FRUIT WASTE MEDIATED SYNTHESIS OF SILVER NANOPARTICLES AND ITS DECOLORIZATION CAPABILITY ON DYES

SARAH SYAFIQAH BINTI JAJULI

UNIVERSITI TEKNOLOGI MALAYSIA

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SARAH SYAFIQAH BINTI JAJULI

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DEDICATION

This thesis is dedicated to my father, Jajuli Bin Marzuki and my mother, Hafsah Binti Rafiee who work hard to get me a proper education, who have been my source of inspiration and strength whenever I feel like giving up and continuously provide moral, spiritual and financial support. In addition, to my family and friends and who always support and give me motivation and encouragement throughout this journey. Without their love and support, this research would not have been made possible.

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ABSTRACT

Conventional synthesized silver nanoparticles (AgNP) often correlated with harmful chemicals and by-products, consumed large amount of energy and involved complex procedure. Therefore, green synthesis of AgNP which offers eco-friendly and cost-effective method in synthesizing AgNP was proposed. This study used banana peel extract (BPE) and pineapple peel extract (PPE) as a reducing agent and stabilizer, and silver nitrate ($AgNO_3$) as a metal salt precursor. Volume of peel extracts, concentration of $AgNO_3$, reaction temperature and incubation time were optimized and the AgNP were characterized. The optimum conditions to produce BPE-AgNP and PPE-AgNP were successfully investigated. Total Phenolic Compound (TPC) analysis showed that BPE has higher phenols content than PPE which is 14.671 mgGAE/g and 8.479 mgGAE/g respectively. Visible spectrum displayed surface plasmon resonance (SPR) peak at 430 nm and 450 nm for BPE-AgNP and PPE-AgNP respectively. X-ray diffraction (XRD) displayed both BPE-AgNP and PPE-AgNP has face centered cubic crystalline nature. FESEM micrograph revealed both BPE-AgNP and PPE-AgNP are almost spherical with the particles size of 30.99 ± 9.47 nm and 21.6 ± 7.01 nm respectively. Energy dispersive X-ray spectroscopy (EDX) analysis showed the highest peak at 3 KeV which is in silver region, confirming the presence of elemental silver. Fourier transform infrared spectroscopy (FTIR) analysis confirmed the functional group responsible as reducing agent in the transformation of Ag^+ to Ag^0 and resulted to the stabilization of AgNP structure. BPE-AgNP and PPE-AgNP showed good potential as a catalyst for methylene blue (MB) and methyl orange (MO) decolorization. Decolorization kinetics observation indicated MB decolorization was best fitted to first order and intraparticle diffusion model for BPE-AgNP and pseudo first order model for PPE-AgNP. Meanwhile, MO decolorization was best fitted to pseudo-second order model for both BPE-AgNP and PPE-AgNP.

ABSTRAK

Kaedah konvensional untuk menghasilkan nanopartikel perak (AgNP) sering dikaitkan dengan penggunaan bahan kimia yang berbahaya, penghasilan produk sampingan yang berbahaya, penggunaan tenaga yang tinggi dan melibatkan prosedur yang rumit. Oleh yang demikian, kaedah sintesis hijau AgNP telah dicadangkan untuk digunakan dalam kajian ini kerana ianya menawarkan kaedah yang mesra alam dan kos efektif. Kajian ini menggunakan ekstrak kulit pisang (BPE) dan kulit nanas (PPE) sebagai sumber reduktor dan penstabil, manakala perak nitrat ($AgNO_3$) digunakan sebagai sumber garam logam. Isipadu BPE dan PPE, kepekatan $AgNO_3$, suhu reaksi dan masa inkubasi dioptimumkan dan AgNP akan dianalisis. Keadaan optimum untuk kedua-dua BPE-AgNP dan PPE-AgNP berjaya dikaji. Analisis kadar fenol di dalam ekstrak mendapati BPE mengandungi fenol lebih tinggi daripada PPE iaitu 14.671 mgGAE/g and 8.479 mgGAE/g. Spektrum spektroskopi menunjukkan sensor permukaan plasmon resonans (SPR) pada 430 nm untuk BPE-AgNP dan 450 nm untuk PPE-AgNP. Kristalografi sinar X (XRD) menunjukkan BPE-AgNP dan PPE-AgNP mempunyai sifat krital kubik berpusatkan wajah. Fesem mikrograf pula menunjukkan BPE-AgNP dan PPE-AgNP mempunyai bentuk bulatan dengan saiz 30.99 ± 9.47 nm and 21.6 ± 7.01 nm. Spektroskopi tenaga serakan sinar X (EDX) menunjukkan puncak tertinggi pada 3 KeV mengesahkan kehadiran elemen perak di dalam BPE-AgNP dan PPE-AgNP. Infra merah transformasi fourier (FTIR) mengesahkan kumpulan yang memainkan peranan dalam penghasilan dan penstabilan BPE-AgNP dan PPE-AgNP. BPE-AgNP dan PPE-AgNP mempunyai potensi yang bagus sebagai pemangkin penghapusan pewarna metilena biru (MB) dan metil oren (MO). Kajian kinetik menunjukkan menunjukkan penyahwarnaan MB oleh BPE-AgNP mengikut model

pertama dan model peneybaran intrapartikel psuedo-first order sementara PPE-AgNP mengikut model semu-pertama. Manakala, penyahwarnaan MO oleh BPE-AgNP dan PPE-AgNP mengikut model semu-kedua.

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LIST OF ABBREVIATIONS

AgNP	-	Silver nanoparticles
$AgNO_3$	-	Silver nitrate
$NaBH_4$	-	Sodium borohydride
$NaCO_3$	-	Sodium carbonate
BPE	-	Banana peel extract
PPE	-	Pineapple peel extract
PPE- AgNP	-	Silver Nanoparticles from banana peel extract
BPE AgNP	-	Silver Nanoparticles from pineapple peel extract
KBr	-	Potassium bromide
MB	-	Methylene blue
MO	-	Methyl orange
FTIR	-	Fourier-Transform Infrared Spectroscopy
XRD	-	X-ray Diffraction Crystallography
FESEM	-	Field Emission Scanning Electron Scanning Microscopy
EDX	-	Energy Dispersive X-ray spectroscopy
TPC	-	Total phenolic content

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CHAPTER 1

INTRODUCTION

1.1 Background Study

Water is essential and an important element in daily life. These days, providing safe and clean water to the public is a challenge due to the growth of the economy, industrial, and technology as it increase the number of pollutants and contaminants in water (Alvarez *et al.,* 2018).

One of the pollutants is a dye where it can be obtained naturally or synthetically with the latter is mainly used (Saha *et al.,* 2017). The dye is used for various industrial purposes and the effluent released is a great concern to the public because the dye absorbs and reflects sunlight which prevents the penetration of light and reduction of dissolved oxygen that consequently results in the generation of oxygen-limiting environment due to the deteriorating rate of photosynthesis and respiration by the aquatic organisms (Singh and Singh, 2017; Kumar *et al.*, 2016).

Most of the dyes are highly stable because they consist of aromatic structures making it hard to degrade and remove from water. Traditionally, the dye is managed through coagulation, filtration, adsorption, reverse osmosis, UV radiation, flocculation, ion exchange, ozonation, and electrochemical reduction. However, these methods are costly because it involves complex procedures and also produce secondary waste (Qing *et al.*, 2017; Saha *et al.*, 2017).

Nanotechnology is a revolutionary science to overcome challenges faced in the water treatment crisis through remediation, purification, detection, and prevention. The key factor in nanotechnology is the exceptionally small size of nanoparticles (1–100 nm). The small size increases the surface area and makes it highly effective (Patanjali *et al.,* 2019). Nanoparticles also have rich surface modification chemistry which allows it to be grafted or functionalize on specific targets. These characteristics give an advantage to nanotechnology compared to conventional methods in wastewater treatment (Guerra *et al*., 2018).

One of the most well-known nanoparticles is silver nanoparticles (AgNP). AgNP possess catalytic activity, robust optical features, and high electrical conductivity. It also has a broad spectrum of antimicrobial, antiviral, and antifungal properties (Sardar *et al.*, 2017). Conventionally, AgNP are synthesized through a chemical and physical pathway. In the chemical pathway, a chemical reduction is the most common practice to form AgNP where three main components are needed which are metal precursors, reducing agents and stabilizing or capping agents (Chouhan, 2015). Meanwhile, heat and pressure are utilized in physical pathway to synthesize AgNP. The common method used is evaporation-condensation and laser ablation (Iravani *et al*., 2019).

Currently, the biological pathway has emerged as a novel method in producing AgNP which utilizes microorganisms and parts of plants. Biological pathway practices a green synthesis approach that is eco-friendly, sustainable, and low cost. Microbes can produce intracellular and extracellular inorganic material and proteins which can act as a metal reduction agent (Alves *et al*., 2019). Meanwhile, plants and its part consist of bioactive compound such as sugar, protein, terpenoid, flavonoid, alkaloids, and phenolic compounds. These bioactive compound can act as reducing and stabilizing agent during AgNP synthesis (Rafi *et al.*, 2018; Parveen *et al*., 2016).

Fruit wastes are the other alternative in a biological pathway which capable of synthesizing AgNP. The rich amount of fiber and nutrients in the fruit waste contributes to its value-added. For instance, fruit waste has been used in food industries as an ingredient in the flour, thickener, and stabilizer and as an edible coating film. In non-food industries, it has been as biosorbent, biocomposite, and as biomass energy (Cheok *et al.*, 2018). Research on the composition of watermelon rind, mango peel, banana peel, and mangosteen pericarp showed a significant number of phytochemicals (Dewi, 2017). These phytochemicals can be used as a reducing and stabilizing agent in AgNP synthesis.

In this study, fruit peels will be utilized to extract the bioactive compound which acts as both the reducing agent and stabilizing agent for the green synthesis of AgNP. The AgNP application will be a focus on its catalytic activity in dye degradation for the water remediation.

1.2 Problem Statement

AgNP are conventionally synthesized through a chemical reduction where it utilized chemicals such as sodium citrate ($Na_3C_6H_5O_7$), sodium borohydride ($NaBH_4$), hydrazine (N_2H_4), N-dimethylformamide (DMF), ascorbate ($C_6H_7NaO_6$), ethylene glycol ($C_2H_6O_2$), and hydrogen dextrose as a reducing agent. Subsequently, stabilizing agents such as surfactants, chitosan, gluconic acid, cellulose and polymers are used to stabilize and avoid agglomeration of AgNP. Regardless, some of these chemicals are expensive, highly reactive, non-biodegradable and even found to be attached to the AgNP surface which hinder its suitability for further application (Silva *et al.*, 2016). Usage of organic solvent during extraction of bioactive compound from plant and its part also raise a concern because it retained in the extract and lead to the presence of unwanted components thus reduced their biological potential (Cvetanović, 2019). Therefore, green synthesis serves as alternative route to minimize usage of chemicals by utilizing fruit peels as a source for natural reducing and stabilizing agent (Dewi, 2017). AgNP has various applications and one of them is, emerging as nano-catalyst in the degradation of the dye due to its high reactivity (Bhattacharya *et al.*, 2015). Usage of green synthesized AgNP to decolorize organic dyes in water bodies will prevent formation of another pollutant. Hence, this study intends to focus on the green synthesis of AgNP using banana peel and pineapple peel extract and its potential as a catalyst in the degradation of methylene blue (MB) and methyl orange (MO).

1.3 Research Objectives

Followings are the objectives proposed for this study: -

(a) To prepare AgNP from banana (BPE-AgNP) and pineapple peel extracts (PPE-AgNP) at optimized condition.

(b) To evaluate the physicochemical properties of BPE-AgNP and PPE-AgNP using instrumental analysis.

(c) To determine the capabilities of BPE-AgNP and PPE-AgNP as a catalyst in decolorization of methylene blue and methyl orange.

1.4 Scope of Study

This research investigated the potential of banana peel extract (BPE) and pineapple peel extract (PPE) in the green synthesis of AgNP and also the catalytic activity of the AgNP for bioremediation. The fruit peels were extracted using solid-liquid extraction and were characterized using total phenolic compound and Fourier-Transform Infrared spectroscopy (FTIR) to determine the functional group responsible for the reduction of Ag^+ to AgNP. The green synthesis of AgNP was done by mixing BPE and PPE with $AgNO_3$. Various parameters were applied to find an individual optimum condition in AgNP synthesis. The parameters were the reaction temperature, volume of extract, concentration of $AgNO_3$, and incubation time. Subsequently, the AgNP were synthesized under optimum conditions and characterized using visible spectroscopy, FTIR, Transfer Electron Microscope (TEM), Energy-Dispersive X-ray spectroscopy (EDX), X-ray Diffraction (XRD), and zeta potential. The instrumental analysis was done to determine the presence and the morphology of AgNP. Lastly, the catalytic activity of BPE-AgNP and PPE-AgNP were applied to the decolorization of methylene blue and methyl orange.

1.5 Significance of Study

The accomplishment of this research is to synthesize AgNP from fruit wastes that will serve another alternative in synthesizing AgNP apart from conventional methods which mainly use expensive and hazardous chemicals as a substrate and solvent. In addition, it avoids the usage of complex tools and can save lots of energy during the process. Not only this research provides green and clean methods of AgNP synthesis, but it also helps in fruit waste management by providing value-added into fruit wastes as a reducing agent and stabilizing agent. Besides, the bioremediation potential of AgNP offers a cheap, eco-friendly, and effective way of removing dye effluent that is harmful to the environment.

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