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EDITORS

Pn. Rosliza Ali

Pn. Nunshaimah Salleh

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Pn. Yanti Yaacob

Pn. Lili Widarti Zainuddin

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Faculty of Applied Sciences,
Universiti Teknologi MARA,
Perak Branch Tapah Campus,
35400 Tapah Road,
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Preface

The Scientific Project Colloquium offers a platform for publishing Diploma Science final year projects (FYP). The objective is to effectively distribute research findings throughout all scientific disciplines. The primary objective of including final year projects into the course curriculum is to encourage students to put their theoretical knowledge into practical applications.

We would like to express our gratitude to our primary establishment, the Faculty of Applied Sciences and Universiti Teknologi MARA, Perak Branch, for their invaluable assistance.

Lastly, we would like to express our gratitude to all of the authors for the tremendous help in preparing the articles, without which this undertaking would not have been completed.

Editors

Rosliza Ali

Nunshaimah Salleh

Norsakina Zurina Zulkifli

Adibatul Husna Fadzil

Yanti Yaacob

Lili Widarti Zainuddin

Universiti Teknologi MARA

Perak Branch Tapah Campus

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SYNTHESIS OF COBALT DOPED IRON OXIDE FOR POLLUTANTS DEGRADATION UNDER SUNLIGHT

¹Jannatul Ma'wa Abdul Rashid, ¹Nur Azmina Mohammad Repin, ^{1,2*}Ahmad Nazeer Che Mat, ¹Mohd Rabani Yaafar, ^{1,2}Nurul Izza Taib

¹Faculty of Applied Science, Universiti Teknologi MARA, Perak Branch, Tapah Campus, 35400 Tapah Road, Perak, Malaysia

²Advanced Materials for Environmental Research Initiative Group, Universiti Teknologi MARA Perak Branch, Tapah Campus, 35400 Tapah Road, Perak, Malaysia

*nazeer@uitm.edu.my

Abstract: A series of different percentage of Cobalt-doped iron(III) oxide namely 1%, 3%, 5%, 7%, and 10% have been successfully synthesized using precipitation method in aqueous solution and tested for photodegradation of methyl orange (MO) under visible light irradiation. The as-prepared photocatalysts were characterized using Ultraviolet-Visible (UV-Vis) and Fourier Transform Infrared (FTIR) spectroscopy for their band gap energy, photocatalytic activity and chemical bonding, respectively. Doping with Co enable the reduction of band gap energy of the as-prepared photocatalyst where 7% Co dopant showed the lowest value of band gap energy (2.40 eV) as compared to the pristine Fe₂O₃ (2.6 eV). The photodegradation of MO has reached the highest activity over 7% Co doped Fe₂O₃ whilst further addition of CO (10%), the degradation of MO remain unchanged.

Keywords: Cobalt doped iron oxide; Precipitation method; Photocatalyst; Methyl orange degradation; Visible light

INTRODUCTION

In Malaysia, we are fortunate to have abundant of natural resources such as water. However, rapid development owing to uncontrolled human activities has polluted water resources such as lakes, rivers, groundwater and oceans with toxic waste from factories, oil spills in the ocean and garbage dumped by local residents into rivers (Al-Taai, 2021; Singh & Gupta, 2016). One of the common pollutants is dye which has been used in various sectors. Dyes can be divided into two types namely natural dyes and synthetic dyes (Tamilarasi & Banuchitra, 2021). Synthetic dyes are significantly more harmful to water as compared to natural dyes due to their complex structures which are difficult to break down, hence persist in the environment (Tamilarasi & Banuchitra, 2021). Some dyes are hazardous to aquatic life, and their colours obstruct sunlight, which harms plant life and the overall ecology. Methyl orange dyes (C₁₄H₄N₃NaO₃S), are major pollutants emitted by the textile, pharmaceutical, printing sectors, creating water contamination and making wastewater treatment difficult. These colours are poisonous in nature, even in low quantities, and may lead to cancer in humans (Alyasi et al., 2023). Methyl orange (MO) dye is resistant in nature and difficult to degrade because the dye is stable, non-biodegradable, and soluble in water. If it is discharged into soil and aquatic resources, it might pose major hazards to the environment and human health. It is also harmful to plant growth (Kishor et al., 2021). Fe₂O₃ is a promising material for various applications such as photocatalyst (Adesibikan et al., 2024), medical purposes especially for drug delivery and cancer treatment (Rezaei et al., 2024), magnetic materials for wastewater treatment (Keshta et al., 2024), and sensors (Muhammad et al., 2024). Fe₂O₃ also has been synthesized using various method for example an α -Fe₂O₃ has been prepared recently through combustion process using glucose and sucrose as organic fuel for methyl orange degradation under Ultraviolet (UV) irradiation by RK Shah. The photodegradation efficiency of methyl orange reached 95.31% over α -Fe₂O₃ using sucrose as compared to glucose (82.17%) as organic fuel (Shah, 2023). Recently, α -Fe₂O₃ has been synthesized biologically using *Trigonella foenum-graecum* seed extract to stabilize Fe nanoparticle for photocatalytic degradation of MO. The degradation of MO over Fe nanoparticles under UV light followed the first order kinetics with rate constant of 0.025 min⁻¹ and 95% photocatalytic efficiency (Radini et al., 2018). Moreover, Sharma et al reported the preparation of a well-crystalline rhombohedral of α -Fe₂O₃ nanoparticle using a facile solution reaction in the presence of NaOH and hexamethylenetetramine (HMTA) and tested for MO photodegradation reaction. Their study revealed that 0.20 g of α -Fe₂O₃ possesses the highest MO photodegradation reaction with 85 % efficiency for 210 minutes under UV light irradiation as compared to other dosage (Sharma et al., 2014). However, as an n-type semiconductor with band gap energy in the range of 1.9 – 2.2 eV, Fe₂O₃ has been implemented as photocatalyst for pollutants degradation for a couple of years owing to its stability and low cost (He et al., 2016). Nevertheless, low surface reaction rates, low carrier mobility (<1 cm²/Vs, high recombination rates of exciton, and a short hole diffusion length retards its overall photocatalytic activity

(Mirbagheri et al., 2014). Scientists address this issue by doping Fe_2O_3 with various metal such as Ti, Zn, Ni, Sn, Al, Mg, Ga, Rh, and Zr (Kumar et al., 2019; Yuan et al., 2017) to modify the physicochemical properties of Fe_2O_3 photocatalysts. The role of metal such as Sn in minimizing the value of band gap energy can be seen by the recent investigation of N.A. Arzaee et al. with different percentage of Sn doped Fe_2O_3 namely 0.5, 3, 5 and 8% of Sn. The band gap value seems to decrease to 2.06, 2.08, and 2.09 eV for Fe_2O_3 doped with 0.5, 3, and 5% Sn and increased to 2.20 eV for 8% Sn. The decrement in the band gap energy indicates that the absorption ability of Fe_2O_3 towards visible light increases leading to formation of more photogenerated charge carriers thus enhanced the direct degradation process of dye through the $\text{OH}\cdot$ degradation mechanism (Arzaee et al., 2023). Amongst the as-prepared Sn doped Fe_2O_3 photocatalyst, 5% Sn was found to exhibit the highest photocatalytic activity towards MO degradation of 100 % efficiency for 120 min in the solution with pH value of 5.3 and 10 ppm MO concentration. Recently, a green synthetic pathway utilizing an extract of *Azadirachta indica* leave as a replacement for toxic chemical reducing agents was used to prepare hematite ($\alpha\text{-Fe}_2\text{O}_3$) which later on was doped with Co for degradation of MO under UV lamp. The study found that, the degradation efficiency of MO over green nanoparticles was higher (89 - 95%) if compared to that of the chemically generated Fe_2O_3 photocatalyst (88–92 %). Moreover, the presence of Co as dopant managed to lower the band gap energy of the photocatalyst in the range of 2.48 – 2.61 eV (Kumar et al., 2023). The present study intent to synthesize Co doped Fe_2O_3 photocatalyst nanoparticle using simple precipitation method in aqueous solution without any chemical reducing agents for photodegradation of methyl orange (MO). The as-prepared photocatalyst nanoparticle will be characterized using FTIR and UV-Vis for the investigations of functional group, band gap energy, and photocatalytic degradation of MO, respectively.

METHODOLOGY

1.0 Materials

Cobalt (II) chloride hexahydrate was purchased from R&M Chemicals, iron(III) chloride, was obtained from Laboratory Reagent and ammonia solution was procured from R&M Chemicals. All of the chemicals were received in a good condition and are utilized without undergoing any further chemical purifications.

2.0 Experimental

2.1 Synthesis of Fe_2O_3 nanoparticle photocatalyst

Precipitation method is used to synthesize Fe_2O_3 nanoparticle photocatalysts. In brief, 6 mmol of iron(III) chloride was completely dissolved in 100 mL of distilled water under continuous stirring at ambient temperature. Later on, a dilute solution of ammonia was added to the solution to yield iron(III) hydroxide followed by gravitational filtration before underwent oven dried at 120 °C overnight. The dried iron(III) hydroxide thereafter was grounded into a fine powder before underwent calcination process at 400 °C for 3 hours.

2.2 Synthesis of Co-doped Fe_2O_3 nanoparticle photocatalyst

In the present study, five different amounts of Cobalt-doped iron(III) oxide nanoparticle photocatalyst have been successfully prepared which are denoted as 1CFO, 3CFO, 5CFO, 7CFO, and 10CFO corresponds to 1, 3, 5, 7, and 10 wt% Co-doped Fe_2O_3 nanoparticle photocatalyst and denoted as 1CFO, 3CFO, 5CFO, 7CFO, and 10CFO, respectively. Concisely, for the preparation of 1CFO, 100 mL of distilled water was used to completely dissolve 13 mmol of FeCl_3 under continuous stirring at room temperature followed by the addition of $\text{CoCl}_2\cdot 6\text{H}_2\text{O}$ (0.3 mmol) to obtain a clear solution. Hydroxide of the respective metal salts were obtained by the addition of diluted ammonia solution followed by filtration before subjected to oven dried at 120 °C for 12 hours. Afterwards, the as-prepared 1CFO photocatalyst was manually grounded using agate mortar and pestle into a fine powder followed by a calcination process at 400°C for 3 hours. The same experimental procedure and conditions were adopted in preparing the other types of Co-doped Fe_2O_3 nanoparticle photocatalyst namely 3, 5, 7, and 10CFO with the appropriate amount of metal salts in aqueous solution.

2.3 Characterizations of Co-Fe₂O₃

The chemical bonding of Co doped Fe₂O₃ was obtained using FTIR-ATR spectrometer in the 4000–250 cm⁻¹ region. Meanwhile, the band gap energy of the material was obtained using Cary 5000 UV-Vis-NIR Spectrophotometer version 1.12, Agilent in the range of 200 – 1000 nm.

2.4 Photocatalytic degradation of Methyl Orange (MO)

Photocatalytic degradation of MO was examined using 20 ppm MO in aqueous solution. Upon photocatalytic degradation of MO, 100 mL of MO as poured into a beaker with continuous stirring in dark for 30 minutes to allow the equilibrium between adsorption and desorption processes. Later on, photocatalytic degradation of MO will be performed for every 30 minutes interval for 2 hours under 150 W UV lamp. The photodegradation of MO over the as-prepared Co-doped Fe₂O₃ nanoparticle will be monitored by taking the samples periodically and examined using UV-Vis spectrophotometer.

FINDINGS

Chemical bonding of the as-prepared Co-doped Fe₂O₃ nanoparticles was shown in Figure 1 below. The weakly intense band at 3416 cm⁻¹ is associated with the stretching mode of hydroxyl, O-H group (Niraimathees et al., 2016). The band near 1628 cm⁻¹ is assigned to the H-O-H bending vibration of water present in atmosphere (Atul et al., 2019; Satheesh et al., 2014). Following that, the observed band at 1402 cm⁻¹ is associated with carbon dioxide vibrations absorbed on the surface of nanoparticles (Parhizkar & Habibi, 2019). The closest value to the reference, the band at 1044 cm⁻¹, clarifies the properties of CoFe₂O₄. This could be due to the residual FeOOH (Parhizkar & Habibi, 2019). The band at 784 cm⁻¹, which is close to the reference value, indicates the formation of Fe-OH-Fe (Du et al., 2020). The band at 540 cm⁻¹ is probably Fe-O stretching vibration of α-Fe₂O₃. The band position of α-Fe₂O₃ is shifted towards a higher wavenumber as the concentration of Co²⁺ increases. The shifting results from Co²⁺ doping disrupting the Fe-O bonds (Suresh et al., 2017).

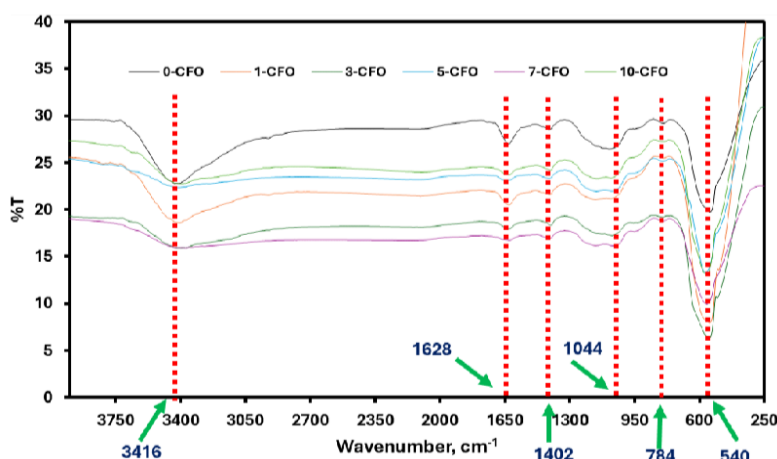


Figure 1. FTIR spectrum of Co doped Fe₂O₃.

The band gap energy of the as-prepared materials were determined by Kubelka-Munk method using UV-Vis-NIR diffuse reflectance spectrometer according to the equation (1) shown below.

$$F(R) = \frac{(1-R)^2}{2R} = \frac{K}{S} \quad (1)$$

where R is the measured reflectance of sample, K is the molar absorption coefficient while S is the scattering coefficient. The band gap energy of the sample can be obtained by plotting $(F(R) hv)^2$ against energy, hv (1240 eV/ wavelength, nm) which is shown in Figure 2 for (a) Fe₂O₃ and (b) 7% Co doped Fe₂O₃, respectively. The band gap energy of the as-prepared photocatalyst slightly decrease after doping with Co with the lowest value of 2.4 eV especially for 7% Co doping as compared to the bare Fe₂O₃ (2.6 eV) which could enhance the photocatalytic activity towards degradation of methyl orange. The photocatalytic degradation of methyl orange for the two hours duration is shown in Figure 3. The photocatalytic degradation of MO was conducted with and without Co in the dark and under light irradiation. In the dark and without the presence of Co, the photodegradation

of MO occurred ineffectively indicated by the higher value of absorbance from UV-Vis analysis. However, the photodegradation of MO underwent efficiently over 7% Co doped Fe_2O_3 (7CFO) as compared to other sample for 2 hours. As the amount of Co increases to 10%, the photocatalytic degradation of MO remains slightly the same as 7% Co doped Fe_2O_3 most probably at higher dosage of Co, the surface properties of Fe_2O_3 were changed owing to surface coverage by Co that might reduce the active site on the surface (Arzaee et al., 2023).

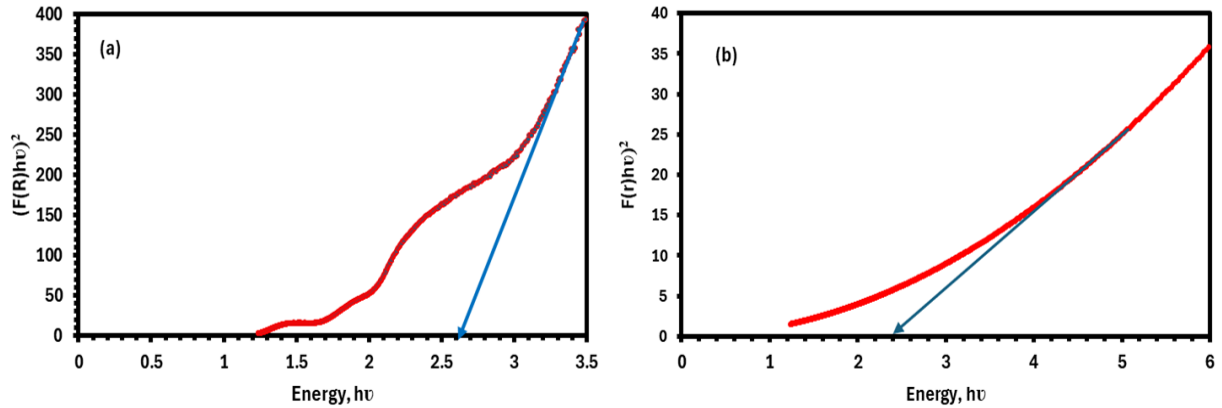


Figure 2. Band gap energy of (a) Pristine Fe_2O_3 ; (b) 7% Co doped Fe_2O_3

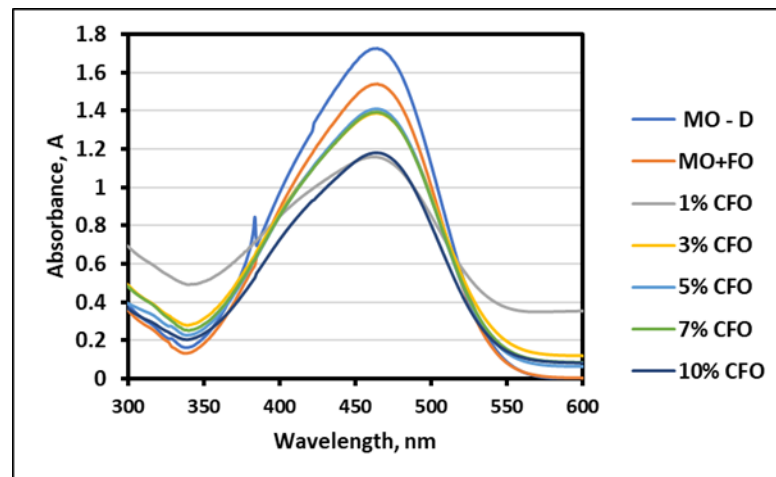


Figure 3. Photocatalytic degradation reaction of methyl orange over bare Fe_2O_3 and 1 – 10% Co doped Fe_2O_3 (CFO).

CONCLUSIONS

The precipitation method was successfully used to synthesize iron(III) oxide and cobalt-doped iron (III) oxide nanoparticles. The peaks indicated by FTIR analysis attest to the formation of Fe-O bond in addition to the formation of CoFe_2O_4 . Doping Fe_2O_3 with Co, showed that the band gap energy slightly decrease indicative of effective photodegradation activity of MO. Furthermore, UV-Vis analysis shows that the absorbance of methyl oranges is decreasing for the duration of two hours indicating the as-prepared photocatalyst were effective towards degradation of MO until it reached the maximum at 7% amount of Co.

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Tarikh : 20 Januari 2023



Prof. Madya Dr. Nur Hisham Ibrahim
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Sekian, terima kasih.

“BERKHIDMAT UNTUK NEGARA”

Saya yang menjalankan amanah,

SITI BASRIYAH SHAIK BAHARUDIN
Timbalan Ketua Pustakawan

nar

Setuju.

27.1.2023

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