WELL-ALIGNED ONE-DIMENSIONAL-LIKE TITANIA-LIQUID CRYSTALS COMPOSITE PHOTOCATALYST SYNTHESIZED UNDER MAGNETIC FIELD

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In the name of Allah, the Most Beneficent and the Most Merciful.

This thesis is dedicated to my beloved parents : Hjh. Mashitoh binti Hj. Ismail and Hj. Abu Bakar bin Chik, parents in law : Azizah binti Ramli and Dzulkifli bin Ariffin, husband : Mohd Farid bin Dzulkifli, son : Izz Adam Irfan bin Mohd Farid, my siblings and friends, especially to Farah, Najidah, Haqzim and Shafiyah.

for their endless support, love, concern, encouragement and continuous prayer for my success in completing this research.

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ABSTRACT

The synthesis of one-dimensional-like titania (TiO_2) and the elucidation of its shape-photocatalytic activity relationship remain a big challenge today. It is hypothesized that this kind of material can be synthesized under magnetic field with the presence of magnetically responsive liquid crystals. This research is considered as a novel work since comprehensive studies have been carried out for the one-dimensional-like TiO_2 and its photocatalytic activity. The importance of the one-dimensional-like TiO_2 should be related to the electronic structures that affect the electron-hole recombination, and hence, photocatalytic activity. In this research, the synthesis of well-aligned one-dimensional-like TiO_2 using liquid crystals as the structure aligning-agent was demonstrated *via* sol-gel method under a magnetic field. The 4-cyano-4'-pentylbiphenyl (5CB) and 4-cyano-4'-octylbiphenyl (8CB) liquid crystals have been used as the structure aligning-agents. Each of the liquid crystals have been mixed with tetra-*n*-butyl orthotitanate (TBOT), 2-propanol and water, and the mixtures underwent slow hydrolysis in a magnetic field (0.3 T) under ambient conditions in the open atmosphere. The obtained TiO_2 composite samples were characterized by scanning electron microscope (SEM), X-ray diffraction (XRD), Fourier transform infrared (FTIR) spectrometer, photoluminescence spectrometer, direct current electrical conductometer and Hall effect analyzer. Interesting results were observed when an external magnetic field was applied during the hydrolysis of TBOT in the presence of the liquid crystals. The TiO_2-5CB composite was more well-aligned in the shape of whiskers compared to the TiO_2-8CB composite when the reaction mixtures were placed under the magnetic field. This is due to the steric size of 8CB which is larger than 5CB. Meanwhile, the TiO_2-5CB and TiO_2-8CB composites have irregular, spherical shape when no magnetic field was applied. Other than that, it is quite surprising that the well-aligned one-dimensional-like TiO_2-5CB showed the anatase peak, considering that no further treatment such as calcination was required. The intensity of the emission peaks in the photoluminescence spectrum of well-aligned one-dimensional-like TiO_2-5CB composite was lower compared to the other composites. This might be caused by electrons transferring from 5CB to TiO_2 in the well-aligned one-dimensional-like TiO_2-5CB during ultraviolet irradiation. The plausible mechanism of electron charge transfer was elucidated by density functional theory (DFT) calculation. Based on these results, the interfacial interaction between the liquid crystal and TiO_2 is the key factor to control the shape of TiO_2 during the hydrolysis process. Furthermore, direct current electrical conductivity and Hall effect studies showed that the well-aligned one-dimensional-like TiO_2 enhanced the electron mobility. Therefore, due to the increasing of electron mobility, the recombination of electrons and holes could be delayed, and hence, the photocatalytic activity of the well-aligned one-dimensional-like TiO_2 in the oxidation of styrene was enhanced. Based on the above results, the structure-photocatalytic activity relationship of well-aligned one-dimensional-like TiO_2 composite, synthesized under magnetic field was clarified in this research.

ABSTRAK

Sintesis titania (TiO_2) yang berbentuk bahan satu-dimensi dan penjelasan mengenai hubungan di antara bentuk dengan aktiviti fotopemangkinan masih menjadi cabaran yang besar pada masa kini. Bahan ini dihipotesiskan dapat disintesis di bawah medan magnet dengan kehadiran cecair hablur yang bergerak balas terhadap magnet. Kajian ini dapat dianggap sebagai penyelidikan yang baharu kerana kajian yang menyeluruh telah dijalankan terhadap bahan TiO_2 satu-dimensi dan aktiviti fotopemangkinannya. Kepentingan TiO_2 yang berbentuk bahan satu-dimensi seharusnya dikaitkan dengan struktur elektronik yang mempengaruhi penggabungan semula elektron-lubang, dan dengan itu, aktiviti fotopemangkinan. Dalam penyelidikan ini, sintesis TiO_2 berbentuk bahan satu-dimensi yang sejajar rapi menggunakan cecair hablur sebagai ejen penjajaran struktur telah dihasilkan melalui kaedah sol-gel di bawah medan magnet. Cecair hablur 4-siano-4'-pentilbifenil (5CB) dan 4-siano-4'-oktilbifenil (8CB) telah digunakan sebagai ejen penjajaran struktur. Setiap cecair hablur telah dicampurkan dengan tetra-*n*-butil ortotitanat (TBOT), 2-propanol dan air, dan campuran ini melalui proses hidrolisis secara perlahan dalam medan magnet (0.3 T) pada keadaan ambien dan terbuka ke atmosfera. Sampel komposit TiO_2 yang diperoleh telah dicirikan dengan mikroskop pengimbas elektron (SEM), pembelauan sinar-X (XRD), spektrometer inframerah transformasi Fourier (FTIR), spektrometer fotopendarcahaya, konduktometer elektrik arus terus dan penganalisis kesan Hall. Hasil yang menarik telah dicerap ketika medan magnet luar digunakan semasa proses hidrolisis TBOT dengan kehadiran cecair hablur. Komposit TiO_2-5CB yang dihasilkan adalah lebih sejajar rapi dalam bentuk misai berbanding dengan komposit TiO_2-8CB apabila campuran tindak balas diletakkan di bawah medan magnet. Hal ini disebabkan oleh saiz sterik 8CB yang lebih besar berbanding 5CB. Sementara itu, komposit TiO_2-5CB dan TiO_2-8CB mempunyai bentuk sfera yang tidak teratur apabila tiada medan magnet digunakan. Selain itu, suatu hal yang agak menghairankan ialah TiO_2-5CB yang berbentuk bahan satu dimensi yang sejajar rapi menunjukkan puncak anatas memandangkan bahawa tiada rawatan lanjut seperti pengkalsinan diperlukan. Keamatan puncak pancaran dalam spektrum fotopendarcahaya bagi komposit TiO_2-5CB berbentuk bahan satu-dimensi yang sejajar rapi adalah lebih rendah berbanding komposit lain. Hal ini berkemungkinan disebabkan pemindahan elektron dari 5CB ke TiO_2 dalam TiO_2-5CB berbentuk bahan satu-dimensi yang sejajar rapi semasa penyinaran ultralembayung. Mekanisme yang sesuai yang boleh dikaitkan dengan pemindahan cas elektron ini dapat dijelaskan dengan pengiraan teori fungsi ketumpatan (DFT). Berdasarkan keputusan kajian, interaksi antara muka di antara cecair hablur dan TiO_2 merupakan faktor utama untuk mengawal bentuk TiO_2 semasa proses hidrolisis. Tambahan lagi, kekonduksian elektrik arus terus dan kajian kesan Hall menunjukkan bahawa TiO_2 berbentuk bahan satu-dimensi yang sejajar rapi dapat meningkatkan keupayaan mobiliti elektron. Oleh itu, peningkatan mobiliti elektron ini dapat menangguhkan penggabungan semula elektron dan lubang dan seterusnya meningkatkan aktiviti fotopemangkinan dalam pengoksidaan stirena. Berdasarkan hasil kajian di atas, hubungan di antara struktur dengan aktiviti fotopemangkinan bagi komposit TiO_2 berbentuk bahan satu-dimensi yang sejajar rapi, yang disintesis di bawah medan magnet telah diperjelas dengan lebih terperinci dalam kajian ini.

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LIST OF ABBREVIATIONS

5CB	-	4-cyano-4’-pentylbiphenyl
8CB	-	4-cyano-4’-octylbiphenyl
MF	-	Magnetic field
LC	-	Liquid crystal
TBOT	-	Tetra-*n*-butyl orthotitanate
TiO_2	-	Titania/titanium dioxide
T	-	Tesla
SEM	-	Scanning electron microscope
DC	-	Direct current
TGA	-	Thermal gravimetric analysis
DR UV-Vis	-	Diffuse reflectance ultraviolet-visible
FTIR	-	Fourier transform infrared spectrometer
XRD	-	X-ray diffraction
PL	-	Photoluminescence
GC-FID	-	Gas chromatography-flame ionization detector
XPS	-	X-ray photoelectron spectrometer
BET	-	Brunauer-Emmet-Teller
a.u	-	Arbitrary unit
Ca.	-	Around, about or approximately
i.e	-	That is
%	-	Percentage
mmol	-	Milimol
eV	-	Electronvolt
^{o}C	-	Degree celcius

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CHAPTER 1

INTRODUCTION

1.1 Background of Research

Photocatalysis is a composite word which consists of two parts, "photo" and "catalysis". The photo is known as light, whereas the catalysis is a process of when a substance contributes in modifying the rate of a chemical conversion of reactants without being altered or consumed in the end (Hermann, 1999; Linsebigler *et al*., 1995; Ohtani, 2017). The substance is identified as the catalyst, which increases the rate of reaction by reducing the activation energy. Meanwhile, photocatalysis is the process where a photocatalyst could be activated under light source, modifies the rate of chemical reaction without being involved itself (Ohtani, 2017). Both catalysis and photocatalysis are commonly studied with the aim to achieve higher activity and product selectivity in chemical reactions (Ohtani, 2017). There is a distinct difference in term of principle between catalysis and photocatalysis. For catalysis, a chemical reaction is accelerated by reducing the activation energy with the presence of active sites in the catalyst (Ohtani, 2010; Ohtani, 2017). On the other hand, photocatalysis is driven by the photoexcitation of a photocatalyst followed by the electron-hole transfer to reactants without involving the active sites in the photocatalysts (Ohtani, 2017).

Among all the photocatalysts, titania/titanium dioxide (TiO_2) has been intensively studied and used in many applications. TiO_2 has been reported to shows the best photostability and highest sustained photocatalytic activity (Fox and Dulay, 1993). Nevertheless, TiO_2 also have strong oxidizing abilities (Nosaka *et al*., 2004;

Jańczyk *et al*., 2006) for decomposition of organic pollutants (Fujishima and Zhang, 2006). The properties of low cost and environment friendliness make TiO_2 a suitable material for many practical applications (Gupta and Tripathi, 2011).

To the best of our knowledge, there are no rigid conclusion on the factors that affect the photocatalytic activity, although many studies have been carried out to modify the surface area (Shah *et al*., 2015; Nikhil *et al*., 2015), pore structure in terms of size, volume and shape (Rasalingam *et al*., 2015; He *et al*., 2015), band gap energy (Shah *et al*., 2015) and crystalline phase (Ouzzine *et al*., 2014) of TiO_2. The enhancement of photocatalytic activity by adjusting these factors remains the focus in the field of TiO_2 photocatalyst (Nakata and Fujishima, 2012). However, the main factor that affects the photocatalystic activity of TiO_2 still remained unclear and becomes the grand challenge in the research field of TiO_2 (Ohtani, 2017). In fact, there is one factor that most of the researchers agree with, which is the electron-hole recombination rate of TiO_2 photocatalyst. When the light of appropriate energy is irradiated to a sensitizer, an electron (e^-) from the valence band will be promoted to the conduction band, leaving an electron deficiency or hole (h^+) in the valence band. Both h^+ and e^-, which are equivalently oxidizing and reducing in properties, respectively, will play the main roles in the redox reactions (Ohama and Gemert, 2011).

Figure 1.1 shows the illustration of the electron-hole recombination in TiO_2 photocatalyst. The highest occupied molecular orbital (HOMO) and lowest unoccupied molecular orbital (LUMO) in TiO_2 is termed as the valence band and conduction band, respectively (Asahi *et al*., 2001; Bahnemann, 2004). Focusing on the electronic process, irradiation of ultraviolet (UV) light will promote the electron to the conduction band leaving a hole in the valence band. This results in the excitation of electron-hole that will be used for next reaction, which is the reduction and oxidation process before the electron-hole recombination is occur.

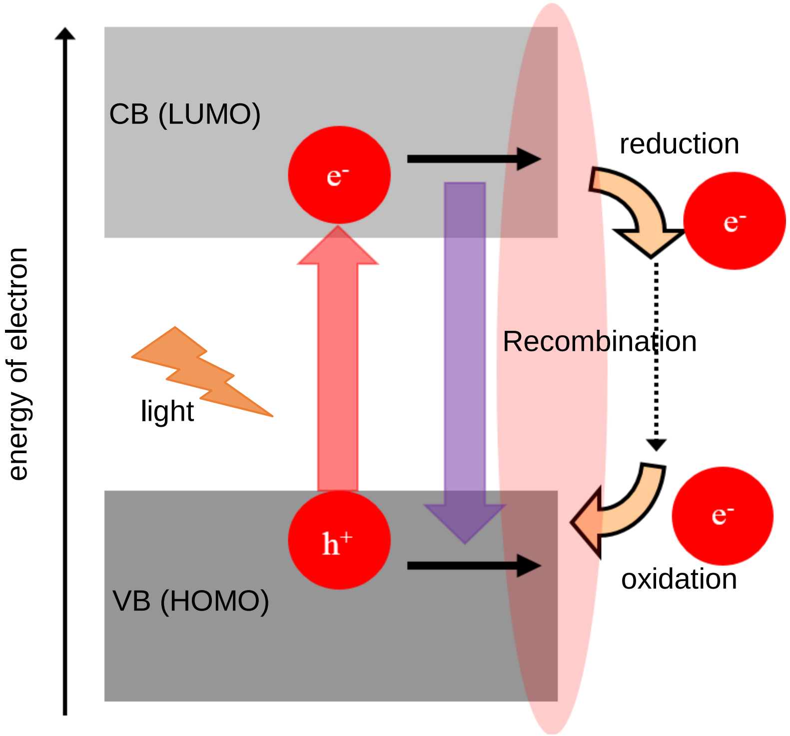

Figure 1.1: Schematic illustration of the formation of photogenerated charge carrier (electron-hole) occurs upon absorption of ultraviolet (UV) light.

Many efforts have been done to decreases the rate of electron-hole recombination in TiO_2 photocatalyst, i.e, addition of noble metals (Rupa *et al.*, 2009; Papp *et al.*, 1993; Wu and Lee, 2004), doping with transition metal cations (Choi *et al.*, 1994; Fox and Dulay, 1993; and Prasad *et al.*, 2009), anions (Diwald *et al.*, 2004; Ao et al., 2010; Yu *et al.*, 2002), metalloids (Xu *et al*, 2009) and structural dimensionality (Feng *et al.*, 2014; Mingzheng *et al.*, 2016; and Xia *et al.*, 2003). As reported by Niu *et al.*, the noble metals such as Ag, Au, Rh and Pt, have been used as co-catalyst of TiO_2. This slightly affects the crystal phase and particle size of TiO_2 (Niu *et al.*, 2016). Xin *et al*. also reported that doping TiO_2 with Fe^{3+} is able to reduce the electron-hole recombination rate, resulting in high photocatalytic activity.

Another example would be the usage of one-dimensional structures, which are in the form of the tubes or fibers. In these structures, the low recombination is caused by the short diffusion of charge carrier (Nakata *et al.*, 2011). Two-dimensional sheets have smooth surfaces and high adhesion (Katsumata *et al.*, 2010). This led to the potential application of self-cleaning coatings (Katsumata *et al*., 2010; Shichi *et al*., 2010). Meanwhile, three-dimensional has the interconnected structure with pores, which provides a significant benefit for efficient diffusion pathways for reactants, such as organic pollutants (Nakata and Fujishima, 2012). Based on the above considerations, it can be considered that the photocatalytic activity of TiO_2 is affected by the shape of the photocatalyst. Figure 1.2 shows the illustration of structural dimensionalities; one-dimensional, two-dimensional and three-dimensional with their properties.

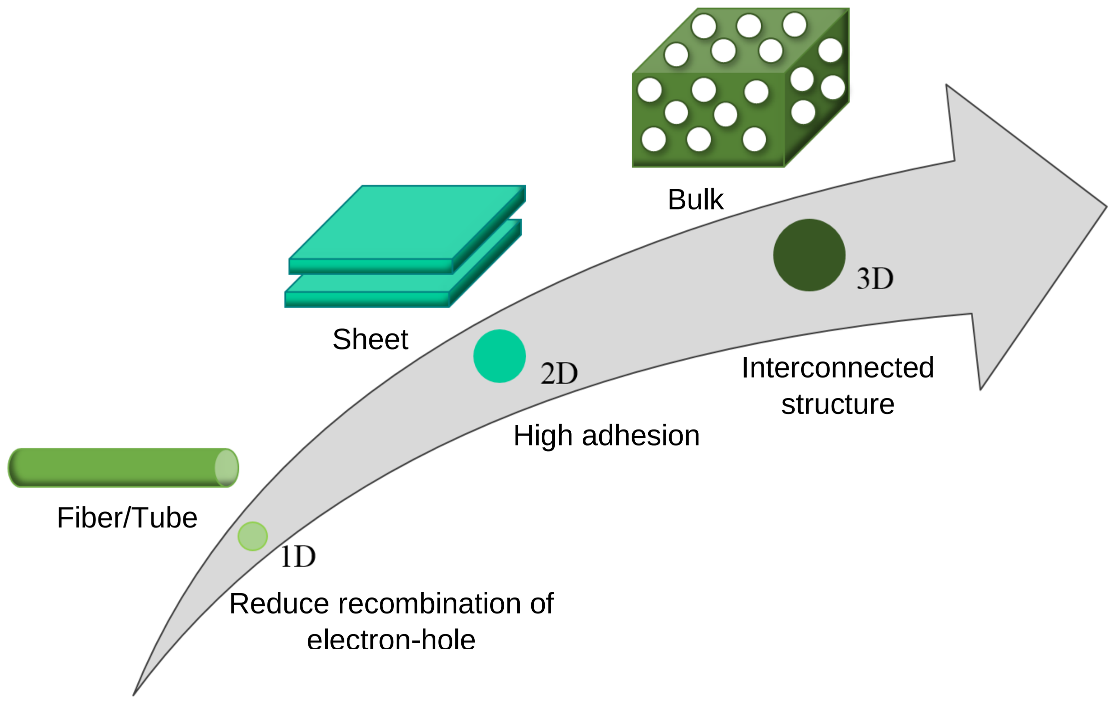

Figure 1.2: Schematic illustration of the structural dimensionality of materials with their properties (Nakata and Fujishima, 2012).

In the past few decades, the one-dimensional structure of TiO_2 has attracted more attention compared to the two-dimensional and three-dimensional structures. It has been reported that the fiber, rod, wire and tube-like materials were considered as the one-dimensional structure (Xia *et al*., 2003, Nakata and Fujishima, 2012; Mingzheng *et al*., 2016). The one-dimensional structure of materials has unique electronic properties since it has been reported that the structure of one-dimensional materials can decrease the electron-hole recombination (Mingzheng *et al*., 2016; Xia *et al*., 2003). In comparison with three-dimensional structure of materials, the probability of electron to recombine with hole reduced ca. 33% since one-dimensional structure has one degree of freedom compared to the three-dimensional structure, which have three degree of freedom. One explains that the density of state for one-dimensional materials, which can be referred as quantum wires, is in such a way, hence the electron and hole are less likely to recombine compared to the two-dimensional and three-dimensional materials since there is no steps in density of state as an increase in the energy of electron (Hicks, 1996; Mao *et al*., 2016).

As stated by Feng *et al*., and Mingzheng *et al*., the one-dimensional TiO_2 showed excellence photocatalytic activity performance and the phenomenon was explained by the electron-hole recombination (Feng *et al*., 2014; Mingzheng *et al*., 2016). Apart from that, various strategies have been designed for the preparation of one-dimensional structure, such as sol-gel template method, chemical vapor deposition (CVD) and hydrothermal method (Lia *et al*., 2009, Wu and Yu, 2004; Attar *et al*., 2009). Hence, this study has been focused on the one-dimensional structure of TiO_2 photocatalyst and its photocatalytic performance in order to clarify the effect of the shape.

In this study, well-aligned one-dimensional-like TiO_2 composite has been synthesized, which possesses one-dimensional-like structure by sol-gel method under magnetic field (0.3 T) with the aid of liquid crystals as the structure-aligning agent. The aim is to prove that the well-aligned one-dimensional-like TiO_2 composite affects the electron-hole recombination's rate, as well as the photocatalytic performance. The well-aligned one-dimensional-like TiO_2 composite possesses unique properties and advantages to the photocatalytic activity due to the higher surface to volume ratio that enables a reduction in the electron-hole recombination rate and high interfacial charge carrier transfer rate, which in-return gave benefit for the photocatalytic activity (Nakata and Fujishima, 2012). The well-aligned one-dimensional-like TiO_2 composite can effectively reduce the recombination rate of electron-hole and distance for charge carrier diffusion (Nakata and Fujishima, 2012).

The magnetic field (MF) technique is used to synthesize well-aligned one-dimensional-like TiO_2 composite since as stated by Yamaguchi and Tanimoto works, all of the materials can be aligned via the magnetic field included diamagnetic materials (Yamaguchi and Tanimoto, 2006). Therefore, this study hypothesized that the well-aligned one-dimensional-like TiO_2 composite can be synthesized under magnetic field using liquid crystal as the structure aligning-agent and this photocatalyst can enhance the photocatalytic activity due to the lower recombination rate of electron-hole.

1.2 Problem Statement

To date, many efforts have been carried out to study the main factor that affects the photocatalytic activity of TiO_2 even though many studies have been carried out on modification of TiO_2. This is the grand challenge in the research field of TiO_2 where the main factors that affect the photocatalytic activity of TiO_2 still remained unclear and discussed until nowadays. In previous study, there are many study on the synthesis of one-dimensional-like material has been done, however, the comprehensive study on the correlation between the material and the photocatalytic performance was not discussed. Therefore, in this study was focused on proving the concept that the aligned structure affects the performance of photocatalytic activity due to the several factors, such as electron-hole recombination rate, surface area and structural phase, and it was clarified using calculation in order to obtain the possible mechanism.

In order to prove this concept, well-aligned one-dimensional-like TiO_2 composite, which has a one-dimensional-like structure has been synthesized. The well-aligned one-dimensional-like TiO_2 composite was synthesized via sol-gel method in magnetic field (0.3 T) with the aid of liquid crystals as the structure aligning-agent. The sol-gel method under the magnetic field is the simplest and cheapest method. The TiO_2 was chosen as the photocatalyst because it has good photocatalytic properties and has being widely studied in photocatalysis field. Liquid crystals was used as the structure aligning-agent since it has magnetic properties and can be aligned under magnetic field.

After the well-aligned one-dimensional-like TiO_2 composite has been successfully synthesized, the photocatalyst was subsequently characterized by several instruments to study their morphology, thermal, structural, interaction and optical properties. These characterizations are important for discussing the relationship of the shape of TiO_2 with its properties. Then, the photocatalytic activity of the well-aligned one-dimensional-like TiO_2 composite was tested out in the oxidation reaction. The testing was carried out to examine the effect of well-aligned one-dimensional-like TiO_2 towards the photocatalytic activity, as well as to prove that the one-dimensional-like structure affects the electron-hole recombination. Figure 1.3 shows the flow of the strategies in this study to overcome the problem.

Synthesis of well-aligned titania using liquid crystals as structure aligning-agent under magnetic field

Concept	• Well-aligned TiO_2 (one-dimensional-like structure) affected the photocatalytic performance due to the less recombination rate of electron-hole.

Prove of concept	• Synthesis the well-aligned TiO_2 with the aid of liquid crystals as structure aligning-agent under magnetic field.
	• Characterizations :
	    • Morphology
	    • Thermal
	    • Structural
	    • Functional group
	    • Interfacial interaction TiO_2/5CB
	• Photocatalytic activity testing – study the relationship with the well-aligned TiO_2 with the aid of liquid crystals as structure aligning-agent.

Hypothesis

- Well-aligned TiO_2 with the aid of liquid crystals as structure aligning-agent synthesized under magnetic field affected the photocatalytic activity through decrease the recombination rate of electron-hole via plausible mechanism of electron charge transfer.

Figure 1.3: The schematic flow of conceptual study and the hypothesis.

1.3 Objective of Research

This study provides comprehensive discussion about the correlation between the physicochemical properties of well-aligned one-dimensional-like TiO_2 composites with its photocatalytic activity. Hence, the main objectives of this study are:

- to synthesize well-aligned one-dimensional-like TiO_2 composites under magnetic field using liquid crystals as structure aligning-agent by sol-gel method.
- to characterize the physicochemical properties of well-aligned one-dimensional-like TiO_2 composites.
- to evaluate the photocatalytic activity of well-aligned one-dimensional-like TiO_2 composites in the oxidation of styrene.

1.4 Scope of Research

In this study, magnetic field was applied to synthesize the well-aligned one-dimensional-like TiO_2 composites with liquid crystal as the structure aligning-agent. The well-aligned one-dimensional-like TiO_2 composites were successfully synthesized by sol-gel method under magnetic field using tetra-*n*-butyl orthotitanate (TBOT) as the TiO_2 precursor in the presence of liquid crystals, with slow hydrolysis process. The liquid crystals used were 4-cyano-4'-pentylcarbonitrile (5CB) and 4-cyano-4'-octylcarbonitrile (8CB), which are in nematic and smectic A phase, respectively (Matsuhashi *et al*., 2002). The liquid crystals acted as structure- aligning-agent since these materials have magnetic properties and can be aligned under magnetic field. The synthesis process was performed with and without magnetic field (up to 0.3 Tesla).

Several techniques were used to characterize the composites sample, such as scanning electron microscopy (SEM), Fourier transform infrared spectroscopy (FTIR), X-ray photoelectron spectroscopy (XPS), X-ray diffraction (XRD) spectroscopy, photoluminescence (PL) spectroscopy, thermal gravimetric analysis (TGA), nitrogen adsorption-desorption analysis, diffuse reflectance ultraviolet visible (DR UV-Vis) spectroscopy, Hall effect studies and direct current (DC) electrical conductivity. The physicochemical properties were examined from the aspects of morphology, surface area, thermal stability, structural analysis and the interactions properties of well-aligned one-dimensional-like TiO_2 composites. The gas chromatography (GC) were used to examine the photocatalytic activity in oxidation of styrene. Besides that, photoluminescence (PL) spectroscopy was used to investigate the recombination rate of electron-hole. Through this PL characterization, the mechanism of the electron charge transfer during the photocatalytic oxidation of styrene could be derived by differential functional theory (DFT) calculation. Therefore, the physicochemical properties of well-aligned one-dimensional-like TiO_2 composites can be correlated to enhance the performance of photocatalytic activity.

1.5 Significance of Research

This study highlighted two significances. First, a new technique to synthesize well-aligned one-dimensional-like TiO_2 using sol-gel method under magnetic field (0.3 T) with liquid crystal as the structure aligning-agent has been developed. This strength of magnetic field was used since the liquid crystals can aligned very well under this strength of magnetic field. Second, the shape dependence of TiO_2 photocatalyst was explored. The novelty of this study is the synthesis of well-aligned one-dimensional-like TiO_2 using a framework of both inorganic precursor and organic aligning-agent under magnetic field effect. From the well-aligned one-dimensional-like TiO_2, it can be examined whether the one-dimensional-like structure affects the performance photocatalytic activity due to the electron-hole recombination.

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