GREEN SYNTHESIS, CHARACTERIZATION AND LASER IRRADIATION OF SILVER NANOPARTICLES AND ITS BIMETALLIC NANOPARTICLES

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A thesis submitted in
fulfilment of the requirements for the award of the
Degree of Master of Science

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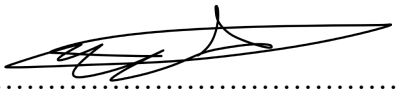
NOVEMBER 2020

I hereby declare that the work in this thesis is my own except for quotations and summaries which have been duly acknowledged.

Student : 

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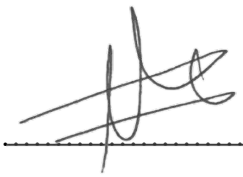
Co-Supervisor: 

DR. ADEL ALI SAEED AL- GHEETHI

This work is dedicated to my family for their support, patience, understanding and prayers throughout the period of this research.

ACKNOWLEDGEMENT

I thank the Almighty who bestowed upon me the grace of reason and religion. The Prophet Muhammad (peace be upon him) said: "Who made you known reward him, if you do not find what you reward him, pray for him until you see that you reward him" (Narrated by Abu Dawood). I commend the praise well to my supervisor Associate Professor Dr. Mohd Arif bin Agam and co-supervisor Prof. Madya Dr. Radin Maya Saphira. I also extend my sincere thanks to those who have spared no effort in helping me in the field of scientific research, especially my co-supervisor Dr. Adel Ali Al-Gheethi for standing by my side and who is credited with guiding me and helping me to compile the research material. I also extend my sincere thanks to my brother Dr. Ammar Saleh Habtoor, who has the highest credit after God for encouraging me to study and help me throughout the stages of the program. All thanks and gratitude to both my beloved mother, my precious wife and my sisters for their patience and prayers and their support for me to achieve my goals and aspiration for my success.

ABSTRACT

Green synthesis of metallic nanoparticles offered an alternative and eco-friendly method for fabricating nanoparticles compared to chemical and physical methods. The current study aimed to manipulate silver nanoparticles (Ag NPs) synthesized in *Murraya koenigii* leaf extract to fabricate nanostructure by using diode laser irradiation. The green synthesized silver nanoparticles (Gre-Ag NPs) used as reducing and stabilizing agent to synthesize silver/copper nanoparticles (Ag/Cu NPs). The Ag NPs was synthesized by mixing Silver nitrate solution ($AgNO_3$) (1, 3 and 5 mM) with *M. koenigii* leaf extract at room temperature and left for two days. In contrast, Ag/Cu NPs were synthesized by mixing Cu solution (1, 3, 5 and 7 mM) with Gre-Ag NPs at room temperature and left for 10 mins. Moreover, the silver nanostructures (Ag NSs) was fabricated by manipulating Gre-Ag NPs with diode laser 450 nm at different times. The Gre-Ag NPs, Ag/Cu NPs and Ag NSs were characterized by advanced analysis Ultra-Violent Visible (UV-Vis), Fourier transform infra-red (FTIR), X-Ray diffraction (XRD), Field emission scanning electron microscope (FESEM) and Atomic Force Microscopy (AFM). UV-Vis analysis showed peaks ranging from (426 to 441 nm) indicating AgNPs formation, as well as the absorbance at 512 nm to indicate the formation of Ag/CuNPs, in addition to a decrease in the absorbance when converting the silver nanoparticles into nanostructures. As for the FTIR, the results showed absorbing peaks for both *M. konigii* extract and AgNPs to indicate that the amino and carboxylic groups are responsible for the formation of AgNPs. The results of FESEM revealed that the Gre-Ag NPs have a spherical shape with diameter parameters between 10 and 80 nm, and XRD appeared that the Gre-Ag NPs have crystalline properties. The silver nanoparticles after being manipulated by diode laser irradiation for different exposure times are deformed into nanostructures as nano prisms, nanocone and nanohexagon examined by FESEM. Preparing metallic nanoparticles using the green method and manipulating them by diode laser irradiation is a simple and easy way to obtain different nanostructures.

ABSTRAK

Teknik Sintesis Hijau nanopartikel logam memberikan kaedah fabrikasi alternatif yang bersifat mesra alam berbanding kaedah fabrikasi kimia dan fizikal. Kajian semasa menumpu kepada fabrikasi bahan nanopartikel seperti nanopartikel Perak (Ag NPs) dan disintesis melalui ekstak daun *Murraya koenigii* menggunakan kaedah pancaran sinar laser berwarna biru. Bahan Ag NPs hijau (Gre-Ag NPs) disintesis melalui proses penurunan kimia dan bahan penstabil dalam penghasilan bahan nanopartikel Perak/Tembaga (Ag/Cu NPs). Ag NPs dihasilkan melalui percampuran larutan Perak ($AgNO_3$) pada varisasi kepekatan (1, 3, 5 mM) dicampurkan kepada ekstrak daun *M. koenigii* dan diperam pada suhu bilik selama 2 hari. Sementara, Ag/Cu NPs difabrikasi dengan percampuran larutan Cu pada kepakatan (1, 3, 5 mM) dengan Gre-Ag NPs pada suhu bilik selama 10 minit. Manipulasi bahan nanopartikel berstruktur Perak melalui manipulasi Gre-Ag NPs dengan pancaran laser biru 450 nm pada durasi berbeza. Sifat Bahan Gre-Ag NPs, Ag/Cu NPs dan Ag NSs dianalisa menerusi peralatan seperti Ultra-Violent Visible (UV-Vis), Fourier transform infra-red (FTIR), X- Ray diffraction (XRD), Field emission scanning electron microscope (FESEM) dan Atomic Force Microscopy (AFM). Analisa puncak-puncak UV-Vis berjulat dari 426 to 441 nm menbuktikan penghasilan bahan AgNPs bersaiz nano. Sementara keputusan analisa melalui FTIR, menunjukkan puncak-puncak penyerapan oleh kedua-dua bahan ekstrak *M. konigii* and logam AgNPs, yang menjelaskan fungsi kumpulan bahan-bahan yang membentuk logam AgNPs. Analisa FESEM menentusahkan Gre-Ag NPs berbentuk sfera dengan julat saiz diantara 10 and 80 nm dan analisa XRD menentusahkan sifat-sifat logamnya. Selepas manipulasi dengan pancaran sinar laser berwarna biru pelbagai bentuk struktur nanopartikel logam diperolehi, antaranya nanoprism, nanocone, nanohexagon, and bahan nano tidak seragam dan kompleks. Kombinasi penghasilan bahan logam bersaiz dan berstruktur nano melalui Teknik Hijau dan manipulasi pancaran Laser berwarna biru

mampu menghasilkan pelbagai bahan berstruktur nano yang berbeza dapat dihasilkan, dicirikan dengan mudah dan berkesan.

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LIST OF SYMBOLS AND ABBREVIATIONS

°	-	Degree
°C	-	Degree Celsius
1D	-	1 dimensional
2D	-	2 dimensional
3D	-	3 dimensional
AFM	-	Atomic Force Microscopy
Ag	-	Silver
Ag-1	-	Silver concentration 1mM
Ag-3	-	Silver concentration 3mM
Ag-5	-	Silver concentration 5mM
$AgNO_3$	-	Silver nitrate
AgNPs	-	Silver nanoparticles
AgNSs	-	Silver nanostructures
Au	-	Gold
BLL	-	Blue Light Laser
Cu	-	Copper
$CuSO_4$	-	Copper Sulfate
FCC	-	Face centred cubic
FESEM	-	Field emission scanning electron microscope
FTIR	-	Fourier transform infra-red
G	-	Gram
Gre-AgNPs	-	Green synthesized silver nanoparticles
JCPDS	-	Joint committee on powder diffraction standard
ml	-	Millilitre
mM	-	Millimolar

MNP	-	Metal nanoparticle
Nd-YAG	-	Neodymium doped Yattrium-Alumnum-Garnet
nm	-	Nano Meter
NP	-	Nanoparticle
NT	-	Nanotechnology
PLAL	-	Pulse Laser Ablation in Liquid
Rel-AgNPs	-	Released silver nanoparticles
SPR	-	Surface plasmon resonance
UV-Vis	-	Ultra-Violet Visible
XRD	-	X- Ray diffraction

CHAPTER 1

INTRODUCTION

1.1 Background

Nanotechnology is one of the most interesting technology due to its entry into many fields such as medicine, industry. The recent directions in the nanotechnology focused on the preservation of the environment, in this chapter, the techniques used in the synthesis of nanoparticles was presented, where the biosynthesis (green synthesis) process in producing Silver nanoparticles (Ag NPs) is proposed. The manipulation technique, including the laser irradiation used to modify nanomaterials as well as the potential applications as catalysts, photocatalysts, electronics and antibacterial materials was also introduced (Lei *et al*., 2018).

Recent advancement of nanotechnology aimed to use the material at the atomic and molecular level to reach new nanometer-scale materials (Mansoori, 2002; Cheng *et al*., 2015). The major properties of nanomaterials such as surface morphologies, crystalline and molecular structure, porosity, electric and magnetic properties as well as solubility of the particles have received more attention are due to their small shapes and sizes that are less than 100 nanometres (Fan & Zhang, 2016).

Nanomaterials can be divided into two main types; the first type has three dimensions within a nanometre range (1-100) called nanoparticle, which may be a polymer, metallic or metal oxide (Gubin, 2009). The second type has at least one dimension within the nanoscale range and is called nanostructure (Han *et al*., 2012). They can be one external dimension (1D) is out 100 nm range, such as Nanorods and

nanowires or two external dimensions (2D) like Nanosheets and Nanofilms (Cao, 2004).

Synthesizing of nanomaterials are divided into two approaches, the "top-down" and the "bottom-up" approach. Top-down, where the size of the bulk material is broken down into smaller materials processed through physical and chemical methods. The Bottom-up is called self-assembly, where nanomaterials are formed by assembling atoms or by clustering molecules to obtaining bigger nanomaterials (Galstyan & Bhandari, 2018).

Currently, nanoparticles have been applied in many aspects of life, such as clothing; architecture; cosmetics as well as the manufacturing of medical instruments, water purification tools, medicine, energy, biotechnology, agriculture and environment (Sanchez & Sobolev, 2010; Mu & Sprando, 2010; Pacheco-Torgal & Jalali, 2011; Raj *et al*., 2012; Saranyaadevi *et al*., 2014; Abdullaeva, 2017; Almeida & Ramos, 2017; Bovi *et al*., 2017).

Chemical and physical methods can produce nanoparticles. These methods produce nanoparticles in large quantities in a short time. However, these methods require high voltage, high temperatures, and the use of non-degradable materials (Kruis *et al*., 2000). Besides, these methods are costly, require high energy, and produce a toxic waste that can harm the environment in general and humans in particular (Mahdieh & Fattahi, 2015). For example, silver nanoparticles (Ag NPs) prepared by the chemical method is synthesized by using silver salt as precursors and sodium borohydride as a reducing agent and colloidal solution as a stabilizing agent (De Sio *et al*., 2015).

Today, biological sources (bio/green nanotechnology) have received high attention as an eco-friendly and alternative for the chemical and physical methods in producing nanoparticles (Singh *et al*., 2016). Biotechnologies have become more attractive because they are environmentally friendly, consume less energy, and have high efficiency in producing nanoparticles with unique properties (Kulkarni & Muddapur, 2014). The biogenic sources of plants and microorganisms are potential for green-synthesis of nanoparticles and have many possible applications (Njagi *et al.*, 2010). The applications of nanoparticles prepared by green methods are many, including drug delivery, antimicrobial, bioimaging and anticancer drugs (Kotcherlakota *et al.*, 2019). Also, nanoparticles prepared by green methods have been used to remove viruses from drinking water (Gusseme *et al.*, 2010). In addition, it is

used to clean up toxic waste sites (Kalaiselvi *et al.*, 2015) and treat environmental pollutions (Sankar *et al.*, 2015). Biogenic sources such as plant leaves of various kinds of plant species do possess a considerable number of biochemical compounds that carry functional groups acting as antioxidants as they are rich in non-double electrons substance (Tilman *et al.*, 1997).

Murraya koenigii leaf is among identified medical plants that have antimicrobial, anti-diabetic, hepatoprotective, anti-inflammatory, antioxidant and anti-carcinogenic properties (Al Harbi *et al.*, 2016). *Murraya koenigii* leaf contains several vital compounds, where compounds such as amino acids that contain amino (NH_2^-) and carboxylic acid ($COOH^-$) are identified as functional groups that are responsible for reducing and stabilizing ions. The brief mechanism can be illustrated as the chemical reaction of silver ions and the leaf extract in (Figure 1), where silver ions transform into clustered atoms until they become nanoscale particles as given by the equation below.

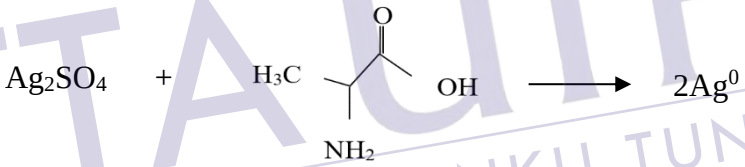

Figure 1.1: Chemical Reduction process in producing Silver nanoparticles

Therefore, the green synthesized NPs using *Murraya koenigii* leaf, where the leaves extract would exhibit antibacterial properties due to their high surface and small particle size, which gives them an increased surface reaction.

Ag NPs are one of the most recently studied nanoparticles due to the high antibacterial activity (Le Ouay & Stellacci, 2015). However, the modification of metallic nanoparticles to obtain many useful properties such as complex structures, purity, stable composition and several other purposes; including reducing the size to nanometre size has been reported (Scaramuzza *et al.*, 2016).

In this research, the green synthesis of silver nanoparticles utilized *Murraya koenigii* leaf extract as a reducing and stabilizing agent to produce silver and silver/ copper nanoparticles. In producing bimetallic silver/copper nanoparticles, the same method are repeated where the *Murraya koenigii* leaf extract is introduced to both silver and copper salt solution. To further manipulate the silver nanoparticles

synthesized from the green-synthesis strategy, the second manipulation strategy of laser irradiation is used to bombard and manipulate the shapes and sizes of the nanoparticles. The ability to manipulate the mono and bimetallic nanoparticles shapes and sizes are vital especially for medicine (antibacterial) and electronic (devices) applications, as different shapes and sizes influence the surface structures that dictate many surfaces and interface interaction.

1.2 Problem statement

Nanostructures that have at least one of its dimensions within the range of 100 nm, and nanoparticles that have three dimensional (3D) within the range of 100 nm are materials of interest in the field of medicine, water purification and electronics. The nanostructures are considered as dimension-less compared to three-dimensional nanoparticles that are having unique shapes and sizes, with higher surface complexities for chemical and biology surface interaction. The high dependence toward chemical and physical methods is characterized with major drawback such as massive production cost and involvement of hazardous materials (toxic), which are the primary concern for scientists and environmentalist. This gap offers an opportunity for the researchers to find alternative methods for nanoparticle synthesis. The green synthesis like plant extracts has vital compounds with functional groups which are used as reducing and stabilizing agent. Therefore, scientists are aggressively looking into mimicking biological synthesis processes in producing materials towards nature's ability in creating nanoparticles.

Moreover, there are many studies on the synthesis of bimetallic nanoparticles by chemical methods in recent years. But, there is no interest in using the green synthesized nanoparticles in the process of reducing ions and stabilizing to obtain different bimetallic nanoparticles. Besides, the conventional methods used in the production of nanostructures are pulsed laser 1064 nm to release nanoparticles in solutions and pulsed laser ablation 532 nm to fabricate nanostructures in solutions. However, these technologies are high cost-oriented, producing by-products that sometimes are toxic and hazardous and need special tools and facilities as well as requires longer time to produce high quality nanostructure. Hence, finding a new method which is easy to use, inexpensive, and able to produce nanostructures is the

theme of this research. In this context, the diode laser 450 nm irradiation for synthesizing nanostructure from silver nanoparticles (AgNPs) is proposed due to its ability to absorb diode laser irradiation. Therefore the current work is focused on the green synthesis of AgNPs and Ag/Cu NPs as well as seek to prove if the diode laser 450 nm irradiation has the potential to generate nanostructure from AgNPs.

1.3 Aim and research objectives

This study aims to synthesize silver and its bimetallic nanoparticles using the *Murraya koenigii* leaf extract and diode laser irradiation as second manipulation technique in producing unique silver nanostructures. The objectives are as follows:

1. To synthesis and characterize of silver nanoparticles using *Murraya koenigii* leaf extract with different concentration of silver nitrate

2. To characterize silver/copper (bimetallic) nanoparticles that synthesized by the reaction of green synthesized silver nanoparticles and copper ions

3. To detect the effect of diode laser irradiation on the characteristic of synthesized silver nanoparticles

1.4 Scope of the study

This research was carried out through three stages. The first stage is the state of preparing materials to synthesis AgNPs, which started with the preparation of *Murraya koenigii* (curry) leaf extract and silver nitrate solution at different concentrations (1, 3, and 5 mM), in addition to preparing copper sulphate solution for use in the second stage. The Ag NPs produced are examine to estimate its forms to be compared with the third stage when the silver nanoparticles are manipulated with laser irradiation. In the second stage, Ag NPs was mixed with copper sulphate solutions to reduce and stabilize copper ions and obtaining the Ag/Cu bimetallic nanoparticles. In the third stage, AgNPs was manipulated by diode laser (JLM45160ZA-O1Y51) irradiation (wavelength450nm, 2000mW) at different periods (1, 10, 15, and 20 mins) to obtain

other structures of the silver nanostructures. This stage is to highlight the ability to manipulate Ag NPs through laser irradiation that can be useful in medical applications, especially the enhanced Ag NPs surfaces when they can be manipulated to form smaller nanostructure.

1.5 Significance of the study

Material development in the field of nanotechnology is one of the most important ways to improve the materials people use to become more efficient. Biotechnology has proven its usefulness and efficiency in the field of nanotechnology, as it has contributed to production of various types of nanoparticles at a lower cost, easier and safer way. Plants are present in every part of the world, and it is easy to get. Nanostructured materials have multiple uses in industrial and health applications, including medical equipment coating and water filters. Therefore, green methods for the production of nanoparticles are classified as reducing the uses of chemicals. Many chemicals are used as reduction and stabilizing agent in producing stabilize nanoparticles, finding a new way in producing nanoparticles by mimicking nature's styles, such as plants in producing nanostructures is becoming more and more important as it reduces the uses of chemicals and its toxicity.

Producing a simple technique in synthesizing bimetallic nanoparticles is another extension to be explored. Understanding the contribution of the first produced nanoparticles, in this case, silver nanoparticles, towards the formation of secondary bimetallic nanoparticles, silver/copper nanoparticles is crucial before exploring manipulation strategies such as laser irradiation.

Manipulating green synthesized silver nanoparticles with a diode laser to obtain silver nanostructures of different dimensions possessing rare electronic, optical, and mechanical properties is needed to be used in medical, water purification, and electronic devices. This type of laser manipulation has less explored in fabricating manipulated metal nanostructures. Nanostructured materials produced by green synthesized nanoparticles and fabricated (manipulated) by diode laser irradiation can be used in coating medical equipment. It can also be used in the water purification filters. Besides, using a diode laser device is very easy and inexpensive.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

In this chapter, a review of studies related to the subject is presented. The nanotechnology and green synthesis and manipulation of silver and silver/copper nanoparticles, as well as their properties, were reviewed. The focus of this chapter is on green synthesis using plant extracts and the fabrication of metallic nanoparticles using laser irradiation.

2.2 Silver Nanoparticles (AgNPs)

Silver is a transitional element and has different properties such as electric and thermal conductivity and has a metallic luster. In the past, silver has many benefits. It is used as a remedy, the manufacture of coins, pots, ointments, and many more (Biswas & Dey, 2015). Recently, interest in nanoscale silver particles has increased in medicine; one of the most widely used silver nanoparticles is antibacterial activity. Antibacterial agents are increasingly desirable at the nanotechnology level. Silver nanoparticles have wide applications in various fields, including water purifiers, surgical instruments, textiles, and cosmetics (Ravishankar Rai & Jamuna Bai, 2011; Thamilselvi & Radha, 2017).

Silver nanoparticles replaced silver sulfadiazine in the treatment of wounds, as they are used as a covering agent in some household appliances and medical surfaces of devices to reduce hospital-related bandages and infections. A silver nanoparticle is prevalent in many health products because of its unique ability to combat infectious

diseases and inhibit the growth of bacteria and germs. The important application of silver nanoparticles is in the medical manufacture, such as topical ointments to stop the infection from burns and open wounds. Nano-silver particles have been reported of having surface plasmon resonance with wave absorption between (400-450 nm), which indicates the possible aggregation of silver NPs due to the reduction and stability of ions in the bio-molecules materials (Thomas *et al.,* 2018; Raja *et al*., 2017) (see Figure 2.1).

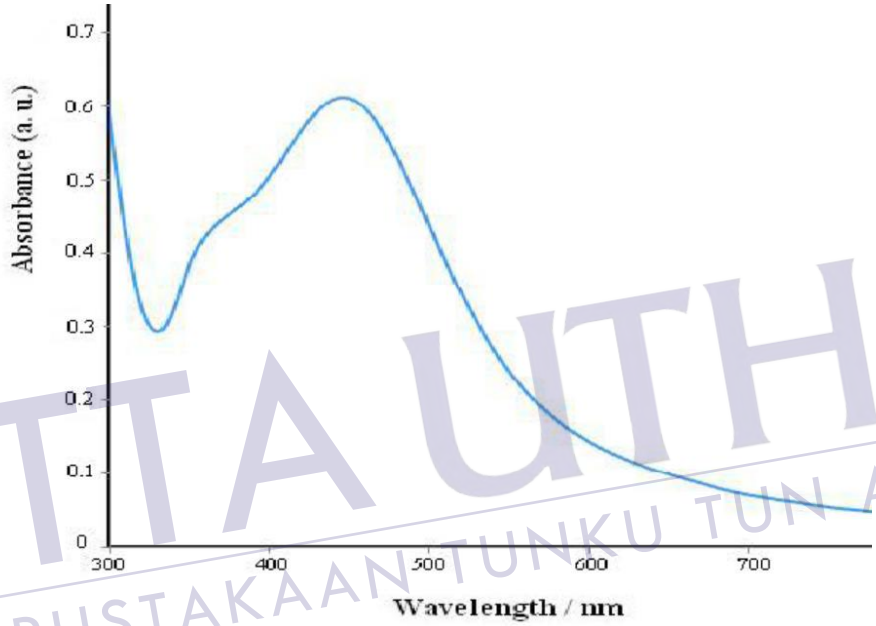

Figure 2.1: UV-Visible spectrum of Ag nanoparticles synthesized by aqueous leaf extract of *Salvadora persica* (Miri *et al*., 2016)

Many researchers have reported Bio-synthesis of inorganic materials, and it is recognized that these methods are considered clean, inexpensive, and non-toxic to humans and the environment. Interest in research and development has grown around silver nanoparticles due to its unique properties that can be applied to bio-nanotechnology (Fayaz *et al*., 2011).

2.3 Silver/Copper bimetallic nanoparticles (Ag/CuNPs)

Bimetallic nanoparticles were produced from two nanometers-sized metals in various structural forms (Liu, 2012). It has geometrical shapes and different patterns that increase its functional capacity, also has more selection, stability, and catalytic activity

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