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Table of contents

Volume 202

2017

[◀ Previous issue](#) [Next issue ▶](#)

**The 4th International Conference on Advanced Materials Science and Technology
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[View all abstracts](#)

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Preface

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[+ View abstract](#) [View article](#) [PDF](#)

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[+ View abstract](#) [View article](#) [PDF](#)

OPEN ACCESS 011003

Maps and Photographs

[+ View abstract](#) [View article](#) [PDF](#)

OPEN ACCESS 011004

Peer review statement

[+ View abstract](#) [View article](#) [PDF](#)

Papers

OPEN ACCESS

012001

Spin-Parity Behavior in the Exchange-Coupled Lanthanoid-Nitroxide Molecular Magnets

T Ishida

[+ View abstract](#)[View article](#)[PDF](#)

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012002

Magneto-Structural Relationship on Strong Exchange Interactions between Chelating Nitroxide Radical and Transition-Metal Spins

A Okazawa

[+ View abstract](#)[View article](#)[PDF](#)

OPEN ACCESS

012003

Fabrication of Mesoporous Silica/Alumina Hybrid Membrane Film Nanocomposites using Template Sol-Gel Synthesis of Amphiphilic Triphenylene

H O Lintang, M A Jalani, L Yuliati and M M Salleh

[+ View abstract](#)[View article](#)[PDF](#)

OPEN ACCESS

012004

Preparation of Low fouling Polyethersulfone Membranes by Simultaneously Phase Separation and Redox Polymerization

A Roihatin and H Susanto

[+ View abstract](#)[View article](#)[PDF](#)

OPEN ACCESS

012005

Crystallinity and Electrical Conductivity of PANI-Ag/Ni Film: The Role of Ultrasonic and Silver Doped

M Diantoro, I N Fitriana, F Parasmayanti, Nasikhudin, A Taufiq, Sunaryono, N Mufti and H Nur

[+ View abstract](#)[View article](#)[PDF](#)

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012006

The Effect of Thickness of ZnO Thin Films on Hydrophobic Self-Cleaning Properties

N Mufti, D Arista, M Diantoro, A Fuad, A Taufiq and Sunaryono

[+ View abstract](#)[View article](#)[PDF](#)

OPEN ACCESS

- Optimalization of Freezing-Thawing Process in Enhancing Magnetic Properties of $\text{Fe}_3\text{O}_4/\text{PAA}/\text{PVA}$ Magnetic Hydrogel Composites 012007
Sunaryono, H Hifdziyah, A Taufiq, M Diantoro and N Mufti
[+](#) View abstract [View article](#) [PDF](#)
-
- OPEN ACCESS** 012008
Fabrication of Magnetite Nanoparticles Dispersed in Olive Oil and Their Structural and Magnetic Investigations
A Taufiq, R E Saputro, Sunaryono, N Hidayat, A Hidayat, N Mufti, M Diantoro, A Patriati, Mujamilah, E G R Putra and H Nur
[+](#) View abstract [View article](#) [PDF](#)
-
- OPEN ACCESS** 012009
Magnetically Separable $\text{Fe}_3\text{O}_4/\text{SnO}_2/\text{Graphene}$ Adsorbent for Waste Water Removal
V Paramarta, A Taufik and R Saleh
[+](#) View abstract [View article](#) [PDF](#)
-
- OPEN ACCESS** 012010
Synthesis of High-Impact Polystyrene Fibers using Electrospinning
A Zulfi, A Fauzi, D Edikresnha, M M Munir and Khairurrijal
[+](#) View abstract [View article](#) [PDF](#)
-
- OPEN ACCESS** 012011
Preparation of PVA/TiO_2 Composites Nanofibers by using Electrospinning Method for Photocatalytic Degradation
Nasikhudin, E P Ismaya, M Diantoro, A Kusumaatmaja and K Triyana
[+](#) View abstract [View article](#) [PDF](#)
-
- OPEN ACCESS** 012012
Synthesis, Single Crystal Structure, and Magnetic Properties of 3-D $\text{Cu}(\text{NITpPy})_2[\text{Cu}(\text{CN})_3] \cdot 2\text{CH}_3\text{OH} \cdot 2\text{H}_2\text{O}$ Complexes
I W Dasna, S Golhen, L Ouahab and Subakti
[+](#) View abstract [View article](#) [PDF](#)
-
- OPEN ACCESS** 012013
Preparation of $\text{MWCNT}-\text{Fe}_3\text{O}_4$ Nanocomposites from Iron Sand Using Sonochemical Route

R Rahmawati, A Melati, A Taufiq, Sunaryono, M Diantoro, B Yulianto, S Suyatman, N Nugraha and D Kurniadi

[+](#) View abstract [View article](#) [PDF](#)

OPEN ACCESS 012014

The Scale Formation of Barite (BaSO_4) from Laminar Flowing Water in The Presence of Tartaric Acid and Ba^{2+} Concentration Variation of Solution

F Fatra, G Ivanto, N S Dera, S Muryanto and A P Bayuseno

[+](#) View abstract [View article](#) [PDF](#)

OPEN ACCESS 012015

Citric Acid Addition to Controlling Crystallization of Barium Sulphate (BaSO_4) in Pipes through Ba^{2+} Concentration Variation in the Solution

G Ivanto, F Fatra, N S Dera, S Muryanto and A P Bayuseno

[+](#) View abstract [View article](#) [PDF](#)

OPEN ACCESS 012016

Phase Transformation of Limonite Nickel Ores with Na_2SO_4 Addition in Selective Reduction Process

W Mayangsari and A B Prasetyo

[+](#) View abstract [View article](#) [PDF](#)

OPEN ACCESS 012017

The Use of Heterogeneous Catalysts of Chitosan Sulfonate Bead on the Esterification Reaction of Oleic Acid and Methanol

H N Chamidy and Riniati

[+](#) View abstract [View article](#) [PDF](#)

OPEN ACCESS 012018

CaSO_4 Scale Inhibition by a Trace Amount of Zinc Ion in Piping System

W Mangestiyono and Sutrisno

[+](#) View abstract [View article](#) [PDF](#)

OPEN ACCESS 012019

Low-Temperature Carbothermic Reduction of Indonesia Nickel Lateritic Ore with Sub-Bituminous Coal

I Setiawan, S Harjanto and R Subagja

[+](#) View abstract [View article](#) [PDF](#)

-
- OPEN ACCESS** 012020
- Preparation of Chitosan/Collagen Blend Membranes for Wound Dressing: A Study on FTIR Spectroscopy and Mechanical Properties
- D J Indrani, F Lukitowati and Y Yulizar
- [+](#) View abstract [View article](#) [PDF](#)
-
- OPEN ACCESS** 012021
- Solid State Reaction Synthesis of Si-HA as Potential Biomedical Material: An Endeavor to Enhance the Added Value of Indonesian Mineral Resources
- Hartatiek, Yudyanto, S D Ratnasari, R Y Windari and N Hidayat
- [+](#) View abstract [View article](#) [PDF](#)
-
- OPEN ACCESS** 012022
- Aging Time Effect on Porous Characteristics of Natural Mud-based Silica Prepared by Hydrothermal-Coprecipitation Route
- A Ubaid, N Hidayat and Munasir
- [+](#) View abstract [View article](#) [PDF](#)
-
- OPEN ACCESS** 012023
- Magnetic Susceptibility and Morphology of Natural Magnetic Mineral Deposit in Vicinity of Human's Living
- S Zulaikah, R Azzahro, S B Pranita, E S Mu'alimah, N Munfarikha, Dewiningsih, W L Fitria and H A Niarta
- [+](#) View abstract [View article](#) [PDF](#)
-
- OPEN ACCESS** 012024
- Preparation of Superparamagnetic $Zn_{0.5}Mn_{0.5}Fe_2O_4$ Particle by Coprecipitation-Sonochemical Method for Radar Absorbing Material
- A Taufiq, S Bahtiar, Sunaryono, N Hidayat, A Hidayat, N Mufti, M Diantoro, A Fuad, Munasir, R Rahmawati, W A Adi, S Pratapa and Darminto
- [+](#) View abstract [View article](#) [PDF](#)
-
- OPEN ACCESS** 012025
- The Effect of Sintering Temperature on The Rolled Silver-Sheathed Monofilament Bi,Pb-Sr-Ca-Cu-O Superconducting Wire
- Hendrik, P Sebleku, B Siswayanti and A W Pramono
- [+](#) View abstract [View article](#) [PDF](#)

-
- OPEN ACCESS** 012026
- Low-Temperature Nitriding of Pure Titanium by using Hollow Cathode RF-DC Plasma
- J M Windajanti, D J Djoko H S and Abdurrouf
- [+](#) View abstract [View article](#) [PDF](#)
-
- OPEN ACCESS** 012027
- Optical Properties Characterization of Gamma Irradiated CeO₂ Nanoparticles Solution
- I Nurhasanah, A Luthfia and Z Arifin
- [+](#) View abstract [View article](#) [PDF](#)
-
- OPEN ACCESS** 012028
- Microstructures and Mechanical Study of Mg Alloy Foam Based on Mg-Zn-Ca-CaCO₃ System
- A Erryani, F Pramuji, D Annur, M I Amal and I Kartika
- [+](#) View abstract [View article](#) [PDF](#)
-
- OPEN ACCESS** 012029
- Phase Analysis and Crystal Morphology of Barium Sulphate Precipitated from The Laminar Flowing Water
- N S Dera, F Fatra, G Ivanto, S Muryanto and A P Bayuseno
- [+](#) View abstract [View article](#) [PDF](#)
-
- OPEN ACCESS** 012030
- Noninvasive and Painless Urine Glucose Detection by Using Computer-based Polarimeter
- Sutrisno, Y A Laksono and N Hidayat
- [+](#) View abstract [View article](#) [PDF](#)
-
- OPEN ACCESS** 012031
- Inexpensive Home-Made Single Wavelength Ellipsometer ($\lambda = 633$ nm) for Measuring the Optical Constant of Nanostructured Materials
- L Z Maulana, K Megasari, E Suharyadi, R Anugraha, K Abraha and I Santoso
- [+](#) View abstract [View article](#) [PDF](#)
-
- OPEN ACCESS** 012032
- Effect of Fe₃O₄ Magnetic Nanoparticle Concentration on the Signal of Surface Plasmon Resonance (SPR) Spectroscopy

M Oktivina, D T Nurrohman, A N Q Z Rinto, E Suharyadi and K Abraha

[+](#) View abstract [View article](#) [PDF](#)

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012033

Effects of Sintering Holding Time on the Structural, Electrical and Magnetic Properties of $Zn_{0.95}Ni_{0.05}O$

M Ginting, D Aryanto, C Kurniawan, A Y Sari, A Subhan, T Sudiro, P Sebayang, E R Tarigan, M N Nasruddin and K Sebayang

[+](#) View abstract [View article](#) [PDF](#)

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012034

Synthesis and Characterization of Ti-6Al-6Mo Prepared by Arc Melting Process

G Senopati, I N G Putrayasa and A C Sutowo

[+](#) View abstract [View article](#) [PDF](#)

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012035

Corrosion Behavior of Magnesium Based Foam Structure in Hank's Solution

P L Franciska, A Erryani, D Annur and I Kartika

[+](#) View abstract [View article](#) [PDF](#)

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012036

The Effect of Substrate Temperature on Surface Modification of Polystyrene by using Nitrogen Plasma

A F Novi, D J D H Santjojo and Masrurroh

[+](#) View abstract [View article](#) [PDF](#)

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012037

Optimizing Heat Treatment Process of Fe-13Cr-3Mo-3Ni Martensitic Stainless of Steel

M S Anwar, S Prifiharni and E Mabruuri

[+](#) View abstract [View article](#) [PDF](#)

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012038

Fe-doped ZnO Supported with Montmorillonite: Synthesis, Characterization, and Photocatalytic Activity

M I Pratiwi, N Afifah and R Saleh

[+](#) View abstract [View article](#) [PDF](#)

-
- OPEN ACCESS** 012039
- Study of Sigma Phase in Duplex SAF 2507
- D M Fellicia, Sutarsis, B A Kurniawan, D Wulanari, A Purniawan and A T Wibisono
- [+](#) View abstract [View article](#) [PDF](#)
-
- OPEN ACCESS** 012040
- Study of Raman Spectra of Aluminum Powder-Substituted Barium Hexaferrite (BaM) $\text{BaFe}_{12-x}\text{Al}_x\text{O}_{19}$ as a Result of Solid State Reaction Process
- S Mustofa, R Rizaldy and W A Adi
- [+](#) View abstract [View article](#) [PDF](#)
-
- OPEN ACCESS** 012041
- Analysis of Distribution of Polyvinyl Alcohol Hydrogel Nanocrystalline by using SAXS Synchrotron
- Sunaryono, A Taufiq, N Mufti, N Hidayat, S Rugmai, S Soontaranon, E G R Putra and Darminto
- [+](#) View abstract [View article](#) [PDF](#)
-
- OPEN ACCESS** 012042
- Crystal Structure, Optical, and Electrical Properties of SnSe and SnS Semiconductor Thin Films Prepared by Vacuum Evaporation Techniques for Solar Cell Applications
- Ariswan, H Sutrisno and R Prasetyawati
- [+](#) View abstract [View article](#) [PDF](#)
-
- OPEN ACCESS** 012043
- Synthesis of Polyvinylpyrrolidone (PVP)-Green Tea Extract Composite Nanostructures using Electrohydrodynamic Spraying Technique
- Kamaruddin, D Edikresnha, I Sriyanti, M M Munir and Khairurrijal
- [+](#) View abstract [View article](#) [PDF](#)
-
- OPEN ACCESS** 012044
- Photodegradation of Rhodamine B by using ZnFe_2O_4 Nanoparticles Synthesized through Precipitation Method
- Karnaji and I Nurhasanah
- [+](#) View abstract [View article](#) [PDF](#)
-
- OPEN ACCESS** 012045
- Effect of Synthesis Parameter on Crystal Structures and Magnetic Properties of Magnesium Nickel Ferrite ($\text{Mg}_{0.5}\text{Ni}_{0.5}\text{Fe}_2\text{O}_4$) Nanoparticles

R Maulia, R A Putra and E Suharyadi

[+](#) View abstract [View article](#) [PDF](#)

OPEN ACCESS

012046

Study on The Influence of Crystal Structure and Grain Size on Dielectric Properties of Manganese Ferrite (MnFe_2O_4) Nanoparticles

Kurnia, Heriansyah and E Suharyadi

[+](#) View abstract [View article](#) [PDF](#)

OPEN ACCESS

012047

Study of Structural and Magnetic Properties of Silica and Polyethylene Glycol (PEG-4000)-Encapsulated Magnesium Nickel Ferrite ($\text{Mg}_{0.5}\text{Ni}_{0.5}\text{Fe}_2\text{O}_4$) Nanoparticles

F Deswardani, R Maulia and E Suharyadi

[+](#) View abstract [View article](#) [PDF](#)

OPEN ACCESS

012048

Effect of Synthesis Temperature and NaOH Concentration on Microstructural and Magnetic Properties of $\text{Mn}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ Nanoparticles

N Siregar, I P T Indrayana, E Suharyadi, T Kato and S Iwata

[+](#) View abstract [View article](#) [PDF](#)

OPEN ACCESS

012049

Effect of Cu-Dopant on the Structural, Magnetic and Electrical Properties of ZnO

D Aryanto, C Kurniawan, A Subhan, T Sudiro, P Sebayang, M Ginting, S M K Siregar and M N Nasruddin

[+](#) View abstract [View article](#) [PDF](#)

OPEN ACCESS

012050

Effect of Growth Time on the Characteristics of ZnO Nanorods

R Idiawati, N Mufti, A Taufiq, H Wisodo, I K R Laila, A Fuad and Sunaryono

[+](#) View abstract [View article](#) [PDF](#)

OPEN ACCESS

012051

Synthesis and Characterization of Magnetic Elastomer based PEG-Coated Fe_3O_4 from Natural Iron Sand

C Kurniawan, A S Eko, Y S Ayu, P T A Sihite, M Ginting, P Simamora and P Sebayang

[+](#) View abstract [View article](#) [PDF](#)

OPEN ACCESS

- Synthesis, Investigation on Structural and Magnetic Behaviors of Spinel M-Ferrite [M = Fe; Zn; Mn] Nanoparticles from Iron Sand 012052
S Bahtiar, A Taufiq, Sunaryono, A Hidayat, N Hidayat, M Diantoro, N Mufti and Mujamilah
[+](#) View abstract [View article](#) [PDF](#)
-
- OPEN ACCESS** 012053
Variation of Carbon Coating on $\text{Li}_2\text{Na}_2\text{Ti}_6\text{O}_{14}$ as Anode Material of Lithium Battery
B Prihandoko, S Priyono, A Subhan and A Mulya
[+](#) View abstract [View article](#) [PDF](#)
-
- OPEN ACCESS** 012054
Optical Properties of Fe_3O_4 Magnetic Fluid from Iron Sand
A Puspitaningrum, A Taufiq, A Hidayat, Sunaryono, N Hidayat and Samian
[+](#) View abstract [View article](#) [PDF](#)
-
- OPEN ACCESS** 012055
High-Performance Silver Nanowire Film on Flexible Substrate Prepared by Meyer-rod Coating
Junaidi, K Triyana, Harsojo and E Suharyadi
[+](#) View abstract [View article](#) [PDF](#)
-
- OPEN ACCESS** 012056
Effect of NiO and Light Intensity on Dielectric Constant of $\text{SiO}_2\text{-B}_2\text{O}_3\text{-Bi}_2\text{O}_3\text{-Na}_2\text{CO}_3$ Glass Based on Silica Gel of Natural Sands
M Diantoro, Z Muniroh, B Zaini, A A Mustikasari, Nasikhudin, A Hidayat, A Taufiq, Sunaryono and N. Mufti
[+](#) View abstract [View article](#) [PDF](#)
-
- OPEN ACCESS** 012057
Composites of $\text{Fe}_3\text{O}_4/\text{SiO}_2$ from Natural Material Synthesized by Co-Precipitation Method
Munasir, A S Dewanto, A Yulianingsih, I K F Saadah, Z A I Supardi, A Mufid and A Taufiq
[+](#) View abstract [View article](#) [PDF](#)
-
- OPEN ACCESS** 012058
Fabrication of TiO_2 /Carbon Photocatalyst using Submerged DC Arc Discharged in Ethanol/Acetic Acid Medium
T E Saraswati, A O Nandika, I F Andhika, Patiha, C Purnawan, S Wahyuningsih and S B Rahardjo
[+](#) View abstract [View article](#) [PDF](#)

-
- OPEN ACCESS** 012059
- Synthesis and Characterization of Microwave Absorber SiO₂ by Sol-Gel Methode
- S Wardiyati, W A Adi and Deswita
- [+](#) View abstract [View article](#) [PDF](#)
-
- OPEN ACCESS** 012060
- Natural Silica Sand/Alumina Ceramic Composites: Promising Candidates for Fuel-Cell Sealants
- N Hidayat, Istiqomah, M Y H Widiyanto, A Taufiq, Sunaryono, Triwikantoro, M Zainuri, M A Baqiya, G Aristia and S Pratapa
- [+](#) View abstract [View article](#) [PDF](#)
-
- OPEN ACCESS** 012061
- Ag/Fe₃O₄/ZrO₂ Composite: Ternary Magnetically Separable UV-light-driven Photocatalyst for Removal Methylene Blue Dyes
- Y Kristianto, A Taufik and R Saleh
- [+](#) View abstract [View article](#) [PDF](#)
-
- OPEN ACCESS** 012062
- Effect of Fe₃O₄ on the Electro-Optic and Magneto-Electric Characteristics of (PANI/Fe₃O₄)-Ag Film
- M Diantoro, D Pradhana, A A Mustikasari, A D Kusumawati, A Taufiq, Sunaryono, N Mufti and H Nur
- [+](#) View abstract [View article](#) [PDF](#)
-
- OPEN ACCESS** 012063
- Effect of Crystallite Structure and Graphene Incorporation on Photocatalytic Performance of LaFeO₃
- N Afifah and R Saleh
- [+](#) View abstract [View article](#) [PDF](#)
-
- OPEN ACCESS** 012064
- Factors Influence the Structural and Magnetic Properties of Ag-Fe₃O₄ Nanocomposites Synthesized by Reduction Method
- F Fajaroh and Nazriati
- [+](#) View abstract [View article](#) [PDF](#)
-
- OPEN ACCESS** 012065

Sonocatalytic Degradation of Methylene Blue with LaMnO_3 Supported by Different Surface Area of Graphene

N Afifah and R Saleh

[+](#) View abstract [View article](#) [PDF](#)

OPEN ACCESS

012066

Nanoneedles of Lanthanum Oxide (La_2O_3): A Novel Functional Material for Microwave Absorber Material

W A Adi, S Wardiyati and S H Dewi

[+](#) View abstract [View article](#) [PDF](#)

OPEN ACCESS

012067

Effect of Thermal Processes on the Electrical and Optical Properties of Fe_2TiO_5 Ceramics

R Fajarin, Widyastuti, M A Baqiya and I Y S Putri

[+](#) View abstract [View article](#) [PDF](#)

OPEN ACCESS

012068

Mechanosynthesis of A Ferritic ODS (Oxide Dispersion Strengthened) Steel Containing 14% Chromium and Its Characterization

A K Rivai, A Dimiyati and W A Adi

[+](#) View abstract [View article](#) [PDF](#)

OPEN ACCESS

012069

Methyl Ester Production via Heterogeneous Acid-Catalyzed Simultaneous Transesterification and Esterification Reactions

S Indrayanah, Erwin, I N Marsih, Suprpto and I K Murwani

[+](#) View abstract [View article](#) [PDF](#)

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012070

Synthesis of Zinc Oxide Nanoparticles using Anthocyanin as a Capping Agent

N L W Septiani, B Yulianto, M Iqbal and Nugraha

[+](#) View abstract [View article](#) [PDF](#)

OPEN ACCESS

012071

Microstructure Transformation of Mg-1.6Gd during Hot Rolled at High Deformation Ratio

O Susanti, M A Mochtar and S Harjanto

[+](#) View abstract [View article](#) [PDF](#)

OPEN ACCESS

012072

The Effect of Molar Ratio on Crystal Structure and Morphology of $\text{Nd}_{1+x}\text{FeO}_3$ ($X=0.1, 0.2, \text{ and } 0.3$) Oxide Alloy Material Synthesized by Solid State Reaction Method

V Zharvan, Y N I Kamaruddin, S Samnur and E H Sujiono

[+](#) View abstract [View article](#) [PDF](#)

OPEN ACCESS

012073

Modification of Pseudobrookite $\text{Fe}_{2-x}\text{Mn}_x\text{TiO}_5$ with Solid State Reaction Method using a Mechanical Milling

Y Sarwanto and W A Adi

[+](#) View abstract [View article](#) [PDF](#)

OPEN ACCESS

012074

Effect of Precursor Concentration Ratio on The Crystal Structure, Morphology, and Band Gap of ZnO Nanorods

A Fuad, A A Fibriyanti, Subakti, N Mufti and A Taufiq

[+](#) View abstract [View article](#) [PDF](#)

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012075

Physical and Magnetic Properties of $\text{La}_{0.5}\text{Ca}_{0.5}\text{Mn}_{0.9}\text{Cu}_{0.1}\text{O}_3$ at Temperature in the Range of 10-100 K

Y E Gunanto, W A Adi, E Steven, B Kurniawan, T Ono and H Tanaka

[+](#) View abstract [View article](#) [PDF](#)

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012076

Effect of Heat Treatment on The Crystal Structur, Electrical Conductivity and Surface of $\text{Ba}_{1.5}\text{Sr}_{0.5}\text{Fe}_2\text{O}_5$ Composite

P Purwanto, WA Adi and Yunasfi

[+](#) View abstract [View article](#) [PDF](#)

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012077

Monitoring Microalgae Population Growth by using Fe_3O_4 Nanoparticles-based Surface Plasmon Resonance (SPR) Biosensor

D T Nurrohman, M Oktivina, E Suharyadi, E A Suyono and K Abraha

[+](#) View abstract [View article](#) [PDF](#)

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012078

The Addition of Graphene and Magnetite Materials in TiO₂/CuO Catalyst for Enhancing Photosonocatalytic Performance and Reusability

A Taufik, A Muzaki and R Saleh

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012079

Synthesis and Characterization of Acrylic-Based Photopolymer as a Candidate for Denture Base Material

S T Wicaksono, Rasyida and H Ardhyanta

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012080

Composite Based Chitosan/Zinc-Doped HA as a Candidate Material for Bone Substitute Applications

S T Wicaksono, A Rasyida, A Purnomo, N N Pradita, H Ardhyanta and M I P Hidayat

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012081

Investigation on the Mechanical Properties of A356 Alloy Reinforced AlTiB/SiC_p Composite by Semi-Solid Stir Casting Method

E I Bhiftime and N F D S Gueterres

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012082

Poly (1,8 Octanediol-co-Citrate) Hydroxyapatite Composite as Antibacterial Biodegradable Bone Screw

P Widiyanti, I Sholikhah, A Isfandiary, NAF Hasbiyani, M B Lazuardi and R D Laksana

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012083

The Effects of the Addition of Silica Mol Fraction (x = 1.5; 2; 2.5) as a Solid Electrolyte on Ion Conductivity of NASICON (Na_{1-x}Zr₂Si_xP_{3-x}O₁₂) Using Solid-State Method

V M Pratiwi, H Purwaningsih, Widyastuti, R Fajarin and H Setyawan

[+ View abstract](#)[View article](#)[PDF](#)**OPEN ACCESS**

012084

Effects of Austenitizing and Forging on Mechanical Properties of MIL A-12560/AISI

4340 Steel

S Herbirowo, B Adjiantoro and T B Romijarso

[+](#) View abstract [View article](#) [PDF](#)

OPEN ACCESS

012085

Influence of Austenitizing Heat Treatment on the Properties of the Tempered Type 410-1Mo Stainless Steel

E Mabruari, Z A Syahlan, Sahlan, S Prifiharni, M S Anwar, S A Chandra, T B Romijarso and B Adjiantoro

[+](#) View abstract [View article](#) [PDF](#)

OPEN ACCESS

012086

Fluorescence Sensing of Nitrite Ions on Polyvinylpyrrolidone/Zinc Oxide Composites Prepared by Impregnation Method

L Yuliati, S Z M So'ad, N S Alim and H O Lintang

[+](#) View abstract [View article](#) [PDF](#)

OPEN ACCESS

012087

Blending of Low-Density Polyethylene and Poly-Lactic Acid with Maleic Anhydride as A Compatibilizer for Better Environmentally Food-Packaging Material

A H Setiawan and F Aulia

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OPEN ACCESS

012088

Mechanical and Thermal Properties of Unsaturated Polyester/Vinyl Ester Blends Cured at Room Temperature

H Ardhyananta, F D Puspawati, S T Wicaksono, Widyastuti, A T Wibisono, B A Kurniawan, H Ismail and A V Salsac

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OPEN ACCESS

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Characteristics of Al-Si-Mg Reinforced SiC Composites Produced by Stir Casting Route

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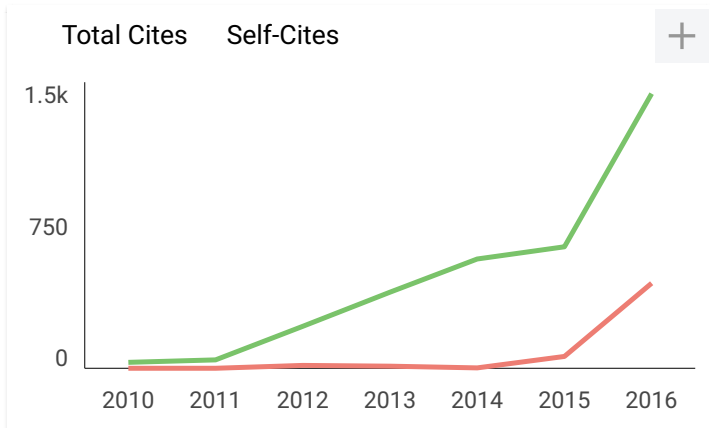
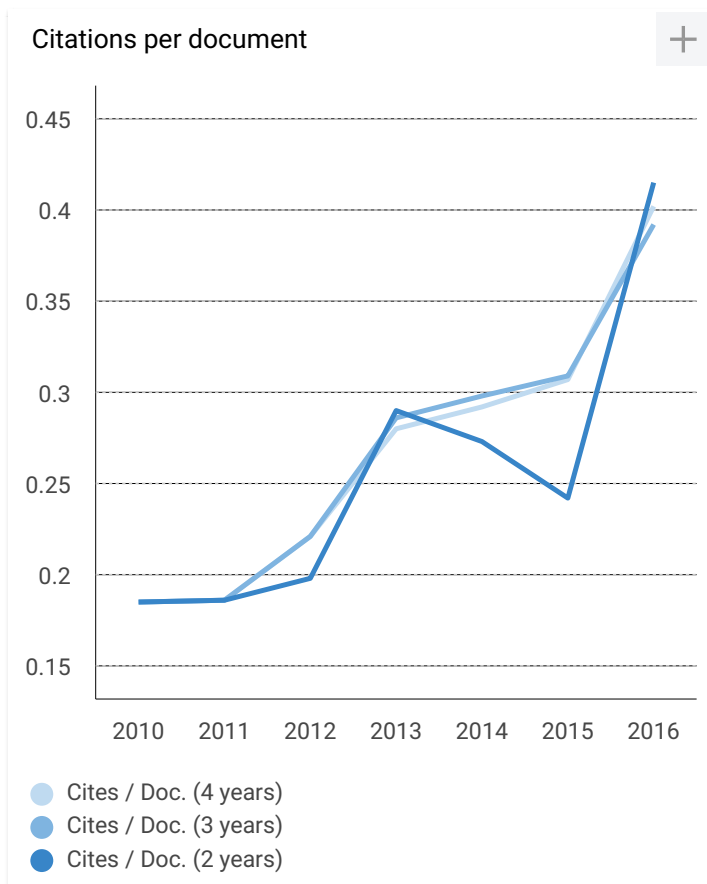
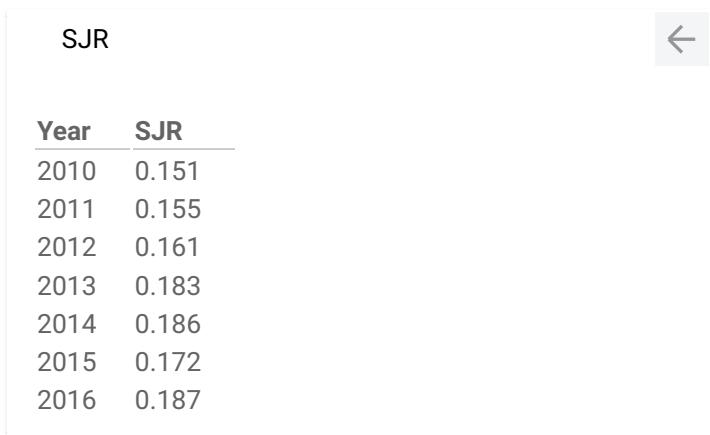
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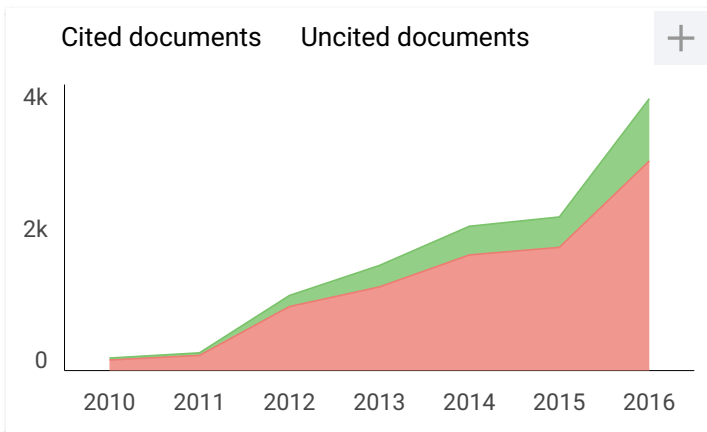
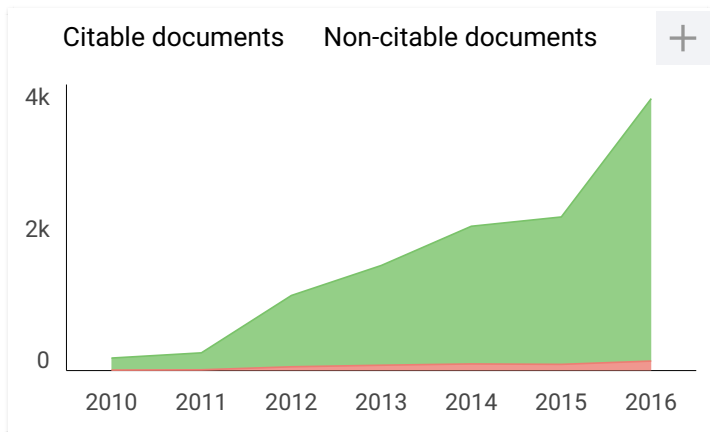
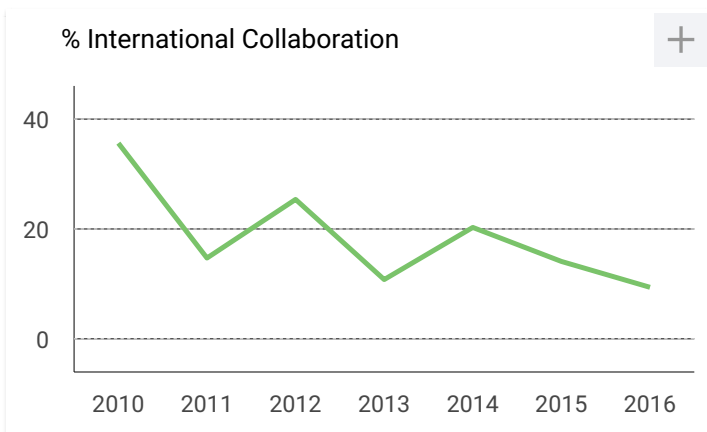
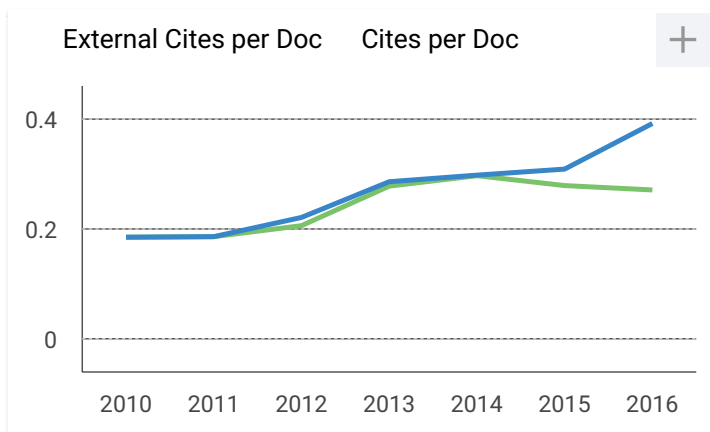
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Fluorescence Sensing of Nitrite Ions on Polyvinylpyrrolidone/Zinc Oxide Composites Prepared by Impregnation Method

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Abstract. A series of polyvinylpyrrolidone/zinc oxide (PVP/ZnO) composites with different loading amounts of PVP was prepared by an impregnation method. Successful formation of the composites was analyzed by the Fourier transform infrared (FTIR), diffuse reflectance ultraviolet-visible (DR UV-Vis) and fluorescence spectroscopies. Prepared composites were then further tested as fluorescence sensors by conducting quenching studies in the presence of nitrite ions (NO_2^-). Among the prepared composites, the ZnO with 1% PVP exhibited the highest sensing performance for the NO_2^- detection ($K_{sv} = 0.07 \mu\text{M}^{-1}$). The efficiency of the composite was *ca.* 1.7 and 1.4 times higher than the bare ZnO and the best composite prepared by the physical mixing method, respectively. These results suggested that impregnation method is a suitable method to prepare the PVP/ZnO composites as fluorescence sensor for the NO_2^- detection.

Keywords: polyvinylpyrrolidone/zinc oxide, composite, impregnation, fluorescence sensor, nitrite ion

1. Introduction

Nitrite (NO_2^-) is commonly used as a preservative and fertilizing agent for food as well as an inorganic fertilizer. Since NO_2^- has a high level of solubility in water, these ions also have high mobility, which has led to constant threats whenever agriculture processes are involved [1]. The emergence of NO_2^- as one of the hazardous contaminants has become a grave concern since NO_2^- can react with secondary or tertiary amines to form nitrosamines, which are carcinogenic compounds [2-4]. Ingestion of NO_2^- might cause endogenous nitrosation that is probably carcinogenic to human [5] and associated with colorectal and stomach cancers [6]. Furthermore, NO_2^- is also known to result in respiratory deficiencies as NO_2^- would react with hemoglobin forming methemoglobin, which disturbs the transport of oxygen [7, 8]. Therefore, it is a must to develop tools or methods that are simple, non-toxic, selective and sensitive to detect the presence of NO_2^- .



For nitrite/nitrate determination, one of the most generally reported methods is spectroscopy method owing to its great limit of detection and easy protocols [1]. Particularly, fluorescence sensors have attracted many attentions since they are simple, their signal can be amplified, they can be fabricated easily into devices, and they can be combined with various outputs [9]. One of the best candidates for a simple, inexpensive, and non-toxic solid material in the detection of NO_2^- is a semiconductor-based sensor. Particularly, zinc oxide (ZnO) has been widely used as a sensor for the detection of various gasses, pollutants, and biomolecules [10-13].

As semiconductor gas sensor, ZnO has been extensively investigated for the detection of gas leaks and environmental monitoring of gaseous pollutants. For instance, ZnO showed excellent sensitivity to detect some flammable gasses as well as corrosive vapors with fast response-recovery characteristic, great selectivity, and stability [10]. ZnO was also reported serving as a sensor for sensing liquefied petroleum gas (LPG) and ethanol [11]. It was observed that ZnO also has good sensitivity at low temperature and it also showed better response and shorter recovery time. The application of ZnO as a sensor for methanal and xylene has also been investigated [12]. In addition, ZnO was also pointed out to improve signal in DNA sensor arrays [13].

In order to enhance the performance of ZnO as a sensor, ZnO can be modified with polymers [14-17]. It is recognized that grafting polymers onto the surface of ZnO was an effective way to improve its dispersion in a polymer matrix and the composites showed improved properties [14]. One of the potential polymers is polyvinylpyrrolidone (PVP), which has been reported to be a potential fluorescence sensor for the detection of nitrate (NO_3^-) [18]. Recently, the addition of PVP to ZnO by a physical mixing method has been reported to be able to increase the sensing efficiency of ZnO towards the NO_2^- detection [15]. In this study, we newly reported a higher enhancement of the fluorescence sensing capability for NO_2^- detection when the PVP/ZnO composites were prepared by an impregnation method.

2. Experimental Method

2.1. Materials

The materials used were zinc acetate dehydrate 99.5% ($\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$, QRëc), sodium hydroxide pellet 99 % (NaOH, QRëc), cetyltrimethylammonium bromide ($\text{C}_{19}\text{H}_{42}\text{BrN}$, CTAB, Fischer Scientific), polyvinylpyrrolidone 45% in H_2O ($(\text{C}_6\text{H}_9\text{NO})_n$, PVP, Sigma-Aldrich), sodium nitrite 99.5% (NaNO_2 , Merck), and distilled water.

2.2. Methods

2.2.1. Preparation of ZnO. Zinc acetate dihydrate, $\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$ was used as a precursor to synthesize the ZnO. The $\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$ (4.5 g) was dissolved in distilled water (100 ml) as solution A. For the preparation of solution B, NaOH (6.4 g) was dissolved in distilled water and followed by the addition of CTAB (7.28 g) into the mixture and stirred for 1 hour to obtain a homogeneous solution. After 1 hour, solution A was added into solution B and heated at 70 °C for 1 hour. The remaining solid was subsequently filtered and washed with distilled water. The solid powder was dried at room temperature and then calcined at 500 °C at a rate of 1.0 °C min^{-1} and further tempered for another 1 h at the same temperature.

2.2.2. Preparation of PVP/ZnO composites. Various PVP/ZnO composites were prepared by an impregnation method, in which the concentration ratios were varied to 0.1, 1 and 3 % (v/w). The samples were labeled as PVP(x)/ZnO, with x that shows the ratio of PVP to the ZnO. For a typical synthesis of PVP (1)/ZnO composite, the ZnO (1 g) was dispersed in distilled water (30 mL). The PVP (0.2020 mL) from 5 % PVP solution was then added dropwise to the mixture containing ZnO and distilled water, followed by stirring and drying at 80 °C on a hot plate.

2.2.3. Characterizations. As for characterizations of the prepared ZnO and PVP/ZnO composites, FTIR spectroscopy (Thermo Scientific, Nicolet iS50) was applied to investigate the functional groups in the prepared ZnO and PVP/ZnO composites. For the measurement, potassium bromide (KBr) pellet technique was used. The FTIR spectra were measured in the range of 400 to 4000 cm^{-1} with 32 scan number. Nitrogen gas was purged into the system before analysis to exclude moisture for better background correction. DR UV-Vis spectroscopy (UV-2600 with an integrating sphere, Shimadzu) was used to measure the absorption spectra of the samples. The absorption spectra were recorded in the wavelengths range from 250 to 500 nm. Scan speed was at the rate of 300 nm min^{-1} and the slit width was set at 1 nm. As a reference, barium sulfate (BaSO_4) was used in the measurement. The fluorescence spectra were recorded on the fluorescence spectrophotometer (FP-8500, JASCO) at room temperature. The emission spectra were measured when the excitation wavelength was 365 nm.

2.3. Sensor tests

Sensor tests for the detection of NO_2^- were carried out based on the quenching study, by monitoring the decreases in the emission intensity of the sensor when various concentrations of the NO_2^- were added into the sensor. Typically, the sensor sample (0.3 g) was transferred into a fluorescence sample holder and placed at the fluorescence instrument. Initial readings for excitation and emission peaks were firstly recorded in the absence of the NO_2^- . As for the quenching test, the selected concentration of NO_2^- (2–10 μM) with a volume of 10 μL was added slowly on top of the sample. The emission spectra were then recorded at an excitation wavelength of 365 nm for ZnO and all the PVP/ZnO composites. The efficiency for the detection of the NO_2^- was further determined by Stern-Volmer plot on each of the prepared sample.

3. Results and Discussion

3.1. Functional groups

The functional groups of PVP, ZnO, and PVP/ZnO composites were examined by using the FTIR spectroscopy. FTIR spectroscopy gives qualitative information on the type of functional groups that present in the samples. As shown in Figure 1(a), PVP gave OH stretching vibration from a water molecule at around 3447 cm^{-1} due to the hydrophilic nature of the PVP, while the peaks observed around 2800–3000 cm^{-1} were assigned to C-H stretching. The characteristic peaks of PVP could be seen at 1655 and 1461 cm^{-1} , which were corresponding to C=O and C-N stretching, respectively. The presence of C-N stretching was also shown at around 1370 cm^{-1} and the peaks below 700 cm^{-1} gave the evidence of C-C chains and N-C=O bending [19].

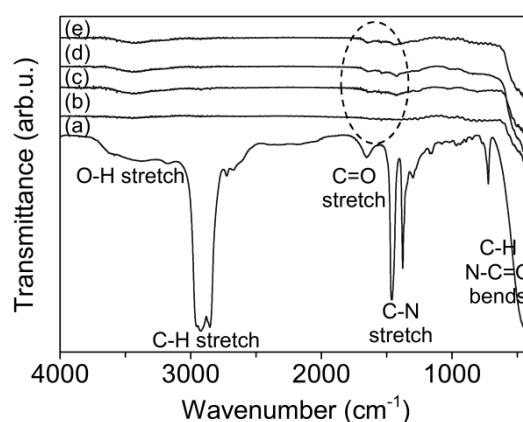


Figure 1. FTIR spectra of (a) PVP, (b) ZnO, (c) PVP(0.1)/ZnO, (d) PVP(1)/ZnO and (e) PVP(3)/ZnO samples

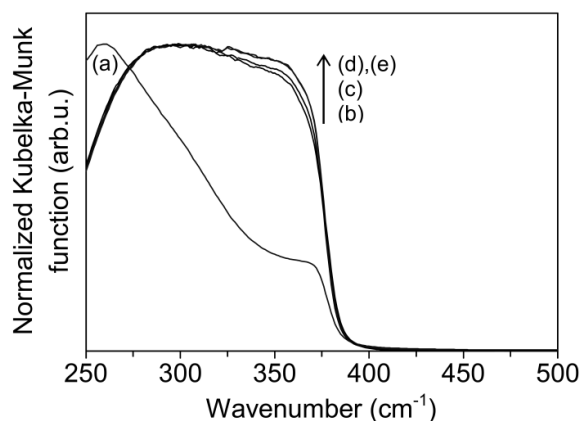


Figure 2. DR UV-Vis spectra of (a) PVP, (b) ZnO, (c) PVP(0.1)/ZnO, (d) PVP(1)/ZnO, and (e) PVP(3)/ZnO samples

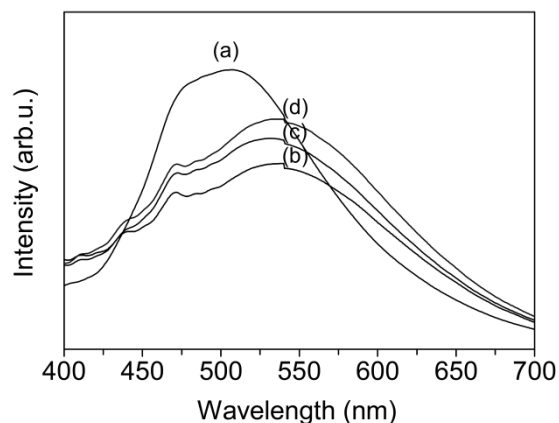


Figure 3. Emission spectra of (a) ZnO, (b) PVP(0.1)/ZnO, (c) PVP(1)/ZnO, and (d) in PVP(3)/ZnO samples

Displayed in figure 1(b) is the FTIR spectrum of ZnO. The ZnO showed a sharp peak at around 420 cm^{-1} that could be assigned as the stretching mode of Zn–O [20, 21]. Figures 1(c)–(e) show the FTIR spectra for the PVP/ZnO composites. All the composite samples gave the presence of Zn–O stretching as well as two peaks appeared at 1647 and 1434 cm^{-1} that could be related to C=O and C–N stretching, respectively. Both of the latter peaks came from the PVP. It has been suggested that the peak shift can be related to the hydrogen bond formation [19, 20]. Therefore, the shifting observed on the PVP/ZnO samples might be the outcome of the hydrogen bonding interaction between the surface hydroxyl of ZnO and the functional group in the PVP. Another responsibility that can be proposed was the existence of a strong coulombic interaction between the ZnO and the polymeric matrix [22]. When the amount of PVP in the PVP/ZnO composite increased, the intensity of C=O stretching and C–N stretching also increased owing to the increment amount of PVP contained in the PVP/ZnO composites. The FTIR spectra suggested the successful preparation of the PVP/ZnO composites.

3.2. Optical properties

The optical properties of the PVP, ZnO, and PVP/ZnO composites were investigated using the DR UV-Vis spectroscopy and are displayed in Figure 2. As shown in Figure 2(a), PVP exhibited intense narrow bands centered at 265 and 370 nm , which might be due to the presence of C=O and N–C groups, respectively [19]. On the other hand, Figure 2(b) shows a strong absorption peak of ZnO in the UV region, which was centered at 330 nm due to the electron charge transfer in the Zn–O linkage [23, 24]. The DR UV-Vis spectra of the PVP/ZnO composites are shown in Figures 2 (c)–(e). All the composite samples showed similar absorption peak to each other, where the main absorption peak of ZnO was clearly observed and the loading amount of PVP also slightly increased the absorption band of PVP. It can be observed that the addition of PVP did not give considerable influence to the band structure of the ZnO.

3.3. Fluorescence properties

Fluorescence spectroscopy is a powerful non-destructive technique to explore the fluorescence properties of the materials. Figure 3 shows the emission spectra of the prepared ZnO and PVP/ZnO composites. Shown in Figure 3(a) is the emission spectrum for ZnO when the excitation wavelength at 365 nm was used. ZnO gave one broad emission peak at 517 nm , which has been recognized as the green emission of ZnO, reflecting the presence of oxygen defect on the surface and deep level of the ZnO [22]. As can be seen from Figure 3(b)–(d), all PVP/ZnO composites have a lower emission intensity as compared to the ZnO. In addition to the reduced fluorescence intensity, the addition of PVP also shifted the emission peak to a longer wavelength. The decrease in fluorescence intensity and

the strong peak shifting indicated that there were interactions existed between the PVP and the ZnO. The most possible interactions between PVP and ZnO would occur via hydrogen bonding and/or coulombic interaction.

3.4. Sensing performance

The sensing performance of the ZnO and the PVP/ZnO composites towards the NO_2^- was evaluated via quenching tests by monitoring the decreases in the emission intensity after the additions of various concentrations of the NO_2^- (0–10 μM) to the ZnO and the PVP/ZnO composites. All samples showed similar results, where the emission intensity was reduced gradually with the increase in the concentration of the NO_2^- . As a representative, the changes of the emission spectra in the absence and presence of NO_2^- for the PVP(1)-ZnO composite are illustrated in Figure 4(a).

The relative emission intensity can be strongly related to the concentration of NO_2^- that investigated by a Stern-Volmer plot. In the plot, the relative emission intensity is expressed as a function of the NO_2^- concentrations, following the Stern-Volmer plot as shown in Equation (1).

$$I_0/I = K_{SV} [Q] + 1 \quad (1)$$

Where I_0 and I show the fluorescence intensity without and with NO_2^- , respectively, Q shows the concentrations of NO_2^- , and K_{SV} is the Stern-Volmer quenching constant. The calculated K_{SV} indicated the efficiency of the sensor. Higher K_{SV} value suggested a larger efficiency of the sensor. All the ZnO and PVP/ZnO composites gave almost linear curves, suggesting that the ZnO and the PVP/ZnO composites can be potential fluorescence sensors in the detection of the NO_2^- . As representative of the samples, Figure 4(b) illustrates the linear Stern-Volmer plots of the ZnO and the PVP(1)/ZnO composite in the presence of NO_2^- .

K_{SV} values of the ZnO and the PVP/ZnO composites are given in Figure 5. The K_{SV} value of the ZnO was determined to be $0.04 \mu\text{M}^{-1}$. It was evident that the addition of PVP from 0.1 to 1% improved the ZnO sensing performance, where the K_{SV} value increased from 0.04 to 0.05 and $0.07 \mu\text{M}^{-1}$, respectively. Unfortunately, the sensing performance decreased to $0.026 \mu\text{M}^{-1}$ when the loading amount of PVP was too high (3%). It was shown that the addition of PVP reduced the ZnO intensity, thus, when the loading of PVP was too high, the reduced emission intensity would decrease the sensitivity of the sensor. This study showed that the PVP(1)/ZnO was the best sample showing the highest efficiency towards the detection of NO_2^- .

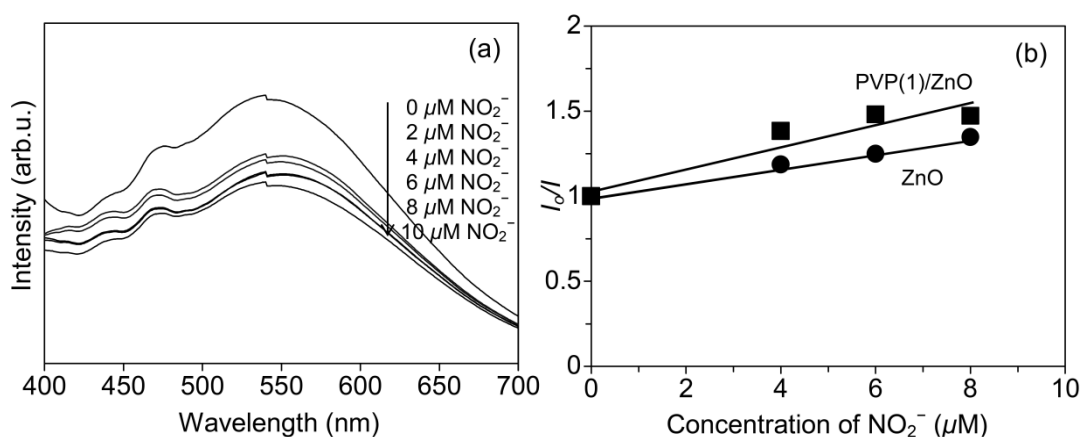


Figure 4. (a) Emission spectra of PVP(1)/ZnO in the absence and presence of different concentrations of NO_2^- and (b) Stern-Volmer plots of PVP(1)/ZnO and ZnO samples

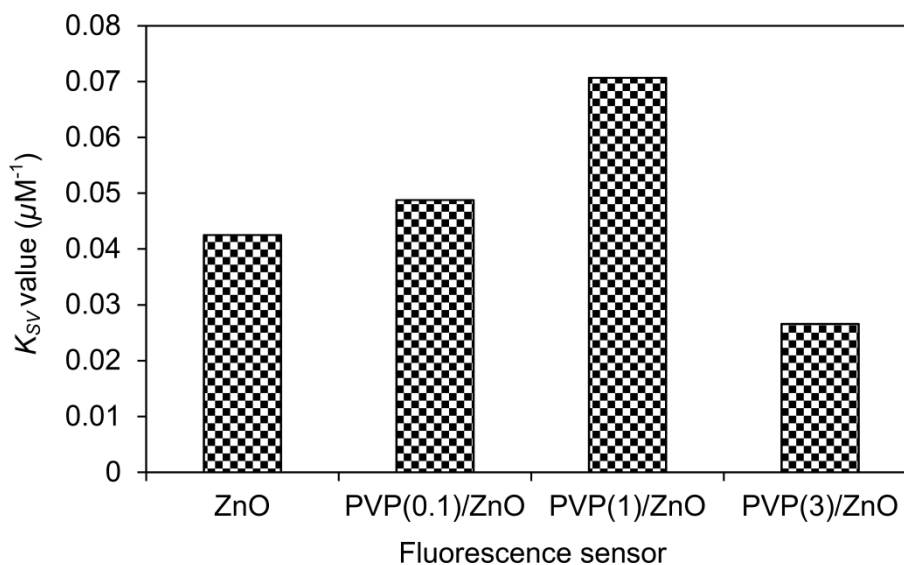


Figure 5. Comparison of K_{SV} values of ZnO and PVP-ZnO composites

It was reported that the best PVP/ZnO composite prepared by physical mixing, the PVP(0.1)/ZnO, gave K_{SV} value of $0.05 \mu\text{M}^{-1}$ [15]. Comparison to the best PVP/ZnO composite prepared by physical mixing showed that the best PVP/ZnO composite developed by the impregnation method gave a higher K_{SV} value of $0.07 \mu\text{M}^{-1}$. It was proposed that better interaction between the PVP and the ZnO prepared by the impregnation method would be the crucial factor to get a higher efficiency. Since the impregnation method employed a sonication step, the PVP would be dispersed better on the ZnO than that prepared by physical mixing. This study demonstrated that the impregnation method was better than the physical mixing to prepare the PVP/ZnO composites as fluorescence sensor of the NO_2^- .

4. Conclusion

The PVP/ZnO composites were successfully prepared by an impregnation method as evidenced by FTIR, DR UV-Vis, and fluorescence spectroscopies. The prepared ZnO and composites gave good linear Stern-Volmer plots with various concentrations of NO_2^- , suggesting the potential capability of these samples as fluorescence sensors for the NO_2^- detection. The best composite, which was PVP(1)-ZnO, gave K_{SV} value of $0.07 \mu\text{M}^{-1}$, which was 1.7 times higher than that of the ZnO.

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