PREPARATION OF SILVER NANOPARTICLES IN AROMATIC LEAVES EXTRACT AND IONIC LIQUID FOR PHOTODEGRADATION OF 2,4–DICHLOROPHENOXYACETIC ACID

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MASTER OF SCIENCE

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We hereby declare that we have checked this thesis and in our opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Master of Science.

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I hereby declare that the work in this thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at Universiti Malaysia Pahang or any other institutions.

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ABSTRAK

Herbisid 2,4–diklorofenolasetik asid (2,4–D) yang digunakan secara meluas dalam industri pertanian adalah penyebab utama berlakunya pencemaran toksik yang mengakibatkan kerosakan ekosistem akuatik dan kesihatan manusia. Oleh ini, kajian berterusan mengenai keberkesanan pemangkin yang dapat membasmi pencemaran ini telah dijalankan sejak kebelakangan ini. Dalam kajian ini, pemangkin perak (Ag) telah disintesis dan dianalisis melalui kaedah elektrokimia yang dilakukan di dalam cecair daun yang telah diekstrak dan cecair ionik. Prestasi pemangkin yang disintesis telah diselidiki melalui kadar kemerosotan 2,4-D dan dioptimumkan oleh Metodologi Permukaan Tindak Balas (RSM). Model kinetik dan penggunaan semula pemangkin yang disintesis juga dikaji. Pertama, daun aromatik telah diekstrak menggunakan kaedah hidro-penyulingan yang dibantu ultrasonik (UAE-HD) dan pengekstrakan menggunakan larutan akueus (AE). Kemudian, nanopartikel Ag telah disintesis oleh kaedah elektrokimia dengan menggunakan larutan daun aromatik yang telah diekstrak dan cecair ionik sebagai media sintesis. Pemangkin kemudiannya dianalisis dengan menggunakan XRD, SEM, TEM, FTIR, BET, UV-Vis DRS dan PL. Kemudian, aktiviti fotokatalitik pemangkin diuji melalui degradasi 2,4-D. Selanjutnya, pengoptimuman proses fotokatalitik dilakukan dengan menggunakan Metodologi Permukaan Tindak Balas (RSM). Kajian kinetik reaksi fotokatalitik juga dianalisis oleh urutan pertama, urutan kedua dan Langmuir Hinshelwood. Akhir sekali, kajian penggunaan smula pemangkin telah dijalankan bagi menentukan kecekapan pemangkin. Keputusan menunjukkan bahawa kaedah UAE-HD dapat menghasilkan kandungan fenolik yang lebih tinggi berbanding dengan kaedah AE. Hasilnya menunjukkan bahawa cecair ionik dan sebatian fenolik dalam ekstrak daun mempunyai kesan sinergi untuk mengurangkan ion perak (Ag^+) ke nanopartikel Ag dan bertindak sebagai agen penutupan dalam pembentukan nanopartikel. Kemudian, prestasi pemangkin diuji terhadap fotodegradasi 2,4-D di dalam reaktor di bawah sinaran cahaya yang boleh dilihat. Keputusan menunjukkan bahawa nanopartikel Ag dapat menghalang rekombinasi lubang elektron untuk memberikan degradasi yang hampir lengkap (96.54%) apabila menggunakan 10 mg L^{-1} 2,4-D pada pH 3 dan 0.01 g L^{-1} pemangkin Ag yang disediakan dalam 1-butil-3-methilimidazolium bis (trifluoromethilsulfonil) imida [BMIM Tf_2N] cecair ionik dan *Orthosiphon stamineus* (OS) daun ekstrak media yang diekstrak oleh kaedah UAE-HD. Telah didapati bahawa jumlah kandungan fenolik dalam ekstrak daun serta rantaian panjang alkil imidazolium dan struktur anion yang besar menyebabkan terhasilnya nanopartikel yang kecil dan seterusnya meningkatkan fotodegradasi 2,4-D. Selain kewujudan sebatian fenolik dan cecair ionik pada permukaan pemangkin, kedua-dua sebatian fenolik dan cecair ionik juga dijelaskan dapat memainkan peranan penting sebagai penerima elektron yang meningkatkan proses pemisahan lubang elektron. Kaedah metodologi permukaan tindak balas (RSM) untuk pemangkin menunjukkan model yang baik dengan nilai kebarangkalian yang rendah (<0.0001) dan pekali penentuan yang tinggi (R^2) dengan 97.80% peratusan optimum 2,4-D degradasi pada pH 3.24, 0.009 g L^{-1} pemangkin Ag dan 8.15 mg L^{-1} daripada kepekatan 2,4-D. Kajian kinetik pemangkin menggambarkan bahawa tindak balas permukaan adalah langkah mengawal proses. Kajian penggunaan semula pemangkin menunjukkan bahawa pemangkin masih stabil selepas 4 reaksi berikutnya. Secara kesuluruhan, kaedah sintesis pemangkin boleh menjadi satu kelebihan besar dalam pembangunan teknologi nanoteknologi untuk kemerosotan pelbagai bahan cemar organik.

ABSTRACT

The 2,4–dichlorophenoxyacetic acid (2,4–D) herbicide used widely in the agricultural industry is the main toxic pollutant that has caused damage to the aquatic ecosystems and human health. Due to this, continuous research on the effectiveness of catalyst for degradation of this recalcitrant pollutant has been conducted in these recent years. In this study, silver (Ag) catalysts were synthesized and characterized via electrochemical methods in leaves extract and ionic liquid. The performance of the synthesized catalyst towards degradation of 2,4–D was investigated and optimized by Response Surface Methodology (RSM). The kinetic model and reusability of the synthesized photocatalyst were also studied. First, aromatic leaves were extracted using ultrasonic–assisted hydro–distillation (UAE–HD) method and classical aqueous extraction (AE). Then, Ag nanoparticles were synthesized by electrochemical methods with aromatic leaf extracts and ionic liquids as synthesis media. The catalysts were then characterized using X–ray diffraction (XRD), Scanning Electron Microscopy (SEM), Transmission electron microscopy (TEM), Fourier transform infrared (FTIR), Surface area analysis (BET), UV–vis diffuse reflectance spectra (UV–vis DRS) and Photoluminescence (PL) studies. Then, the photocatalytic activity of the catalyst was tested by degradation of 2,4–D. Furthermore, the optimization of the photocatalytic process was carried out by using Response Surface Methodology (RSM). The kinetic study of the photocatalytic reaction was also analyzed by first order, second order and Langmuir Hinshelwood. Lastly, the reusability study was conducted to determine the efficiency of the catalyst. The results indicated that UAE–HD method was able to yield a higher amount of phenolic content as compared to AE method. The results revealed that ionic liquids and phenolic compounds in leaf extract have synergistic effects to reduce silver ions (Ag^+) into zero–valent Ag nanoparticles and act as capping agents in the nanoparticles formation. Then, the performance of the catalysts was tested towards the photodegradation of 2,4–D in a batch reactor under visible light irradiation. The results showed that the Ag nanoparticles were able to inhibit electron–hole recombination to give a nearly complete degradation (96.54%) of 10 mg L^{-1} 2,4–D at pH 3 when using 0.01 g L^{-1} of Ag catalyst prepared in 1–butyl–3–methylimidazolium bis(trifluoromethylsulfonyl) imide [BMIM Tf_2N] ionic liquid and *Orthosiphon stamineus* (OS) leaves extract media extracted by UAE–HD method. It was found that high amount of phenolic content in leaf extract as well as a long alkyl chain of imidazolium cation and large structure of anion led to diminutive and discrete nanoparticles, which enhanced the photodegradation of 2,4–D. Besides, the existence of the phenolic compound and ionic liquid on the surface of the catalysts play important roles as electron acceptors that enhanced the electron–hole separation process. The response surface methodology (RSM) analysis of the catalysts showed a good significance of model with low probability values (<0.0001) and a high coefficient of determination (R^2) with 97.80% of the optimum percentage of 2,4–D degradation at pH 3.24, 0.009 g L^{-1} of Ag catalyst and 8.15 mg L^{-1} of 2,4–D concentration. The kinetic studies of the catalysts illustrated that the surface reaction was controlling the step of the process. A reusability study showed that catalysts were still stable after 4 subsequent reactions. Significantly, the synthesis method of the catalysts could be a great advantage in the future development of nanotechnology for degradation of various organic pollutants.

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LIST OF SYMBOLS

e^-	Photogenerated electron
h^+	Photogenerated hole
OH•	Hydroxyl radical
C_0	Initial concentration of the 2, 4-D
C	Concentration at any time
C_t	Concentration of 2, 4-D solution after t hours
D	Mean size of the ordered (crystalline) domains
k	Dimensionless shape factor with a typical value of 0.9
λ	X-ray wavelength
β	Full width at half the maximum intensity in radians
θ	Bragg angle
r	Initial photocatalytic degradation rate
k_r	Reaction rate constant
K_{LH}	Adsorption equilibrium constant
$1/k_r$	Intercept
$1/k_rK_{LH}$	Slope of the plot
k_1	First-order rate constant
k_2	Second-order rate constant
E_{EO}	Electrical energy
P	Power
V	Volume

LIST OF ABBREVIATIONS

2,4–D	2,4–Dicholorophenoxyacetic acid
Ag	Silver
TPC	Total phenolic content
OS	*Orthosiphon stamineus*
PC	*Petroselinum crispum*
PM	*Polygonum minus*
CN	*Cymbopogon nardus*
AC	*Allium Cepa*
ILs	Ionic liquids
[BMIM Tf_2N]	1–butyl–3–methylimidazolium bis(trifluoromethylsulfonyl) imide
[BMIM BF_4]	1–butyl–3–methylimidazolium tetrafluoroborate
[BMIM $EtSO_4$]	1–ethyl–3–methylimidazolium ethyl sulphate
UAE–HD	Ultrasonic–assisted hydro–distillation
AE	Classical aqueous extraction
XRD	X-ray diffraction analysis
SEM	Scanning Electron Microscopy
TEM	Transmission electron microscopy
BET	Brunnauer–Emmett–Teller
FTIR	Fourier Transform Infrared
PL	Photoluminescence
UV–Vis DRS	UV–vis diffuse reflectance spectra
VB	Valence band
CB	Conduction band
CCD	Central composite design
RSM	Response surface methodology
PD	Potassium dichromate
IP	Isopropanol
SO	Sodium oxalate
NaOH	Sodium hydroxide
HCL	Hydrochloric acid

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