FABRICATION AND CHARACTERIZATION OF FLY ASH HOLLOW FIBRE CERAMIC MEMBRANE FOR LOW LEVEL RADIOACTIVE WASTE TREATMENT

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ABSTRACT

Hollow fibre ceramic membrane has excellent properties that could perform in high pressure, harsh chemical and thermal resistance environment. However, ceramic material is quite costly and could become a drawback in the fabrication of ceramic membrane. The abundance and low cost of fly ash are the major factors for the present study to fabricate the fly ash hollow fibre ceramic membrane. Three fly ash loadings which were 40 wt %, 45 wt % and 50 wt % were fabricated as fly ash hollow fibre ceramic membrane (FA-HFCM). All the three different loadings were sintered at three different sintering temperatures which were 1150 ºC, 1250 ºC, and 1350 ºC to be characterised by employing three point bending test and scanning electron microscopy to evaluate their strength and morphology. All the three fly ash loadings had optimum characteristics at 1250 ºC sintering temperature. Surface roughness and pore size distribution for all three fly ash loading at 1250 ºC sintering temperature were characterized using atomic force microscopy and mercury intrusion porosimetry (MIP). The volumetric porosity, shrinkage, and density for all three loadings also were determined to support the result of bending strength. From the results obtained, the 45 wt % FA-HFCM was found to be the optimum fly ash loading due to its considerable strength of 37 MPa, less surface roughness and acceptable pore size distribution which was in the range of 0.22 μm to 10.33 μm, resulting in a better water permeability which was 29.02 L/m^{2}hr. Next, the 45 wt % FA-HFCM was sintered at three different sintering temperatures of 1150 ºC, 1250 ºC, and 1350 ºC for the investigation on crystallinity and pore size distribution by X- ray diffraction and MIP. The results show that higher pore volume for 45 wt % FA-HFCM at sintering temperature 1250ºC could provide higher surface area for the adsorbent to attract adsorbate and help reduce the resistance of water permeation. In addition, there was presence of phase crystallization of Mullite, Anorthite and Pyrophyllite which contributed to the ionic charge of silica and alumina to perform in the adsorption activity. The treatment of low level uranium in water was performed by 45 wt % FA-HFCM that was sintered at 1250 ºC in dual function mode which were adsorption and filtration methods. The result of zeta potential shows that FA-HFCM and uranium were present in negatively charged, but adsorption of uranium onto FA-HFCM in adsorption method shows that adsorption activity occurred between ionic charges of uranium and membrane. Removal of uranium was investigated by batch, and in the effect of pH from 3 – 8, both methods achieved higher removal of uranium at pH 3. Meanwhile, in the effect of contact time, the removal of uranium in filtration method achieved 98% efficiency in 4 hr, and adsorption method achieved 92 % efficiency after 3 hr. The study of kinetic models for adsorption activity was fitted with the pseudo-second order, and the FA-HFCM achieved adsorption capacity with 2.00 mg/g of uranium. This study concluded that characteristics of FA-HFCM as adsorbent membrane, successfully treated the low level uranium employing both adsorption and filtration method.

ABSTRAK

Membran seramik berongga mempunyai ciri-ciri istimewa yang membolehkan ia bertindak dengan baik dalam persekitaran bertekanan tinggi, bahan kimia berbahaya dan rintangan haba yang tinggi. Namun, kos bahan seramik yang tinggi menghadkan fabrikasi membran seramik. Limpahan abu arang dengan kos yang rendah menjadi faktor utama kepada kajian ini untuk fabrikasi membran seramik yang berongga berasaskan abu arang (FA-HFCM). Tiga berat abu arang iaitu 40 % berat, 45 % berat dan 50 % berat telah difabrikasikan sebagai membran seramik berongga. Ketiga-tiga membran telah disinter pada tiga suhu iaitu 1150 ºC, 1250 ºC dan 1350 ºC serta dicirikan dengan menggunakan ujian lenturan tiga titik dan mikroskop elektron imbasan untuk menilai kekuatan dan morfologi membran. Ketiga-tiga membran memperoleh sifat yang optimum pada suhu 1250 ºC. Kekasaran permukaan dan taburan saiz liang bagi ketiga-tiga FA-HFCM pada suhu 1250 ºC dikaji dengan menggunakan mikroskop tenaga atom dan porosimetri penerobosan merkuri (MIP). Isipadu keliangan, pengecutan dan ketumpatan bagi ketiga-tiga fabrikasi membran juga ditentukan untuk menyokong keputusan kekuatan lenturan. Berdasarkan keputusan yang diperolehi, FA-HFCM 45 % berat telah didapati menjadi muatan abu terbang optimum disebabkan kekuatan yang boleh dipertimbangkan iaitu 37 MPa, permukaan yang kurang kasar serta isi padu liang yang boleh diterima iaitu dalam julat 0.22 μm hingga 10.3 μm, dan menghasilkan kebolehan alir air yang lebih baik iaitu 29.02 L / m^2hr. Seterusnya, FA -HFCM 45 % berat disinter pada tiga suhu yang berbeza iaitu 1150 ºC, 1250 ºC dan 1350 ºC untuk mengkaji penghabluran yang berlaku dengan menggunakan pembelauan sinar-x dan taburan saiz liang dengan menggunakan MIP. Berdasarkan keputusan yang diperolehi, dengan isi padu liang yang lebih tinggi, FA-HFCM 45 % berat bukan sahaja menyediakan luas permukaan yang lebih tinggi bagi penjerap untuk menarik bahan terjerap, tetapi juga mengurangkan rintangan untuk kadar alir air. Di samping itu, terdapat kehadiran fasa penghabluran Mullite, Anorthite dan Pyrophyllite yang menyumbang kepada cas ionik silika dan alumina untuk melaksanakan aktiviti penjerapan. Air yang mengandungi uranium berkadar rendah telah dirawat oleh FA-HFCM 45 % berat yang telah disinter pada suhu 1250 °C dengan keadaan dwifungsi iaitu penjerapan dan penapisan. Keputusan keupayaan zeta menunjukkan FA-HFCM and uranium bercas negatif, namun terjerapnya uranium kepada FA-HFCM dalam kaedah penjerapan, membuktikan berlakunya aktiviti penjerapan diantara cas ionik uranium dan membran. Penyingkiran uranium dikaji dengan cara berkala, bagi kesan pH dari julat 3 – 8, kedua-dua kaedah mencapai penyingkiran uranium tertingggi pada pH 3. Manakala, bagi kesan masa rawatan, penyingkiran uranium dari kaedah penjerapan mencapai 92 % keberkesanan setelah 3 jam dan kaedah penapisan mencapai 98 % keberkesanan setelah 4 jam. Model kinetik dalam aktiviti penjerapan adalah mengikut tertib pseudo-kedua, dan FA-HFCM mencapai kapasiti jerapan sebanyak 2.00 mg/g per uranium. Kajian ini dapat disimpulkan bahawa sifat FA-HFCM sebagai membran penjerap, berjaya merawat uranium berkadar rendah dengan menggunakan kaedah penjerapan dan penapisan.

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LIST OF ABBREVIATIONS

AFM	-	Atomic Force Microscopy
Al_2O_3	-	Aluminium Oxides
CEC	-	Cation Exchange Capacity
DT	-	Decision Trees
EDX	-	Energy Dispersive X-ray
EDTA	-	Ethylenediaminetetraacetic acid
FA-HFCM	-	Fly Ash Hollow Fibre Ceramic Membrane
HFOMZ	-	Hydrous Ferric Oxide Modified Zeolite
HPGe	-	High- Purity Germanium
ILM – CEUF	-	Inorganic Ligand Modified, Colloid – Enhanced Ultrafiltration
SEM	-	Scanning Electron Microscopy
MIP	-	Mercury intrusion porosimetry
MWCO	-	Molecular Weight Cut Off
MWCNT	-	Multi- Walled Carbon Nanotubes
NMP	-	N-methylpyrrolidone
NORM	-	Naturally Occurring Radioactive Materials
PDADMAC	-	Poly (diallydimethylammonium) chloride
PESf	-	Polyethersulfone
pHpzc	-	Point zero charged of pH
TENORM	-	Technologically Enhanced Naturally Occurring Radioactive Materials
TGA	-	Thermal Gravimetric Analysis
TCC	-	True Coincidence Correction
TGR	-	Terrestrial Gamma Radiation
USEPA	-	United States Environmental Protection Agency
UNSCEAR	-	United Nations Scientific Committee
XRF	-	X–Ray Fluorescence
XRD	-	X–Ray Diffraction
YSZ	-	Yttria-Stabilized Zirconia
ZFAM	-	Zeolite–Iron Oxide Magnetic Nanocomposite

LIST OF SYMBOLS

Bq - Becquerel
Bq/L - Becquerel per litre
˚C - Celsius
Cfu/mL - Colony forming unit per millilitre
Ci - Curies
Da - Dalton
g - Gram
g/cm^3 - gram per centimetre cube
g/mg.hr - gram per milligram per hour
hr - Hour
Jw - Water flux
L/m^{2}hr - Litre per meter square per hour
mGy/hr - milligray per hour
mGy/min - milligray per minute
mg/g - milligram per gram
mg/L - milligram per litre
mCi/L - millicurie per litre
mBq/L - millibecquerel per litre
MPa - Mega Pascal
mV - Millivolts
nGy/hr - nanogray per hour
pCi/kg - picocurie per hour
ppb - part per billion
ppm - part per million
wt % - weight percentage

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CHAPTER 1

INTRODUCTION

1.1 Background

Nowadays, radioactive were successfully applied into developing industry especially in medicine, pharmaceutical, agriculture, research and education (Parfitt, 2007). The increasing demand of radioactive sources have indirectly increased the risk of radioactive pollution to the water. However, the risk could be reduced with a proper handling of the radioactive wastes. The radiation of the radioactive is determined by the value of Terrestrial Gamma Radiation (TGR) that is from direct measurement of adsorbed dose rate radiation from air and soil. According to the United Nations Scientific Committee (UNSCEAR, 2000), the recommended radiation value for the effect of atomic radiation was less than 0.1 mGy/min or 6 mGy/hr. Meanwhile for the maximum contaminated limit of uranium in water was stated by USEPA must be approximately not more than 0.03 ppm or 1 Bq/L (27 pCi/kg).

In Johor, the mean value for TGR dose rate was in the range from 9 nGy/hr to 1237 nGy/hr (Saleh *et al*., 2015). Meanwhile, in Pahang, report that the (TGR) dose rate from 640 locations in Pahang state had ionizing radiation with the mean value 176±5 nGy/hr (Gabdo *et al*., 2016). These ionizing radiations still under in recommended radiation. Specifically, the radioactive become more dangerous if exposed directly either by consume or inhaled, and indirectly which exposed to the human skin by radiation. Nevertheless, to avoid the effect of exposure from radiation, a proper procedure, such as the type of covering material used from the radioactive sources, the distanced from sources and contact time in handling the sources could protect human beings (Asfahani, Aissa and Al-Hent, 2016; Favre-Reguillon *et al*., 2008; Shen and Schafer, 2014). From UNSCEAR (2000), the report of Carcinoembryonic Antigen, was also claimed that uranium radiation is a hundred times weaker than its chemical toxicity, which is no different from the chemical danger imposed by common heavy elements such as Chromium, Lead and others.

When concentration level of radioactive increased from their original level natural sources due to human activities, it was classified as low-level radioactive pollution or Technologically Enhanced Naturally Occurring Radioactive Materials (TENORM). The TENORM (Hrichi, Baccouche and Belgaied, 2013). As could be seen, the concentration of uranium increased when the groundwater passed through the soil, sedimentary rock and granites, since all of them are the host for accumulations of uranium (Raff and Wilken, 1999). In addition, the TENORM of uranium wastes usually comes from several industries sector, particularly from industries that utilize radioactive material, uranium and metal mining, phosphate ore processing, and industry of petroleum (Egidi and Hull, 1999). Effect of radioactive exposure to human body could affect for lung disease and kidney failure (Bleise, Danesi and Burkart, 2003). The chronic physical illness is not apparent in short term due to it slow pathogenesis, but the diseases may appear many years later if the current risk is not properly addressed and controlled.

In Malaysia, there had two places that famous with bauxite mining which are Teluk Rumunia Johor and Kuantan, Pahang. In Kuantan, there are four water treatment plants located in the area of bauxite which are Bukit Goh, Bukit Sagu, Bukit Ubi, and Semambu Water Treatment Plant (Abdullah, 2016). Besides that, every year Malaysia will import rock phosphate, or phosphorite, to be used as feedstock for fertilisers. According to the Department of Statistics Malaysia, in 2018, Malaysia imported 357,531 tonnes of fertilisers containing phosphorite at a cost approximately RM114.93 million (Water Leach Purification a sustainable alternative for imported rock phosphate, Lynas says, 2019). Still, in Malaysia, there are no report for higher concentration of radioactive as other hazard metal, but some prevention such as water treatment for low level radioactive need to be consider before water pollution due to radioactive activities becomes worse. As reported by Friesen *et al*. (2009) that bauxite dust exposure could trigger for cancer, respiratory and circulatory disease.

Therefore, there are various method for treatments of radioactive wastewater included the conventional process such as adsorption, filtration, ion exchange, chemical treatment and evaporation was operated. However, many process and operation in conventional process attributed to unsatisfactory and unfriendly in removing the liquid radioactive waste (Ambashta and Sillanpaa, 2012; Grazyna and Chmielewski, 2004; Rahman *et al.*, 2011; Rana *et al.*, 2013). Thus, membranes technology such as ultrafiltration, nanofiltration, reverse osmosis and membrane distillation present a simple process in treatment of wastewater with low decontamination. (Oliveira, Barbosa and Afonso, 2013; Favre-Reguillon *et al.*, 2008; Zakrzewska-Trznadel and Harasimowicz, 2002; Liu *et al.*, 2016b; Montana *et al.*, 2013; Svittsov, Khubetsov and Volchek, 2011).

Next, other reviews also successfully proved the treatment of that uranium and other nuclides in wastewater by adsorption process (Villalobos-Rodriguez *et al.*, 2012; Nekhunguni, Tavengwa and Tutu, 2017; Gladys-Plaska, Grabias and Majdan, 2017; Sprynskyy, Kovalchuk and Buszewski, 2010; Wang *et al.*, 2016; Crawford, Lofts and Liber, 2017; Shuibo *et al.*, 2009; Abdi *et al.*, 2017). The reviews concluded that had several factors contributed to the increased of higher adsorption capacity of radionuclides, such as the amount of large surface area of adsorbent, efficiency of complexing agent, presence of strong ionic charge. Additionally, the effect of different pH and temperature also attributed to the increased of adsorption activity by increasing the electrostatic interactions between adsorbent and adsorbate. Nevertheless, all the adsorbent that successful adsorbed radionuclides demanded for post treatment to separate the clean water and pollutants.

Other than that, there also present some membrane filtration that need complexing agent as pre-treatment before going further for membrane filtration, either as ultrafiltration process (Wylie *et al.*, 2014; Babu *et al.*, 1994; Roach and Zapien, 2009) , nanofiltration process (Raff and Wilken, 1999), and reverse osmosis process (Shen and Schafer, 2014; Chen, Chu and Shieh, 1992). Most of them have declared that, to remove uranium by membrane filtration, it was needed a pre-treatment to the uranium wastewater before proceeding for filtration. Next, there also present some study that accomplished to remove heavy metal from water in one step process, which by using a one membrane to perform dual function as adsorbent membrane and membrane filtration. As had been proved by Hubadillah *et al*. (2017a). they were using rice husk hollow fibre ceramic membrane in the process to treat heavy metal and Adam *et al*. (2018), they were using zeolite hollow fibre ceramic membrane in order to remove chromium in water.

Advantages of membrane filtration as a low energy consumption, simple and easy to be manage can be used for almost any kind of separation (Van Rijn, 2004). Various driven forces were performed by membrane filtration in the way to accomplish their function in wastewater treatment such as temperature, pressure, electrical potential gradient and concentration gradient. According to Parfitt (2007) in his Technical Manual Book with title "Identification of Radioactive Source and Device", pressure-driven membrane processes are very well developed in nuclear industry for their radioactive waste treatment, meanwhile the present study introduced the adsorption and filtration method, which not only by pressure driven forces but also the effects of electrostatic interactions.

Other than that, there have two type of synthetic membrane which are organic and inorganic membranes. However, characteristics of organic membrane easily to deform and decompose when experienced a higher pressure and temperature (Ismail & Li, 2008b). Thus, scientific community make an alternative by developing the inorganic membrane in the form of ceramic, metal, graphite and glass (Richard W. Baker, 2012). Advantages of ceramic membrane can overcome the limitation of polymer membrane, but the elevated cost of ceramic material also been an issue in the production of ceramic membrane. Thus, there have a few researchers introduced a low-cost ceramic membrane material such as zeolite, bauxite, rice husk and fly ash (Bouzerara *et al*., 2009; Ghouil *et al*., 2015; Gorgojo *et al*., 2008).

1.2 Problem Statement

Utilization of radioactive help to development of technology in medical, education, and power plant. The increasing demand of radioactive sources have indirectly increased the risk of radioactive pollution to the water and effect to toxic food chain from animal and aquatic plant to the human body. It was causing the important organs in human body especially lung and kidney to lose its function.

Membrane technology had successfully reduced the radioactive in water as review by Rana *et al*. (2013). Nevertheless, all the membrane filtrations were made from a polymer material in which they were lose their properties after a long contact time with ionizing radiation then, undergo degradation with fast ageing (Grazyna and Chmielewski, 2004). In response to this problem, several studies had introduced fabrication of membrane filtration from ceramic membrane such as silicic carbide (Rosell, 2015), mesoporous clay (Abubakar *et al*., 2016). However, ceramic material is a costing and it could become a drawback to the fabrication of ceramic membrane.

Therefore, a few studies replaced the expensive ceramic material to the natural ceramic product such as bauxite, natural clay, kaolin and fly ash. The abundant and low cost of fly ash were the major factor for the present study to fabricate the hollow fibre ceramic membrane based on fly ash. Besides that, the higher of silica content in fly ash, directly, gives high value added to fly ash, to act as an adsorbent by the active sites of silanol. Unfortunately, some reviews claimed that fly ash consists an unburned carbon, it could affect the low mechanical strength of ceramic product as in case study of Valderrama, Agredo and Gutiérrez (2011). Therefore, in fabricating the FA-HFCM, the present study investigated the different fly ash loading and different sintering temperature, before the unburned carbon could affect the strength and porosity of FA-HFCM.

1.3 Objectives

The main objective of this study is to develop the hybrid adsorptive hollow fibre ceramic membrane based on fly ash via phase inversion technique for the treatment of low level uranium in water. Hence, the specific objectives of the study are outlined as follow: -

i. To produce a porous hollow fibre ceramic membrane based on waste mineral fly ash via phase inversion technique.

ii. To characterize the fly ash hollow fibre ceramic membrane in terms of phase transformation, morphological structures, pore size and porosity, permeability as well as physicochemical properties.

iii. To evaluate the performance of fly ash hollow fibre ceramic membrane for treatment of low level uranium in water via adsorption and filtration process.

1.4 Scopes of Study

In order to achieve the objectives of this study, the following scopes are drawn in order to achieve the objectives mentioned above: -

Fabrication of porous hollow fibre ceramic membrane based on waste mineral fly ash via phase inversion technique.

i. The fly ash powder with < 36 μm particle size was used in fabrication fly ash hollow fibre ceramic membrane (FA-HFCM) and the presence of chemical elements in fly ash powder was determined by using X-ray fluorescence (XRF) test.

ii. The hollow fibre ceramic membrane based on fly ash (FA-HFCM) were fabricated via dry wet spinning in phase inversion technique by using three different loadings of fly ash which are 40 wt %, 45 wt % and 50 wt % with three different sintering temperature which are 1150 °C, 1250 ºC and 1350 ºC.

In characterization of fly ash hollow fibre ceramic membrane in terms of phase transformation, morphological structures, pore size, porosity and permeability as well as its physicochemical properties.

i. The cross-sectional morphology and the mechanical strength of three loading of FA-HFCM at three different sintering temperature were characterized by using Scanning Electron Microscopy (SEM) and three-point bending strength, respectively.

ii. The three different loading of FA-HFCM at optimum sintering temperature were characterized for surface roughness by Atomic Force Microscopy (AFM), water permeability, volumetric porosity, and pore size distribution by Mercury porosimeter.

iii. The optimum loading in three different sintering temperature were characterized for phase transformation by X-ray diffraction (XRD), pore size distribution by mercury porosimeter and permeability of water flux.

iv. The presences of electrostatic charged on optimum loading of FA-HFCM at optimum sintering temperature was analyzed from zeta potential test.

In evaluation the performance of fly ash hollow fibre ceramic membrane for treatment of contaminated water by uranium ore as adsorption and filtration process.

i. The low level uranium in water was prepared by dissolving the 120 g of uranium ore for two weeks in order to achieve low level radioactive treatment which less 10^7 Bq. The electrostatic charged of uranium was determined from zeta potential test.

ii. The optimum loading of FA-HFCM at optimum sintering temperature was used for removal of uranium in adsorption and filtration method by varying the effect of pH in the range 3 to 8.

iii. The best pH condition for treatment of low level uranium solution was studied further for effect of contact time in the range 1 hr to 6 hr.

iv. The adsorption capacity and kinetic model that suited adsorption of uranium onto FA-HFCM was calculated.

1.5 Significance of Study

The significance of this study is to give a high value added in fly ash by utilizing it as ceramic material for hollow fibre ceramic membrane and increased the case study of ceramic membrane in research world. At the same time, utilization of fly ash also reduces water pollution from the abundant fly ash waste which is possible to contribute in water pollution due to its leaching process. By applying hybrid membrane for the treatment of radioactive waste, it reduces the treatment step especially by combining adsorption and filtration processes in a single application. Lastly, this project also gives contribution to the industries involving radioactive materials as they can shift their waste treatment with low cost and long-lasting membrane operation.

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