# SUGARCANE GREEN CERAMIC HOLLOW FIBRE MEMBRANE FOR OILY WASTEWATER SEPARATION

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*This thesis is dedicated to my father, who taught me that the best kind of knowledge to have is that which is learned for its own sake. It is also dedicated to my mother, who taught me that even the largest task can be accomplished if it is done one step at a time.*

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# ABSTRACT

Oily wastewater is one of the greatest problems in the world and needs an urgent solution. Therefore, this work describes the preparation of green ceramic hollow fiber membranes derived from waste sugarcane bagasse ash (WSBA) using phase inversion and sintering techniques. The first step in the production of WSBA was the calcination process of sugarcane bagasse at 800 °C followed by characterization of the microstructure, phases present, and thermal behavior using transmission electron microscopy (TEM), X-ray fluorescence (XRF), X-ray diffraction (XRD), thermogravimetric analysis (TGA), and Fourier-transform infrared spectroscopy (FTIR). This process was followed by the preparation of ceramic dope suspension consisting of WSBA powder as the main material, NMP as the solvent, PESf as the binder, and Arlacel P135 as the dispersant. In this work, various spinning parameters (effect of WSBA contents, bore fluid flow rate, air gap, and sintering temperature) were evaluated. By varying these parameters, significant effects on the membrane structure and mechanical strength were observed. The preliminary performance tests show that the green ceramic hollow fiber membrane prepared with a WSBA content of 60 wt%, bore fluid flow rate of 10 mL/min, air gap of 10 cm, and sintering temperature of 1000 °C induced a stable permeate water flux (PWF) of ~466.2 L/m$^2$h at the beginning of the filtration process. The investigation was further towards development of superhydrophobic and superoleophilic green ceramic hollow fibre membrane (ss-CHFM/WSBA) grafted with TEOS and MTES through sol-gel method for separation oily wastewater. Various grafting parameters such effect of grafting time, grafting cycle and calcination temperature were conducted towards membrane performance and morphology. The newly-developed ss-CHFM/WSBA induced excellent wettability properties with contact angle value of 161.1° at 60 min of grafting time, 3 cycle of grafting cycle and calcination temperature at 400 °C. In addition, it was also found that these parameters gave evenly nano-silica particle distribution on the surface. As a result, excellent oil flux (134.2 L/m$^2$h ) and oil separation efficiency (99.9%). Therefore, the optimization of oil condition in term of pH, oil concentration and oil temperature on the ss-CHFM/WSBA performance (oil flux and oil separation efficiency) was evaluated by using response surface methodology (RSM). The optimum ss-CHFM/WSBA performance was predicted at pH 10, 10.01 ppm for oil concentration and 69.04 °C for oil temperature. The verification result was found in acceptable average error at 4.71% for oil flux and 0.746% for oil separation efficiency.

# ABSTRAK

Sisa kumbahan berminyak merupakan masalah terbesar di dunia dan memerlukan penyelesaian segera. Justeru, kerja ini menerangkan penyediaan membran serat berongga seramik yang diperbuat daripada sisa buangan hampas tebu (WSBA) dengan menggunakan teknik penyongsangan fasa. Langkah pertama dalam penghasilan WSBA adalah proses kalsinasi sisa hampas tebu pada suhu 800 °C dan pencirian dari segi mikrostruktur dan fasa bahan menggunakan TEM, XRF, XRD, TGA, dan FTIR. Proses ini diikuti dengan penyediaan campuran seramik yang terdiri daripada WSBA sebagai bahan utama, NMP sebagai pelarut, PESf sebagai pengikat, dan Arlacel P135 sebagai pelincir. Parameter utama seperti kesan kandungan WSBA, kadar aliran bendalir, jurang udara, dan suhu sintering dikaji. Suhu sinter yang berbeza menghasilkan liang-liang berbentuk dalam struktur membran kerana pertumbuhan leher dan mekanisme lebur. Ujian prestasi permulaan menunjukkan bahawa membran seramik berongga hijau seramik yang disediakan dengan kandungan WSBA sebanyak 60% berat, menanggung kadar aliran bendalir 10 mL / min, jurang udara 10 cm, dan suhu sintering 1000 ° C disebabkan air berkadar stabil fluks (PWF) ~ 466.2 L/m$^2$h pada permulaan proses penapisan. Penyiasatan itu terus ke arah membran membran seramik berongga seramik hijau *superhydrophobic* dan *superoleophilic* (ss-CHFM / WSBA) yang dicelup dengan TEOS dan MTES melalui kaedah sol-gel untuk pengasingan air buangan berminyak. Pelbagai parameter cantuman seperti kesan mencantum, kitaran cantuman dan suhu penalaan dilakukan terhadap prestasi membran dan morfologi. Ss-CHFM/WSBA yang baru dibangunkan menunjukan sifat kelembapan yang sangat baik pada nilai sudut hubungan 161.1° pada 60 minit masa cantuman, 3 kitaran kitaran cantuman dan suhu kalembapan pada 400 ° C. Di samping itu, juga didapati bahawa parameter ini memberikan pengedaran zarah nano-silika sama rata pada permukaan. Hasilnya, fluks minyak yang sangat baik (134.2 L /m$^2$h) dan kecekapan pemisahan minyak (99.9%). Oleh itu, prestasi ss-CHFM / WSBA terhadap air sisa sebenar berminyak dari efluen kilang minyak sawit (POME), tempat cuci kereta dan restoran dipelajari selanjutnya. Oleh itu, pengoptimuman keadaan minyak dari segi pH, kepekatan minyak dan suhu minyak pada prestasi ss-CHFM / WSBA (kecekapan minyak dan kecekapan pemisahan minyak) telah dinilai dengan menggunakan kaedah permukaan respon (RSM). Prestasi optimum ss-CHFM / WSBA diramalkan pada pH 10, 10.01 ppm untuk kepekatan minyak dan 69.04 ° C untuk suhu minyak. Hasil pengesahan didapati dalam kesilapan purata yang boleh diterima pada 4.71% untuk fluks minyak dan 0.746% untuk kecekapan pemisahan minyak

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# LIST OF ABBREVIATIONS

| | | |
|---|---|---|
| AFM | - | Atomic force microscopy |
| COD | - | Chemical oxygen demand |
| CA | - | Contact angle |
| FTIR | - | Fourier-transform infrared Spectroscopy |
| MF | - | Microfiltration |
| NMP | - | N-Methyl-2-pyrolidone |
| PESf | - | Polyethersulfone |
| DMAc | - | Dimethylacetamide |
| RO | - | Reverse osmosis |
| SEM | - | Scanning electron microscopy |
| TEM | - | Transmission electron microscopy |
| TGA | - | Thermogravimetric analyzer |
| WHO | - | World Health Organization |
| WSBA | - | Waste sugarcane bagasse ash |
| CHFM | - | Ceramic Hollow Fibre Membrane |
| ss- CHFM | - | Superhydophobic and Superoleophilic |
| XRD | - | X-ray diffraction |

# LIST OF SYMBOLS

| | | |
|---|---|---|
| $A$ | - | Effective membrane area ($m^2$) |
| $R$ | - | Rejection (%) |
| $J$ | - | Permeate flux ($kg/m^2h$) |
| $\sigma$ | - | Mechanical strength (MPa) |
| $R_a$ | - | Surface roughness (µm) |
| $t$ | - | Time (min) |
| $L$ | - | Effective membrane length |
| $C_{m,f}$ | - | Solute concentration at feed solution (ppm) |
| $C_{b,f}$ | - | Solute concentration at bulk solution (ppm) |
| $\alpha_w$ | - | Membrane activity |
| $T$ | - | Temperature |

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# CHAPTER 1

# INTRODUCTION

## 1.1 Background of Study

Water is an essential element for human life that used in domestic usage, industry, agriculture, and many other fields. As the population and economies of developing countries expand, the demand of water will become greater. Competition for water resources among sectors and fields are already occurring. About 40% of the world's populations live in area that directly competes for shared transboundary water resources [1]. In the upcoming years, the global population is expected to grow and the demands on water resources will continuously grow. Clean water supply is the most important aspect in improving the health of the community. According to Gro Harlem Brundtland in World Health Organization (WHO) in World Water Day Report, some of the human rights include the safe water supply and adequate sanitation to protect health. Ensuring their availability would contribute infinitely to health and productivity for development [2].

The rate at which industries generate billions of gallons of wastewater containing a high concentration of organic matters is inevitable. This rate will continue to be persistent as more and more volumes of wastewater are produced during extractions, transport, in petrochemical plants, oil refineries, metalworking plants and specifically, in oil terminals during washing of reserving tank [3, 4]. The generated water effluent often contains micrometre-sized oil droplets dispersed in water, forming a stable oil-in-water emulsion even without any stabilizer [5]. Oil is an organic matter in wastewater and an important contaminants in wastewater that affected the environment severely [6]. It was reported that more than 2000 million tons of wastewater is produced by oil refineries in the world [7]. The conventional methods such as gravity separation, adsorption, oil skimmers, biological treatment and sedimentation in a centrifugal field has been applied in oily wastewater treatment [8]. However, these conventional methods have disadvantages such as low efficiency, high operation costs, and corrosion and recontamination problems. For instance, the method of using oil skimmers contaminate environment easily and the method of combustion also results in secondary pollution of water [9].

Superhydrophobicity is one of the extreme wetting states. Whereas, superoleophilicity is referred to the oil absorption capabilities of certain material. It is worth mentioning here that both of these behaviours are related to each others. Herein, this behaviour repels water strongly and exhibits an intimate relationship with oil-water separation. In recent years, with the in-depth studies of superhydrophobic and superoleophilic surface, researchers have started to explore materials and design coatings to fabricate oil-water separation surfaces with special wettability. In 2004, Feng et al. coated copper mesh with polytetrafluoroethylene (PTFE) to fabricate a mesh for oil-water separation as the first team [10]. Based on the inspiration, a lot of methods have been explored to solve the oil-water separation problem, for example, template method [11], sol-gel process [12], chemical etching [13], electrospinning [14, 15] and chemical vapor method [16, 17]. Herein, copper meshes are chosen as oil-water separation meshes by researchers widely because of its availability, low cost, and its porous structure with special properties. Unfortunately, one of the most serious disadvantages of copper mesh is its susceptibility to corrosion, that is, oxidation [18], resulting a shorter life.

Therefore, non-metallic substrates such as sponges, and textiles were chosen, replacing the copper mesh. For example, melamine sponges were used as core material for preparing porous materials possessing superhydrophobic and superoleophilic property for continuous separation of oils and organic solvents from water [19, 20] Zhang et al. fabricated smart textiles with switchable superoleophobicity and superoleophobicity by grafting a block copolymer comprising pH-responsive poly(2-vinylpyridine) and oleophilic/hydrophobic polydimethylsiloxane blocks on these materials [20]. Unfortunately, most sponges and textile fibres become completely rotten in three to five weeks. Due to this, recent studies have been focused on the stainless steel mesh as superhydrophobic and superoleophilic substrates for oily wastewater separation [21-23]. Because of the unique ability of stainless steel to resist corrosion, heat damage and chemical damage, high strength duplex grades provide added strength, allowing for a reduced thickness in the material, providing a high upfront cost than other types of substrates.

Membranes are the selective barriers normally used to separate two phases which allow certain substances to pass through and restrain other unwanted substances in a selective manner. Membrane technologies are more favourable as substrates as they are cost-effective and fast, highly selective, and flexible to be integrated with other processes. Many approaches taken by the researchers in the disinfection of water and found that the membrane technology is the best and suitable process for the treatment in comparison to others technique [24]. The separation process of the membrane will not change the phase and requires less space. It allows one component from a mixture to permeate the membrane freely, while hindering permeation of other components. In addition, researchers were reported that membrane separation at microfiltration stage is one of the method widely used geared toward the removal of oil impurities to minimize the adverse effect of oily wastewater. In general, membrane separation processes are classified into micro-filtration (MF), ultrafiltration (UF), nanofiltration (NF), reverse osmosis (RO). To be noted, MF are low-pressure driven operations and has been widely used for the separation process of oil from oily wastewater [25]. A number of researchers have explored the use of these processes for the removal of oil from oily wastewater [26]. In addition, microfiltration offers high flux but possesses a high risk of oil breakthrough during oily wastewater separation. Another researcher reported that MF has proven to be an efficient method in the treatment of oily wastewater mixtures due to its suitable pore sizes, sieving mechanism and the capability of removing emulsified oil droplets without any de-emulsification processes [27].

Among all MF membrane types, ceramic membranes present interesting advantages such as high thermal and chemical stability, high surface area and extended lifetime in oily wastewater treatment [28]. Many researchers reported that ceramic membranes are majority fabricated from particles of ceramic oxides such as $Al_2O_3$, $TiO_2$, $ZrO_2$ and $SiO_2$ [29, 30]. Early research in ceramic membrane fabrication is focused towards the utilization of α-alumina which is an expensive precursor to fabricate the membrane [31]. In addition, alumina has been used widely in ceramic membrane fabrication towards various separation technology. This is due to alumina membrane has received considerable attention for its use in separation processes because of its thermal, chemical and mechanical stability [28]. However, alumina membrane is considered to be in high cost. It was reported that alumina membrane were cost around \$1200-2000/$m^2$ which is more than half the price of polymeric membranes [29]. Still, ceramic membrane was in high consideration when come to other advantages.

Powder-like clay, dolomite, apatite, fly ash, natural raw clay and kaolin which are categorized as cheaper raw material have been used in ceramic membrane fabrication in order to reduce the cost production [31, 32]. Interestingly, recent research is orientated to the use of agricultural waste such as fly ash and rice husk ash for fabrication of green ceramic membrane. [32, 33]. Notably, most agricultural waste has been converted into ashes through calcination process and disposed of in landfills. However, the increasing refusal of communities to have landfills nearby, as well as the increased pressure from environmental agencies for proper waste management is creating the need to alternative final disposal consistent with environmental needs at a rational cost [34]. In fact, utilisation of ashes from agricultural waste for energy production, alternative materials replacing cement and extraction of silica have become increasingly important.

In ceramic membrane, there are three types of grafting process, which are immersion, chemical vapour deposition (CVD) and sol gel method. Immersion method is the simplest method but do not possessed any oil absorption capabilities. Meanwhile, CVD is dangerous method due to the thermal process involved. From literatures, it was reported that grafting via sol gel method is always applied for the application of oily wastewater separation. As stated by Pierre (2013), there are many definitions of sol gel process exist. For instance, sol gel process takes into account multicomponent oxides that are homogeneous at the atomic level. In fact, the term “sol gel” is restricted to the gels synthesized from alkoxides in which from colloidal dispersion or from metal alkoxides. In other word, grafting process through sol gel method can be defined as a colloidal route used to synthesize ceramics with an intermediate stage including a sol and/or gel state.

## 1.2 Problem Statement

Oily wastewater is generated by various industrial process operations such as petroleum industries, chemical and petrochemical plant, and oil refineries terminal during washing of reserving tank and metal working plants. This kind of pollution can affect groundwater, seawater, crop production drinking water as a result of the percolation of contaminants in produced water into the water resources, endangering aquatic resources, atmospheric pollution, destructing the natural landscape, and arising safety concern due to oil burner coalescence. In recent years, with the development of technology and imaging means, the lotus effect and water repellent legs of water striders have been revealed. These studies represent the starting point for the development of a plethora of artificial superhydrophobic surfaces by mastering the surface topography and chemical composition of various materials.

Among all hydrophobization process, sol−gel process on porous substrates has attracted a good deal of attention because of its advantages of superior homogeneity, low cost, low processing temperature, and simple operation process. Unfortunately, conventional substrates such as copper meshes, stainless steel, sponge and textile show drawbacks. Remarkably, ceramic membranes especially in hollow fibre configuration show potential advantages such as high thermal and chemical stability and long lifetime. However, commercially available ceramic membranes are made up from ceramic oxide such as alumina, zirconia, titania and mullite. To be noted, these materials induced high melting point, thus resulting in high sintering temperature. Recently, alternative materials from agricultural wastes such as rice husk, corn cob, palm oil fuel ash and sugarcane bagasse waste have received a significant amount of research attention. At the same time, environmental problem associated with the open dumping of agricultural waste can be minimized if it isutilized in proper way.

Sugarcane is an agricultural tree-free plants renewable resource, carbon neutral, higher rate of energy conversion etc. The lateral product of sugarcane after extracting minerals and milled is light yellowish particles known as bagasse composed of water, small cellulous fibres and cube sugar soluble mineral as well as fire cause due to fibres produced methane gas at certain circumstances. The bagasse is burned with various temperatures to prepare the ash properties for the development of composition, the increasing in temperature shows the percentage weight of $SiO_2$ is

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