POLYSTYRENE/TITANIUM DIOXIDE NANOCOMPOSITE PREPARED BY SOL-GEL METHOD AT LOW TEMPERATURE FOR SMALL ENERGY BANDGAP

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DEDICATION

To my mother and father and family

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ABSTRACT

Titanium dioxide (TiO_2) nanomaterial is broadly employed as an effective photocatalysis semiconductor due to its comprehensive energy band structure. Unfortunately, the efficiency of this material as a photocatalyst with respect to solar spectrum or visible light as the energy source is highly hindered by its wide bandgap and crystal structure. The synthetic methodology for controlling the crystal structure and tuning the TiO_2 bandgap has been investigated in this study. Moreover, results showed that TiO_2 crystalization is first tunable by polystyrene (PS) acting as a precursor, preventing TiO_2 nanomaterials from aggregating. Later, it was found that the bandgap of TiO_2 was very much tuneable with the variation of hydrogen peroxide (H_2O_2) volume (0 mL, 6 mL, and 12 mL) added during the synthesis process. The further process is focused on using only 12 mL of H_2O_2 has positively depleted the bandgap of PS/TiO_2 from 3.49 eV to 3.3 eV. Additional depletion of TiO_2 bandgap values to 2.6 eV, 2.52 eV, and 2.47 eV after calcination and hydrothermal treatment of 450°C, 600°C, as well as 750°C were performed. In this research, 100% rutile crystalization of TiO_2 was successfully fabricated at a lower temperature [750°C] compared to current literature, where the calcination and hydrothermal treatment transformation of the PS/TiO_2 to TiO_2 crystal structure from mixed-phase to single-phase rutile matches with the X-ray diffraction (XRD) and Raman Spectroscopy analysis. This study concludes that PS as precursor material prevents agglomeration of TiO_2 nanomaterials, while H_2O_2 and Tetrahydrofuran (THF) assist the binding of TiO_2 nanomaterials. On the other hand, calcination and hydrothermal controlled the sizes and bandgaps of TiO_2 nanomaterials. The potential application of this research finding includes photocatalyst material and strategies for tuning the crystallisation of nanomaterials.

ABSTRAK

Bahan nano TiO_2 telah digunakan secara meluas sebagai bahan fotokatalisis dari semikondutor yang berkesan kerana memiliki nilai jurang tenaga yang komprehensif. Namun demikian, keberkesanannya sebagai bahan fotokatalisis dibawah cahaya tampak atau spektrum solar sebagai sumber tenaga terjejas akibat jurang tenaga yang agak lebar dan ketidakupayaan mengawal pembentukkan struktur kristalnya. Di dalam kajian ini, method sintisis diperhalusi terutama dalam usaha mengawal proses pengkristalan dan penjajaran jurang tenaga bahan. Hasil dapatan menunjukkan bahawa pengkristalan TiO_2 dapat dikawal dimana PS sebagai bahan pemula yang menghalang aglomerasi bahan nano. Jurang tenaga TiO_2 mampu dijajar dengan variasi jumlah bendalir H_2O_2 (0 mL, 6 mL, and 12 mL) yang ditambah semasa proses sintesis. Focus pada penambahan 12 mL H_2O_2 memberi kesan positif penurunan jurang tenaga PS/TiO_2 dari 3.49 eV kepada 3.3 eV. Pengurangan jurang tenaga TiO_2 kepada 2.6 eV, 2.52 eV, 2.47 eV melalui proses kalsinasi and hyrothermal pada suhu-suhu terpilih 450°C, 600°C, 750°C. Di dalam kajian ini, 100% pengkristalan pada struktur rutile TiO_2 berjaya difabrikasi pada suhu rendah [750 °C] berbanding kajian semasa, di mana proses kalsinasi dan hydrothermal telah mentransformasikan PS/TiO_2 kepada kristal TiO_2 dari fasa-bercampur kepada struktur fasa rutile yang dibuktikan berpadanan dengan data X-ray diffraction (XRD) and analisa Raman Spectroscopy. Kesimpulannya, PS sebagai bahan pengawal pemulaan yang menghalang agglomerasi bahan nano TiO_2, H_2O_2 dan THF membantu proses pengikatan bahan nano TiO_2 serta kalsinasi dan hidroterma mengawal saiz dan jurang tenaga bahan nano TiO_2. Potensi dapatan kajian ini adalah termasuk bahan fotokatalis dan strategi mengawal pengkristalan bahan nano.

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LIST OF SYMBOLS AND ABBREVIATIONS

°C	-	Degree celsius
3D	-	Three dimensional
A	-	Anatase
Ag	-	Silver
Al_2O_3	-	Aluminium oxide
a	-	Absorbtion coefficient
C	-	Carbon
CdTe	-	Cadmium telluride
Ce	-	Cerium
CIGS	-	Copper Indium Gallium di-selenide
cm	-	Centimeters
CNT	-	Carbon Nanotube
Co	-	Cobalt
$CoCl_2$	-	Cobaltv(II) chloride
CoO	-	Cobalt oxides
Cr_2O_3	-	Chromium (III) oxide
$CrCl_3$	-	Chromium (III) chloride
CrO_2	-	Chromium dioxide
CrO_3	-	Chromium trioxide
Cu	-	Copper
Cu_2O	-	Copper (I) oxide
CuO	-	Copper (II) oxide
CuO	-	Copper oxide
CVD	-	Chemical Vapour Deposition
CZTS	-	Copper Zinc Tin Sulphide
D	-	Grain size

DRS	-	Diffuse Reflectance Spectroscopy
DSSC	-	Dye-Sensitized Solar Cell
DTA	-	Differential Thermal Analysis
DTG	-	Differential Thermal Analysis
EDS	-	Energy Dispersive X-ray Spectroscopy
Eg	-	Energy Bandgap
EPS	-	Expanded Polystyrene
eV	-	Electron Volt
Fe	-	Iron
FESEM	-	Field Emission Scanning Electron Microscopy
FTIR	-	Fourier Transformed Infrared Spectroscopy
FWHM	-	Full Width at Half Maximum
H	-	Hydrogen
h	-	Blanck constant
H_2O_2	-	Hydrogen peroxide
H_2SO_4	-	Sulfuric acid
Li	-	Lithium
m	-	Meter
Mn	-	Manganese
$MnCl_2$	-	Manganese (II) chloride
MnO	-	Manganese oxide
MnO_2	-	Manganese dioxide
MOFs	-	Metal-Organic Frameworks
N	-	Nitrogen
Nb_2O_5	-	Niobium pentoxide
NCs	-	Nanocomposites
NiO	-	Nickel oxide
NPs	-	Nanoparticles
O	-	Oxygen
PMMA	-	Poly(methyl methacrylate)
PS	-	Polystyrene
PVD	-	Physical Vapour Deposition

R	-	Rutile
R.T.	-	Room Temperature
SG	-	Sol-Gel
SHE	-	Standard Hydrogen Electrode
SiO_2	-	Silicon dioxide
SnO_2		Tin (IV) oxide
SSA	-	Specific Surface Area
ST	-	Solvothermal
TCO	-	Transparent Conducting Oxide
TEM	-	Transmission Electron Microscopy
TGA	-	Thermogravimetric Analysis
THF	-	Tetrahydrofuran
Ti	-	Titanium
$Ti(OC_4H_9)_4$	-	Titanium (IV) butoxide
$TiCl_3$	-	Titanium trichloride
$TiCl_4$	-	Titanium tetrachloride
$TiO(NO_3)_2$	-	Titanyl nitrate
TiO_2	-	Titanium dioxide
TTIP	-	Titanium (IV) Isopropoxide
UV-Vis	-	Ultraviolet-Visible Spectroscopy
V	-	Vanadium
W	-	Tungsten
WO_3	-	tungsten trioxide
XRD	-	X-Ray Diffraction
Zr	-	Zirconium
ZrO_2	-	Zirconium dioxide

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CHAPTER 1

INTRODUCTION

1.1 Background of the study

When a semiconducting material is exposed to light, photocatalysis occurs, resulting in the formation of an electron-hole pair. Additionally, Doerffler and Hauffe (1964) were the first to describe carbon dioxide (CO) oxidation on zinc oxide (ZnO) under illumination. Subsequently, photocatalysis attained the attention of researchers worldwide after studying the photocatalytic water-splitting properties of titanium dioxide (TiO_2) (Doerffler and Hauffe 1964). Also, (Fujishima & Honda, 1972), as quoted by (Navarro, Guil, and Fierro 2015). Since then, the method of photocatalysis has been applied in several domains. As noted by (Zhu and Wang 2017), the two most significant domains are the energy-related applications from the water-splitting capability of photocatalytic semiconductors and the photocatalytic breakdown of organic pollutants.

The continuous bands' creation of permissible energy states for easily excited electrons inside photocatalytic semiconductor materials is linked to the mobility and convolution of atomic orbitals. In a semiconductor, the valence band, which is the greatest occupied band, and the conduction band, which is the lowest unoccupied band, are separated by a relatively tiny energy gap or bandgap (E_g). The material's Fermi energy level is located in the bandgap (Jasieniak, Califano, and Watkins 2011). Suppose this semiconductor material absorbs a light photon having energy $E > E_g$. In that situation, an electron might be upgraded from the valence band to the conduction band while simultaneously producing a hole in the valence band.

The absorption threshold is the radiation frequency at which the electron is excited from the valence band to the conduction band, causing the photoconductivity phenomena (Tanguy 1996). Exciton refers to the combination of an excited electron and hole. Therefore, it was able to move around the substance without restriction. Recombination or relaxing of the electron into the valence band hole, on the other contrary, may occur if the material possesses large bulk defects. Subsequently, the exciton could move to the material's surface and either be recombined or transferred to any available electron donors and acceptors. In this light, Yersin (2012) argued that the oxidation reactions on the surface of a semiconductor could be reduced by the hole and electron occurrence. Moreover, band bending emerges as the band energy levels change as it advances towards the surface. This phenomenon affects the propensity with respect to the photoexcited holes and electrons to be transferred at the surface. For example, TiO_2 is an active photoanode due to its tendency to show slight downward band-bending, which then causes a greater negative surface charge.

Apart from that, it is crucial to mention that different TiO_2 crystal faces could act as photocathodes adeptly to allow reduction and oxidation reactions to emerge at the same time on the material's surface (Agrawal et al., 2018). Recently, nanoscale materials have predominantly been used as catalysts. This is because they have a high surface area and quantum effects, altering behaviour by shrinking the particles' dimensions (Klaine et al., 2008). One example is (TiO_2), which can excite electrons in the bulk material to change the light to chemical potential energy. As mentioned in (Navarro, Guil, and Fierro 2015), (Fujishima & Honda, 1972) were among the first studies that observed this trait and generated interest in photocatalysis.

Moreover, Hamidi and Aslani (2019) mentioned that most studies had used TiO_2 considering its low cost, accessible bandgap energy, high chemical stability and non-toxic property compared to other materials with photocatalytic activity. While there are many studies on (TiO_2), the development of new TiO_2-based photocatalysis is still ongoing. Subsequently, Umebayashi et al. (2002) stipulated that most studies have focused on narrowing the bandgap of TiO_2 so that it may absorb more light from ambient sources, for instance, sunlight.

This enhances the duration of excited electronic states (Kleverlaan et al., 2000) as well as the production of well-defined particles with known surface properties. Furthermore, TiO_2 is a metal oxide semiconductor having the formula TiO_2. It is thermally stable, hard, and chemical resistant like other metal oxides (Yoo et al., 2018).

Industrially, TiO_2 has been created as a whitener and pigment since the early twentieth century (Gázquez et al., 2014). Its photocatalytic behaviour was first noticed as chalking, which causes the photodegradation of binders in paint. Furthermore, Herrmann (2012) shows that, based on its early history, heterogeneous photocatalysis is a type of heterogeneous catalysis. Titanium (Ti) is currently the most well-studied photocatalytic material (Konstantinova et al., 2007). It sees use in several fields, for instance, organic pollutant remediation, water splitting, self-cleaning surfaces, as well as sensors, among others (Lazar, Varghese, and Nair 2012).

Brookite, anatase, as well as rutile are the three TiO_2 found in nature (Mohamad et al., 2015). The three phases comprise octahedra, which are bound in various ways. The most thermodynamically stable TiO_2 polymorphs are rutile, which form long chains by sharing the edges of octahedra. In the meantime, as described in Chapter 2, anatase is inclusive of point-sharing octahedra as well as brookite, a mix of edge and point-sharing octahedra. According to Kubacka, Fernández-García, and Colón (2012), rutile and anatase are the most studied photocatalytic applications phases. Brookite, on the other hand, is notoriously difficult to synthesise reliably (Morishima et al., 2007) and is generally photocatalytically inactive (Li and Gray, 2007). In this light, this study will not study brookite in detail.

Rutile, a direct-bandgap semiconductor having a 3.0 eV bandgap, denotes the stable and most typical TiO_2 type. Regardless of having the narrowest bandgap than other TiO_2 polymorphs, it could be used for photocatalysis due to its higher electron-hole recombination rate as well as durability, especially when silver (Ag^+) and hydeogen peroxide (H_2O_2) are present (Sclafani and Herrmann 1996). Otherwise, while used anatase TiO_2 is technically less stable as opposed to rutile, it continues to have a tremendous impact as it is metastable up to 600-1000°C. Anatase is an indirect bandgap semiconductor. Despite possessing a 3.2 eV bandgap, which limits its activity threshold to UV light, it is the most often utilised titania phase with respect to photocatalytic applications (Etacheri et al., 2011). According to (Dobrosz-Gómez et al., 2015), due to its propensity towards hole-trapping on the particle surface, it has the potential to greatly enhance the photoexcited electron-hole pairs' lifespan in the material. The excited electrons on anatase (E = −0.66 eV vs. Standard Hydrogen Electrode (SHE) have sufficient energy to take part in H^+ reduction to hydrogen, while holes (E = 2.54 eV vs. SHE) may oxidise water to oxygen (O_2) (Mohajernia 2020) and help the degrade several organic pollutants.

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