بسم الله الرحمن الرحيم

MONA MAGHRABY

شبكة المعلومات الجامعية

التوثيق الالكتروني والميكروفيلم

MONA MAGHRABY

شبكة المعلومات الجامعية

التوثيق الإلكتروني والميكروفيلم

جامعة عين شمس

التوثيق الإلكتروني والميكروفيلم

قسم

نقسم بالله العظيم أن المادة التي تم توثيقها وتسجيلها
علي هذه الأقراص المدمجة قد أعدت دون أية تغيرات

يجب أن

تحفظ هذه الأقراص المدمجة بعيدا عن الغبار

MONA MAGHRABY

Ain Shams University **Faculty of Science**

Synthesis of some nanocomposites based on WO_3 and their photocatalytic applications

A Thesis submitted for

By

Eman Ali Mohamed Torad

M.Sc. Inorganic Chemistry – Ain Shams University (2015)

For the requirement of the Ph.D. Degree of Science in Chemistry

Under Supervision of

Prof. Dr. Mostafa Mohamed Khalil

Prof. of Inorganic Chemistry, Faculty of Science, Ain Shams University

Prof. Dr. Eman Hameed Sayed Ismail

Prof. of Inorganic Chemistry, Faculty of Science, Ain Shams University

Prof. Dr. Mohamed Mokhtar Mohamed

Prof. of physical Chemistry, Faculty of Science, Benha University

Dr. Gehad Mohamed Ahmed Attia

Lecturer of Chemistry, Faculty of Science, Ain Shams University

To

Department of Chemistry

Faculty of Science, Ain Shams University

2021

Ain Shams University

Faculty of Science

APPROVAL SHEET FOR SUBMISSION

Title of thesis:

"Synthesis of some nanocomposites based on WO_3 and their photocatalytic application"

By

Eman Ali Mohamed Torad

M.Sc. Inorganic Chemistry Ain Shams 201٥

The thesis has been approved for submission by the supervisors:

Signature

Prof. Dr. Mostafa Mohamed Khali ……

Prof. of Inorganic Chemistry, Faculty of Science,
Ain Shams University

Prof. Dr. Mohamed Mokhtar Mohamed ……..

Prof. of physical Chemistry, Faculty of Science, Benha University

Prof. Dr. Eman Hameed Sayed Ismail ……..

Prof. of Inorganic Chemistry, Faculty of Science, Ain Shams University

Dr. Gehad Mohamed Ahmed Attia ……..

Lecturer of Chemistry, Faculty of Science, Ain Shams University

Head of Chemistry Department

Prof. Dr. Ayman Ayoub Abdel- Shafi

Ain Shams University

Faculty of Science

Researcher Data

Name: Eman Ali Mohamed Torad

Date of Birth: 06/03/1990

Academic Degree: MSc. Degree of Science

Field of specification: Inorganic Chemistry

University issued the Degree: Ain Shams University - Faculty of Science- Inorganic Chemistry Department

Graduation year: 2010

Date of M.Sc. Degree: 2015

ACKNOWLEDGMENT

Words are not enough to describe my deep thank to ***Prof. Dr. Mostafa Hassan Khalil,*** *Professor of Analytical chemistry, Faculty of Science, Ain Shams University and* ***Prof. Dr. Mohamed Mokhtar Mohamed,*** *Professor of Physical Chemistry, Faculty of Science, Benha University, for suggesting the point of the research and for management of this work, their guidance and supervision in the course of the work, and for their stimulating criticisms, support and help in the preparation of the thesis throughout the course of this work. They taught me how I can be a student seeks to research and knowledge.*

I would like to express my deep thanks and gratitude to ***Prof. Dr. Eman Hamed Sayed,*** *Professor of Analytical chemistry, Faculty of Science, Ain Shams University and* ***Dr.Gehad Attia****, lecturer of Analytical chemistry, Faculty of Science, Ain Shams University, for their valuable help and support throughout this work.*

Also, I offer my thanks and appreciations to all of those who supported me in any respect in the Chemistry Department during the completion of this thesis.

Last but not Least, my thanks to my family, especially my father, my mother and my husband, for their support and encouragement that gave me the strength to finish this work.

Eman Ali Mohamed

Materials Research Bulletin 137 (2021) 111193

Contents lists available at ScienceDirect

Materials Research Bulletin

journal homepage: www.elsevier.com/locate/matresbu

Tuning the redox potential of $Ag@Ag_2O/WO_3$ and $Ag@Ag_2S/WO_3$ photocatalysts toward diclofenac oxidation and nitrophenol reduction

Eman Torad [a], Eman H. Ismail [a], Mohamed Mokhtar Mohamed [b,*], Mostafa M.H. Khalil [a,*]

[a] Chemistry Department, Faculty of Science, Ain Shams University, Abbassia, 11566, Cairo, Egypt
[b] Chemistry Department, Faculty of Science, Benha University, Benha, 11566, Egypt

ARTICLE INFO

Keywords:
Ag_2O/WO_3
Photo-fenton
Diclofenac oxidtion
Nitrophenol reduction
Visible light

ABSTRACT

WO_3 nanoplates modified with either Ag_2O or Ag_2S nano-architectures were synthesized by a deposition-hydrothermal route (180 °C for 5 h). They were characterized using X-ray powder diffraction, N_2 sorptiometry, Transmission electron microscopy, UV–vis diffuse reflectance spectroscopy, Photoluminescence spectroscopy, and X-ray photoelectron spectroscopy. The photo-catalytic ($\lambda > 420$ nm, 160 W) degradation of Diclofenac (DCF; 60 mg/l), was achieved using H_2O_2 (1×10^{-4} M) with either $Ag@Ag_2O/WO_3$ ($K = 32.0 \times 10^{-3}$ min^{-1}) or $Ag@Ag_2S/WO_3$ ($K = 7.3 \times 10^{-3}$ min^{-1}) catalysts. In the case of DCF degradation using radical scavengers, $^{\bullet}O_2^-$ played a key role in the degradation process whilst $^{\bullet}OH$ and holes acted moderately minor roles. The possible DCF degradation paths and intermediates were assessed by LC–MS. Both $Ag@Ag_2O/WO_3$ and $Ag@Ag_2S/WO_3$ catalysts were used in the photo-reduction of 4-nitrophenol (4-NP; 1.8×10^{-4} M) to 4-aminophenol (4-AP) with rate constants equal 8.3 **x** 10^{-3} min^{-1} and 1.6 **x** 10^{-3} min^{-1}, respectively.

1. Introduction

Water purification includes water decontamination and disinfection using photocatalysis is widely studied [1–3]. Pharmaceuticals exhibit a class of water pollutants which are potentially harmful even in trace amounts [4]. Diclofenac (DCF), is generally exploited as anti-aching, anti-rheumatic and analgesic agent. Following DCF human intake, 15 % of it is excreted without any change [5]. It is found to be regularly abundant in municipal wastewaters because its bio-degradation in waste-water remedy plants is restricted [6]. As many of these pharmaceutical pollutants are not remediated by biological methods, diclofenac is widely present all over surface waters in Europe [7], and thus leads to severe environmental risks. Lately, great efforts are made to provide effective and new treatment processes for DCF removal, including sonolysis oxidation [8], ozonation [9], photocatalysis [10] and photo-Fenton catalysis route [11].

Therefore, the DCF degradation alone or along with other drugs, provoked by various advanced oxidation processes (AOPs) has been inspected [12–15]. Among these AOPs, visible light photocatalysis techniques have shown an effective degradation of water contaminants via harvesting solar/visible-light irradiation energy that is cost-effective than the UV energy. The DCF degradation rate constant increased gradually with increasing pH from 8 to 12 [14–15]. Thus, using an oxidizing agent as $^{\bullet}OH$, generated from hydrogen peroxide activation, is preferred than $SO_4^{\bullet -}$, generated from persulfate ($S_2O_8^{2-}$) activation for DCF degradation because alkalinity of water samples is negatively influences the efficiency of the $S_2O_8^{2-}$ moieties [16–17].

Aromatic amines are organic contaminants created as sideways outcomes or intermediates of diverse industrialized products such as pharmaceuticals, make-ups, agro-chemicals, photographic compounds and complex mediators [18–21]. Many attempts of investigations for the reduction of p-nitro-phenol have been achieved in the last few years [22, 23]. The hydrogenation of aliphatic [24] or aromatic [25] nitro-compounds, via electrolytic reduction and metal/acid reduction, is hard to be achieved when reactive substituents such as Cl, CH_3 and OH are attached. Thus, investigators aimed to use modern techniques such as photo-catalysis via visible-light reduction to change the organic functionality.

Among the various AOPs, tungsten trioxide (WO_3), a photo-chromic n-type semiconductor with a band gap ranging between 2.4–2.8 eV is an appropriate challenger for sunlight photo-catalytic applications [26,27]. It is thought to be a promising photo-catalyst due to its low cost, chemical inertness, non-toxicity, distinctive electronic and optical properties and low density. It has a strong oxidizing power and good

* Corresponding authors.
E-mail addresses: mohmok2000@yahoo.com, mohamed.mokhtar@fsc.bu.edu.eg (M.M. Mohamed).

https://doi.org/10.1016/j.materresbull.2020.111193
Received 8 October 2020; Received in revised form 18 December 2020; Accepted 19 December 2020
Available online 31 December 2020

Contents

Contents