# INTEGRATION OF CERAMIC MEMBRANE THROUGH 3D PRINTING TECHNOLOGY

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# INTEGRATION OF CERAMIC MEMBRANE THROUGH 3D PRINTING TECHNOLOGY

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*I strongly dedicated this project to my beloved father (Lim Kiok Meng), mother (Rina Lestari) and my siblings (Lim Chin Siang and Lim Hui Kheng), with their sincere prayers and endless support afforded me to successfully accomplish this thesis.*

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## ABSTRACT

In this paper, 3D printing technology was being presented for its compatibility with ceramic materials due to its competitive process in terms of cost and speed, especially for the small quantities production. There were four types of ceramic membrane samples used in this study, which differ in their powder particle sizes and membrane shapes. They were 72µ without hole (1a), 72µ with hole (1b), 133µ without hole (2a), and 133µ with hole (2b). This paper presents the research effort that focuses on integration of ceramic powder with 3D printing technology in order to produce an effective ceramic membrane and characterize them on its physical, structural, and functional properties. Sample 1 has small particle size that results in small open (0.806cm$^3$) and closed porosity (0.808cm$^3$), which causes a higher bulk density (1.362g/cm$^3$) if compared with sample 2, which has the open porosity (0.919cm$^3$), closed porosity(1.127cm$^3$) and bulk density (1.351g/cm$^3$). The smaller particle forms an interconnecting structure that can trap the water molecules and increases the water absorption. The water absoption was 36.67% in sample 1 higher than that (33.24%) for sample 2. The 3D printing produces a ceramic membrane with an inhomogeneous structure which cause a deviation in its filtration rate. However, the membrane hole shape enhances the filtration rate by more than 50%, which is from 107.4ml/min to 171.1ml/min. The filtration rate was decreased with the treatment duration from 1 to 5 minutes due to the accumulation of particulate matters. The ceramic membrane with hole (1b and 2b) can improve the decreasing of filtration rate by 64.85% to 70.64% for particle size between 72µ to 133µ. The cleaning of the membrane was characterize by spectra detected by EDX and it shows an effectiveness in order to remove the accumulation of the particular matters after the backwash process. Among the samples, the ceramic membrane 1b has a higher efficiency in terms of chemical oxygen demand (COD) and total suspended solid (TSS), which were achieved 98.33% reduction in COD and 46.15% in TSS.

# ABSTRAK

Dalam kertas ini, teknologi percetakan 3D telah dibentangkan untuk keserasian dengan bahan-bahan seramik kerana proses ini kompetitif dari segi kos dan kelajuan, terutamanya untuk pengeluaran kuantiti yang kecil. Terdapat empat jenis sampel membran seramik digunakan dalam kajian ini, ia berbeza dari segi saiz zarah serbuk dan bentuk membran. Ia terdiri daripada 72μ tanpa lubang (1a), 72μ dengan lubang (1b), 133μ tanpa lubang (2a), dan 133μ dengan lubang (2b). Kertas kerja ini membentangkan usaha penyelidikan yang memberi tumpuan kepada integrasi serbuk seramik dengan teknologi percetakan 3D untuk menghasilkan membran seramik yang berkesan dan menguji membran dengan sifat-sifat fizikal, struktur, dan fungsi. Sampel 1 mempunyai saiz zarah yang kecil yang menyebabkan keliangan terbuka (0.806cm$^3$) dan keliangan tertutup (0.808cm$^3$) yang kecil, ia menyebabkan ketumpatan pukal yang lebih tinggi (1.362g/cm$^3$) jika dibandingkan dengan sampel 2 yang mempunyai keliangan terbuka (0.919cm), keliangan tertutup (1.127cm$^3$) dan ketumpatan pukal (1.351g / cm$^3$). Zarah yang lebih kecil mempunyai struktur bersambung yang boleh memerangkap molekul air dan meningkatkan penyerapan air. Penyerapan air adalah 36,67% dalam sampel 1 (72μ) lebih tinggi daripada sampel 2 (133μ) yang mempunyai penyerapan air sebanyak 33.24%. Percetakan 3D menghasilkan membran seramik dengan struktur tak homogen yang menyebabkan penyelewengan dalam kadar penapisan itu. Walau bagaimanapun, bentuk lubang membran meningkatkan kadar penapisan lebih daripada 50%, iaitu dari 107.4ml/min ke 171.1ml/min. Kadar penapisan telah berkurangan dengan tempoh masa rawatan tersebut daripada 1 hingga 5 minit disebabkan oleh pengumpulan partikulat. Membran seramik dengan lubang (1b dan 2b) boleh meningkatkan penurunan kadar penapisan daripada 64.85% kepada 70.64% untuk saiz zarah antara 72μ untuk 133μ. Pembersihan membran dicirikan oleh spektrum yang dikesan oleh EDX dan ia menunjukkan keberkesanan dalam usaha untuk menghapuskan pengumpulan partikulat tertentu selepas proses pencucian terbalik. Di

antara sampel-sampel itu, membran seramik 1b mempunyai kecekapan yang lebih tinggi dari segi Keperluan Oksigen Kimia (KOK) dan Jumlah Pepejal Terampai (JPT), iaitu boleh mencapai pengurangan sebanyak 98.33% untuk COD dan 46.15% untuk TSS.

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## LIST OF SYMBOLS

| | |
|---|---|
| 3DP | - Three Dimensional Printing |
| AM | - Additive Manufacturing |
| BSEs | - Backscattered Electrons |
| BSEs | - Backscattered Electrons |
| CAD | - Computer Aided Design |
| CAM | - Computer Aided Manufacture |
| COD | - Chemical Oxygen Demand |
| DLP | - Digital Light Processor |
| EDX | - Energy Dispersive X-ray |
| EDXA | - Energy Dispersive X-ray Analysis |
| EDXMA | - Energy Dispersive X-ray Micro Analysis |
| EPA | - Environmental Protection Agency |
| FDM | - Fused Deposition Modeling |
| FESEM | - Field Emission Scanning Electron Microscopy |
| HPDC | - High Pressure Die Casting |
| LRV | - Log Reduction Value |
| PLM | - Product Lifecycle Management |
| SEs | - Secondary Electrons |
| SiC | - Silicon Carbide |
| SLS | - Selective Laser Sintering |
| STL | - Stereolithography |
| TSS | - Total Suspended Solids |

# LIST OF APPENDICES

# CHAPTER 1

# INTRODUCTION

## 1.1 Background of Study

Three-dimensional printing (3D printing) is a technology which can convert 3D images from drawing into a physical object by using a printer. This technology has opened up the world to exciting possibilities. A lot of previous researchers have provided many benefits from the application of 3D printing technology such as product design, education, manufacturing, architecture, medical, and pharmaceutical sector. 3D printing has become a competitive strategy which involves product designing, customization, rapid prototyping, and creating a specific product. 3D printing technology application is further enhance by the decreasing costs of 3D printers, wide types of materials available, and the availability of 3D printing devices from different manufacturers (Brooks et al., 2014).

3D printable models can be created by a computer aided design (CAD) package or a 3D scanner. 3D printing can be used to produce a complex model such as printing toys, human implants, space flight components, and replacement parts. In this study, the ceramic membrane was produced by Z-Corporation 3D printer and additional indirect binder application method was applied to create sufficient strength for the green parts. The ceramic membrane was investigated for its efficiency through the water influent and effluent analysis. The material, kaolinite clay, is available in powder form, making them ideal to be used in powder-based 3D printing system.

This study explores the potential to fabricate the membrane from clay powder, wet it with printed liquid to turn it into its plastic form, before it dries to its green state. The green part produced can be increased its strength by applying the binder indirectly and further sintering up to 1300°C (Reay et al., 2011). The strength of bonded ceramic membrane is important to resist the pressure differential for the water medium across the ceramic membrane.

3D printing gives porosity to the ceramic membrane which builds up from the layer by layer basis. The effect of porosity may be influenced by its geometrical structure and microstructural properties (Beall et al., 2012). A high level of porosity may result in high specific area, high permeability, and high tortuosity. Porosity structure can be determined through its microstructural analysis. The porosity, particle density, and distribution structure will determine different filtration performance, thus should fully consider the structural factors.

The parameters used in this study were the powder sizes and membrane shapes. Powder sizes can be obtained from the original clay powder after sieving process while the membrane shapes can be obtained from the Unigraphic design software. Porosity of membrane is depending on the size of the powder. Different porosity will lead to different filtration rate and efficiency of the treatment. Moreover, filtration performance also depends on membrane shapes as it will lead to different surface areas.

## 1.2 Problem Statement

3D printing is a technology that employs an additive manufacturing process in which the printer deposits the materials based on the layer by layer manufacturing technologies in order to build up a different geometrical shape of 3D components (Withell et al., 2012). It is differ from the conventional subtractive manufacturing where 3D printing builds up components from nothing, to layer by layer, until the part is complete, whereas the conventional subtractive manufacturing removes the material from a solid block to create the desired part.

In the aspect of design and production for the materials which are supplied as powders, the material characteristics are the critical concern. The powder characteristics such as powder particle size, shape, and distribution will influence the resulting microstructure, which impacts the material properties (Petrick & Simpson, 2013). These has become a barrier to achieve a finished quality ceramic parts. Various factors such as powder sizes and membrane shapes are considered as independent variables to evaluate the ceramic membrane efficiency in this experimental work.

The traditional production method which can generate shape and microstructure of finalizing part consumes a lot of raw materials, labor, energy, and impose a higher cost for the shop-volume production (Simonis & Basson, 2012). Particularly, the casting or molding method requires the need for producing the molds and tooling as well as inflexible to create, modify, and improve the shape for various applications. Final parts can be limited by the capabilities of the tools used in the manufacturing processes.

Furthermore, the use of pore former to produce porous ceramic membrane provide additional cost of material to the manufacturers. As the manufacturing cost is high, the treatment and replacement cost for wastewater treatment raised as well. 3D printing technology suits to produce a low cost porous ceramic membrane and flexible to produce different membrane shape for different water treatment system application.

Water contamination is another problem, especially at rural areas and underdeveloped regions where the communities are lacking of capacity to receive the clean water. The environmental degradation occurs when pollutants are directly or indirectly discharge into the water bodies without adequate treatment to remove harmful components. The contaminants may include organic and inorganic substances. Some contaminants such as pathogens can produce waterborne diseases in either human or animal hosts. The ceramic membranes are effective to remove the bacteria and protozoa (Bielefeldt et al., 2010). They have been proven to remove 99.9% of microbiological contaminants (Malapane et al., 2012).

There are 780 million people around the world who are lacking in access to potable water and approximate 3.4 million people die each year from drinking unsanitary water supplies. According to Cooley et al. (2014), Asian rivers are three times higher than the global average results in bacteria levels from sewage and industrial waste. High demand for water treatment system from consumers is driven by the imposition of  government environment legislation.

Several water filtration technologies have led to educational initiatives, government, and non-government organizations to resolve the potable water contamination. Millions of these porous clay ceramic membrane are in use at numerous location in Africa, Asia, and South America (Plappally et al., 2011). 3D printing technology may provides benefits in the manufacture of porous ceramic membranes because it is easier to transport and set up in a short period of time and reduce the treatment costs.

## 1.3 Objective of The Research

The objective is crucial in providing a clear purpose and as a guide to the assessment strategies to achieve the goal of studies. There are three main objectives for this study, which are:

i. To determine the feasibility of 3D printing technology in order to produce a high efficiency ceramic membrane.

ii. To analyze the membrane structure and physical properties.

iii. To investigate the effect of powder sizes and membrane shapes towards the membrane efficiency.

## 1.4 Research Question

The research questions in this study are:

i. Is the 3D printing technology compatible with clay powder to produce a ceramic membrane with a better filtration efficiency?

ii. What are the membrane structures and physical properties of ceramic membrane produced by 3D printing technology?

iii. Is the clay powder sizes and membrane shapes has a significant effects on the membrane efficiency?

## 1.5 Scope

The scope of this study focused on:

1. The preparation of the ceramic membrane by using two different sizes of clay powders.
2. Characterization of different sizes of clay powders by Malvern Zetasizer Nano S.
3. Characterize the surface morphological of ceramic membrane using the field emission scanning electron microscopy (FESEM).
4. Identify the bulk density, water absorption, porosity, and filtartion rate of ceramic membrane fabricate at different parameters.
5. Chemical compositional analysis to characterize the backwash effect bu using the energy dispersive x-ray spectroscopy (EDX).
6. Testing the ceramic membrane efficiency by water filtration process in terms of chemical oxygen demand (COD) and total suspended solids (TSS).

## 1.6 Significant of Study

3D printing is a modern technology that can work with low cost ceramic materials to produce ceramic membrane in this study. The ceramic membranes are significantly to remove bacteria and protozoa (Bielefeldt et al., 2010). The ceramic membrane also have been historically proven to be effective in removing 99.9% of microbiological contaminants (Malapane et al., 2012). Once the contaminated water is introduced to one side of a ceramic membrane, the 3D printer will produce a porous structure of the ceramic material which could blocks the passage of anything larger than the pore size.

Moreover, to the best of the knowledge, the ceramic membranes are useful to remove the bacteria and protozoa from processed water in the microfiltration application, but still has a limited functionality of the membranes. The limitation is caused by the undesired growth of the bacteria cells through the membrane pores during long filtration times (Kroll et al., 2010). The efficiency may not be sustained after subsequent high levels of bacteria passing through the membranes. Some preventive measures for bacteria growth are suggested to be used for prolonged periods.

As refer to Figure 1.1, the collection efficiency for the printed ceramic membrane may significantly influenced by clay powder size and treatment particle size. Choi et al. (2014) demonstrated that the collection efficiency increased with increasing the treatment particle size and decreasing the silicon carbide (SiC) powder size at the filtration velocity of 1m/min. They found that the samples made with SiC100 and SiC 200 powders have low collection of efficiencies if compared with SiC10, 25, and 50. The results can be improved by avoiding the formation of pinholes and cracks for the samples.

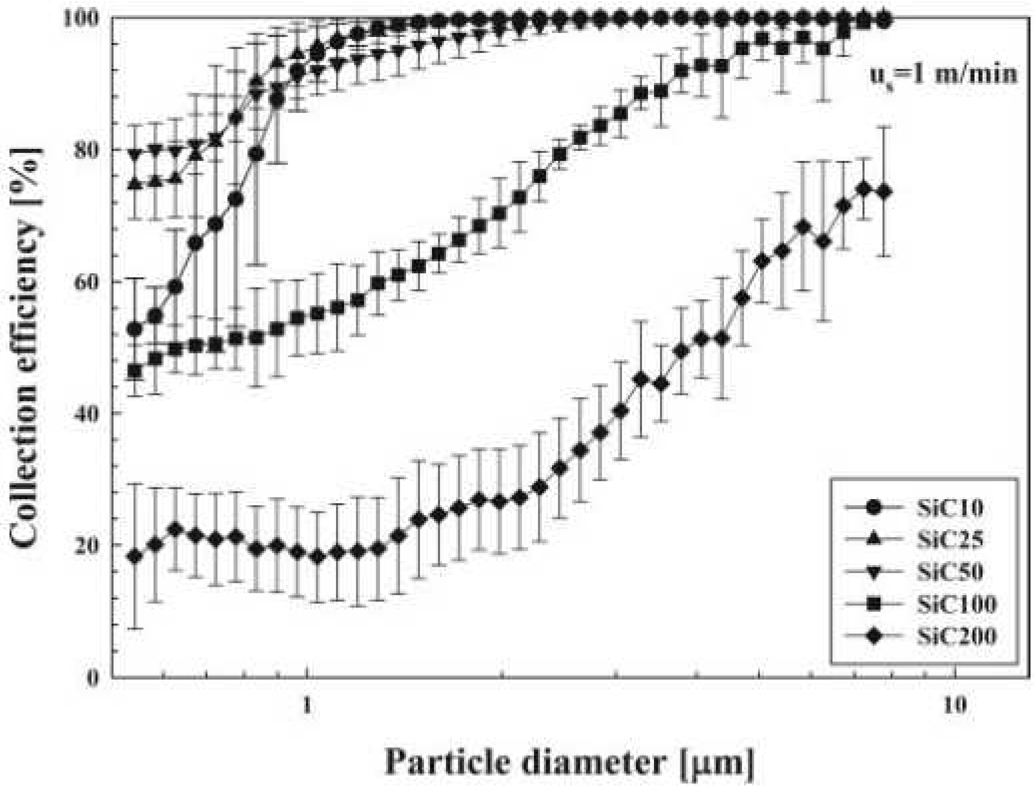


**Figure 1.1:** Collection efficiencies of ceramic membranes prepared with SiC powders of various sizes.

The significant of this study is due to the use of the low cost method which is one of the most promising technique to produce the ceramic membrane which can provide clean, safe, and affordable water for residential, industrial, and institutional use. The processing method for ceramic membrane which is simple and easy to set up benefits for commercial and pilot plant. The 3D printing requires fewer materials and less energy for the shaping process compared to the conventional casting and molding method. It is also sustainable because the materials can be reused and recycled.

A study made by Ulbrich et al. (2012) showed that 3D printing was faster and cheaper in terms of process time and material cost, compared to other rapid prototyping process. A comparison to describe the material cost consumed and processing time for each rapid prototyping technology is shown in Table 1.1. In today fast growing and competitive industry, the manufacturers favor to minimize their cost and time in order to maximize their profit, so that, the application of 3D printing technology will become another alternative trend.

**Table 1.1:** Comparisons for each rapid prototyping processes in terms of total material cost and processing time.

| No | Rapid prototyping processes | Total material cost | Processing time |
|---|---|---|---|
| **1** | Selective Laser Sintering (SLS) | $6.75 | 04hours07min |
| **2** | Fused Deposition Modeling (FDM) | $20.39 | 13hours47min |
| **3** | Digital Light Processor (DLP) | $15.22 | 06hours41min |
| **4** | Three Dimensional Printer (3DP) | $6.75 | 01hours00min |
| **5** | Polyjet | $42.90 | 03hours50min |

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