

## Preliminary study on Preparation and Characterization of Ceramic Membrane for Water and Wastewater Filtration

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**ABSTRACT.** Ceramic membranes are widely used in filtration as it was high chemical resistant, able to withstand high temperature and pressure, and has long life span. In this study, multilayer ceramic membrane (MLCM) was produced in three layers using titanium dioxide (TiO<sub>2</sub>), alumina and porous porcelain as top, intermediate and support layer, respectively. The thickness of top layer was vary by using different concentration/amount of TiO<sub>2</sub> ranging from 6 to 12 wt.%. Interlayer of MLCM was observed by using SEM while density and porosity was measured using Archimedes principle. Physical observation on samples shows a good formation of multilayer membrane without formation of cracks. SEM micrograph revealed that higher concentration of TiO<sub>2</sub> produce thicker top layer which is decrease in pore sizes. Density was increased while porosity was decrease with increment of TiO<sub>2</sub> concentration. As a conclusion, multilayer ceramic membrane without any cracks was successfully produced and greatly potential to be used for water and wastewater filtration.

**Keywords:** Ceramic membrane, Multilayer, Titanium dioxide, Filtration;

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### 1. INTRODUCTION

Water pollution was an increasing problem over the last few years. This is because of increasing in human population and increasing industrial activities. The progress of textiles, leather, surface treatment, mining, automotive and general chemical process industries lead to increase the concentration of colorant and dangerous ions of heavy metals not only in the wastewaters but even in drinking ones [1]. It can be quickly passed along the food chain and can cause various problems. Meanwhile, dyeing wastewater from the textile industry contains large amounts of dyestuff together with significant amounts of suspended solids, salts and trace metals. This dyeing wastewater can cause serious environmental problems due to its high color, large amount of suspended solids and high chemical oxygen demand. The best ways to solve this problem is by using the materials such as membrane filtration that can separate waste and water, and be reused for other benefits. Ceramic membrane can be designed in single or multilayers structure.

In multilayer ceramic membrane (MLCM), alumina normally used as support due to excellence mechanical and chemical properties. Ceramic membranes normally have an asymmetrical structure composed of at least two, mostly three, different porosity levels [2]. In order to reduce the surface roughness, a mesoporous has been applied first before applying the active micro porous top layer. The macro porous support ensures the mechanical resistance of the nano filtration process. These elements are grouped together in housings and these membrane modules can withstand high temperatures, extreme acidity or alkalinity and high operating

pressures that will make it suitable for many applications where polymeric and other inorganic membranes cannot be used. The development of such multilayer configuration includes, shaping of an appropriate support material, formation of mesoporous interlayers, and microporous top layer. Alumina, titania, zirconia or silica are considered as the main ceramic materials for the formation of the multi-layer structures [3]. Ceramic membrane permeability depends primarily on its porosity, pore tortuosity and morphology.

The effectiveness of the membrane is influenced by its structural properties for separation during the filtration process and it can be controlled during the fabrication of the membrane. The function of MLCM can be enhanced by incorporating  $\text{TiO}_2$  as top layer.  $\text{TiO}_2$  layer will act as organic pollution degradation by photocatalytic action in presence of light and give advantage over conventional filtration membrane. The aim of this research was to prepare and characterize the multi-layer ceramic membrane for water and wastewater filtration by using of  $\text{TiO}_2$  as top layer. The effect of different concentration of  $\text{TiO}_2$  toward physical and morphological structure of MLCM will be investigated in this study.

## 2. MATERIALS AND METHODS

### 2.1 Support layer

Commercial ready mixed porcelain cake which is supplied by Abbe Bay Sdn. Bhd. was used in this research. Porcelain cake was ground into powder and added with appropriate amount of water to form dough and shape to tubular form by using the extrusion method. The sample then dried at room temperature for 24 hours to ensure homogenous drying before heated in oven for  $70^\circ\text{C}$  for another 24 hours. Samples were sintered at  $900^\circ\text{C}$  for 2 hours holding time to produce tubular alumina support.

### 2.2 Interlayer Membrane

An intermediate layer was coated on the support layer in order to increase filtration ability. The powder suspension technique was being used to prepare the alumina ( $\text{Al}_2\text{O}_3$ ) intermediate layer. A deflocculated suspension of alumina can be obtained by mixing the alumina powder, binder and dispersing agent (aqueous solution). The ratio of the alumina powder to water containing binder and dispersing agent is 30:70 wt.%. The sample was being prepared by using dip coating method. After the sample was dried at room temperature for one day, the sample was heated in oven for  $70^\circ\text{C}$  to make sure the sample was totally dried before sintered at  $1000^\circ\text{C}$ .

### 2.3 Top Layer

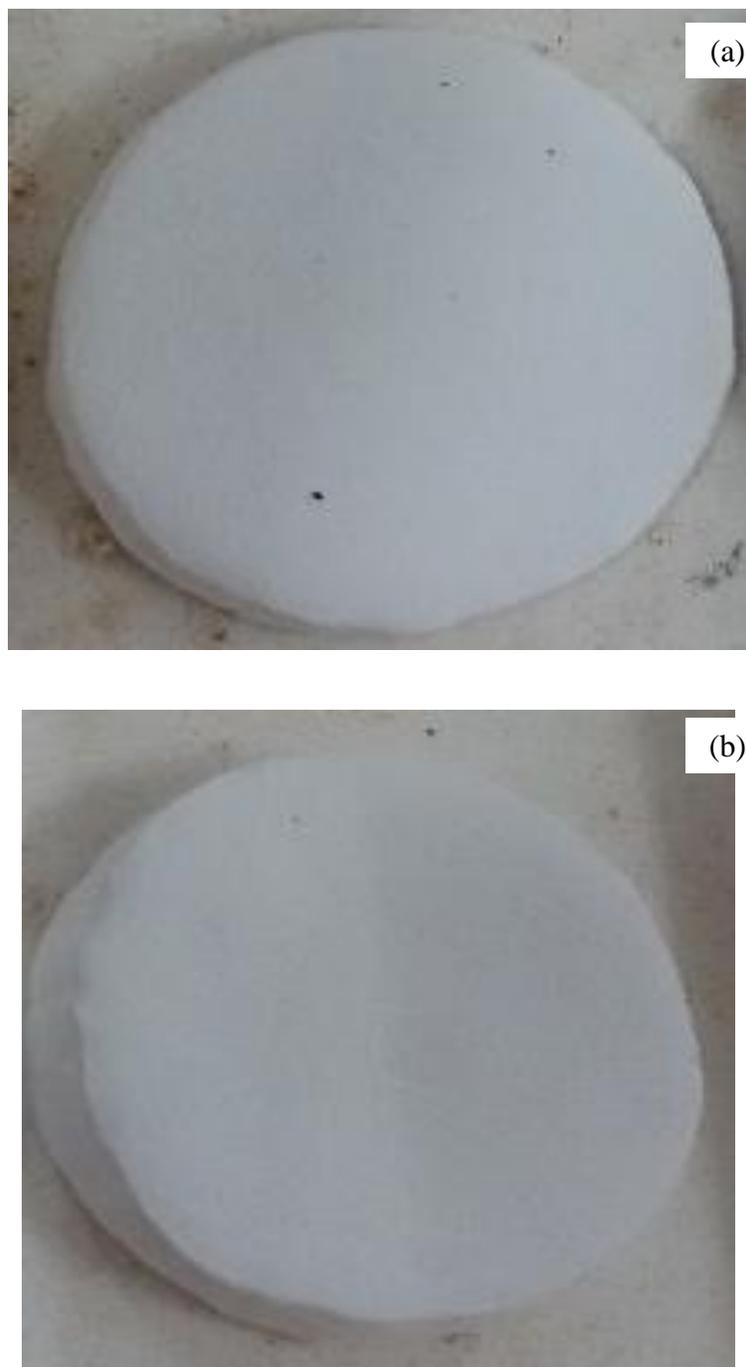
The preparation of top layer has been prepared by using pouring method with different concentration of  $\text{TiO}_2$  (6-12 wt.%) in 100 ml of solution containing binder and dispersant. The thickness of the layer is controlled by pouring for 1 min on the top of intermediate layer, the remaining solution was discarded. Sample then dried at room temperature for 24 hours and in oven for  $70^\circ\text{C}$  for another 24 hours before sintered at  $1000^\circ\text{C}$  as 2 hours holding time. The MLCM then characterized for density and porosity using Archimedes principle and morphological observation using SEM.

## 3. RESULTS AND DISCUSSION

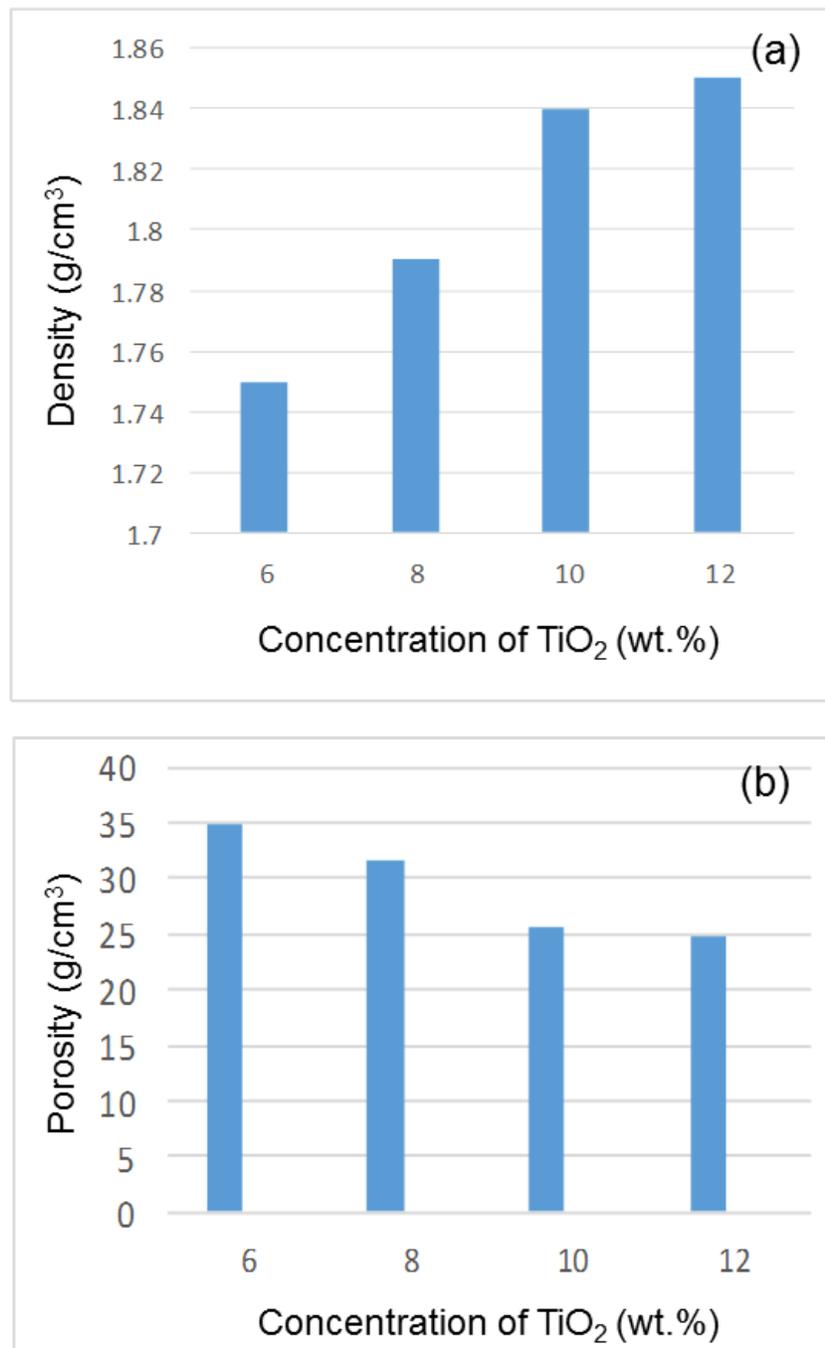
A good multilayer ceramic membrane was obtained without cracks for samples, example of sample produced at 6 and 12 wt.% of  $\text{TiO}_2$  are shown in Fig 1. It is clearly seen a good bonding between support, intermediate and top layers.

Density and porosity of MLCM was determined using Archimedes principle. It was found that the density of MLCM was increased from 1.75 to 1.85  $\text{g/cm}^3$  with increasing concentration of  $\text{TiO}_2$  from 6 wt.% to 12 wt.% as shown in Fig. 2(a). This is due to the increment in amount of  $\text{TiO}_2$  which contributed to mass and better packing arrangement of particles at higher concentration of  $\text{TiO}_2$  [4]. As the density increases, the amount of porosity of MLCM was decreased from 35% to 25% as shown in Fig. 2(b).

The amount of porosity in MLCM will influence the filtration of the membrane. For better filtration, the pore size distribution of the multi-layer ceramic membrane must be larger and this makes the volume of water more collected at the highest porosity [5].



**Fig. 1** Example of MLCM produces at (a) 6 wt.% and (b) 12 wt.% of  $\text{TiO}_2$



**Fig. 2** (a) Density and (b) Porosity of MLCM at different concentration of TiO<sub>2</sub>

Fig. 3 shows the morphology of fracture surface of MLCM. It can be seen the good bonding between layers was obtained without cracks. The thickness of the top layer was found increase with increasing concentration of TiO<sub>2</sub>. At 6 wt.% TiO<sub>2</sub> the thickness of top layer was around 20.6-20.7  $\mu\text{m}$ , compared to 41.0-56.2  $\mu\text{m}$  for 12 wt.% TiO<sub>2</sub>. The required thickness for membrane are normally between 1-50  $\mu\text{m}$  [6]. The thickness of the

layer can be controlled by the percentage of the powder added to the suspension and the coating time. The pore size of MLCM with 6 wt.% was larger (20-200  $\mu\text{m}$ ) compared to 12 wt.%  $\text{TiO}_2$  which is around 30-70  $\mu\text{m}$ . As previously describe, higher concentration of  $\text{TiO}_2$  produce better particle packing and also reduce the pore sizes and pore volume.

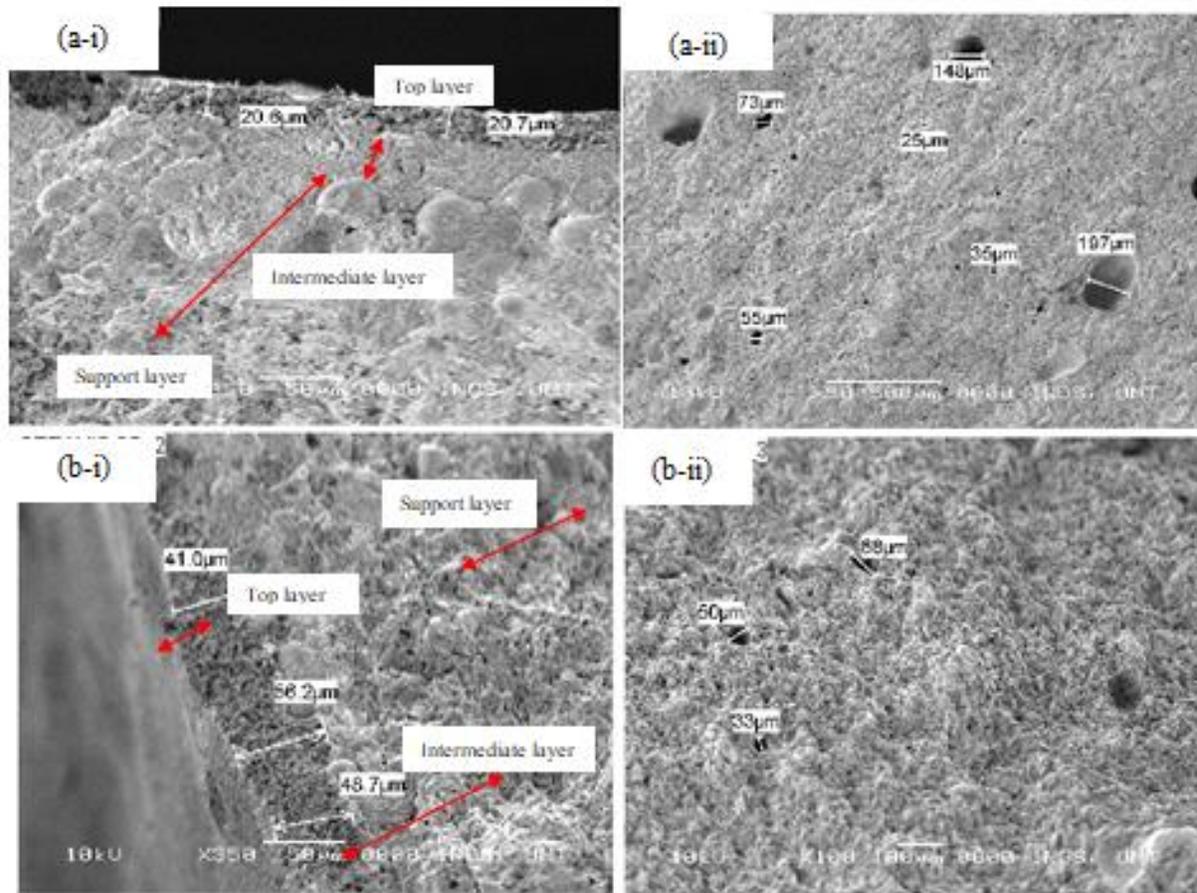


Fig. 3 Morphology of MLCM (a) 6 wt.%  $\text{TiO}_2$  and (b) 12 wt.%  $\text{TiO}_2$

#### 4. SUMMARY

MLCM was successfully produced without any crack between its interlayer. Increasing the concentration of  $\text{TiO}_2$  give better particles packing and increase the density of MLCM. 12 wt.%  $\text{TiO}_2$  produce smaller pore size and amount of porosity which is suitable for filtration under high pressure in the future study.

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