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MASTER DEGREE THESIS

Synthesis of ZnO micro-flowers assisted by plant-mediated strategy and their photocatalytic application

By

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**A THESIS SUBMITTED IN FULFILLMENT OF THE REQUIREMENTS FOR THE
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CERTIFICATION

I, **Jiale Huang**, hereby certify that I have read this manuscript and recommend it for acceptance by the Xiamen University as a dissertation entitled “*Synthesis of ZnO micro-flowers assisted by plant-mediated strategy and their photocatalytic applications*” in fulfillment of degree of Master of Engineering at Xiamen University, People’s Republic of China.

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ORIGINAL STATEMENT

The research described in this thesis Masters of Engineering was conducted under the supervision of **Associate Professor Jiale Huang** at the Department of Chemical and Biochemical Engineering, Xiamen University. I hereby declare that the work submitted is my own and that appropriate credit has been given where reference has been made to the work of others. I also confirm that it has not been previously or concurrently submitted for any other degree, diploma or any other qualifications at Xiamen University, P.R China or other institutions.

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ABSTRACT

The harmful effects of organic pollutants especially dyes have received significant concerns due to their impact on human lives and the environment. Photocatalytic degradation of these dye pollutants with the assistance of semiconductors has been well established as efficient means of removing these pollutants. Different semiconductors have been applied as photocatalyst for the degradation of these pollutants, among them; ZnO has attained tremendous attention from researchers nowadays due to its strong adsorption ability, non-toxicity, high catalytic efficiency, wide band gap, chemical stability, large exciton binding energy and inexpensive nature. The above mentioned properties make ZnO a good photocatalyst. The performance of ZnO photocatalyst is strongly dependent on their size, surface area and morphology. As such the synthesis and shape control of ZnO micro/nanostructures have attracted much attention. Compared with traditional chemical and physical methods of fabricating ZnO micro/nanostructures, synthesis based on the use of plants have emerged as cost effective approach to attain highly stable and less toxic ZnO structures.

In this thesis, we report the use of precipitation assisted by *C. camphora* leaf extract to synthesize ZnO micro-flowers (ZnOMFs). The results showed that the ZnOMFs consisted of several nanoplates connecting together under the direction of the plant extract which acted as a bio-templating and stabilizing agent. FTIR analysis of the sample showed that biomolecules mainly polyols assisted the formation and guided the evolving shape of the ZnOMFs.

Characterization techniques such as UV–Vis diffuse reflectance absorption Spectrum (DRS), Energy-dispersive X-ray (EDX), Scanning electron microscopy (SEM), Transmission electron microscopy (TEM), Photoluminescence analysis (PL) and X-ray diffraction (XRD) were used to verify the nature of the obtained ZnOMFs. Well-defined ZnOMFs with statistical particle diameter of $0.81 \pm 0.2 \mu\text{m}$ could be synthesized by reacting 30 mL of aqueous $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (0.2 M) with 10 mL of *C. camphora* leaf extract, adjusting the pH of the mixture to 12 and annealed at 450 °C for 4 h. The average crystalline size was found to be 33 nm as per Scherrer's equation and BET surface area was 13 m²/g. XRD analysis confirmed the purity and high crystalline nature of the ZnOMFs and EDX spectrum verified that pure ZnO was

produced as it showed only peaks of Zn and O.

Also, adjusting the synthesis parameters such as the precursor concentration, dosage of leaf extract, and pH of the reaction solution were effective in tuning the morphology of the as-formed ZnO microstructures (ZnOMSs). Flowerlike ZnOMSs could be formed upon varying the plant extract from 5 to 20 mL but the formation of the ZnOMFs was not possible without the plant extract. ZnOMFs were only attained at pH of 12 but other pH levels led to the formation of different morphologies.

Photocatalytic property of the ZnOMFs was investigated by photodegradation of methyl orange (MO) under UV illumination. The ZnOMFs showed excellent photocatalytic activity for the degradation of MO. A very low catalyst loading of 0.05 g of ZnOMFs could degrade MO of initial concentration of 24 mg/L under 45 minutes of UV light exposure. Several operating variables such as initial MO concentrations, the ZnOMFs loading and pH of the reaction solution were found to have significant influence on the photocatalytic activity.

Keywords: Semiconductors, Zinc oxide, Micro-flowers, Microstructures, Photocatalyst, Photocatalytic degradation, Organic pollutants, Methyl orange, *C. camphora*

摘要

有机污染物中特别是染料，对人类生活和环境的不利影响，已经受到了社会的广泛关注。在半导体存在的条件下，光催化降解已成为一种除去染料污物的有效手段。许多不同的半导体已经被应用于光催化降解的过程中，其中，ZnO 由于其吸附能力强、无毒性、高催化效率、带隙宽、化学稳定性、激子结合能大和廉价等特点，已经引起了研究者极大的关注。而上述性质决定了 ZnO 具有良好的光催化性质。氧化锌的光催化性能与其尺寸、表面积和形貌有关，因此 ZnO 的微/纳米结构合成和形貌控制备受关注。与传统的化学和物理方法相比，利用植物法合成高度稳定的 ZnO 成本效益更高。

在本文中，我们采用芳樟叶浸出物合成氧化锌微花 (ZnOMFs)。结果表明，该氧化锌微花由几个纳米片组成，纳米片在植物浸出物溶液中，按一定方向组合在一起，其中植物浸出物作为生物模板和稳定剂。该氧化锌微花的 FTIR 分析表明，主要是生物分子主要是多元醇促使氧化锌微花的形成并引导其形状变化。

用各种表征技术如 UV-Vis 光谱、能量色散 X 射线 (EDX)、扫描电子显微镜 (SEM)、透射电子显微镜 (TEM)、光致发光分析 (PL) 和 X 射线衍射 (XRD) 来表征合成的氧化锌微花的性质。用 30 mL $Zn(NO_3)_2 \cdot 6H_2O$ (0.2M) 的水溶液与 10 mL 芳樟叶浸出物反应，调节混合物的 pH 值至 12，在 450°C 焙烧 4 小时制得的氧化锌微花粒径介于 $0.81 \pm 0.2 \mu m$ ，形貌规整。根据 Scherrer 公式得到，合成的氧化锌微花平均晶体尺寸是 33 nm，BET 表征测得比表面积为 $13 m^2/g$ 。XRD 分析证实了氧化锌微花纯度高以及结晶性良好。EDX 谱图只有 Zn 和 O 的峰，表明合成的为纯的 ZnO。

另外，通过改变合成参数，如前驱体的浓度、芳樟叶浸出物的用量和反应溶液的 pH 值，可以有效地改变氧化锌微结构的形貌。加入植物浸出液的浓度为 5-20 mL 时，可以合成花状的 ZnOMSs，但是不加入植物浸出物，则无法合成 ZnOMFs。只能在 pH 值为 12 时可获得 ZnOMFs，而其它的 pH 值下会得到不同形貌 ZnO。

该 ZnOMFs 对甲基橙 (MO) 有很好的光催化降解活性。在 UV 光照射下，仅用 0.05g ZnOMFs 催化剂，45 分钟就能将初始浓度为 24 mg/L 的 MO 降解完全。此外，其它条件如 MO 初始浓度、ZnOMFs 用量和反应液的 pH 值均对 ZnOMFs 光催化活性影响显著。

关键词： 半导体，氧化锌，微花，微结构，光触媒，光催化降解，有机污染物，甲基橙

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LIST OF ABBREVIATION, SYMBOLS AND TERMS

SEM	Scanning Electron Microscopy
TEM	Transmission Electron Microscopy
UV-Vis	Ultraviolet-Visible
XRD	X-ray Diffraction
FTIR	Fourier Transform Infrared spectroscopy
SAED	Selected Area Electron Diffraction
EDX	Energy-disperse X-ray
g	Gram
nm	Nanometre
h	Hour
M	Molar
mL	Milliliter
μ L	Microlitre
rpm	Revolution per minute
a.u	Arbitrary units
NPs	nanoparticles
μ m	Micrometer
NSs	Nanostructures
ZnO	Zinc oxide
TiO ₂	Titanium oxide
Fe ₂ O ₃	Iron II oxide
ZnS	Zinc sulfide
CdS	Cadmium sulfide
V ₂ O ₅	Vanadium pentoxide
eV	electron volt
MPa	MegaPascals
3D	3-dimensional
Zn(NO ₃) ₂ ·6H ₂ O	Zinc nitrate hexahydrate
ZnCl ₂	Zinc Chloride
ZnSO ₄	Zinc Sulfate
Zn(CH ₃ COO) ₂ ·2H ₂ O	Zinc acetate dihydrate
ZnOMFs	Zinc Oxide microflowers
ZnOMSs	Zinc Oxide microstructures
ZnOSPs	Zinc Oxide microsponge
NaOH	Sodium hydroxide

List of Abbreviations, Symbols and Terms

KOH	Potassium hydroxide
CTAB	exadecyltrimethylammonium bromide
TMAH	Tetramethylammonium hydroxides
ZEH	Zinc 2-ethylhexanoate
SDS	Sodium dodecyl sulfate
TEA	Triethanolamine
PVP	Polyvinylpyrrolidone
ZnONPs	Zinc oxide nanoparticles
C ₁₂ H ₂₇ N	Dodecylamine
C ₃ H ₉ N	Propylamine
(NH ₄) ₂ CO ₃	Ammonium carbonate
KBr	Potassium bromide
PL	Photoluminescence
MO	Methyl orange
DSSCs	Dye Sensitized Solar Cells
e ⁻ / h ⁺	Electron hole pair
OH [·]	Hydroxyl radicals
FWHM	Full width at half maximum
mg/L	milligram per litres

Chapter 1 Literature review

1.0 General Introduction

Recent increases in human society and intensification of industrial activities have brought about excessive discharge of organic pollutants into water bodies therefore causing environmental problems. Among these pollutants, organic dyes which are frequently used in textile, paint, plastic and cosmetic industries have received more attention as prominent environmental contaminants due to their harmful effects on humans.¹ Besides the toxicity, carcinogenic, and non-biodegradability nature of these dyes, they also affect the nature of water bodies as it obstructs sunlight penetration and reduce photosynthetic activity important for flora and fauna lives.² As such these organic chemicals found in wastewater effluents must be removed or destroyed before discharging them into the environment. However, the conventional techniques such as coagulation, filtration, adsorption by activated carbon and air stripping have been reported not efficient since they only transfer the pollutant from one phase to other and are relatively high cost techniques.³ It is therefore necessary to find an effective method to degrade organic pollutants from wastewater. Many researchers have reported photocatalytic oxidation by semiconductors as new cost effective and rapid technique for the removal of organic dye pollutants from water. Various semiconductors such as ZnO,⁴ ZnS,⁵ TiO₂,⁶ CdS,⁷ Fe₂O₃,⁸ V₂O₅,⁹ etc have been well reported to be good photocatalyst. Among these photocatalysts, Zinc oxide (ZnO) micro/nanostructures with low toxicity and production cost, have been of special interest because of their distinctive properties like excellent chemical/thermal stability, wide direct band gap of 3.37 eV, high catalytic efficiency, high exciton binding energy of 60 meV, and strong adsorption ability have been found to be a promising and potential photocatalyst for degradation of various organic pollutants.

Researchers have proven that the activity of photocatalysts is strongly dependent on their microstructures/nanostructures and morphology i.e., the arrangements of atoms in their crystal structures. Anisotropic nanostructures have been well documented to have enhanced performance over their bulk counterparts due to their large surface area to volume ratio and

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