

**FABRICATION OF SAND/ZINC OXIDE-BASED  
NANOCOMPOSITE VIA SOL-GEL  
IMMERSION METHOD FOR  
PHOTOCATALYSIS  
APPLICATION**

**NUR JANNAH BINTI IDRIS**

**UNIVERSITI PENDIDIKAN SULTAN IDRIS**

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## ABSTRACT

This study aimed to fabricate sand/zinc oxide (ZnO) nanorods (NRs)-based nanocomposite via sol-gel immersion method with titanium dioxide (TiO<sub>2</sub>) and graphene oxide (GO)-based materials for methylene blue (MB) dye degradation. The nanocomposite photocatalyst was initially fabricated by growing ZnO via sol-gel immersion followed by synthesizing TiO<sub>2</sub> using hydrothermal method on the sand as a substrate. Different concentration and synthesis time were used as parameters for the fabrication. These nanocomposites were then hybridized with GO and GO\_multi-walled carbon nanotubes (MWCNTs) hybrid solution via immersion method. Prior to hybridization, the initial GO was synthesized using electrochemical exfoliation method assisted by commercially available single-tail sodium dodecyl sulphate surfactant and was further mixed with MWCNTs to form GO\_MWCNTs hybrid solution. The sand/ZnO, sand/ZnO/TiO<sub>2</sub> nanocomposites, and sand/ZnO/TiO<sub>2</sub>/GO-based photocatalyst materials were then characterized by using ultraviolet (UV)-light irradiation within three-days interval for MB dye degradation, field emission scanning electron microscopy (FESEM), micro-Raman spectroscopy and ultraviolet-visible spectroscopy (UV-vis). The finding, sand/ZnO NRs (4h) presented the highest photocatalysis performance (92.64%) as compared to sand/ZnO/TiO<sub>2</sub> nanocomposite and sand/ZnO/TiO<sub>2</sub>/GO-based photocatalyst materials. This was due to high density and active sites presented by sand/ZnO NRs (4h) which lead to higher adsorption of MB molecules on its surfaces. As for the conclusion, sand/ZnO NRs (4h) demonstrated a potential ability to be applied as a photocatalyst material to degrade MB solution. The implication of this study is a novel, simpler, low-cost and green approach for the production of sand/ZnO, sand/ZnO/TiO<sub>2</sub> nanocomposites, and sand/ZnO/TiO<sub>2</sub>/GO-based photocatalyst materials for photocatalysis application.





## FABRIKASI NANOKOMPOSIT BERASASKAN PASIR/ZINK OKSIDA MELALUI KAEDAH RENDAMAN SOL-GEL UNTUK APLIKASI FOTOPEMANGKINAN

### ABSTRAK

Kajian ini bertujuan untuk menghasilkan nanokomposit berasaskan pasir/zink oksida (ZnO) nanobatang (BtG) melalui kaedah rendaman sol-gel dengan titanium dioksida ( $\text{TiO}_2$ ) dan bahan berasaskan grafin oksida (GO) untuk degradasi pewarna metilena biru (MB). Nanokomposit fotopemangkin pada awalnya dihasilkan dengan menumbuhkan ZnO melalui rendaman sol-gel diikuti dengan mensintesis  $\text{TiO}_2$  menggunakan kaedah hidroterma di atas pasir sebagai substrat. Kepekatan dan masa sintesis yang berbeza digunakan sebagai parameter untuk fabrikasi. Nanokomposit ini kemudiannya dihibridisasi bersama GO dan GO\_nanotiub karbon berbilang dinding (NTKBD) larutan hibrid melalui kaedah rendaman. Sebelum hibridisasi, awalnya GO disintesis dengan menggunakan kaedah pengelupasan elektrokimia yang dibantu oleh komersial rantaian tunggal sodium dodesil sulfat dan selanjutnya dicampurkan dengan NTKBD untuk membentuk larutan hibrid GO\_NTKBD. Pasir/ZnO, pasir/ZnO/ $\text{TiO}_2$  nanokomposit, dan pasir/ZnO/ $\text{TiO}_2$ /berasaskan-GO kemudiannya dicirikan dengan menggunakan sinaran sinar ultraviolet (UV) selama tiga hari untuk degradasi pewarna MB, pemancaran medan mikroskopi imbasan elektron (FESEM), spektroskopi Raman-mikro dan spektroskopi ultraungu-tampak (UV-vis). Dapatan kajian, pasir/ZnO BtG (4j) menunjukkan prestasi fotopemangkin yang tertinggi (92.64%) berbanding dengan bahan fotopemangkin pasir/ZnO/ $\text{TiO}_2$  nanokomposit, dan pasir/ZnO/ $\text{TiO}_2$ /berasaskan-GO. Ini disebabkan oleh kepadatan dan tapak aktif yang tinggi ditunjukkan oleh pasir/ZnO BtG (4j) yang membawa kepada penjerapan molekul MB yang lebih tinggi pada permukaannya. Sebagai kesimpulan, pasir/ZnO BtG (4j) menunjukkan kemampuan potensi untuk digunakan sebagai bahan fotopemangkin untuk degradasi larutan MB. Implikasi kajian ini adalah pendekatan baru, lebih mudah, kos rendah dan hijau bagi penghasilan bahan fotopemangkin pasir/ZnO, pasir/ZnO/ $\text{TiO}_2$  dan pasir/ZnO/ $\text{TiO}_2$ /berasaskan-GO untuk aplikasi fotopemangkin.



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## LIST OF ABBREVIATION

$\text{Bi}_2\text{WO}_6$	Russellite
C	Carbon
CB	Conduction Band
CBD	Chemical Bath Deposition
CdS	Cadmium Sulfide
$\text{Cl}^-$	Chloride Ion
$\text{Co}_3\text{O}_4$	Cobalt (II,III) Oxide
CVD	Chemical Vapor Deposition
D	Defect and Disorder Peak
DI-water	De-Ionized Water
DWCNTs	Double-Walled Carbon Nanotubes
EDX	Energy Dispersive X-Ray
$E_g$	Band Gap Energy
eV	Electron Volt
FESEM	Field Emission Scanning Electron Microscopy
G	Crystalline Graphite Peak
GO	Graphene Oxide
$\text{H}^+$	Hydrogen Ions
HCl	Hydrochloric Acid
HMT	Hexametylenetramine
$I_D/I_G$	Ratio of D and G peak

M	Molarity
MB	Methylene Blue
MO	Methylene Orange
MWCNTs	Multi-Walled Carbon Nanotubes
NH <sub>3</sub>	Ammonia
NRs	Nanorods
NFs	Nanoflowers
OH <sup>-</sup>	Hydroxide Ions
<i>OH</i> ·	Hydroxide Radicals
ppm	Parts Per Millions
PVD	Physical Vapor Deposition
RhB	Rhodamine Blue
SDS	Sodium Dodecyl Sulfate
SnO <sub>2</sub>	Tin Oxide
SrTiO <sub>3</sub>	Strontium Titanate
SWCNTs	Single-Walled Carbon Nanotubes
TBOT	Titanium Butoxide
TiO <sub>2</sub>	Titanium Dioxide
UV	Ultraviolet
UV-Vis	Ultraviolet Visible
VB	Valence Band
wt%	Weight Percentage
ZnO	Zinc Oxide
Zn(NO <sub>3</sub> ) <sub>2</sub>	Zinc Nitrate
ZrO <sub>2</sub>	Zirconium dioxide

## LIST OF APPENDICES

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## CHAPTER 1

### INTRODUCTION

This chapter discusses the research background of zinc oxide (ZnO), titanium dioxide (TiO<sub>2</sub>) and ZnO/TiO<sub>2</sub> nanocomposite as the sand-based photocatalysts for photocatalysis application. The hybridization of sand-based photocatalysts with graphene oxide (GO) and GO\_multi-walled carbon nanotubes (MWCNTs) are also described clearly. Research problem, objectives, scope and limitation of this study and thesis organization are also presented in this chapter.



## 1.2 Research Background

Nowadays, clean water sources are limited due to the pollution from industry that released waste disposal into fresh water sources without treating them properly. Textile wastewater consists of various types of heavy metal and non-biodegradable organic dye which caused a very serious problem for human health (Saravanan, Gracia, & Stephen, 2017). Furthermore, most of organic dye are rich with hazardous chemicals which make it difficult to treat and harmful to the environment or biological health as they are toxic by nature (Katheresan, Kansedo, & Lau, 2018; Maučec et al., 2018). Methylene blue (MB) is one of organic dye that have a complex aromatic structure and difficult to decompose (N.Sun et al., 2018). Water will be highly coloured, difficult to treat and decolorize whenever it mixed with MB dye (Basturk & Karatas, 2015). Therefore, it is imperative to find a way to degrade the organic dye so that clean and fresh water source can be obtained.

Recently, methods for treating dye effluent received the limelight as clean water sources possibly begin to diminish rapidly if there is no reliable solution is found. There are several methods that can be used to treat dye effluent, such as coagulation, biodegradation, adsorption, and membrane process. Unfortunately, these methods are inefficient enough to completely degrade the dye in the polluted water (Anjum, Miandad, Waqas, Gehany, & Barakat, 2016). Moreover, these methods present some disadvantages from the environment aspect such as the generated secondary pollution and high energy requirement to operate (Anjum et al., 2016; Fei et al., 2018; Katheresan et al., 2018). A simple method, semiconductor-based photocatalysis process then



considered as promising method to treat dye polluted waste water (Banerjee, Benjwal, Singh, & Kar, 2018; Khataee & Kasiri, 2010; Martins et al., 2018).

Photocatalytic phenomenon has been discovered in 1972 by Honda and Fujishima where they have splitted water into oxygen and hydrogen under ultraviolet (UV) light on a TiO<sub>2</sub> electrode (Fujishima & Honda, 1972b). After that discovery, they published their finding in 'Nature' that lead to the beginning of a new era in heterogeneous photocatalysis (Fujishima & Honda, 1972b). Then, this phenomenon began to evolve as Frank and Bard used TiO<sub>2</sub> photocatalysis for environmental purification by destroying the pollutants in polluted water (Frank and Bard, 1977).

Photocatalysis is a chemical reaction where it uses light radiation to activate the catalyst in order to accelerate the reaction rate. In other word, photocatalysis is an acceleration of a photoreaction in the existence of catalyst where photon energy is converted into chemical energy (Ibhadon & Fitzpatrick, 2013; Lacombe & Keller, 2012). Many researchers have been attracted by the effects of the photocatalysis as it is one of the cleanest and environment-friendly for water purifying (Basturk & Karatas, 2015; Khojasteh, Salavati-Niasari, & Sangsefidi, 2018). In addition, it also becomes a low cost method since it only consumes low level of energy for operation (Ehrampoush, Moussavi, Ghaneian, Rahimi, & Ahmadian, 2011). In order to activate the photon excitation, semiconductor materials are clearly needed. Various semiconductor photocatalysts that commonly used in photocatalysis process were ZnO, TiO<sub>2</sub>, cadmium sulphate (CdS), tungsten oxide (WO<sub>3</sub>), tin oxide (SnO<sub>2</sub>), strontium titanate (SrTiO<sub>3</sub>), and russellite (Bi<sub>2</sub>WO<sub>6</sub>), zirconium dioxide (ZrO<sub>2</sub>) (Gaya, 2014).



It has been reported that ZnO is considered as an ideal semiconductor photocatalyst for photocatalysis process owing to its high-electron mobility, high chemical and physical stability, high photostability, good oxidizing power and has high surface area (Anjum et al., 2016; Adnan, Julkapli & Hamid, 2016; Gupta & Tripathi, 2011; Nalumaga, 2017). However, the agglomeration between ZnO particles would decreased the number of active surface sites and surface area thus decreased its photocatalysis performance (Azmina et al., 2017). In addition, ZnO photocatalysts was inefficient in powder form as it will dispersed in water and produced milky solution which hindered the photocatalyst activation under UV light (Eddy et al., 2015). A substrate then clearly needed in order to activate the photocatalyst and perform the photocatalysis process. Several types of substrate usually used for photocatalyst were clay, glass, zeolite, silica, sand, and fly ash. Among them, sand offers several advantages such as has porous morphology, high density, locally available, low cost, and chemically inert (Abdel-Maksoud, Imam, & Ramadan, 2018; Hadjltaief, Zina, Galvez, & Costa, 2016; Shan, Ghazi, & Rashid, 2010).

Type of ZnO nanostructures also play a crucial role for the efficiency of photocatalysis performance. ZnO nanostructures can be divided into zero- (0D), one- (1D), two- (2D) and three-dimensional (3D). The most commonly used ZnO nanostructures as photocatalyst is 0-D such as nanoparticles, nanospheres and quantum dot owing to their large surface area (Zhou, Wen, Zhao, & Anderson, 2017). However, 0-D ZnO nanostructures possess several limitations as mentioned before. Recently, 1-D and 3-D nanostructures such as nanorods (NRs) and nanoflowers (NFs) of ZnO has been used as photocatalyst (Yin, Chen, Zhang, Cai, & Wang, 2014). NRs nanostructures is an ideal candidates as a photocatalyst due to its photostability under





both UV-light and visible light irradiation, simple procedure formation and attachable to any types of substrate (Azam & Babkair, 2014; Baruah, Jaisai, Imani, & Nazhad, 2010; N. Huang et al., 2015; Nikoofar, Haghghi, Lashanizadegan, & Ahmadvand, 2014; Zhou et al., 2017). Meanwhile, NFs nanostructures possessed large surface-to-volume ratio which offer higher active sites which offer larger contact area between dye and the catalyst thus resulted in the maximum dye adsorption (Ong et al., 2014; Peter, Praveen, Vignesh, & Nithiananthi, 2017; Zhou et al., 2017). Moreover, high oxygen vacancies offered by NFs nanostructures could also enhanced the photocatalysis performance (Tripathi et al., 2014).

N. Huang et al. (2015) showed that ZnO NRs possessed high dye removal of rhodamine blue (RhB) and methylene orange (MO) which almost completely degraded both dyes within 100 minutes as compared to ZnO nanosphere under UV-light-radiation. This result also in a good agreement with Yimin Wang, Hangbo Zhang, Qu, & Su (2016) which showed that faster RhB dye degradation was achieved by ZnO nanorods (within 60 minutes) as compared to ZnO nanosphere. Meanwhile, Mohammad, Kapoor, & Mobin (2016) claimed that ZnO-NFs successfully degraded 99.46% of MO in just 50 mins as compared to commercial ZnO (76.86%) within 130 mins. Ameen, Akhtaer & Shin (2017) showed that, ZnO-NFs presented high removal of bromophenol dye with 96% within 120 mins.

On the other hand, since ZnO also possess limitation in photocatalysis process caused by the high recombination rate between electron-hole pair (Nalumaga, 2017; N.Sun et al., 2018). In order to overcome this limitation, ZnO photocatalyst must be composited with other semiconductor, metal, non-metal, or carbon based materials





(Banerjee et al., 2018; N. Sun et al., 2018). It have been reported that the combination between semiconductor-semiconductor nanocomposite such as ZnO/TiO<sub>2</sub> showed excellent photocatalysis effect as compared to the pure semiconductor (P. Cheng et al., 2014; Habib et al., 2013). Longer lifetime of the photogenerated electron/hole was achieved when ZnO and TiO<sub>2</sub> were composited together, as the TiO<sub>2</sub> act as a trap site which can prevent electron-hole recombination (Hadjltaief et al., 2016; Nalumaga, 2017). Moreover, the incorporation of ZnO into TiO<sub>2</sub> can decreased the band gap value and extend the light absorption range (Bai, Kou, Gong, & Zhao, 2013; Cirak et al., 2018; Wetchakun, Wetchakun, & Sakulsermsuk, 2019). Cirak et al. (2018) showed that by compositing ZnO and TiO<sub>2</sub>, 95% dye degradation was achieved compared to the pure TiO<sub>2</sub> (65%). In addition, P. Cheng et al. (2016) and C. Cheng et al. (2014) also showed that ZnO/TiO<sub>2</sub> nanocomposite performed high photocatalysis activity than pure ZnO and TiO<sub>2</sub>. These results were in a good agreement with prior work Hadjlataief et al. (2016) which showed that ZnO-TiO<sub>2</sub>/clay nanocomposite achieved higher dye degradation of 98.7% as compared to of TiO<sub>2</sub>/clay (87.2%).

To date, there are several available methods that can be used in order to synthesize ZnO/TiO<sub>2</sub> nanocomposite such as thermal evaporation, vapor liquid solid, electrospinning, chemical and physical vapor deposition (CVD/PVD) (Lim, 2010; Siwinska-Stefanska et al., 2018). However, these methods consumed high temperature, pressure and energy, complex procedures and utilized expensive materials (Benkara & Zerkout, 2010; Zhou, Wen, Zhao, & Anderson, 2017). Typically, photocatalysis performance are strongly depend on the crystallite size and morphology of the photocatalysts. Therefore, synthesis method play a vital role in order to synthesize nano sizes, uniform and highly distributed ZnO/TiO<sub>2</sub> nanocomposite on the substrate (Lim,





2010; Rosnan, Haan, & Mohammad, 2018). Moreover, low-cost, facile, low energy and low pressure synthesis method are being demanded in order to grow ZnO/TiO<sub>2</sub> nanocomposites.

In particularly, sol-gel and hydrothermal methods offered uniform size distribution, various synthesized morphology, low temperature operation, simple procedures and resulted high purity and crystallinity of nanocomposites (Adnan et al., 2016; Ba-abbad et al., 2013; Kołodziejczak-Radzimska & Jesionowski, 2014; Lee et al., 2010; Ong, Ng, & Mohammad, 2018; Rosnan et al., 2018; Wetchakun et al., 2019; Zhou et al., 2017). Sol-gel method also has proven in promoting good purity, good dispersing and homogeneity (Bodson et al., 2010). P.Cheng et al. (2016) synthesized ZnO-TiO<sub>2</sub> nanocomposites by using two steps hydrothermal method and resulted in complete degradation of MO dye within 25 mins. Siwińska-Stefańska et al. (2018) synthesized TiO<sub>2</sub>-ZnO nanocomposite by using sol-gel method has successfully degraded 93.4% of C.I. basic violet 10 dye within 120 mins. Meanwhile, Hakki, Allahyari, Rahemi, & Tasbihi (2019) synthesized TiO<sub>2</sub>-ZnO on glass by using sol-gel dip coating method and effectively removed the MB dye (97.3%) within 360 mins. Siwińska-Stefańska et al. (2019) fabricated TiO<sub>2</sub>-ZnO by using hydrothermal method and exhibited high photocatalysis performance in removing C.I. Basic Violet 10 (95%) within 180 mins. Therefore, owing to excellent properties proposed by sol-gel and hydrothermal method thus it were chosen in order to fabricate ZnO and TiO<sub>2</sub>, respectively.

In order to further enhance the photocatalysis performance, the hybridization of photocatalyst material are also done with other carbon based-material, such as graphene





oxide (GO) and multi-walled carbon nanotubes (MWCNTs) owing to their large surface area and high electron mobility (Mahmoodi, 2013; Saleh, 2013; Tayel, Ramadan, & Seoud, 2018). The  $sp^2$  hybridization of carbon atoms on GO allow a fast electron transfer thus improve the charge separation which can enhance the efficiency of photocatalysis (Jeanmonod, Rebecca, & Suzuki, 2018; Sun et al., 2018; Tayel et al., 2018). The utilization of GO and MWCNTs with the photocatalyst are proven to further improve the photocatalysis process (Dalt, Alves, & Bergamann, 2016; Zhang et al., 2016).

Chaudhary, Singh, Vankar, & Khare (2018) showed that ZnO/MWCNTs achieved 93% of MB degradation compared to pure ZnO (48% of MB degradation). Meanwhile Raliya, Avery, Chakrabarti, & Biswas (2017) showed that the hybridization of GO with  $TiO_2/ZnO$  nanocomposite have enhanced the photocatalytic performance from 40 to 44%. These results were in a good agreement with Dalt et al, (2016) which showed that the incorporation of MWCNTs with  $TiO_2-ZnO$  achieved the highest photocatalytic activity compared to  $TiO_2-ZnO$  nanocomposite.

GO is commonly synthesized via Hummers' method which produced high quality of GO (Kumar, Madaria, & Zhou 2010). However, this method presents several drawbacks, such as the utilization of high hazardous chemical that can lead to environmental damage, plenty of procedures, and long-time of production (Brodie, 1859; Hummers & Offeman 1957). A simpler electrochemical exfoliation assisted by surfactant becomes a promising solution to synthesize GO in large scale production (Fatiatun, 2018, Muqoyyanah, 2019; Suriani, Muqoyyanah, Othman, Mamat, et al 2018; Suriani, Muqoyyanah, Othman, Rohani, et al., 2019). In addition, this process also





environment-friendly, economic, can be operated at ambient pressure and temperature, less procedures and less hazardous chemical usage (Fatiatun, 2018, Muqoyyanah, 2019; Suriani, Muqoyyanah, Othman, Mamat, et al., 2018; Suriani, Muqoyyanah, Othman, Rohani, et al., 2019).

The transfer process of GO-based materials onto the ZnO/TiO<sub>2</sub> nanocomposite can be done via spray coating, spin coating, dip coating and immersion method. Spray coating method is simple and provide a large surface area coverage onto the desired substrate (Y. Liu, 2017, Y. Chen et al., 2018). However, the uniformity of the coverage is relatively poor and there are material wastage or loss during the process (Y. Liu, 2017). Moreover, this method also required very expensive apparatus. Meanwhile, dip coating is relatively a slow coating process. Spin coating is easy to handle, cheap and provide high uniformity. However, spin coating also experienced coating material wastage same as spray coating, non-uniform layer of and only applicable for flat substrate. Therefore, immersion method was chosen in order to transfer GO-based materials onto the sand/ZnO/TiO<sub>2</sub> owing to its simplicity, low-cost, time-saving, large scale production and obtainment of homogenous substrate (Latthe, Gurav, Maruti, & Vhatkar, 2012; Poorebrahimi & Norouzbeigi, 2015)

To the best of our knowledge method to synthesize ZnO/TiO<sub>2</sub> nanocomposite by combining sol-gel immersion and hydrothermal method, respectively are not well explored. In addition, based on the literature, the utilization of immersion method to transfer GO-based materials onto the ZnO/TiO<sub>2</sub> nanocomposite also rarely explored. Therefore, in this work, the novel combination of ZnO and TiO<sub>2</sub> synthesized by sol-gel immersion and hydrothermal method, respectively was used as sand-based





photocatalyst materials applied for photocatalysis application. Meanwhile, the novel hybridization of the GO-based materials with sand-based photocatalysts were done via immersion.

### 1.3 Research Problems

ZnO is the most common semiconductor material utilized as photocatalyst applied for photocatalysis application. This was due to its unique characteristics such as high stability, eco-friendly and cheap (P. Cheng et al., 2016). However, photocatalysis performance of individual ZnO were limited due the aggregation of the powder form of ZnO and difficulties to separate from the treated water the due to its nanosize (Mousavi, Davar, & Loghman-Estarki, 2016). Moreover, its powder form is inefficient as it is produced milky solution and easily dispersed (Katheresan et al., 2018).

In order to hinder these drawbacks, the ZnO photocatalyst must be supported on a substrate in order to immobilize it in the solution. There are several substrates that are commonly used such as zeolites, glass, clay, silicon, sand and quartz (Hadjltaief et al., 2016). In this work, sand is selected as a substrate due to its availability, high density, inexpensive and chemically inert (Abdel-Maksoud et al., 2018)

ZnO possesses high rate of recombination between electron and hole (Cirak et al., 2018; Eddy et al., 2015). In addition, self-oxidation of ZnO also lead to the inefficient photocatalysis performance (Cirak et al., 2018). Therefore, ZnO was composited with other semiconductors materials, such as TiO<sub>2</sub> in order to enhance its



performance. It has been reported that the coupling between these two semiconductors showed excellent photocatalysis performance (C. Cheng et al., 2014; Gita, Hussan, & Choudhury, 2017; Gupta & Tripathi, 2011; Habib et al., 2013).

ZnO was synthesized on the sand substrate via sol-gel immersion method due to the low energy and cost consumption, low temperature operation, simple procedure, has a good control over the physical characteristics and morphology and resulted high purity and crystallinity of ZnO (Adnan et al., 2016; Ba-abbad et al., 2013; Kolodziejczak-Radzimska, & Jesionowski, 2014; Ong et al., 2018; Wetchakun et al., 2019). Meanwhile, ZnO/TiO<sub>2</sub> nanocomposite was synthesized by growing TiO<sub>2</sub> on the prior synthesized sand/ZnO via hydrothermal method which offer simple equipment and large scale production (Adnan et al., 2016; Wetchakun et al., 2019)

Other materials that can be utilized to further improve the photocatalysis performance is carbonaceous materials such as GO and MWCNTs. These materials possess high conductivity, extraordinary strength, provide large surface area, and could stabilize the electron/hole separation by acting as electron transfer and acceptor (Mallakpour & Rashidimoghadam, 2019; Saleh, 2013). GO was commonly synthesized by Hummer's method due to its high-quality GO production (Kang et al., 2016). However, this method utilizing hazardous chemicals and strong acids which lead to the serious environmental pollution, consuming long time production and causing metal ion impurities on the GO sheets (Pei, Wei, Huang, Cheng, & Ren, 2018; Suriani et al., 2018). Therefore, electrochemical exfoliation method was chosen to synthesize high production of GO owing to its simplicity, economic, environment friendly, and can be operated at ambient pressure and temperature (Kakaei & Hasanpour, 2014; Parvez et



al., 2014). This process utilized water-based electrolyte assisted by commercially single-tail sodium dodecyl sulphate (SDS) surfactant that is functioning in stabilizing the GO dispersion (Md Disa et al., 2015).

Hence, in this work, sand/ZnO photocatalyst materials were fabricated with different nanostructures which are NRs and NFs before compositing it with TiO<sub>2</sub> NRs. The hybridization of sand/ZnO/TiO<sub>2</sub> nanocomposite were also done with GO and GO\_MWCNTs hybrid solution as the photocatalyst materials. Afterward, the fabricated sand-based photocatalyst materials are used to investigate their effectiveness for photocatalysis application (MB degradation). To the best of our knowledge, this work presents a novel combination between ZnO with TiO<sub>2</sub> on the sand as a substrate via sol-gel immersion and hydrothermal method, respectively. Meanwhile, the hybridization of the sand/ZnO/TiO<sub>2</sub> nanocomposite was also done with GO-based solution via immersion method.

#### 1.4 Research Objectives

The research objectives of this study are:

- 1) To fabricate sand/ZnO and sand/ZnO/TiO<sub>2</sub> nanocomposites as photocatalyst materials via sol-gel immersion and hydrothermal method, respectively.
- 2) To fabricate sand/ZnO/TiO<sub>2</sub>/GO-based photocatalyst materials via immersion method.





- 3) To investigate the photocatalysis performance of the fabricated sand/ZnO, sand/ZnO/TiO<sub>2</sub> nanocomposites and sand/ZnO/TiO<sub>2</sub>/GO-based photocatalysts for MB dye degradation.

## 1.5 Scope and Limitation of Study

This study focuses on the fabrication of various type of sand-based photocatalysts and its hybridization with carbon-based materials in order to improve the photocatalysis performance. The nanostructures used for ZnO and TiO<sub>2</sub> were limited to NRs and NFs only. Meanwhile, the crystalline phases used for ZnO and TiO<sub>2</sub> were only focused on wurtzite and rutile phase, respectively. The surfactant used for GO production was only focused on commercially available single-tail SDS surfactant and MWCNTs based on waste cooking palm oil. In addition, the investigation of photocatalysis performance are limited by specifying the photocatalyst mass (30 g) and the usage of low MB dye concentration (5 ppm).

The fabricated sand-based photocatalysts and its hybridization with carbon-based materials were characterized using several instrumentations. FESEM and EDX were utilized to investigate the surface morphology and elements compound of various fabricated sand-based photocatalysts. Meanwhile, micro-Raman spectroscopy and UV-Vis were used to investigate the structural properties and measure the absorption of the treated water after dye degradation test was performed, respectively.



## 1.6 Thesis Organization

This thesis consists of five chapters which presents the details work regarding photocatalysis application. Chapter 1 explains the research problem, research objectives, and scope and limitations of the study. Chapter 2 presents the literature review of the fundamental theories, including photocatalyst, photocatalysis and previous study related to photocatalysis application. The research methodology which cover the fabrication, synthesis process and characterization techniques of various sand-based photocatalysts are explained in Chapter 3. Chapter 4 presents the results and discussion including the morphology, structural and performance of the fabricated sand-based photocatalysts. Finally, the last chapter, Chapter 5 summarizes the results, conclusions and suggestion for the future work.