

## Development & Characterization of Ceramic Membranes

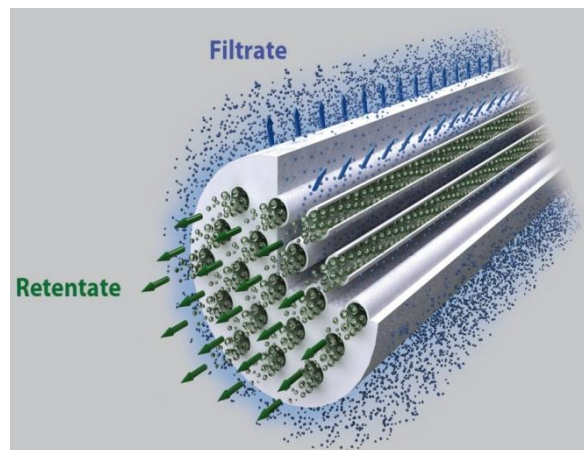
Manohar

**Abstract:** Ceramic membranes are used for water and gas filtration of its chemical inertness, low temperature preparation, good mechanical and porous properties. Important characteristics of ceramic membranes are high porosity, high permeability, defined pore size and good mechanical properties. In this study, fabrication and characterization of ceramic membranes preparation was carried out.

For example, Zirconia ceramic samples in the form of tubes are prepared by extrusion. The effect of hydrothermal reactions of zirconia oxynitrate for Zirconia membrane preparation as a starting powder was analyzed. Zirconia coating is applied on the SiC tube. It is mainly carried out by hydrothermal reaction. Zirconia solution prepared from zirconia oxynitrate and water. This hydro thermal test is carried out in an oven at 150°C for 16hrs. Finally we got good zirconia coating on SiC base. Found zirconia coated body is then dried at 100°C and fired at 800°C. Fired samples are characterized and tested for density, porosity, permeability, & S.E.M. Obtained product shows optimum porosity (40-45%) and good permeability for the membrane application like water filtration.

### I. INTRODUCTION

Membrane separation has become an important unit operation in water operation in water and wastewater treatment. Different degrees of water quality can be obtained with removal of particles, dissolved organic compound, protein and ions. However, two dominant areas are removal of organic compound in water clarification and osmotic desalination. The overall principle in any membrane separation is sketch in figure.



The principle in membrane separation.

#### 1.1 Membrane operation

The first thing to recognize is that regardless the use of different designs the separation depend on the physical – chemical properties of the material to be separation and the membrane used. The separation is driven by a difference in chemical potential across the membrane. The force needed to establish such chemical potential difference across the membrane. Pressure related membrane separation is usually divided into four operations: Microfiltration, ultra filtration, nanofiltration and reverse osmosis- each having different characteristic.

Table shows that separation of small particles/molecules requires a high trans- membrane pressure. In addition, the rate of separation, the flux, decreases. New membrane materials with different properties are developed all the time simply to fit the need for the membrane operation just mentioned.

Tabel1.a.1: Pressure drop for different membrane processes

Membrane process	$\Delta p$ (bar)	Size Range ( $\mu m$ )	$J(l/m^2 \cdot h \cdot bar)$
Microfiltration	0.1 -2.0	0.1-10	>50
Ultrafiltration	1.0-5.0	0.02-0.3	10-50
Nanofiltration	5.0-20	0.001-0.01	1.4-12
Reverse osmosis	10-100	<0.001	0.05-1.4

### 1.2 Classification of membranes

Membrane are classified according to separation mechanism, physical morphology, membrane geometry and physical-chemical properties. This can sometimes be confusing but one must bear in mind that scientific people have different interest in membrane technology. Some are only interested in membrane synthesis, others in membrane module design. A good exercise is to ask our self: which kind of separation mechanism is used in this membrane operation? What is the membrane morphology? etc.

Essentially, there are three type of separation mechanism:

1. Separation by size- the sieve effect: This requires porous membranes with rather large pores. Terms like macropores, mesopores and micro pores (figure 1.a.2) are used to describe the pore size in membrane for microfiltration ultra-filtration and nanofiltration.
2. Separation by different in solubility and diffusivity of material: This principle is used in operations like reverse osmosis and requires the membrane used to be dense (nonporous).
3. Separation by charge: An ion exchange membrane separates compounds of different charges. Ion-exchange membranes are used in operation like electro dialysis and are generally nonporous.

### II. Polymer Membranes:

For the sake of simplicity, only two very common organic membrane materials will be discussed here. Details about other materials can be found in Table.

One of the most common materials used is cellulose and derivatives. Cellulose esters like cellulose acetate and ethyl cellulose are all repeating units of glucose, connected by glycoside bonds, where the hydroxyl groups on each glucose unit participate in ester formation. The membranes are hydrophilic but only sparingly soluble in water. Polarity is compared to the other materials in table (except polyacrylonitrile) relatively high. Cellulose esters resist cleaning with chlorine, but hydrolyze easily, like other ester compounds, when exposed to acid or base. In addition, temperature and biological activity may cellulose membranes.

#### Summary of the most widely used porous membrane materials.

Material	Physical-chemical properties	Primary membrane operation area in water treatment
Cellulose esters (CE) primary cellulose acetate (CA)	Low chemical stability towards acid/base, temperature and biological activity. Resist chlorine cleaning. Relatively polar. Very hydrophilic.	Desalination, softening, disinfection and clarification using micro, ultra and nano filtration and reverse osmosis.
Aliphatic and aromatic polyamides (PA)	Higher hydraulic and thermal stability than CE, but sensitive towards oxidation e.g. chlorine. Hydrophilic.	Reverse osmosis but also ultra and micro filtration applications.
Polyacrylonitrile (PAN)	Less hydrophilic than CE's and PA's but very polar.	Ultra filtration applications in general
Polysulfone (PSF) and Polyethersulfon (PES)	Weak hydrophobic with some polar tendency. Good chemical and thermal stability.	Ultra filtration, support material for several composite membranes.
Polytetrafluoroethylen (PTFE), polyvinylidene fluoride (PVDF), and polypropylene (PP).	Hydrophobic non-polar membranes. High thermal and solvent resistance very chemically inert. Adsorb only weakly.	Micro and ultra-filtration applications.

The above table is a very qualitatively way to list of physical-chemical membrane properties. It is often difficult to do otherwise with this kind of properties, but in the next section a tool to physical characterization will be presented and it will make the classification more quantitatively.

### Ceramic membranes



Plant Engineers and plant operators demand, with respect to membrane installations, process stability, high availability and low requirements for preliminary treatment and minimum need for support and maintenance.

In contrast to many polymer membranes the ceramic membranes are a perfect match for this demand due to their extremely high chemical and physical stability, their outstanding separation characteristics and their long working life.

Ceramic materials are generally very stable chemically, thermally and mechanically, and in addition are frequently bio inert. They are therefore ideal materials for many applications in the chemical and pharmaceutical industry or in water and waste water processing.

The medium to be filtered flows through the channels of the membrane carrier. Particles are retained if their size exceeds the radius of the membranes pores, building up the concentrate. The filtrate permeates through the pores and it is subjected to subsequent process stages.

Many are the advantages deriving from the ceramic membranes use:

- No ageing, long life and reliability.
- High temperature stability.
- Mechanical stability under large pressure gradient.
- Stability over a large pH range.
- 0.3 – 100 efficiency in gas separation.
- Ability to be backwashed and abrasion resistance (steam sterilization and high flash capability )
- Incentive to bacterial action.
- Can process highly viscous fluid.
- Good control of pore dimension fouling.
- They are ecologically friendly and more favorable than separation technologies.
- No additives are necessary and the process temperature is not limited

Filtration with ceramics is a mild, highly selective process without phase transformation. Running costs are limited by closed production cycles and continuous processes.

Disadvantages are:

- Brittle character needs configuration supporting system.
- Relatively high capital installation cost.
- Relatively high modification costs in case of defects.
- Sealing technology for high temperature application may be complicated.

### Scope of porous ceramic membranes



Inorganic membranes are versatile they can operate at elevated temperatures with metal membranes stable at temperatures ranging from 500 to 800°C and with many ceramics membranes stable at over 1000°C they are also much more resistance to chemical attack because of the wide variety of materials that may be used in the fabrication of our inorganic membranes resistance to corrosive liquids and gases even at elevated temperatures can be realized.

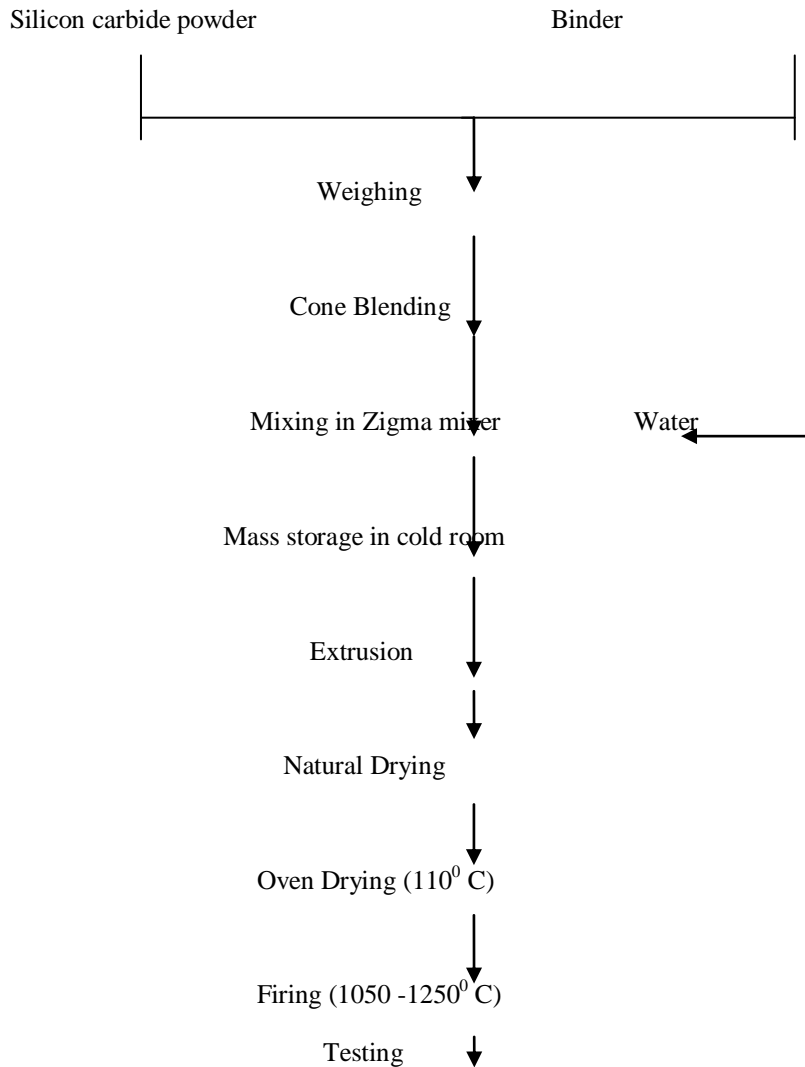
Inorganic membranes completed with organic membranes for commercial use in many of the harsh operational environmental listed above organic membranes offer need solution.

### Applications

Initially, ceramic membranes were used in waste water technology. Meanwhile, successful and possible applications cover all industries where media were filtered.

- Chemical industry :
  - Product separation and cleaning.
  - Concentration of polymer suspensions and metal hydroxide solutions.
  - Separation of catalysts.
  - Recovery of dyes and pigments.
  - Desalination of products.
  - Cleaning and recycling of organic solvents.
  
- Metal industry / Surface engineering :
  - Recycling and disposal of degreasing and rinsing bathes.
  - Treating of oil /water emulsions.
  - Recovery of heavy metals.
  - Cleaning of waste water from grinding processes.
  - Treatment of waste water form glass and glass fiber production.
  
- Textiles / Pulp and paper industry :
  - Concentration fractionation, isolation and sterilization for antibiotics, enzymes, proteins, amino acids and vitamins.
  - Separation, concentration and dewatering of biomass and algae.
  - Disposal of fat emulsions.
  - Separation of yeast.
  - Desalination.
  
- Food and beverages
  - Clarification of juice and beer.
  - Concentration of juice.
  - Sterilization of milk and whey.
  - Desalination of whey.
  - Dewatering of products.
  - Purification of drinking water.
  
- Recycling and environment.
  - COD /BOD reduction.
  - Oil / water separation.
  - Recovery of pharmaceuticals and pesticides.
  - Retention of microorganism.
  - Retention of heavy metals and radioactive substances.
  - Recycling of water form swimming pools.
  - Purification of the drain of sewage plants.

**PREPARATION OF RODS AND TUBES:** Flow chart for tubes & rods preparation



**Raw materials used**

**Silicon carbide:**

The Acheson process is used to produce large quantities of Sic. This process carbothermally reduces SiO<sub>2</sub> to give SiO and CO (g) in a resistance furnace. Low purity silicon carbide is used in abrasion and refractory application. High quality silicon carbide is used for reaction bonded ceramics that require strength at high temperature, high thermal conductivity, high thermal shock resistance, and allow thermal expansion co-efficient.

**Graphite (Black Lead):**

It is a crystalline form of carbon. It occurs in nature either as microcrystalline earthy lumps or in a lobular force. Natural graphite is classified on the basis of its physical properties, nature. Ex:-flakes, lump and amorphous (micro crystalline) type. It is very and usually has metallic structure and a greasy feel.

Occurrence: Graphite occurs in a Andhra Pradesh, Lohit (Arunachal Pradesh), Pala man (Bihar), Karnataka, samba lour and Bolangir (Orissa), Ernakulum (Kerala), Doomra(Rajasthan) and in Baramulla(J and K).

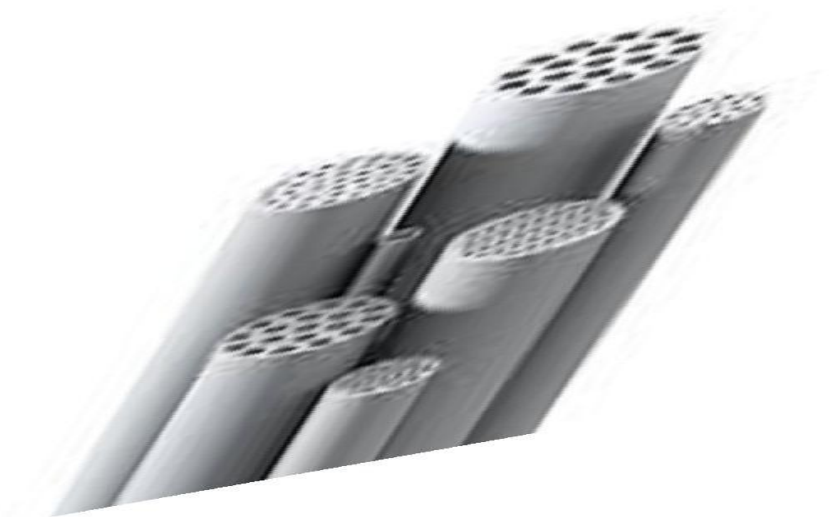
**Binders:** Substances, which hold ceramic raw material and bodies together during the initial stage of processing, are called binder.

1. Lignin has been used for tempering all type body fabricated by ceramic process it increases the strength of the weak body.
2. Tannic acid has long been employed to increase the particles of days, but it is not effective as extract in increasing the dry strength.
3. Ordinary paraffin's, with a small percentage of carnauba wax, is a good binder in some cases and can be molded cold with a reduction in molding time.

**Procedure for rods and tubes preparation:**

The silicon carbide powder and binder (5%) were weighed according to the composition accurately and then the batch was dry mixed in cone blender. The weighed batch was charged to cone blender. The purpose of cone blending is to mix the titania powder and the binder homogeneously. This blending was carried out for a period of 2 hour. The batch is weighed and charged to zigma mixer. The water is slowly added during mixing until required consistency mass is obtained and the amount of water is noted down.

The mixed mass is kept in cold room for ageing. After ageing the mass is feed to the extruder where simultaneously de-airing and compaction is carried out. In the extruder kerosene is used as lubricant. The die is fixed accordingly i.e. this depends up on rod or tubes to be extruded. Then the pieces are extruded in aluminum channels and kept for natural drying. The natural drying is carried out for a couple of days. Then the naturally dried pieces are kept for drying in electrical dryer where temperature is initially kept at 40<sup>0</sup> C for about 3 hours then temperature is raised to 60<sup>0</sup> C and kept for about 3 hours and then 80<sup>0</sup> C and 100<sup>0</sup> C in the same way. Care should be taken while drying the rod or tubes must be kept between metal supports. Then the dried pieces are carried for firing operation. The firing ids carried out at different temperature 1050, 1100, 1150, 1200<sup>0</sup> C.The fired pieces are tested fir the following tests: %apparent porosity, bulk density, %water absorption, SEM, Permeability etc.



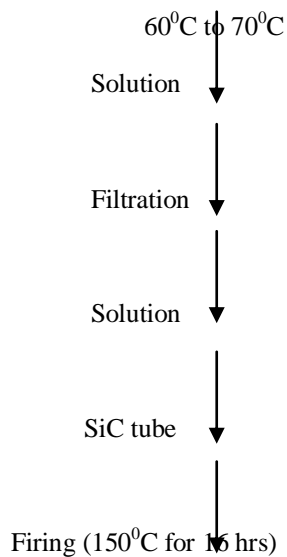




**Zirconia coating on SiC tube**

Flow chart for hydrothermal test

300ml of H<sub>2</sub>O + 45 gms of zirconia oxy nitrate



### Zirconium Oxide(ZrO<sub>2</sub>)

Zirconia is an extremely refractory material. It offers chemical corrosion inertness to temperatures well above the melting point of alumina. The material has low thermal conductivity. It is electrically conductive above 600<sup>0</sup> C and is used in oxygen sensor cell and as the susceptor (heater) in high temperature induction furnaces. With the attachment of platinum lead, Nernst glowers use in spectrometers can be made as a light emitting filament which operated in air.

### Key Properties of Zirconium Oxide

- ✓ Use temperatures up to 2400<sup>0</sup>C
- ✓ High density
- ✓ Low thermal conductivity (20% that of alumina)
- ✓ Chemical inertness
- ✓ Resistance to molten metals
- ✓ Ionic electrical conduction
- ✓ Wear resistance
- ✓ High fracture toughness
- ✓ High hardness

### Typical Uses of ZrO<sub>2</sub>

- ✓ Precision ball valve balls and seats
- ✓ High density ball and pebble mill grinding media
- ✓ Rollers and guides for metal tube forming
- ✓ Thread and wire guides
- ✓ Hot metal extrusion dies
- ✓ Deep well down-hole valve and seats
- ✓ Powder compacting dies

- ✓ Marine pump seals and shaft guides
- ✓ Oxygen sensors
- ✓ High temperature induction furnace susceptors
- ✓ Fuel cell membranes
- ✓ Electrical furnace heaters over 2000<sup>0</sup>C in oxidizing atmospheres

### General Zirconia Oxide information:

Pure zirconia exists in three crystal phases at temperatures. At very high temperatures (>2370<sup>0</sup>C) the material has a cubic structure. At intermediate temperatures (1170<sup>0</sup>C to 2370<sup>0</sup>C) it has a tetragonal structure. At low temperatures (below 1170<sup>0</sup>C) the material transform to the monoclinic structure. The transformation from tetragonal to monoclinic is rapid and is accompanied by a 3 to 5 percent volume increase that causes extensive cracking in the material. This behavior destroys the mechanical properties of fabricated components during cooling and makes pure zirconia useless for any structural or mechanical application. Several oxides which dissolve in the zirconia crystal structure can slow down or eliminated these crystal structure changes. Commonly used effective additives are MgO, CaO, and Y<sub>2</sub>O<sub>3</sub>. With sufficient amounts added; the high temperature cubic structure can be maintained to room temperature. Cubic stabilized zirconia is a useful refractory and technical ceramic material because it does not go through destructive phase transition during and cooling.

The controlled, stress include volume expansion of the tetragonal to monoclinic inversion is used to produce very high strength, hard, tough varieties of zirconia available from accuratus for mechanical and structural applications. There are several different mechanisms that lead to strengthening and toughness in zirconias that contain tetragonal grains. These depend on the grain sizes, the thermal history and the kind and amount of stabilizing additive in the body. These variation lead to two strong, commercially available partially stabilized zirconia (PSZ) microstructure identified as TTZ (tetragon ally toughened zirconia) and TZP (tetragonal zirconia polycrystalline) ceramic. The TTZ is an MgO partially stabilized zirconia often designated MgTTZ or MgPSZ consisting of uniformly dispersed tetragonal precipitates in larger cubic phase crystals. The secondary thermal aging process requiring tight manufacturing controls for proper micro structural development has limited the supplier base for the tetragonal phase, very fine grain mateial stabilized with rare earth oxides, primarily yttria and less commonly ceria. They are often designated YTZP for the yttria stabilized product and CeTZP for the ceria stabilized product. The TZP material has found uses in cutting and wear resistant application due to its reliable and outstanding hardness and toughness. TZP properties degrade rapidly when the material is exposed to water vapor at 200 to 300<sup>0</sup>C, so controlled use condition are important for good performance. All of the toughened zirconia show a degrading of properties with increasing temperature, and this class of high strength, tough material is generally limited to use temperatures below 800<sup>0</sup>C.

### History of titania (TiO<sub>2</sub>):

The element titania was discovered in 1791 by William Gregory in England. Gregory spent much of his time studying mineralogy, which lead him to his discovery. This happened when he discovered a sample of a black sandy material in his neighborhood. He studied this substance and after he was assured that it was a mineral he called it menachanite. Four years later a man named Martin h klaproth recognized that there was a new chemical element in this mineral. He later named it titanium after the titans which are numerous monsters that ruled the world in Greek mythology. Martin h klaproth was not able to make the pure element of titanium however he was only able to produce titanium dioxide.

### Occurrence

The element occurred with a number of mineral deposits. Principally rutile ilmenites which are widely distributed in earth crust and lithosphere and it is found in almost in all living things, rocks, water bodies and soil. The metal is extracted from its principle mineral ore via kroll process or hunter process.

### Processing methods for manufacture of titanium dioxide

There are 2 distinct technologies for manufacture of titania

- Sulphate process
- Chloride process

#### Sulphate process

The sulphate process has been in existence for more than 70 years. The process is carried out in the following steps.

Digestion of ilmenite or titania slag in sulphuric acid

Reduction and crystallization

Hydrolysis

Calcinations

Dry milling



The product obtained by sulphate process is usually anatase which can be further treated to obtain rutile. The feed stocks obtained used for sulphate process are mainly ilmenite or titania slag.

The major advantage of this process is that large quantities of waste product are generated in the form of spent acid and copper. The problem of this copper can be reduced by using titania slag as feed instead of ilmenite.

**Chloride process**

The chloride process was developed by du-pont around 1959. The main process steps are as follows,

- Chlorination of synthetic rutile or other raw material.
- Purification of titanium tetrachloride.
- Oxidation of pure titanium di-oxide.

The main advantages of chloride process are as follows,

- Product obtained is rutile and is of better quality.
- Process is continuous.
- The waste products obtained are in small quality.

A technical composition of sulphate & chloride process indicates that the chloride process gives a product which is in finer in particle size, low in impurity & less waste products are generated. As a result more & more manufactures are shifting to chloride process. Presently about 55% of titania is produced by sulphate process.

**Physical and mechanical properties of titania**

Density	4 gm/cc
Porosity	0 %
Modulus of rupture	140 Mpa
Compressive strength	680 Mpa
Poisson's ratio	0.27
Fracture toughness	3.2 Mpa.m <sup>-1/2</sup>
Shear modulus	90 Gpa
Modulus of elasticity	230Gpa
Micro hardness(HV 0.5)	880
Resistivity(25 <sup>0</sup> C)	10 <sup>12</sup> ohm.cm
Resistivity(700 <sup>0</sup> C)	2.5×10 <sup>4</sup> ohm.cm
Dielectric constant(1 MHz)	85
Dissipation factor(1 MHz)	5× 10 <sup>4</sup>
Dielectric strength	4 kV/mm
Thermal expansion(RT-1000 <sup>0</sup> C)	9×10 <sup>-6</sup>
Thermal conductivity(25 <sup>0</sup> C)	11.7Wm/K

**Optical properties of titania**

Forms	RI	Density	Crystal structure
Anatase	2.49	3.84	Tetragonal
Rutile	2.903	4.26	Tetragonal

**Procedure for Hydrothermal test to prepare zirconia membrane:**

First we take 300 ml of water in beaker and then heated by using magnetic stirrer. Add 45 gms of zirconia oxy nitrate to water and maintained temperature at 60 -70<sup>0</sup>C. After completion of heating, filtering process is carried out to get a filtered solution. Then take autoclave apparatus containing Sic tubes and add 3/4<sup>th</sup> filtered solution into the autoclave apparatus. Autoclave apparatus is kept in oven at 150<sup>0</sup>C for 16 hrs. After completion of hydrothermal test, the apparatus is put in water for cooling. Finally we got a good coated zirconia on Sic tube.

A zirconia coated tubes is dried naturally and then send to calcined process at 800<sup>0</sup>C for 2 days. After calcined tubes are tested like permeability, SEM, density porosity, water absorption etc.

**CHARACTERIZATION AND TESTING**

**1. Density**

Bulk density is defined as the mass per unit volume of a material including the volume of open pores.

**2. True density**

True density is the mass per unit volume of a material including all forms of pores i.e. open and closed pores.

### 3. Porosity

Apparent porosity is the percentage relationship between the open pores to that bulk volume.

### 4. Water absorption

The water of absorption is expressed as the %age relationship of weight of water absorbed by the dry specimen.

#### Experimental procedure:

The specimen to be tested is kept in Dryer at 110<sup>0</sup> C until it reaches constant weight, then dry weight (D) of the specimen is noted down. Then the specimens are kept in boiling water for 2 hours, then they are allowed cooled to room temperature and the saturated weight (W) of the test specimens are noted down, then the specimens are suspended in water and the suspended weight (S) of the specimens are noted down and calculated using the formula given below.

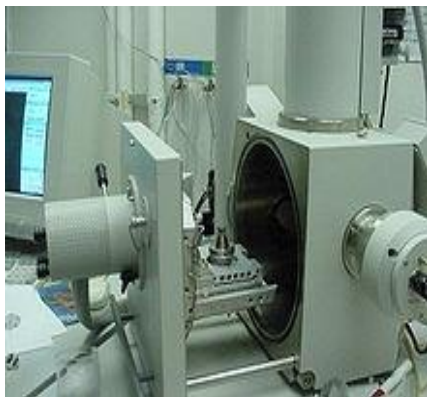
$$\text{Bulk density} = \frac{\text{Dry weight}}{\text{Saturated weight} - \text{suspended weight}}$$

$$\text{App. Density} = \frac{\text{Dry weight}}{\text{Dry weight} - \text{suspended weight}}$$

$$\% \text{ Apparent porosity} = \frac{\text{Saturated weight} - \text{dry weight}}{\text{Saturated weight} - \text{suspended weight}} \times 100$$

$$\% \text{ Water absorption} = \frac{\text{Saturated weight} - \text{dry weight}}{\text{Dry weight}} \times 100$$

#### SEM analysis



The scanning electron microscope (SEM) is a type of electron microscope that images the sample surface by scanning it with a high- energy beam of electrons in a raster scan pattern. The electrons interact with the atoms that make up the sample producing signals that contain information about the sample's surface topography, composition and other properties such as electrical conductivity.

The types of signals produced by an SEM include secondary electrons; back scattered electrons (BSE), characteristic X-rays, light (cathodoluminescence), specimen current and transmitted electrons. These types of signal all require specialized detectors for their detection that are not usually all present on a single machine. The signals result from interactions of the electron beam with atoms at or near the surface of the sample. In the most common or standard detection mode, secondary electron imaging or SEI, the SEM can produce very high –resolution images of a sample surface, revealing details about 1 to 5 nm in size Due to the way these images are created, SEM micrographs have a very large depth of field yielding a characteristic three – dimensional appearance useful for understanding the surface structure of a sample. This is exemplified by the micrograph of pollen shown to the right. A wide range of magnifications is possible, from about x 250,000, about 250 times the magnification limit of the best light microscopes. Back- scattered electrons (BSE) are beam electrons that are reflected from the sample by elastic scattering. BSE are often used in analytical SEM along with the spectra made from the characteristic X-rays. Because the intensity of the BSE signal is strongly related to the atomic number

(Z) of the specimen, BSE images can provide information about the distribution of different elements in the sample. For the same reason BSE imaging can image colloidal gold immuno-labels of 5 to 10 nm that would otherwise be difficult or impossible to detect in secondary electron images in biological specimens. Characteristic X-rays are emitted when the electron beam removes an inner shell electron from the sample, causing a higher energy electron to fill the shell and release energy. These characteristic X-rays are used to identify the composition and measure the abundance of elements in the sample.

**Permeability :**



Permeability apparatus

$$\text{Permeability} = \frac{\text{Permit flux}}{\text{Pressure difference}}$$

$$\text{Area of tube} = \pi dl$$

Where,

d is the diameter of tube

l is length of the tube

$$\text{LMH} = \frac{\text{litre}}{\text{m}^2 \cdot \text{hr.}}$$

**RESULTS**

Sample No.	Dry wt.(gms)	Suspended wt.(gms)	Saturated wt.(gms)	Bulk density (gm/cc)	% Porosity	% Water absorption
U-3-1	6.806	4.513	8.653	1.64	44.61	27.13
U-3-2	8.986	5.919	11.422	1.63	44.26	27.10
U-3-3	9.967	6.595	12.605	1.65	43.89	26.46
T-3-1	8.102	5.353	10.312	1.63	44.56	27.27
T-3-2	6.818	4.515	8.658	1.64	44.41	26.98
T-3-3	6.107	4.042	7.734	1.65	44.06	26.64

U –Uncoated samples

T – Titania coated sample

**Water Flux**

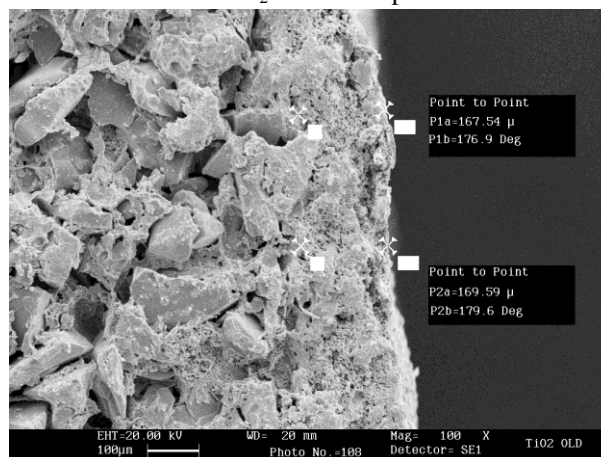
Membrane size : 13 mm OD, 9 mm ID, L=106 mm

Sample no	Composition	Area(m <sup>2</sup> )	Inlet pressure (kg/cm <sup>2</sup> )	Permit flux ml/min	LMH (l/m <sup>2</sup> .hr)
3	100% titania sol	0.00319	0.5	7	131.66
			1	10	188.09
			1.5	13	244.514
			2	14	263.32
			2.5	16	300.32
6	45gms of zirconia oxynitrate + 300ml of water	0.0046	0.5	8	104.35
			1	11	143.48
			1.5	15	195.65
			2	17	221.74
			2.5	21	273.913
5	Titania sol + 5% titania powder(50ml)+ 50ml of slurry	0.00319	0.5	16	300.47
			1	20	375.59
			1.5	28	525.82
			2	41	769.95
			2.5	44	826.29
13	Titania slurry+ 15% sol	0.0032	0.5	9	168.75
			1	12	225
			1.5	17	318.95
			2	23	431.52
			2.5	26	487.8
14	Titania slurry+ 15% sol	0.0047	0.5	27	344.83
			1	38	485.31
			1.5	48	613.03
			2	60	766.28
			2.5	70	893.99
15	5% of titatnia sol + 95ml of slurry	0.003	0.5	40	800
			1	53	1060
			1.5	60	1200
			2	61	1220
			2.5	62	1240
16	5% of titatnia sol + 95ml of slurry	0.0042	0.5	24	342.86
			1	31	442.86
			1.5	34	485.71
			2	35	500
			2.5	36	514.29
6	210grams of zirconia oxynitrate + 700ml of water	0.0043	0.5	10	138.50
			1	12	166.20
			1.5	15	207.80

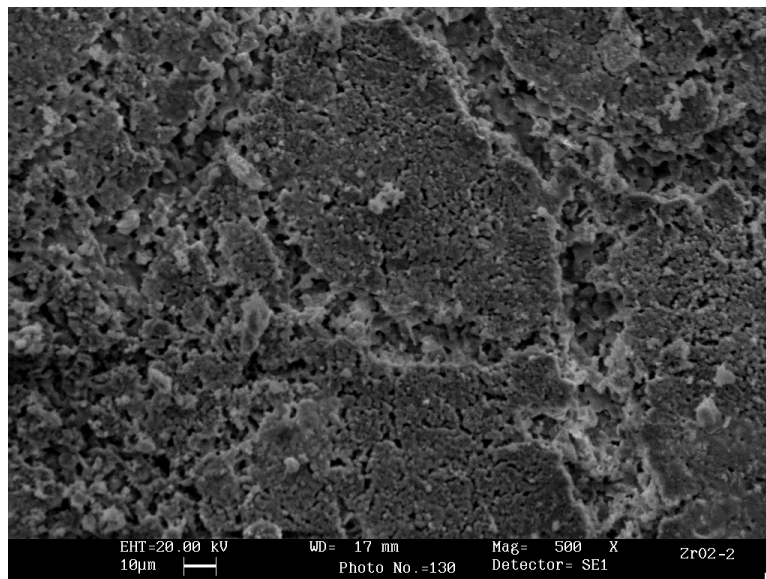
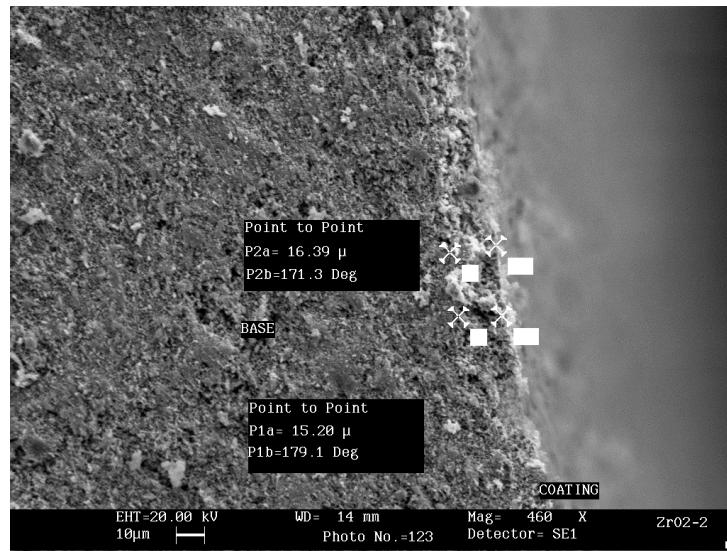
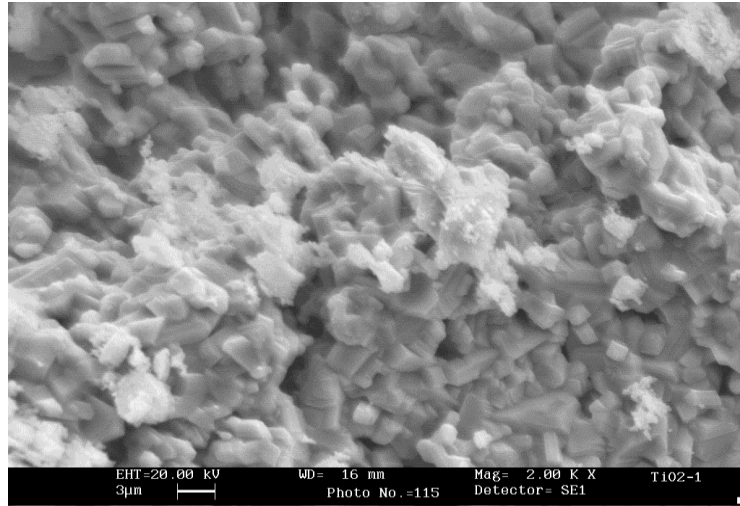
			2	16	221.60
			2.5	17	235.46
21	210grams of zirconia oxynitrate + 700ml of water	0.00302	0.5	22	437.37
			1	23	457.26
			1.5	27	536.78
			2	28	556.66
			2.5	30	596.42
22	210grams of zirconia oxynitrate + 700ml of water	0.003	0.5	23	460
			1	25	500
			1.5	28	560
			2	29	580
			2.5	32	640
14	200ml of zirconia oxynitrate solution + 100ml of water	0.0046	0.5	22	286.96
			1	34	443.28
			1.5	40	521.51
			2	42	545.45
			2.5	45	586.70
23	200ml of zirconia oxynitrate solution + 100ml of water	0.0032	0.5	24	433.99
			1	25	452.08
			1.5	32	578.66
			2	34	614.83
			2.5	37	669.08
24	200ml of zirconia oxynitrate solution + 100ml of water	0.0031	0.5	22	425.78
			1	28	541.90
			1.5	30	580.61
			2	32	619.31
			2.5	34	658.02

**SEM analysis:**

1. TiO<sub>2</sub> coated samples







2. ZrO<sub>2</sub> coated samples



### Conclusions

Zirconia coating is applied on the SiC tube. It is mainly carried out by hydrothermal reaction. Zirconia solution prepared from zirconia oxynitrate and water. This hydro thermal test is carried out in an oven at 150<sup>0</sup>C for 16hrs. Finally we got good zirconia coating on SiC base. Found zirconia coated body is then dried at 100<sup>0</sup>C and fired at 800<sup>0</sup>C. Obtained product shows optimum porosity (40-45%) and good permeability for the membrane application like water filtration.

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