# SYNTHESIS AND TEXTURAL STUDY OF POROUS MEMBRANE

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## Summary

The technique Sol-Gel is a process of materials elaboration which is consists on hydrolysis – condensation reactions indirectly to form a veritable lattice of oxide from molecular precursors .
The whole chemical reactions and experimental conditions ( concentration , temperature , nature of precursors )influence strongly structural , morphologic characteristics of solid phase , then solid textural control ( surface area , porosity ) is much easy than in solid chemical classical synthesis at high temperature.
The surface study by nitrogen adsorption –desorption allowed us to observe the variation of the surface area , porous volume and pore diameters according to temperature.
We observe an increasing of BET surface area and porous volume up to 600°C probably due to the progressive creation of pores in $SiO_2$ matrix.
From 600°C on, we notice a decreasing of BET surface area and porous volume which can be explained by an removal of inorganic products which leads to widening of pores and then an increasing pore diameters.
We notice that thermal treatment creates diameters widening which is in agreement with surface area and porous volume variations

## Introduction

The aim of this study allows on the one have to know obtaining conditions and characteristics of vitreous ceramic material by Sol-Gel process from precursors and on the other hand to contribute to obtain thin and microporous layers .
At present , these processes are applied at the synthesis of material in thin microporous layers shapes, intended to separation , concentration or purification of chemical species.
The microporous ceramics have taken in additional or in substitution of organic membrane, a dominating space in the process put into play the selective separations .

## I-Study of the system $Al_2O_3$-$SiO_2$ synthesis

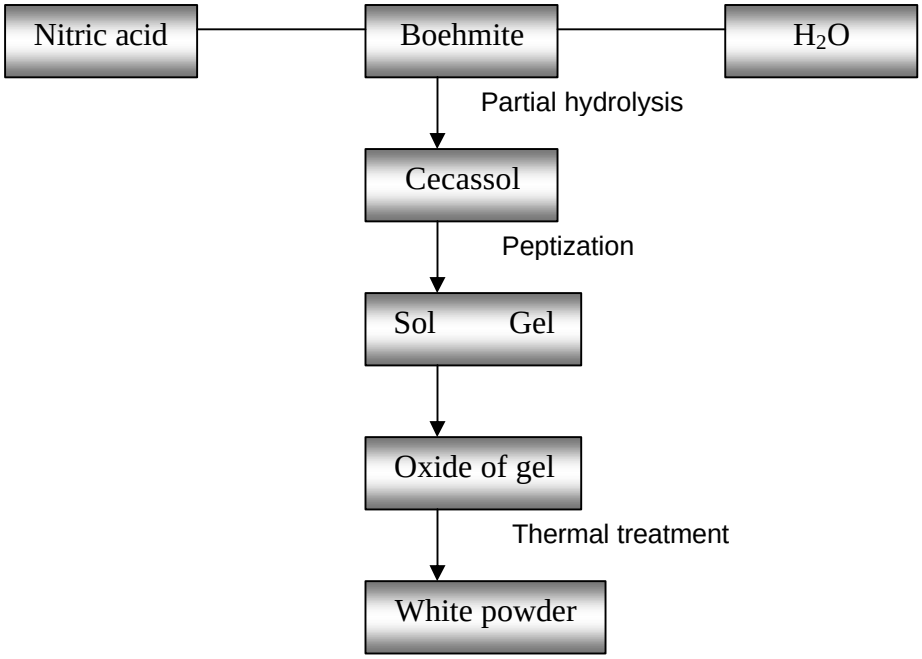
The precursors used to obtain these oxide system are the following :

- Boehmite pural SB AlO(OH)
- Cecassol ( sobret france ) 30% $SiO_2$

The synthesis difficulty lies in the difference of two precursor reactivity as regards of hydrolysis and condensation reactions.
Our fist preoccupation is to pass from the powder suspension to the colloidal solution.Thus, we have chosen the destabilization of colloidal solutions process.

In a first step, we have done a partial hydrolysis of boehmite before to add cecassol.
This allowed us to solve initial difficulty, and to have a good peptization of suspension.

The different steps to this process are representing in following schema:




*Preparation steps of $AL_2O_3$-$SiO_2$*

## *II-Structural characterization of mixed oxides $Al_2O_3$-$SiO_2$*

## *II-1-By RX diffraction*

The RX diffraction results of powders treated at different temperatures.
The powder presents amorphous structure up to 600°C .The powder treated at 900°C , gives a RX diagram characterizing ? alumina.
The powder treated at 1340°C presents a RX diagram corresponding at compound $Al_6Si_2O_{13}$ ( mullite) .This temperature is lower than that was mentioned in litterature 1550°C ( figII-1-a ) ( fig II-1-b) .

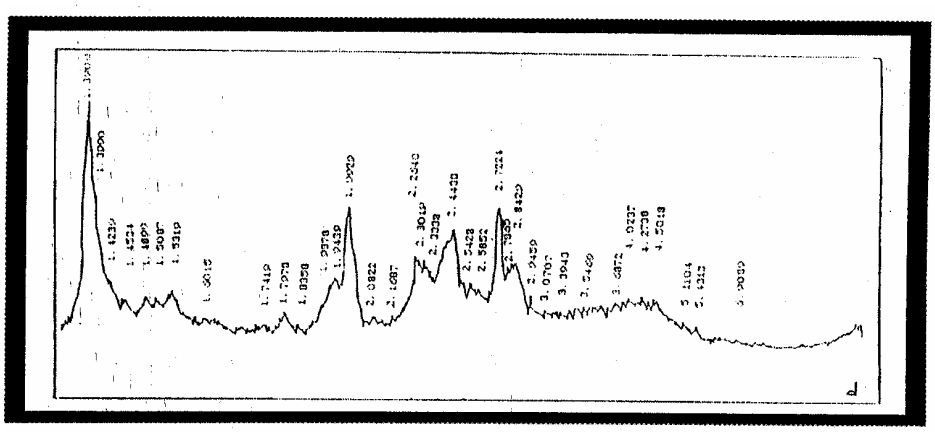


***Figure II-1-a : X-Ray spectra of powder treated at 600°C***

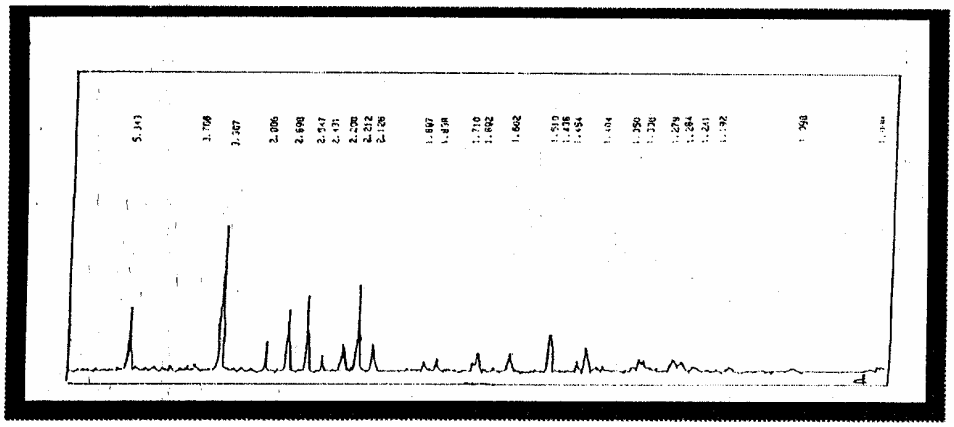


*Figure II-1-b : X-ray spectra of powder treated at 900°C*

## II-2-By spectroscopy adsorption IR

We have recorded a spectra infra-rouge for every powder treated at different temperature ( figII-2).

The bands observed at 1140cm$^{-1}$ – 670cm$^{-1}$ and 500cm$^{-1}$ correspond to the connection vibrations Si-O-Al. We notice up to 600°C , only the bands characterizing metallic connection M-O-M and O-M-O between 400 and 1200cm$^{-1}$ . M is the silicon or alumina.

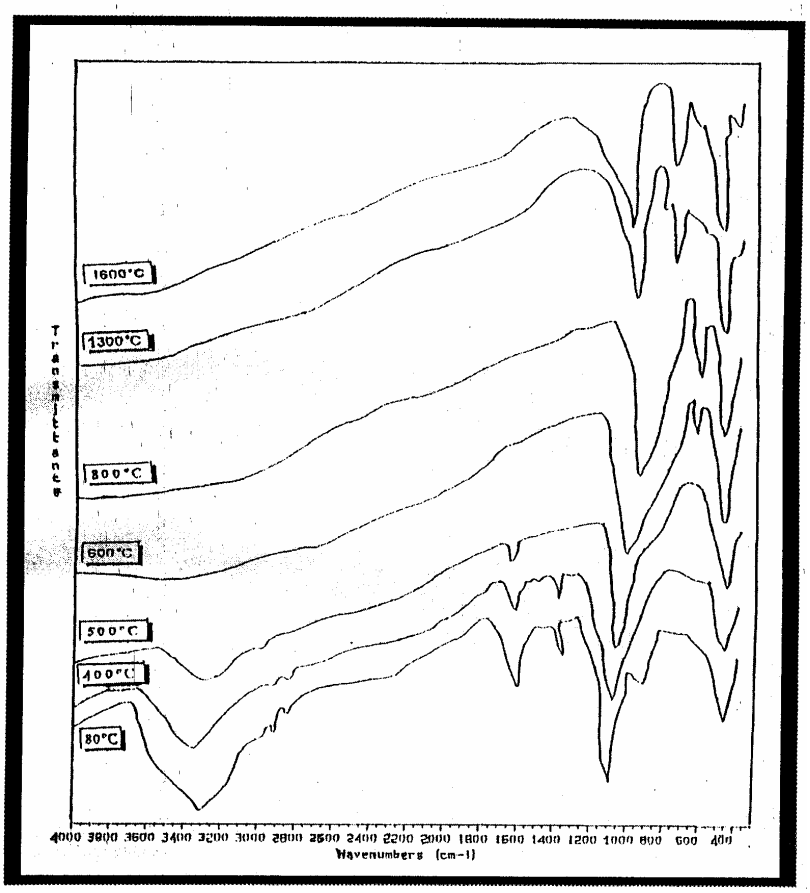



*Figure II-2 : Infrared spectra of powders treated at different temperatures*

## *III- Textural characterization of $Al_2O_3$-$SiO_2$ mixed oxides by adsorption/desorption of nitrogen*

We have drawn adsorption isotherms for five samples treated at different temperatures .
We notice that desorption isotherm does not coincide with adsorption isotherm, there is an appearance of a characteristic hysteresis of a capillary condensation.The observed isotherm for five samples is type IV characterising mesoporous bodies ( fig III-a) .
The surface study of the samples treated at different temperatures allows to observe the temperature influence treatment of surface BET, microporous volume and pore diameters.
In the range of temperature from 500 to 800°C, we observe a decrease of BET surface (s) and microporous volume (w) due to the densification and the consolidation of system ( fig III-b)(figIII-c)
In fact , densification process leads to reduction of interior energy and creates a decrease of porosity.
The increase of microporous volume can only be explained by removal of residual products occupying one part of pores and thus leaving vacuum cavities.

We notice in figure (III-d) that thermal treatment creates pore diameters widening which is in agreement with specific surface and porous volume variations.

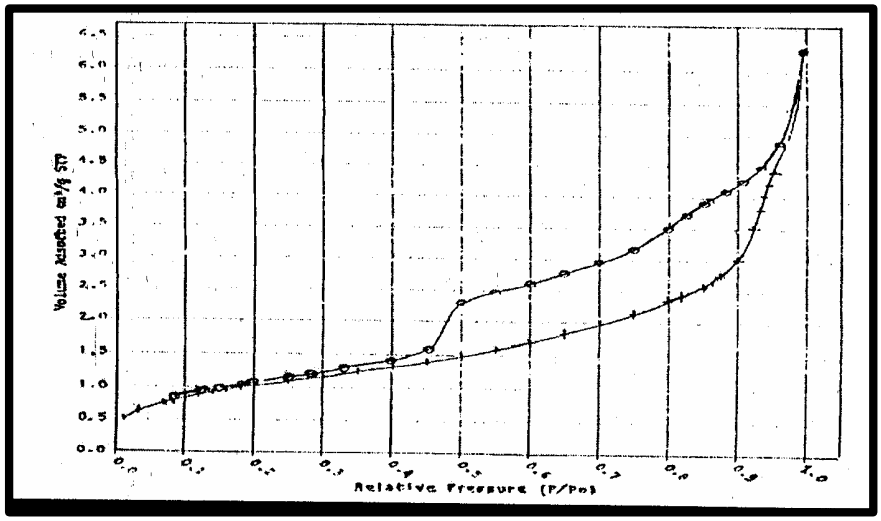



***Figure III-a- : Adsorption isotherm of oxyde powder $Al_2O_3$-$SiO_2$***
***+ : adsorption, 0 : desorption***

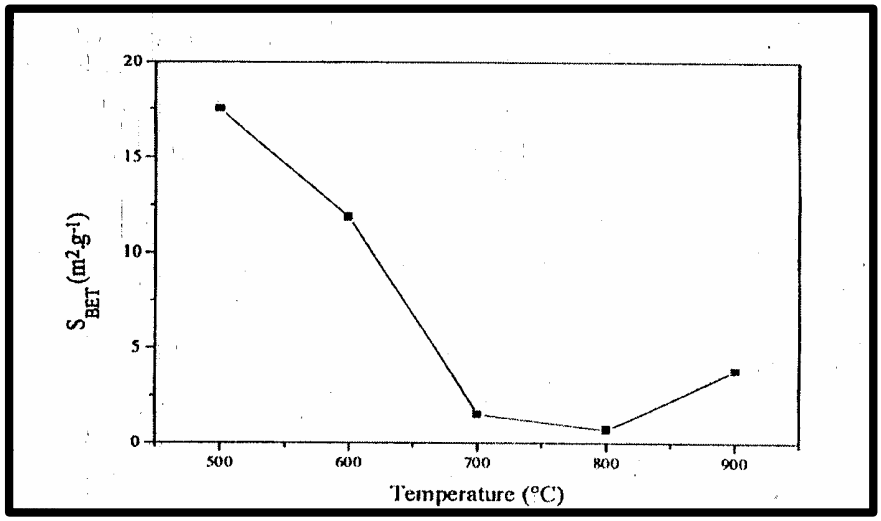



***Figure III-b- : Affect of treatment temperature on the surface area of $Al_2O_3$-$SiO_2$ Powder***

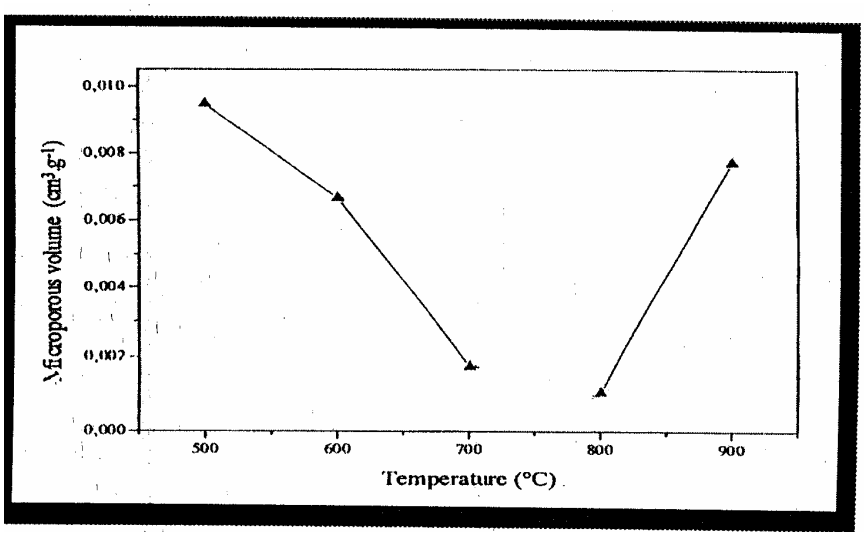
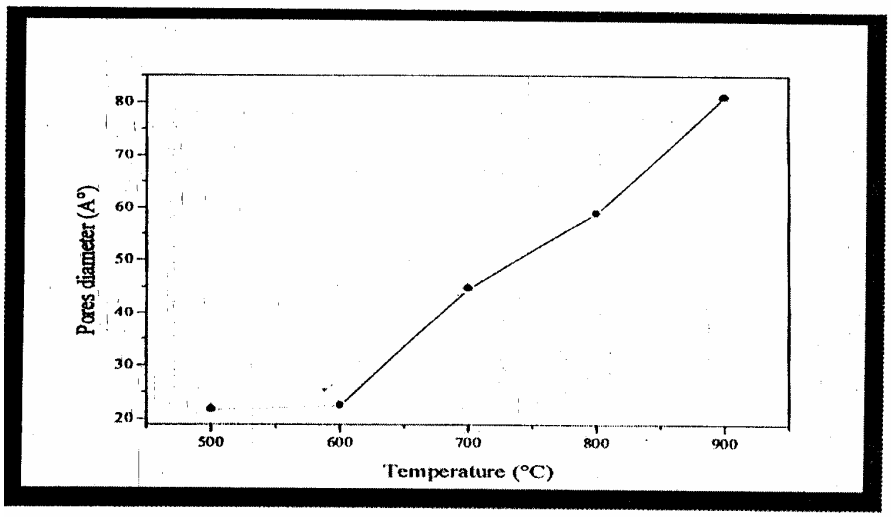



***Figure III-c: Affect of treatment temperature on the pore volume of $Al_2O_3$-$SiO_2$ Powder***




***FigureIII-d: Affect of treatment temperature on the pore diameter of $Al_2O_3$-$SiO_2$ powder***

## *IV- Deposits of thin microporous layers*

The last aspect of this study has been devoted to offered possibilities by $Al_2O_3$-$SiO_2$ system in a realization of thin microporous layers .These layers present interest in a domain of inorganic membranes as far as they have characteristics with high chemical and mechanical resistances .

These layers have been deposited in porous support a alumina presenting the pore diameters of 0.2um on the usable face for the deposit .

The membrane deposit has been realised with a sol prepared with polymerization of molecular entities process.

Scanning electron spectroscopy observation showed homogenous layers without cracking with a thickness of 2um ( fig IV-a) .We observe increasing of pore diameters according to temperature.We obtain at 600°C a pore diameter average to 100nm(figIV-b).

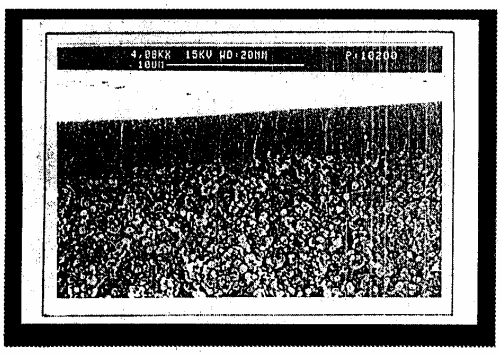
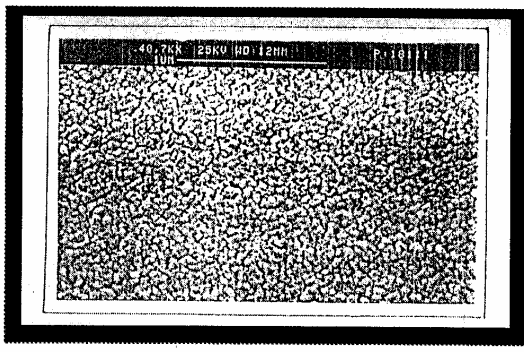


*Figure IV-a: Cross –section of the layer Obtained at 600°C*



*FigureIV: Micrography of the layer obtained at 600°C*

## Conclusion

We have chosen to study this system mixed of oxides $Al_2O_3$-$SiO_2$ for their interesting properties and interest which may bring in the domain of inorganic membranes chemical and mechanical high resistance.

The obtained results by adsorption /desorption of nitrogen showed that textural characteristics are linked to structural changes.

The deposit tests of thin layers in the macroporous suppot have showed homogeneous layers , without cracking with a thickness of 2um.

The sol-gel processes are particularly well suited at the realization of thin microporous layers.

The formation of $Al_6Si_2O_{13}$ (mullite) compound has been observed at a temperature 1350°C, lower than this obtained by classical method of powders(1550°C).