	0.1–1.4 μ m	Tube
		Al ₂ O ₃ /Al ₂ O ₃	0.1–1.2 μ m	Tube/Monolith
		+ TiO ₂ /ZrO ₂ /Al ₂ O ₃	5–100 nm	
NGK		Al ₂ O ₃ /Al ₂ O ₃ + SiO ₂	0.2–5 μ m	Tube/Monolith
Whatman	ANOPORE®	Al ₂ O ₃	20 nm–0.2 μ m	Disk
Gaston County	UCARSEP®	ZrO ₂ /C	4 nm	Tube
Du Pont/Carré		Zr(OH) ₄ /SS	0.2–0.5 μ m	Tube
TDK	DYNACERAM®	ZrO ₂ /Al ₂ O ₃	≈ 10 nm	Tube
		Al ₂ O ₃ /Al ₂ O ₃	50 nm	
ATECH		SiC/SiC	0.05–1.0 μ m	Tube/monolith
		Al ₂ O ₃		
Asahi Glass		Glass	0.1–1.4 μ m	Tube
Fuji Filters		Glass	4–90 nm	Tube
		Glass	0.25–1.2 μ m	
Fairey	STRATA-PORE®	Ceramics/Ceramics	1–10 μ m	Tube/Plate
	MICROFILTRIX®	SS	0.2–1 μ m	Tube/Plate
Osmonics	HYTREX®	Ag/None	0.2–5 μ m	Tube/Plate
	CERATREX®	Ceramics/Ceramics	0.1 μ m	
Ceramem		Ceramics/Cordierite	0.05–0.5 μ m	Honeycomb
Trideltafiltration		Al ₂ O ₃ /Al ₂ O ₃	0.1–7 μ m	Tube/Monolith
Hoogovens		Al ₂ O ₃ /Al ₂ O ₃	0.1–1 μ m	Tube
Steeneker		Al ₂ O ₃	0.4 μ m	Tube
NOK		Al ₂ O ₃ /Al ₂ O ₃	0.2–6 μ m	Tube
TOTO		Al ₂ O ₃ /Al ₂ O ₃	0.1–0.2 μ m	Tube/
		ZrO ₂ /Al ₂ O ₃	5–30 nm	Monolith
Carré		ZrO ₂ /SS		Tube

Tab. 2 Inorganic membranes commercially available

4. Gas membrane application

Inorganic membranes are increasingly being explored to separate gas mixtures. Besides having well known thermal and chemical stabilities, inorganic porous membranes usually have much higher gas fluxes as compared to polymeric membranes.

More than 90% of the membrane gas separation business involves the separation of inorganic gases such as nitrogen from air, carbon dioxide from methane and hydrogen from nitrogen, argon or methane (Baker, 2002).

Tab. 3 shows all possible fields of application of gas membrane (Abedini 2010, Venkataraman 1992).

Common gas separation	Application
O ₂ /N ₂	Oxygen enrichment, inert gas generation
H ₂ /Hydrocarbons	Refinery hydrogen recovery
H ₂ /N ₂	Ammonia Purge gas
H ₂ /CO	Syngas ratio adjustment
CO ₂ / Hydrocarbons	Acid gas treatment, landfill gas upgrading
H ₂ O/ Hydrocarbons	Natural gas dehydration
H ₂ S/ Hydrocarbons	Sour gas treating
He/ Hydrocarbons	Helium separation
He/N ₂	Helium recovery
Hydrocarbons/Air	Hydrocarbons recovery, pollution control
H ₂ O/Air	Air dehumidification

Tab. 3 Gas membrane application

5. Ceramic membranes

Ceramic material is an inorganic, non-metallic solid prepared by the action of heat and subsequent cooling (unlike glass that is amorphous, ceramics are crystalline materials). Ceramics are compounds of metallic and non-metallic elements such as aluminium and oxygen (Al₂O₃), zirconium and oxygen (ZrO₂) or silicon and carbon (SiC). These compounds occur naturally in clays and other minerals (Gitis 2016).

As stated in par.3 the first modern industrial application of ceramic membranes was separation of U-238 and U-235 isotopes for making nuclear weapons and fuels in the 1940 and 1950. Many aspects of that work (the so called Manhattan Project) are still classified; some information can be derived from several patents in the 1970s. As proposed for the polymer membranes in 1962, it appeared that ceramic membranes could be made by number of layers like onions. Generally, the skin layer determines the separation and the support layer the mechanical strength.

Full scale installations for water and waste water purifications started in Japan in 1998.

Companies such as Atech Innovations, Orelis, Veolia Water, Hyflux, Kubota, TAMI Industries, Inoceramic GmbH, Metawater, Mitsui, Meidensha, Jiangsu Jiuwu, ervatech and Ceraver (now Pall Corporation) now advance ceramic membranes in new fields such as water and wastewater treatment, food nad beverages, chemical, pharmaceutical, electronic, petrolchemical and energy sectors. Fig. 5 sketches a brief of ceramic membrane history and their entry into various industrial sectors.

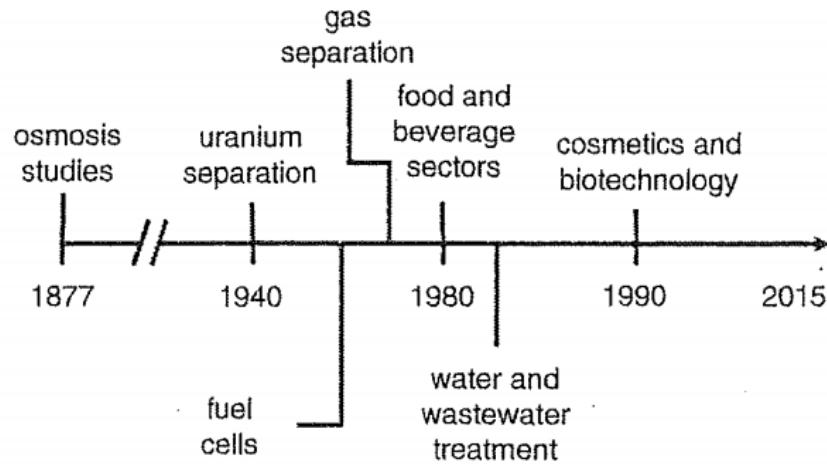


Fig. 5 Ceramic membranes history (Gitis 2016)

Today ceramic membranes are established in modern separation techniques.

The unique thermal, chemical and mechanical properties of ceramic membranes give them significant advantages over polymeric membranes, stainless steel membranes and conventional filtration techniques in many applications.

Ceramic membranes are operated in cross (tangential) flow filtration mode, which has the benefit of maintaining a high filtration rate for membrane filters compared with the direct-flow filtration mode of conventional filters (Sondhi 2003).

In the future, ceramic membranes will be employed always in more applications with clear advantages in chemical and thermal stability, longer lifetime, higher flux and higher recoveries.

Fig. 6 shows an asymmetric microporous ceramic membrane at scanning electron microscope. It is possible to see the three layers: a thick support macroporous layer, a mesoporous intermediate layer and a skin microporous top layer with separating capacities.

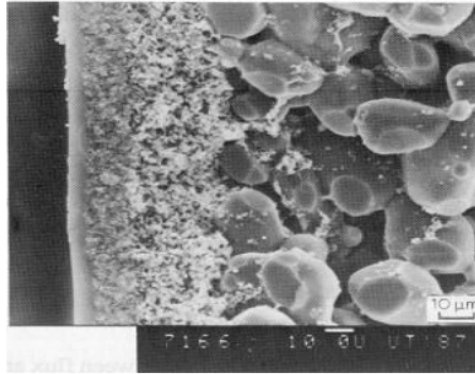


Fig. 6 Ceramic membrane at scanning electron microscope (Sensidoni 2012).

Ceramic membranes are often formed into an asymmetric, multi-channel element. These elements are grouped together in housings, and these membrane modules can withstand high temperatures extreme acidity or alkalinity and high operating pressures, making them suitable for many applications where polymeric and other inorganic membranes cannot be used.

Fig. 7 shows four different sections of commercial ceramic membranes (Sensidoni 2012).

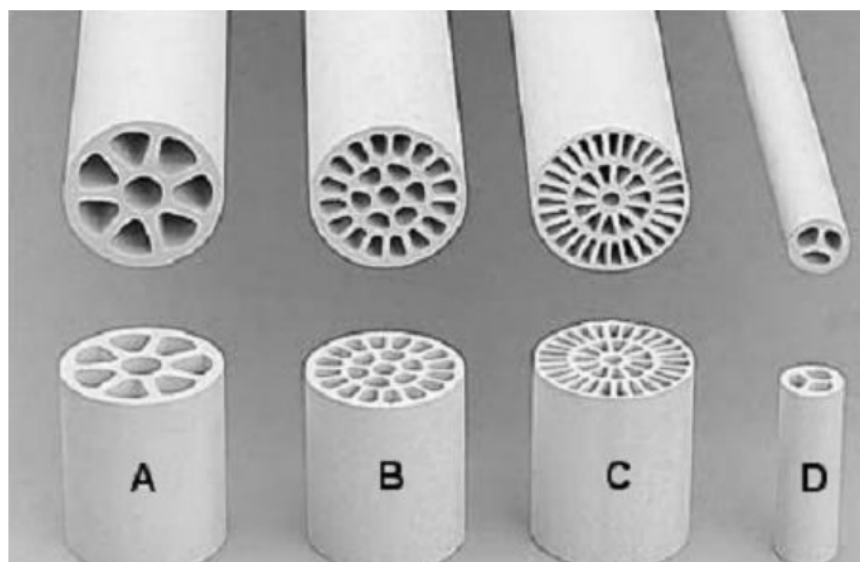


Fig. 7 Different sections of commercial ceramic membranes (Tami Industries, Nyons- F)

The multichannel monolithic form of membrane elements offers the highest filtration packing density (filtration area per unit volume) among commercial ceramic membranes. In the monolithic shape, the

selective membrane layer usually is in the innermost layer at the circumference of the channel while most solid portion of the element is the porous bulk support.

Feed streams usually enter the channels at one end of the monolith and the retentate exits at the other end. In this crossflow configuration, the permeate passes through the membrane and the subsequent support layers.

Ceramic membrane elements have sealing gaskets attached at each end and are then assembled within housings, available in 316L SS, polyvinylidene and other alloys (see fig. 8). A typical industrial installation will have several of these modules arranged in series and/or parallel configuration.



Fig. 8 Ceramic membrane housings

Ceramic materials are generally very stable chemically, thermally and mechanically, and in addition are frequently bio inert. The medium to be filtered flows through the channels of the membrane carrier. Particles are retained if their size exceeds the radius of the membrane pores, building up the concentrate. The filtrate permeates through the pores and is subjected to subsequent process stages.

6. Mechanisms for gas permeation across porous membranes

Permeation of gases through micro-porous solid is a complex phenomena including interactions between the pore wall and diffusing molecules as well as interactions between diffusing molecules themselves.

Mechanism governing the transport of gases in micro-porous media depends of physical properties of the material, experimental conditions and of permeating gas.

Experimental conditions influencing the permeation process are mainly temperature and pressure which determine the mean free path of the molecule λ (Venkataraman 1992, Lu 2007):

$$\lambda = \frac{3 \eta}{2 P} \frac{(\pi RT)^{1/2}}{2M}$$

where η is the viscosity of the gas, R the universal gas constant, T the temperature, M the molecular weight, an P the pressure.

Fig. 9 gives a schematic representation of the mechanisms for the permeation of gases through a porous membrane.

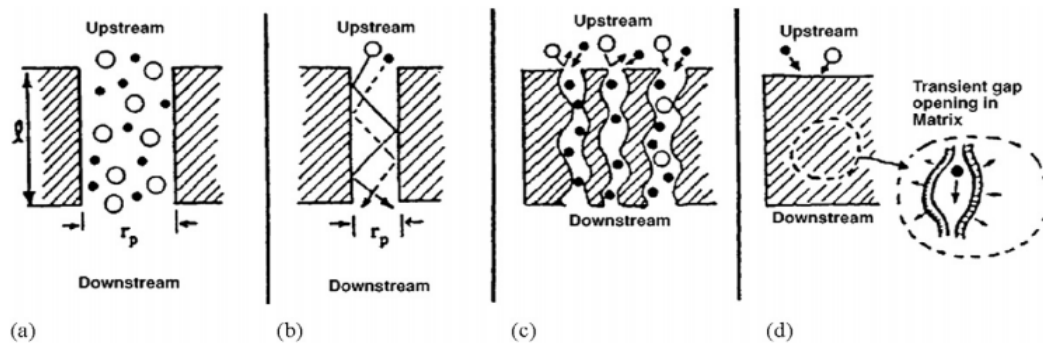


Fig. 9 Various gas separation mechanisms a) viscous flow, b) Knudsen diffusion, c) molecular sieving and d) solution diffusion (Lu 2007)

There are four molecular transport mechanisms through membranes as summarized below:

- (a) viscous flow (or Poiseuille regime), no separation is achieved. In this regime $\lambda/r \ll 1$ (where r is the pore radius and λ the free path of the gas molecules)
- (b) Knudsen flow regime. In Knudsen flow r/λ is much less than one, then there are more collisions with the pore walls than with other gas molecules. At every collision with the pore walls the gas

molecules are momentarily absorbed and then reflected. The separation of gas mixture is achieved because different gas species move at different velocities.

For Knudsen flow the selectivity ratio for binary gas mixtures can be estimated from the inverse square root ratio of the molecular weights A and B; separation factor:

$$\alpha_{AB} = \left(\frac{M_B}{M_A} \right)^{1/2}$$

- (c) Micropore molecular sieving (or activated diffusion), separation is based on the much higher diffusion rates of the smallest molecule, but adsorption capacities may be important factors for similarly sized molecules
- (d) Solution-diffusion regime, separation is based on both solubility and mobility factors in essentially all cases.

Table 4 presents the ideal separation factors for various pairs of gases under the Knudsen mechanism (Pandey 2001).

Gas pair	Separation factor
H2/N2	3,73
H2/CO	3,73
H2/H2S	4,11
H2/CO2	4,67
H2/SO2	5,64
N2/O2	1,07
O2/CO2	1,7

Tab. 4 Calculated separation factor based on Knudsen flow for selected binary gas mixtures

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