REMOVAL OF OIL IN OILFIELD PRODUCED WATER USING PHOTOCATALYTIC GRAPHITIC CARBON NITRIDE NANOFIBERS DEPOSITED ON CERAMIC MEMBRANE

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ABSTRACT

Oil and gas exploration and production generates billions of barrels of oilfield produced water (OPW) annually, thus making OPW the largest abundant by-product in oil and gas industry. Therefore, an efficient and effective separation of OPW has become a major challenge as it is a cornerstone to water management process that needs to meet regulatory standard for discharge and disposal to the environment. Membrane separation technology has previously delivered reliable performance of separation for treatment of OPW. However, severe fouling issues on the membrane has called for alarming an urgent technological advancement on membrane filtration. To address this concern, this study successfully synthesized a novel and potential membrane material to perform efficient and sustainable separation performances. Asymmetric ceramic hollow fiber membranes from Al_2O_3 precursor were fabricated using spinning technique based-phase inversion followed with high temperature sintering process. Al_2O_3 hollow fiber membranes were further coated with graphitic carbon nitride (GCN) incorporated polyacrylonitrile (PAN) nanofibers via direct electrospinning technique. GCN in bulk (bGCN) and nanosheets (nsGCN) configurations were synthesised from facile thermal decomposition of urea precursor. Meanwhile, bGCN was converted to nsGCN via liquid exfoliation method using isopropanol (IPA) to improve the photocatalytic properties of GCN. Decent morphological structure, well dispersed GCN, high specific surface area of nanofibers and large opening of nanofibers mesh that permitted oil droplet to permeate have been identified as the factors contributing to the excellent photodegradation of GCN nanofibers even at low loading of GCN at 1:10 to polymer ratio. Narrowed band gap energy of nsGCN as compared to bGCN, demonstrated enhancement on percentage degradation of NF-nsGCN on OPW under UV light irradiation at 96.6%. The results also revealed that synergetic effects of concentrate and degradation of oil molecules were the major important factors to obtain the high photodegradation efficiency of OPW. Due to outstanding features showcased by NF-nsGCN, in this study, self-supported photocatalytic nanofiber was coated on Al_2O_3 hollow fiber membrane surface using newly designed electrospinning technique to form hybrid photocatalytic nanofiber-coated membrane. Interestingly, NF-nsGCN/Al_2O_3 membrane established the highest pure water flux (PWF), OPW permeate flux, and oil rejection percentage at 816 $Lm^{-2}h^{-1}$, 640 $Lm^{-2}h^{-1}$, and 99%, respectively in 180 min-filtration. These findings concluded that sparse mesh-structure, high water affinity, and smooth morphology of nanofiber coatings, were the plausible parameters that significantly improve membrane performances. On top of that, NF-nsGCN/Al_2O_3 membrane also sustained the highest permeate flux (577 $Lm^{-2}h^{-1}$) and oil rejection (97%) in three cycle of filtrations, which confirmed the excellent cleaning performance of membrane in prolonged membrane operation. Excellent photodegradation ability of NF-nsGCN nanofiber permitted the nanofibers coating to in situ degrade the adsorbed oil contaminants under UV irradiation. Hence, it sustained high permeate flux and oil rejection of the membrane in repeating filtration system. In conclusion, this study recommends the potential application of the NF-nsGCN-coated Al_2O_3 hollow fiber membrane as the highly potential novel membrane for the treatment of industrial OPW.

ABSTRAK

Penerokaan dan pengeluaran minyak dan gas menjana berbilion tong air hasilan lapangan minyak (OPW) setiap tahun, menjadikan OPW sebagai produk sampingan terbesar dalam industri minyak dan gas. Oleh itu, pemisahan OPW yang cekap dan berkesan telah menjadi cabaran utama kerana ia merupakan asas kepada proses pengurusan air yang perlu memenuhi piawaian pengawal seliaan sebelum dilepaskan atau dilupuskan ke persekitaran. Teknologi pemisahan membran sebelum ini telah memberikan prestasi pemisahan yang berkesan untuk rawatan OPW, bagaimanapun, isu kotoran ke atas membran yang membimbangkan memerlukan teknologi penapisan membran yang lebih canggih dan segera. Untuk menangani masalah ini, kajian ini telah berjaya mensintesis membran baharu yang berpotensi untuk melakukan pemisahan yang cekap dan lestari. Membran asimetri gentian geronggong seramik dari prapenanda Al_2O_3 telah dibangunkan menggunakan teknik pemintalan penyongsangan fasa dan proses pensinteran pada suhu tinggi. Seterusnya, membran gentian geronggong Al_2O_3 disalut dengan gentian nano poliakrilonitril (PAN) bercampur grafiti karbon nitrit (GCN) melalui teknik langsung pemintalan elektro. Konfigurasi GCN secara pukal (bGCN) dan kepingan nano (nsGCN) disintesis daripada penguraian mudah termal oleh prapenanda urea. Sementara itu, bGCN ditukar kepada nsGCN melalui kaedah pengelupasan cecair menggunakan iso-propanol (IPA) untuk meningkatkan sifat fotobermangkin GCN. Struktur morfologi yang baik, penyebaran GCN yang baik, luas permukaan gentian nano yang tinggi dan bukaan gentian nano yang luas yang membolehkan titisan minyak dibenarkan untuk meresap, telah dikenalpasti sebagai penyebab kepada fotoperosotan sangat baik oleh gentian nano GCN walaupun pada kandungan rendah GCN pada nisbah polimer 1:10. Jalur jurang tenaga yang kecil oleh nsGCN berbanding dengan bGCN, menunjukkan peningkatan pada peratusan perosotan NF-nsGCN ke atas OPW di bawah sinaran cahaya UV pada 96.6%. Keputusan kajian juga menunjukkan, kesan sinergi oleh pemekatan dan perosotan molekul minyak adalah faktor utama untuk mendapatkan fotoperosotan OPW yang tinggi. Disebabkan ciri-ciri cemerlang yang dipamerkan oleh NF-nsGCN, dalam kajian ini, fotobermangkin gentian nano tanpa sokongan, disalut pada permukaan membran gentian geronggong Al_2O_3 menggunakan teknik pemintalan elektro yang baharu direka untuk menghasilkan membran hibrid bersalut fotobermangkin gentian nano. Menariknya, membran NF-nsGCN/Al_2O_3 menghasilkan fluks air tulen (PWF), flux resapan OPW, dan peratus penolakan minyak yang tertinggi, masing-masing pada 816 $Lm^{-2}h^{-1}$, 640 $Lm^{-2}h^{-1}$, dan 99% dalam masa penapisan selama 180 min. Penemuan ini menyimpulkan bahawa struktur jaring, afiniti air yang tinggi dan morfologi licin gentian nano, adalah parameter yang meningkatkan prestasi membran. Di samping itu, membran NF-nsGCN/Al_2O_3 juga mengekalkan fluks resapan yang tertinggi (577 $Lm^{-2}h^{-1}$) dan penolakan minyak (97%) dalam tiga kitaran penapisan dan mengesahkan prestasi pembersihan membran yang sangat baik. Keupayaan fotoperosotan yang sangat baik oleh gentian nano NF-nsGCN yang membolehkan salutan gentian nano untuk memperosot secara *in situ* cemaran minyak di bawah sinaran UV. Oleh itu, ia mengekalkan fluks resapan yang tinggi dan degradasi minyak dalam sistem penapisan membran yang berulang. Kesimpulannya, kajian ini mengesyorkan aplikasi gentian geronggong Al_2O_3 yang disaluti NF-nsGCN sebagai membran baharu yang sangat berpotensi untuk rawatan OPW di industri.

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LIST OF ABBREVIATIONS

AOPs	-	advanced oxidation processes
BET	-	Brunauer-Emmett-Teller
BETEX	-	benzene, toluene, ethylbenzene and xylene
BJH	-	Barrett, Joyner, Halenda (BJH) method
CB	-	conduction band
DTG	-	derivative thermogravimetric
EOR	-	enhance oil recovery
FESEM	-	field emission scanning electron microscopy
FTIR	-	Fourier transform infrared spectroscopy
GC-MS	-	gas chromatography-mass spectrometry
GCN	-	graphitic carbon nitride
GO	-	graphene oxide
LED	-	light-emitting diode
MD	-	membrane distillation
MF	-	microfiltration
MW	-	molecular weight
NF	-	nanofiltration
†N.A	-	not available
OPW	-	oilfield produced water
PAN	-	polyacrylonitrile
PES	-	polyethersulfone
PI	-	polyimide
PL	-	photoluminescence
PP	-	polypropylene
PMR	-	photocatalytic membrane reactor
PPM	-	part per million
PSA	-	polysulfone amides
PSF	-	polysulfone
PVA	-	polyvinyl alcohol
PVC	-	polyvinyl chloride

PVDF - polyvinylidene fluoride
PVP - polyvinylpyrrolidone
RhB - reactive black 5
RO - reverse osmosis
SEM - scanning electron microscopy
SiO_2 - silicon dioxide
SMPR - submerged membrane photocatalytic reactor
TEM - transmission electron microscopy
TGA - thermal gravimetric analysis
TiO_2 - titanium dioxide
TMP - transmembrane pressure
TOC - total organic carbon
UF - ultrafiltration
USEPA - united states environmental protection act
UV - ultraviolet
UV-Vis - ultraviolet visible
VB - valence band
XRD - x-ray diffraction
ZnO - zinc oxide
ZnS - zinc sulphide

LIST OF SYMBOLS

A - area of nanofibers
h_v - absorption of efficient photons
A_t - absorbance of OPW solution at initial time
A_t - absorbance of OPW solution at time t
T_{bulk} - average thickness of the bulk
E_g - band gap energy
ρ - density
F_y - drag force
D - droplet size of liquid medium
A_m - effective membrane area
ΔH_{mix} - enthalpy of mixing
C_i - concentration of OPW solution at initial
C_t - concentration of OPW solution at time t
J - flux membrane
L - length of nanofibers
F_L - lift force
γ_{lv} - liquid-vapor surface tension
w_d - mass of dry nanofiber
w_w - mass of wetted nanofiber
d_m - mean pore diameter
v_F - permeate rate of liquid medium
τ_w - shear stress of liquid medium
γ_{sl} - solid-liquid surface tension
γ_{sv} - solid-vapor surface tension
S_{BET} - specific surface area
δ - square root of the component surface energy
Δt - time
I_o - TOC intensity of OPW solution at initial
I_t - TOC intensity of OPW solution at time
V_{total} - total pore volume

η - viscosity of the liquid medium

ΔV - volume

ϕ - volume fraction

W - Watt

λ - wavelength of UV lamp

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Petroleum exploration and production (E&P) is associated with a major environmental challenge in handling large ‘so-called’ oilfield produced water (OPW) that is co-produced from subsurface geological formations (Zsirai *et al.*, 2016; Dickhout *et al.*, 2017; Johari *et al.*, 2020). Production of OPW is considered as the largest waste effluent for most oilfield platforms. Typically, the ratio of water to oil is approximately 3:1 for oil producing wells, and the ratio is higher for gas wells (Neff *et al.*, 2006). It is also reported that, twenty-one billion barrels of OPW were generated in 2007 by global onshore and offshore facilities with 87% from oil production activities (Pardue *et al.,* 2014). Overall it is estimated that more than 88 billion barrels of wastewater are produced yearly in the world from gas and oil production Rezakazemi *et al.*, (2017).

OPW contains an intricate composition of organic and inorganic mixtures, nitrogen compounds (nitrate, nitrite and ammonia), total dissolved solids, total suspended solids, naturally occurring radioactive materials (NORM), and biocides which is the chemical used to prevent fouling of oil pipelines (Alley *et al.*, 2011; Pardue *et al.*, 2014). Increasing volume of global OPW and effect of discharging large volume of OPW has become a significant environmental concern and more stringent environmental regulatory standard has been implemented. The annual average limit for discharge of dispersed oil for produced water into the sea has been set by the convention for the Protection of the Marine Environment of the North-East Atlantic (OSPAR Convention) at 40 mg/L (OSPAR, 2013).

Therefore, a holistic view on this matter has alarming the needs of efficient technologies to treat the OPW before it can be safely discharged to the environment. Furthermore, treating the vast amounts of OPW in a cost-effective way on sometimes remote locations (such as offshore platforms) also demands smart solutions so the water can be safely re-used for other purposes (Dickhout *et al.*, 2017).

Numerous physical, chemical biological methods, and combinations of these methods have been employed to treat the OPW (Macedonio *et al.*, 2014). Among the said methods, membrane technology has been claimed as a promising technology because it offers several advantages of being robust, lighter weight, small footprint, thus lower its installation costs (Duhon, 2012; Haiqal *et al.*, 2019; Johari *et al.*, 2020a). Moreover, membrane technology has emerged as a favorable separation approach due to its efficient separation on various contaminants especially on wastewater treatments. Membranes have been designed and configured in various shapes depending on its applications. However, from the industrial point of view, hollow fiber membranes own advantageous over flat sheet membranes due longer lifespan and high surface are per volume ratio by high packing densities (Huang and Arning, 2019). Besides that, hollow fiber membranes also easy to be handled in cross flow mode as compared to flat sheet membranes (Wan *et al.*, 2017). Hence, due to these advantages, hollow fiber membranes able to play a prominent role in the development of membrane technology for wastewater treatment applications.

Despite excellent properties, several pitfalls on membrane materials such as low mechanical, chemical, thermal resistance and limited mass transport has limit the membrane’s lifetime substantially (Chemistry, 2008; Hakami *et al.*, 2020). Furthermore, conventional membrane filtration is also facing serious challenges especially on membrane fouling and ineffective disposal of rejected organics (Wang *et al.*, 2013) (Yu *et al.*, 2016; Hammami *et al.*, 2017; Pourbozorg *et al.*, 2017; Pandey *et al.*, 2018; W. Li *et al.*, 2018; Fu, 2019; Pan *et al.*, 2019; Sun *et al.*, 2019; Tanudjaja *et al.*, 2019; Zhang *et al.*, 2020). In recent years, photocatalytic membranes have attracted enormous interests in the industry and academia due to their highly desirable multi-modal functionalities and innovative designs which can potentially overcome the drawbacks associated with conventional membranes (Dzinun *et al.*, 2016; Mamba, 2016; Zhang, *et al.*, 2016a; Zhang *et al.*, 2016; Molinari *et al.*, 2017; Argurio *et al.*, 2018a, 2018b; Tran *et al.*, 2019). Photocatalytic membrane integrates photocatalysis and membrane filtration techniques to produce a high quality of permeate. Photocatalysis is one of the advanced oxidation processes (AOPs) that has been intensive studied since the discovery of electrochemical photolysis of water by Fujishima and Honda in 1972 (Fujishima and Honda, 1972). In ideal photocatalysis process, photogenerated reactive oxygen species (hydroxyl radicals, superoxides) completely degrade most of organic pollutants. Rapid innovation in advancing photocatalytic membranes had successfully improved degradation and separation of organic pollutants from wastewater (Koe *et al.*, 2020; Liu *et al.*, 2020).

Moreover, it also shows a great potential as a low-cost, environmental friendly and sustainable treatment technology for industrial wastewater (Huang *et al.*, 2018; Guo *et al.*, 2019). Titanium dioxide, TiO_2 commonly used as photocatalyst to be incorporated as polymer-inorganic hybrid membrane (Ong *et al.*, 2014; Ong *et al.*, 2015; Dzinun *et al.*, 2015; Nor *et al.*, 2015, 2016; Mohamed *et al.*, 2016; Nor *et al.*, 2017). However, TiO_2 can only be activated at band gap (3.2eV for anatase crystal phase) and it requires activation by UV irradiation. In contrast, graphitic carbon nitride (GCN) has experienced a renaissance as a highly active photocatalyst although it being the oldest materials described by chemical literature (Liu *et al.*, 2016a). The versatile features, highly responsive to visible light and facile synthesis of bulk and nanosheet GCN, had spurred the interest to employ GCN for photocatalytic degradation of organic pollutant in wastewater (Tahir *et al.*, 2014; Xu *et al.*, 2015b; Azuwa *et al.*, 2018; Azuwa *et al.*, 2019; Azuwa *et al.*, 2019).

Emerging development of nanoscience and nanotechnology over the last two decades has discovered the importance of nanofibers which by virtue having extremely large surface area-to-volume ratio, high porosity, interconnectivity and can be an excellent material for membrane separation. Electrospinning specifically has been regarded as the most efficient technique to fabricate continuous nanofibers on a large scale and the diameter of the fiber can be altered from micrometers to nanometers (Fang *et al.*, 2011; Xin and Song, 2015). Previously, Xu *et al.*, (2015b) had reported that, electrospun photocatalyst nanofibers showed three times higher photocatalytic activities as compared to photocatalyst powders due to nanofibers meso-porosity and catalyst nanoparticle alignment and well dispersed in nanofibers. This was due to the efficient charge separation through inter-particle charge transfer along the nanofiber frameworks. Besides, polymer-based electrospun nanofibers also can acts as carrier for photocatalyst which provide dual effect of organic pollutant removal through physical adsorption or chemisorption and electrostatic attraction mechanisms. Hence, it is envisaged that an excellent treatment of OPW can be achieved using photocatalytic membranes. Development of photocatalytic membrane may include the electrospun photocatalyst polymer-based nanofibers coated on hollow fiber ceramic membrane surface. In a meantime, the optimization study of the photocatalytic membrane characteristics is essential towards development of efficient treatment of OPW.

1.2 Problem Statement

Massive production of oilfield produced water (OPW) from oil and gas production facilities has alarming an urgent need on effective technology to treat the OPW. Besides that, the ever-evolving and increasingly stringent regulatory standards imposing on OPW discharge limit that pose colossal environmental and economic implications, also demanding on more cost-effective treatment technology (Alzahrani and Wahab, 2014). The existing conventional membranes to treat OPW do not adequately satisfy the petroleum industry requirements in compliance with discharge and reuse standards. Furthermore, membrane fouling, which arises from the nonspecific interaction between membrane surface and oil contaminant, significantly impedes the efficient application of membrane technology (Drioli *et al.*, 2015). Therefore, preparing antifouling membranes is always a key fundamental strategy to deal with pervasive fouling problems from a variety of types of OPW.

In recent years, major advancements have been devoted in photocatalytic membranes preparation techniques and in elucidating the antifouling mechanisms of photocatalytic membrane processes, which integrates filtration and degradation of oil contaminants (Zangeneh *et al.*, 2018; Golshenas *et al.*, 2020; Liu *et al.*, 2020; Barati *et al.*, 2021; Zong *et al.*, 2021). In comparison with ceramic membranes, chemical stability of photocatalytic membranes from polymer material are poor in term of UV irradiation and resistances to the concomitant attack by photogenerated hydroxyl radicals (Athanasekou *et al.*, 2014). Therefore the outstanding features of ceramic membranes such as superior chemical stability and thermal resistance is necessary to maintain the robustness of the photocatalytic membrane system (Ahmad *et al.*, 2018; Golshenas *et al.*, 2020).

Graphitic carbon nitride (GCN) has drawn much research fascination as a nanomaterials based-membranes owing to its unique physicochemical characteristics (Seyyed *et al.*, 2020; Wang *et al.*, 2020). Immobilization of GCN photocatalysts in the membrane substrates have resulted in significant loss of photoactivity, caused by limited direct light absorption by the entrapped photocatalyst (Zhu and Wang, 2017; Argurio *et al.*, 2018). Furthermore, although membrane permeability were reported influenced by dosage of the entrapped photocatalyst (Ong *et al.*, 2015), high photocatalyst loading tends to decrease membrane permeability due to agglomeration. This agglomeration further forms clusters and occupy membrane pores and finally reduce the membrane pore sizes and decrease the flux (Ong *et al.*, 2016b; Xu and Antonietti, 2017; Zheng *et al.*, 2017; Salim *et al.*, 2018).

In order to bridge this research gap, electrospinning technique can be used to disperse GCN photocatalyst into polymer-based nanofibers, thus increase the active surface sites and enhance the photoactivity of GCN (Liang *et al.*, 2015; Jang *et al.*, 2018; Seyyed *et al.*, 2020). Polyacrylonitrile (PAN) is a compatible polymer with GCN that has a good spinnability due to its flexible polymer chain (Rahaman *et al.*, 2007; Zhang *et al.*, 2014; Sailah *et al.*, 2019; Soulis *et al.*, 2020). Moreover, previous works by GCN/PAN nanofibers have showed great performance on treatment of dyes such as Rhodamine B (Zhang *et al.*, 2015; Xu *et al.*, 2015b), and methylene blue (Suchitra and Udayashankar, 2017; Bairamis *et al.*, 2019). On top that, several excellent progress also have been reported on photocatalytic nanofiber coated membranes (Efome *et al.*, 2016; Nor *et al.*, 2016; Dobosz *et al.*, 2017; Su *et al.*, 2018). However, none has reported on development of GCN/PAN nanofibers coated hollow fiber ceramic membrane, thus there are still lack of inspiring and comprehensive research studies.

Therefore, this study is intended to close the gap on the development of excellent performance of hybrid GCN/PAN nanofibers coated Al_2O_3 hollow fiber membrane which will further contribute important insight towards the efficient technology for treatment of OPW.

1.3 Objective of Study

Based on the aforementioned research background and problem statements, the objectives of this study are:

1. To investigate the effects of synthesis condition of GCNs powder (bulk (bGCN) and nanosheets (nsGCN)), and GCN/PAN nanofibers towards physicochemical and thermal properties
2. To examine the photocatalytic performance of the produced GCNs powders and GCN/PAN nanofibers on OPW treatment.
3. To assess the membrane separation performance of the fabricated nanofibers coated ceramic hollow fiber membranes on OPW treatment.
4. To evaluate the prolonged separation performance of the fabricated hybrid GCN nanofibers coated Al_2O_3 hollow fiber membrane on OPW treatment.

1.4 Scope of Study

In order to fulfil the objectives of the study, the scopes of study have been outlined as below:

1. Synthesising two different configurations of GCN, bulk GCN (bGCN) and nanosheets GCN (nsGCN). The bGCN was synthesised via thermal decomposition of urea at different calcination temperature, 300 °C, 400 °C, 500 °C and 600 °C. Meanwhile, nsGCN were synthesised via liquid exfoliation of bGCN using five different solvents, isopropanol (IPA), N-methyl-

pyrrolidone (NMP), dimethylformamide (DMF), ethanol and water. The synthesised samples were characterized their physicochemical and thermal properties using FESEM, TEM, XRD, Raman spectroscopy, nitrogen adsorption/desorption analysis, FTIR, UV-vis-VIS, photoluminescence spectrophotometers and TGA. GCNs nanofibers were fabricated from polymer dope solution containing polyacrylonitrile (PAN) at different loading of bGCN (0 wt%, 10 wt%, 15 wt% and 20 wt%) via Nanofibers Electrospinning Unit. The physicochemical and thermal properties of the fabricated GCN/PAN nanofiber samples were investigated using FESEM, TEM, FTIR and TGA. The distribution of nanofibers diameter was analysed using Gaussian function approximation (Origin software).

2. Determining the photocatalytic performance of bGCN and nsGCN powder and nanofibers on the hydrocarbon removal from OPW by evaluating the adsorption and degradation rate of organic compound of the treated OPW using the UV-vis spectrophotometer. Photocatalytic experiment was carried out in a suspension mode photocatalytic reactor. The suspension solution containing synthetic OPW sample at 1000 ppm and photocatalysts were placed inside the beaker and further irradiated using two type of light irradiation sources, these are ultraviolet (UV) lamp (Vilber Laurmat, $\lambda = 312$ nm, 30 watt) and white light-emitting diode (LED) flood light (CSFL-30W, $\lambda > 420$ nm, 30 watt).

3. Assessing the membrane separation performances of the fabricated nanofibers coated Al_2O_3 hollow fiber membranes. Asymmetric Al_2O_3 hollow fiber membranes were fabricated from Al_2O_3 via spinning technique based-phase inversion followed with sintering process at 1400 °C in a tubular furnace. The morphological structure and wettability analysis of the fabricated Al_2O_3 hollow fiber membrane were examined using FESEM and contact angle goniometer. After that PAN and GCNs nanofibers were further coated with Al_2O_3 hollow fiber membranes via a newly designed direct electrospinning technique. The surface and cross-section morphology of nanofiber coated Al_2O_3 hollow fiber membrane were assessed using FESEM. The membrane separation performances were assessed based on the hydrocarbon removal from OPW (1000 ppm) using a benchtop cross-flow membrane filtration setup. The membrane stability were evaluated in three cyclic filtrations at pressure of 2

bar and 180-min duration for each of the cycle. Membrane cleaning were performed under UV irradiation before the new cycle was started. The oil rejection percentage were examined using UV-vis spectrophotometer and chromatography–mass spectroscopy (GC–MS).

4. Evaluating the photocatalytic-separation performances of the fabricated hybrid GCN nanofibers coated Al_2O_3 hollow fiber membranes using a self-fabricated hybrid photocatalytic membrane filtration system for removal of hydrocarbon in OPW. The PWF and OPW permeate flux of NF-nsGCN/Al_2O_3 membrane were operated using filtration with UV and without UV irradiation. Lastly, prolonged membrane filtration performance under UV irradiation were conducted using the same filtration setup. The OPW permeate flux and oil rejection were evaluated using different OPW feed concentrations of 100, 200, 500 and 1000 ppm at operating pressure of 2 bar for 5 h duration. The oil rejection percentage were examined using UV-vis spectrophotometer.

1.5 Significance of Study

The ever-evolving and increasingly stringent environmental rules and regulations on the discharge of OPW have been applied, since it poses colossal environmental and economic implications. Hence, there is a sense of urgency alarming and oil producers are also being compelled to establish a promising solution using membrane technology in order to facilitate the treatment of immensely volume of OPW discharged yearly. Adopting by massive development on nanomaterials, GCN as the oldest material describe in the chemical literature has recently experienced renaissance as a highly active photocatalyst with visible-light photo-responsive which led to tremendous endeavours on its potential to degrade organic pollutants. Along with that, the remarkable morphological properties of electrospun nanofibers which possess high surface area, high porosity, ultrafine continuous fibres, and variable pore-size distributions which can serve as the best supported photocatalyst for GCN has consequently overcome the disadvantageous of agglomeration and subsidence of GCN powders and resulted in significant photocatalytic capability. Inspired by pioneering works on photocatalytic membranes, nanoarchitecture of a novel hybrid of GCN nanofibers/alumina hollow fiber photocatalytic membrane will be an intensive practical medium to enhance the hydrocarbon rejection by employing integrated photocatalytic and separation processes with a potential application under sunlight.

Thus, this research may assist in advancing the industrialization of membrane technologies for treatment of OPW by shifting the current view from OPW as merely a source of pollution to its role as a renewable water resource which particularly associated with water-stress countries. The significant findings of this research also will help to address the current challenges as well as provide critical perspectives for the future development of membrane technology for treatment of OPW especially in onshore and offshore facilities.

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1. **Alias, N. H,** Jaafar, J., Samitsu, S., Ismail, A. F., Mohamed, M. A., Othman, M. H. D., A Rahman, M., Othman, N. H., Nor, N. A. M., Yusof, N., Aziz, F. (2020) 'Mechanistic insight of the formation of visible light responsive nanosheet graphitic carbon nitride embedded polyacrylonitrile nanofibres for wastewater treatment', *Journal of Water Process Engineering*, 33, pp. 101015. **(Q1, IF : 3.173)**

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7. Syafikah Huda Paiman, Mukhlis A.Rahman, Tetsuo Uchikoshi, Nik Abdul Hadi Md Nordin, **Nur Hashimah Alias**, Norfadhituladha Abdullah, Khairul Hamimah, Abas, Mohd Hafiz Dzarfan Othman, Juhana Jaafar, Ahmad Fauzi, I. (2020) 'In situ growth of α-Fe_2O_3 on Al_2O_3-YSZ hollow fiber membrane for oily wastewater', *Sep. and Pur. Tech.*, 236, p. 116250. **(Q1, IF: 5.107)**

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