

## CHAPTER II

### LITERATURE REVIEW

#### 2.1 Reviewed Papers of Ceramics Membrane

Yoldas [3] characterized the effect of acid concentration on sol formation and the acid type which is suitable for the peptization of the hydroxide in 1975. It is shown that aluminum monohydroxide which was formed by the hydrolysis of aluminum alkoxide were peptizable to a clear sol when at least 0.03 mole of acid per mole hydroxide was added, and heat treatment temperature was above 80 °C for a sufficient time. In addition, the acid used must meet two general requirements: first, the anion of the acids must be noncomplexing with aluminum ions at these low concentrations. second, the acid must have sufficient strength to produce the required charge effect at low concentrations. Nitric, hydrochloric, perchloric, acetic, chloroacetic and formic acids meet these requirements.

In 1982, Yoldas [4] also showed that molecular structural variations can be introduced in oxide systems by controlled hydrolysis and polymerization reactions during the oxides formation from metal alkoxides: such as aluminum alkoxides,  $Al(OR)_3$ , were prepared by a nonliquid hydrolysis by atmospheric

moisture, hot-water hydrolysis (about 80 °C) , and cold-water hydrolysis (about 25 °C) followed by 24-hr aging at room temperature. These processes yield an amorphous monohydroxide, a crystalline monohydroxide, and a trihydroxide, respectively. The chemical and stoichiometric makeup of hydroxides and oxides is not fixed and can change rapidly as a function of molecular size in the region where the size is extremely small. The internal structural variations significantly alter the sintering behavior of oxide powders and affect their subsequent crystalline transformations.

In 1986, Leenaars and others [5] studied the effect parameters of a gel layer formed. They discussed that no gel layer was formed under any of the conditions when the sol concentration was lowered to 0.1 mol/litre. The effect of the pore size of the support and the type of acid used, are shown in Table 2.1. When boehmite particles were relatively large and the pore sizes of the support were relatively small, gel formation took place whereas this did not occur when the particles were small and the pores were large. The gel layer thickness increased linearly with the square root of the dipping time which was caused by increasing membrane layer thickness and for temperature from 400 °C up to 800 °C, pore growth was small and then remained constant after a long sintering period.

Table 2.1 Influence of Support (Pore Size) and Peptizing Acid on the Formation of Gel Layers During Dipping

Support Pore Size (type)	Peptizing Acid Used		
	HCl	HNO <sub>3</sub>	HClO <sub>4</sub>
1 (0.12 μm)	yes	yes	yes
2 (0.34 μm)	yes	yes	no
3 (0.80 μm)	yes	no	no

In 1988, Terstra and others [6] prepared the tubular ceramic membrane system which consisted of a substrate that was made by means of extrusion, and a microfiltration layer which was about 30 μm thick and which was applied by a film coating technique. This two layer system could also be used as a substrate for an ultrafiltration or gas separation membrane layer which was then also applied by a coating technique. The layer thickness could be adjusted by adjusting the suspension viscosity and increased with the square root of the contact time. The porosity of this layer which were characterized by mercury porosimetry was about 0.51. The permeabilities of these gases through a three layer membrane is a function of the mean pressure in the membrane.

Anderson and others [7], in 1988, also made TiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> ceramic membranes and analyzed the effect of adding chemicals in sols. Both TiO<sub>2</sub>

membranes were obtained from DCS and PMU sols. From the experiment, the DCS sols would be stable if the molar ratio of  $H^+$  (from the acid) to titanium was between 0.1 and 1.0. And the water concentration in the initial solution could control the viscosity of the PMU sol: the more water, the more viscous the solution. From Table 2.2, it indicates that the influence of water concentration upon gelling volume and gelling time.

Table 2.2 Influence of Water Concentration on Gelation

no.	$H_2O/Ti$ (mole ratio)	$H^+/Ti$ (mole ratio)	$C_3H_7OH/Ti$ (mole ratio)	Gelling time (hr)	Gelling volume (cm)
1	1	0.025	28	19.5	1.80
2	1.5	0.025	28	14.5	1.85
3	2	0.025	28	11.0	1.90
4	2.5	0.025	28	5.0	1.95
5	3	0.025	28	4.0	2.00
6	3.5	0.025	28	3.5	2.00
7	4	0.025	28	1.5	2.00

Unsupported  $Al_2O_3$  membranes which were obtained from DCS sols used sodium nitrate ( $NaNO_3$ ) as an inert electrolyte and phosphoric acid ( $H_3PO_4$ ) as a bridging anion. Pore size distribution (PSD) results showed that sodium nitrate concentration up to 0.01 M and phosphoric acid concentration up to  $10^{-3}$  M did not have any effect to the pore structure.

In 1992, Yu and Klein [8] prepared alumina membranes by using a sol-gel process. Thin gel layers of these membranes were grown onto porous alumina support tubes by using the permeation of water through the tube to

control the rate of hydrolysis and also studied effect of three alcohols (ethanol, 2-propanol and 2-butanol) by varied volume ratio of Aluminum-sec-butoxide solution to alcohol. This concluded that the optimum coating conditions were found to be an ABS to 2-propanol volume ratio of 1:25, for coating time between 10 and 15 sec, and partial drying times between 2.5 and 3 min, depending on whether the water was flowing in the tube or stored in the tube. The coatings remained crack-free and adherent when fired to 600 °C.

In 1993, Anderson and Sheng [9] prepared two methods of producing small particles and intended to avoid peptizing the alkoxide at high temperature. One method was based on the hydrolysis of alumina tri-sec-butoxide (ATSB) at temperature below 80 °C. After hydrolysis, the reactant was quickly heated to above 80 °C. The another one was begun by mixing the water phase into ATSB. From the experiment, it is indicated that both of these methods will produce colloidal sols containing a suspension or solution of very small alumina particles. Furthermore, the first method showed that sol was stable and clear but the second was not completely stable.

## **2.2 Reviewed Papers of $\alpha$ -Amylase Enzyme**

In 1984, Pichet Itkor [10] studied the production process of alpha amylase from *Bacillus amyloliquefaciens* KA 63 showed *B.amyloliquefaciens* KA 63 produced alpha amylase with the optimum pH for the activity at 6.0 in

0.02 M phosphate buffer at 55 °C. The suitable composition for the enzyme production contained 3% cassava starch and 4% defatted soybean as appropriate carbon and nitrogen sources, respectively and 0.15%  $\text{KH}_2\text{PO}_4$  , 0.01%  $\text{CaCl}_2$  , and 0.02%  $\text{MgSO}_4$  were also required. The optimal temperature and incubation time for cultivation were 30 °C and 72 hours, respectively, whereas the initial pH of the medium can be varied from 6.0 to 8.0. Under these conditions, one millilitre of fermented broth contained 465 units of saccharifying activity and 646 units of dextrinizing activity.(The unit of enzyme is amount of enzyme saccharify 1 mg of glucose within 1 minute at 50 °C, pH 6.9).

In 1989, Tsvetkov and Emanuilova [11] isolated  $\alpha$ -amylase from a continuous culture of a thermophilic strain of *Bacillus brevis* using ultrafiltration, ammonium sulphate precipitate, and gel filtration chromatography, respectively. This enzyme was purified eightfold and achieved 30 % yield . The pH and temperature optima of the purification enzyme were between pH 5.0 and 9.0 and at 80 °C.

In 1990, Bealin-Kelly and others [12] studied the purification of an  $\alpha$ -amylase from *Bacillus caldovelox* which produced both an  $\alpha$ -amylase and an  $\alpha$ -glucosidase and the ratio of extracellular  $\alpha$ -amylase/ $\alpha$ -glucosidase was highest when grown on the above optimized medium at 55 °C for 25 hours. Resolution and purification of the  $\alpha$ -amylase involved initial ammonium sulphate

precipitation, ultrafiltration followed by fast protein liquid chromatography Superose 12 gel filtration. Then the  $\alpha$ -amylase was purified 163-fold.

In 1990, Shah and others [13] studied about an economical  $\alpha$ -amylase preparation which was obtained from a high yielding stable mutant of *Bacillus subtilis* by qualitative and quantitative optimization of the ingredients of its production medium as well as production parameters. Further, it was upgraded by filtration and 15-fold concentration was obtained through an ultrafiltration membrane. Finally, its stabilization during storage at ambient temperature was attained using 20% sodium chloride and 0.1% calcium chloride as stabilizers. Results of the concentration preparation with stabilizers showed that 63% of the original activity after 26 weeks of storage could be obtained at ambient temperature, while the concentrated preparation without stabilizer showed less than 5% stabilization during the same period under identical conditions of storage. This stabilizer could be substituted by maintenance at 10 °C for comparable stabilization of concentrated  $\alpha$ -amylase preparation for 26 weeks. The stabilized enzyme preparation afforded efficient liquefaction of 35% tapioca starch at 76 °C in 30 minutes, comparable to the fresh preparation with or without concentration.