

Biosynthesis of silver nanoparticles using plant extracts and their application in water treatment

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Dissertation submitted in fulfilment of the requirements for
the degree of *Master of Science in Applied Chemistry* at the
North-West University

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Graduation May 2018

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DECLARATION

I, Tanzimjahan Saiyed, declare that this dissertation entitled 'Biosynthesis of silver nanoparticles using plant extracts and their application in water treatment' and the work that has been presented are my own work.

I confirm that this work was done mainly while as a candidate for a research degree at NorthWest University (Mafikeng campus).

I declared that this dissertation has not been submitted for the award of any other Degree, Diploma, Associateship or Fellowship or any other title in this University or any other University.

I have also consulted the other published works and the source is also always given. With the exception of some quotations, entirely this thesis is my own work.

I have acknowledged all main sources of help.

Signed:

Date:

CERTIFICATE OF ACCEPTENCE FOR EXAMINATION

This work entitled 'Biosynthesis of silver nanoparticles using plant extracts and their application in water treatment' submitted by Tanzimjahan Saiyed, student number 24458910 of the Department of Chemistry in the Faculty of Agriculture, Science and Technology (FAST) is hereby recommended for acceptance for examination.

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ACKNOWLEDGEMENT

With boundless love and appreciation, I would like to extend my heartfelt gratitude and appreciation to all who made this dream a reality. I would like to thank ALLAH for the wealth of life to achieve this Master's programme. In a very special way, I want to thank the following:

Firstly, my supervisor, Dr. D. C. Onwudiwe, a man with a heart of gold without whom this work couldn't get to this stage. I recalled all your encouragements, motivations and fatherly advice which spurred me to take this work to this level. I have learnt a lot from you which would guide me throughout my future endeavors; may God reward you immensely.

I would like to thank my former supervisor Prof. David Isabirye who guided me to choose

Dr. Onwudiwe as my supervisor to begin my Master's programme. All his comments and advice during this study were indelible.

I would also like to thank my Co-supervisor Dr. E. E. Elemike, for all the help he rendered to me in shaping my proposal and dissertation. I worked day in and out with him to be able to acquire most of my data. I am greatly indebted to you sir. Special thanks to my Inorganic and Material Chemistry research group especially, Madam F. F. Bobinihi who always encouraged me when I felt down, a lady who didn't even think twice and gave me her laptop during my proposal writing and O. J. Adeyemi, who always helped me in times of distress and assisted in recovering my forgotten password and lost files or data.

My sincere gratitude also to Dr. Jeje Osuntokun who guided me through article writing and data collection and also gave me my new name as Tanzim Tanzim that always inspired me to double my effort. He had a great sense of humor. I want to thank the lab technician, Kagiso Mokalane, whose help was infinite during my research and the whole Department of Chemistry. My special thanks to all my friends who cared to share a smile with me.

I thank the North-West University for admitting me to study at the university; the NorthWest University postgraduate bursary, and the funds they gave me to help me with the studies.

I would also like to thank my lecturer in India, Dr. G. M. Malek and the management of Navyug Science College who allowed me to do part of my laboratory work during my holiday times.

I would like to thank all my in laws for enduring my absence, when I was far from my children and family. Sincere thanks to my father in law who always encouraged me to finish up my work and mother in law who always stood next to me in my difficult times and took great care of my two little daughters Alvina and Aryana. I am also thankful to my whole family and friends in India whose phone calls can make me more energetic.

DEDICATION

This work is dedicated to each member of my household and especially to a person, who has been a constant source of support and encouragement during the challenges of my studies and life. I can't even imagine standing and holding my degree without you, my dearest husband. Thank you for being in my life and allowing me fulfill our dreams together. I love you Afaq Hussain Syed.

ABSTRACT

The field of nanoscience cuts across many areas of research and researchers continue to explore this interesting area, with the objective of developing materials or improving the properties of the existing ones. Nanomaterials have promising applications in electronics, agriculture, medicine, photo devices and environment. Phytonanotechnology which involves the use of plants has carved out an interesting niche in the field of nanoscience making most Indian greeneries and spices important substrates for the synthesis of nanoparticles (NPs). Umbrella plant, Welsh onion and Guar plants were used for the synthesis of silver NPs in this work. The aqueous plant extract and different concentrations (1 mM, 2 mM and 5 mM) of the precursor compound (AgNO_3) were prepared. In the synthesis of the NPs, two volume ratios of 1:5 and 1:10 of the substrate to the precursor were made from each of the precursor (AgNO_3) concentration (1 mM, 2 mM and 5 mM), and the rate of formation of silver NPs was monitored using UV-vis spectroscopy. The biosynthesized NPs which were represented as UmAgNPs, WoAgNPs and GuAgNPs (reflecting the type of plants involved in their synthesis: Umbrella plant, Welsh onion and Guar plants respectively) were further characterized using Fourier transform infrared (FTIR) spectroscopy, transmission electron microscopy (TEM) and powder X-ray diffraction (pXRD) techniques. The results of the FTIR analysis revealed the presence of biomolecules such as sugars, amino acids, flavonoids on the surface of the NPs, therefore confirming the role of the plants extracts as passivating/capping agents in addition to their reducing effect. The particle sizes of all the NPs: UmAgNPs, WoAgNPs and GuAgNPs, were below 20 nm, with the NPs synthesized from 1 mM AgNO_3 using Welsh onion plant (WoAgNPs) showing the highest average particle size of 15.72 nm. Averagely, GuAgNPs obtained from 2 mM AgNO_3 using 1:10 volume ratio has the lowest particle size among all. However, particles mediated by umbrella plant (UmAgNPs) and prepared from 2 mM AgNO_3 using both of 1:5 and 1:10 volume ratio gave the most stable NPs with high degree of monodispersity.

The potential application of the green synthesized silver NPs in the treatment of water polluted with organic dyes was evaluated by studying their ability to photo-catalytically degrade methyl red, and their antimicrobial potency against some bacteria (majorly the water borne). The UmAgNPs showed the greatest percentage of degradation of about 29.72%. The antimicrobial properties of the NPs against common microbes: *Staphylococcus auerus*, *Bacillus cerues*, *Kleb. pneumonia* and *Echerichia coli* displayed interesting results with WoAgNPs having the greatest growth inhibition against the studied microbes.

Key words: Nanoparticles; Phytonanotechnology; plant extract; inhibition; antimicrobial.

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LIST OF ACRONYMS

AgNPs	:	Silver NPs
CNT	:	Carbon nanotubes
ESBL	:	Extended spectrum beta-lactamases
FTIR	:	Fourier transform infrared
GuAgNPs	:	Guar plant extract capped silver NPs
HPLC	:	High performance liquid chromatography
MIC	:	Minimum Inhibition Concentration
NPs	:	Nanoparticles
pXRD	:	Powder X-ray diffraction
S.D	:	Standard Deviation
SPBs	:	Surface plasmon bands
TEM	:	Transmission electron microscopy
TOC	:	Total Organic Carbon
UV-Vis	:	Ultraviolet visible
UmAgNPs	:	Umbrella plant extract capped silver NPs
WHO	:	World Health Organization
WoAgNPs	:	Welsh onion plant extract capped silver NPs
XRD	:	X-ray diffraction
ZnTAPc	:	Zinc tetraamino phthalocyanine

LIST OF EQUATION

$$A = \epsilon bc \quad 17$$

$$\% \text{ Dye degradation} = \frac{c_0 - c_t}{c_0} \times 100 \quad 17$$

CHAPTER ONE

INTRODUCTION

The study of nanomaterials is a rapidly growing area due to their wide application in different areas of science and technology, for the benefit of manufacturing novel materials with improved properties [1]. Research involving the nanoparticles (NPs) obtained from noble metal has been the subject of increasing focus as a result of their unique optical, electronic, physical and chemical properties that are significantly different from those of their respective bulk materials [2]. Among the NPs of the noble metals, silver NPs have gained wide interests because of their unique characteristics such as chemical stability, catalytic, and biological activities. Silver NPs have been used in cancer diagnosis and as well as treatment [3, 4]. The unique properties of silver NPs could also be incorporated into composite fibers, cryogenic superconducting materials, cosmetic products, food materials, and electronic components for enhanced functional activities [5, 6].

Silver is a soft, semi-liquid, white, shining transition metal which possesses high electrical and thermal conductivity. Due to medical and therapeutic benefits of silver, it has been known longer than the recorded history [7]. Silver is being utilized for different purposes such as coins, vessels and foils. It can also be used in form of colloids as lotions or ointments. It is one of the first generation of materials used as therapeutic agent to combat infectious diseases in medicine [8]. Silver nanoparticles could be obtained from its salts like silver nitrate and silver chloride.

Different techniques such as chemical, physical and biological methods have been used for the synthesis of silver in nanoparticulate form. The different synthetic methods have been summarized in Figure 1.1. The most important physical routes are the evaporation-condensation and laser ablation. The physical synthetic methods have advantage of absence of solvent contamination in the prepared thin films and uniformity of NPs distribution, compared with the chemical processes. However, the physical methods have some disadvantages which include high energy consumption and time demands required to achieve thermal stability [9, 10]. The environmental issues are also great. The most common method for the synthesis of silver NPs is the chemical methods, where organic and inorganic capping agents are used for reduction of silver ions (Ag^+) in aqueous or nonaqueous solutions [11]. Other methods may include photochemical and electrochemical reduction. But there are many problems associated with these methods such as the use of toxic solvents, nonhygienic, expensive and generation of hazardous by-products [12]. Synthesis of NPs via the green route is inevitable because it is harmless, inexpensive, hygienic, eco-friendly and involves benign reaction media and procedures that pose no hazard [13, 14].

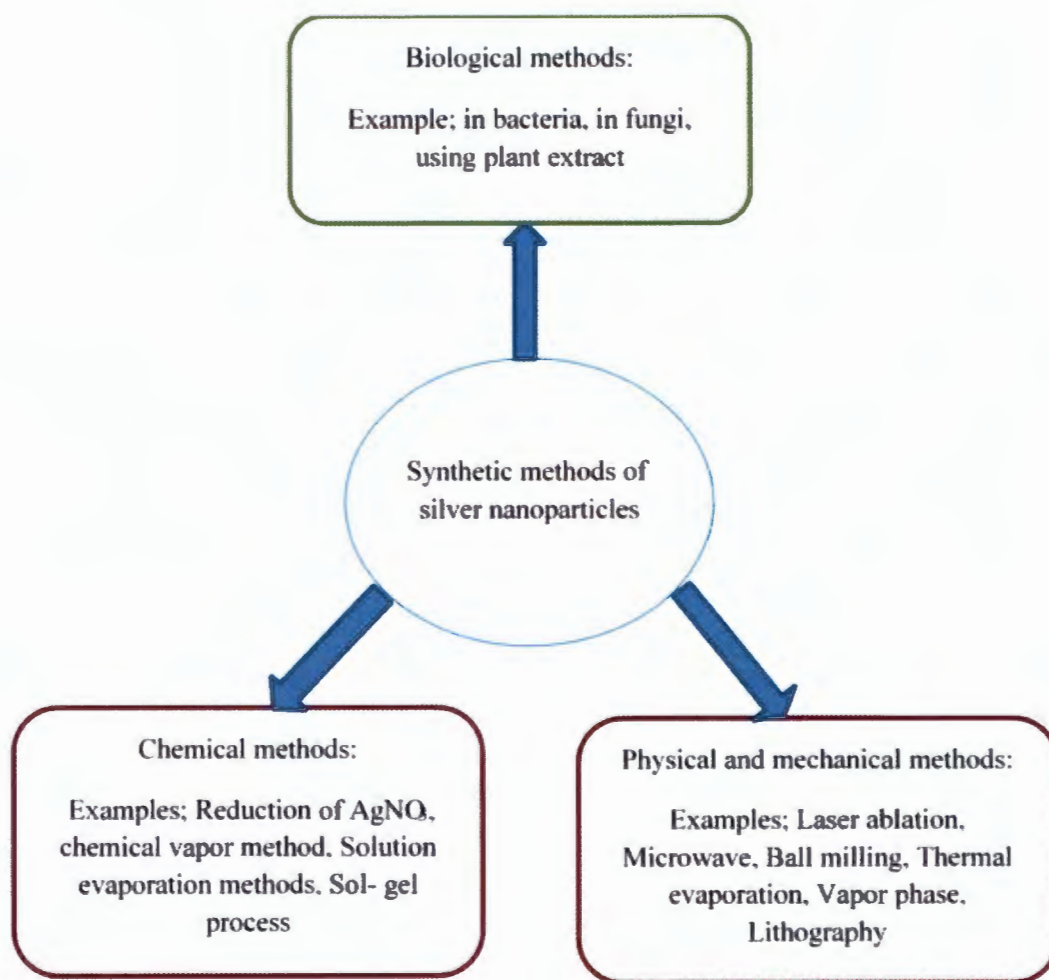


Figure 1.1: Different methods of synthesis of silver NPs [15].

The development of environmentally benign processes for the synthesis of NPs is evolving into an important branch of nanotechnology [16]. An increasing awareness towards green chemistry and utilization of green route for production of metal NPs, especially Ag-NPs was led by a desire to develop eco-friendly methods.

Green synthesis can be grouped into two categories: (a) utilization of microorganisms like fungi, yeasts (eukaryotes), bacteria, and actinomycetes (Prokaryotes), and (b) use of plants and plant extracts [17]. Inorganic materials are produced by bacteria either extra or intracellular. This makes them potential bio-factories for the formulation of noble metal NPs.

Ag-NPs are known to be biocompatible, although some bacteria are known to be Ag resistant [18]. Therefore, these bacteria can aggregate Ag on the cell walls. Fungi have potential for the synthesis of metallic NPs due to metal bioaccumulation capacity and their tolerance, high binding capacity, and

easy to handle in a research facility as compared to bacteria [19]. The development of these microorganisms synthesis methods have low rate, produced limited number of size and shape distribution of NPs, and also tricky compared to the utilization of plants. The synthesis of Ag-NPs using plants or plant extract have advantages over other biological routes. This method overcomes the slow synthetic route of using microorganisms, produced more silver NPs and prevents aggregation of silver NPs. Other advantages of synthesis from plant extracts are provision of hygienic working environment, health and environment shielding, reduced wastages and synthesis of more stable products [20].

1.1 Problem associated with waste water.

Most countries today have drinking water problems. The recommendation of World Health Organization (WHO) is that water meant for human consumption should contain fecal and total coliform counts of 0, in any 100 mL sample [21]. Another concern of waste water is organic pollutants that contain toxic molecular compounds which are capable of causing significant diseases in human life and wildlife [22]. These organic compounds originate from a variety of industrial products such as detergents, petroleum hydrocarbons, plastics, pesticides, dyes and they can be found in diverse environments [23]. Most of these organic compounds, particularly dyes, are not biodegradable and persist within the ecosystem for a protracted period of time.

Immediate investigative action should be taken whenever a contamination in the quality of water occurs. The problem of population growth which causes significant increase in volumes of waste water has also been highlighted. All these make it important to develop effective and affordable technologies for waste water treatment [24].

1.2 Different methods of water treatments.

Different techniques have been used for the treatment of water. Some common treatment includes the use of chemical and physical agents such as chlorine and its derivatives, ultraviolet light [25], boiling, low frequency ultrasonic irradiation [26], distillation, use of water sediment filters (fiber and ceramic) and activated carbon. Other methods include adsorption, precipitation, ion exchange, reverse osmosis, electrochemical treatments, membrane filtration, oxidation, biosorption (used for removing organic cations like Na^+ , K^+ or Ca^{2+} , NH_4^+ from the water) [27]. These physicochemical processes have major disadvantages; they are either very expensive or could not be used at high scale level.

Thus, new methods and technologies which are capable of degrading dye molecules into nontoxic compounds are of importance. Nanotechnology has a great potential in wastewater treatment because it offers advantages such as low cost, reuse and are highly efficient in removing dye pollutants from water [28]. Various forms of nanomaterial such as catalysis, absorption, polymeric and nanocomposite membrane, bioactive NPs and thin film composite have been used in waste water treatment. Diverse organic chemicals [29] are absorbed more efficiently by using carbon nanotubes (CNT) than activated carbon. Metal oxides such as iron oxides, titanium dioxide and alumina are effective, low cost adsorbents for heavy metals and radio nuclides [30-32]. Polymeric nanomaterials like dendrimers have the ability of discarding both organics and heavy metals [33]. Nano-adsorbents are used as powder, beads or porous granules loaded with nano-adsorbents. Membranes with nanofibres are capable of removing micro-size particles from aqueous phase with a high elimination rate, without considerable fouling [34].

Noble metals such as silver (Ag), gold (Au), copper (Cu), platinum (Pt) and palladium (Pd) are used in the water treatment [35]. Among these NPs, silver NPs are the most widely used in water treatment because it is cheaper than gold, platinum and palladium. Compared to copper, it has higher thermal activity and electrical conductivity [28]. Silver NPs (AgNPs) are highly toxic to microorganisms including viruses [36], bacteria [37], and fungi [38]. It exhibits a very high degradation activity under visible light source, widely used in the waste water treatment [39].

1.3 Research aim and objectives

1.3.1 Aim

The aim of this research is to synthesize silver nanoparticles using environmentally friendly approach for application in waste water treatment.

1.3.2 Objectives

The objectives are to:

- synthesize silver nanoparticles using *Cyamopsis tetragonoloba*, *Allium fistulosum* and *Schefflera arboricola* aqueous extract.
- characterize the silver nanoparticles using UV-vis, XRD, TEM, and FTIR.
- study the photocatalytic reduction of selected organic pollutants using the synthesized AgNPs.
- study the kinetics of the photocatalytic reduction of the organic pollutants in water.
- compare the photocatalytic properties of AgNPs with other literature reports.

CHAPTER TWO

LITERATURE REVIEW

2.1 Details of the plants used for the study

Three different types of plants were used for the study. They were sourced from India and South Africa and properly identified by botanist. The plants are welsh onion, umbrella plant and cluster bean. These plants are known to be rich in phytochemicals which are able to serve as both reducing and capping agents.

2.2 Umbrella plant (*Schefflera arboricola*)

2.2.1 Origin and geographical distribution of plant

Umbrella plant originates from the islands of Taiwan and Hainan. It is an ornamental plant which belongs to the Araliaceae family. Its elongated leaves is liken to the shape of an umbrella. Thus, its nickname. It is an evergreen shrub which can grow up to 3-4 m [40]. The plant also goes by many names such as Dwarf Umbrella Tree, Parasol Plant and Octopus Tree.



Figure 2.1: Digital image of Umbrella plant [41].

Table 2.1: Botanical name details of Umbrella plant [40]

Scientific name	<i>Schefflera Arboricola</i>
Common name	Dwarf umbrella tree
English name	Umbrella plant
Genus	<i>Schefflera</i>
Family	Araliaceae

2.2.2 Composition and structure

Many components were isolated from oils of *Schefflera arboricola* Hayata, which contain volatile organics in different percentages such as 20.40% 4-terpineol, 16.01%(-)-spathulenol, and 8.90% caryophyllene oxide [42].

2.2.3 Health benefits of the plant

Pharmacological studies [43] showed that the ethanolic leaf extract of *S. arboricola* exhibited sedative, hypnotic, analgesic, anticonvulsant and smooth muscle relaxant effects. Previous phytochemical investigation on a mixture of leaves and stems of *S. arboricol* has led to the isolation of the two oleanolic acid glucuronoides cynarasaponin H and olaxoside [44]. The species *Schefflera* is used as an antipyretic, anti-inflammatory, analgesic and tonic to treat liver diseases.

2.3 Welsh onion (*Allium fistulosum*)

2.3.1 Origin and geographical distribution of the plant

Welsh onion plant originates from china, and it is one of the cultivated species of *Allium*. Although native to China, it is cultivated in other parts of the world such as Eurasia and North America [45]. It is a perennial specie and the leaves which can be freshly consumed all year to end, have nutritional value [46].



Figure 2.2: Digital image of Welsh onion [46].

Table 2.2: Botanical name details of Welsh onion [45]

Scientific name	Allium fistulosum
Common name	Spring onion
English name	Welsh onion
Genus	Allium
Family	Amaryllidaceae

2.3.2 Composition and structure

The plant has sulphur containing compounds, flavonoids and fatty acids. The sulfurcontaining compounds make up about 87% and 82% of the total volatiles in the distilled oils of Welsh onions. Carbonyl compounds (i.e., aldehydes and ketones) constitute only 3.4% and 7.4% of the total volatiles in these two oils. The compound, methyl ethyl trisulphide is the most abundant among all the volatile compounds isolated from Welsh onions [47]

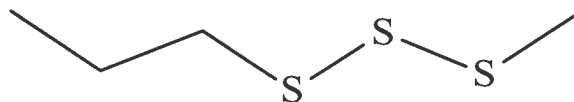


Figure 2.3: Structure of methyl propyl trisulphide [48].

2.3.3 Health benefits of the plant

The Welsh onion has medicinal properties as antifungal and antioxidant due its sulphur containing compounds [45]. There are many therapeutic qualities attributed to the welsh onion especially in Chinese medicine. It is used to increase the activities of internal organs, metabolism, eyesight, aid digestion, perspiration and to prolong life. It can also enhances recovery from headaches, heals wounds and festers sores [49]. The bulb of welsh onions has antibacterial, antiseptic, diaphoretic, diuretic, galactagogue, stomachic and vermifuge (an anthelmintic medicine) properties. It is also used in the treatment of colds [50]. Tea made from the roots of welsh onion is helpful for sedative treatment in children [51].

2.4 Cluster bean (*Cyamopsis tetragonoloba*):

2.4.1 Origin and geographical distribution of the plant

Guar is a plant which originates from Africa, but it has been cultivated throughout Asia especially in India. It is grown there mainly for its green fodder and pods which are used for food. The seeds are used for feeding livestock [52]. It is called cluster bean because of the manner in which its pods are clustered together. It is a coarse, upright, bushy, droughtresistant summer annual plant with height which ranges from 2-4 feet in height. Guar plant, an annual shrub is also grown in other parts of the world, viz. Sudan, Australia, Brazil, South Africa, Pakistan and parts of United States of America [53]. It has soil-enriching properties since it is a legume.



Figure 2.4: Digital image of Cluster bean [14].

Table 2.3: Botanical name details of Cluster bean [53]

Scientific name	Cyamopsis tetragonoloba
Common name	Guar bean
English name	Cluster bean
Genus	Cyamopsis
Family	Fabaceae

2.4.2 Composition and structure

This plant has been much appreciated for its gum obtained from endosperm of the seeds which has many applications in paper, food, mining, cosmetics, textile, oil and pharmaceutical industries across the world [54]. Its flavonoid contains Quercetin, daidizein, kaemferol, and its 3-arabinosides [55]. Guar seeds contain various flavonols such as diadzein, genistein, quercetin and kempherol [56]. The main composition of guar gum is big molecular weight polysaccharides of galactomannans. These are linear chain of (1 → 4)-linked β -D-mannopyranosyl units which contain (1 → 6)-linked α -D-

galactopyranosyl residues as side chains as shown in Fig. 2.5 These galactose and mannose groups represent the galactomannan part of seed endosperm.

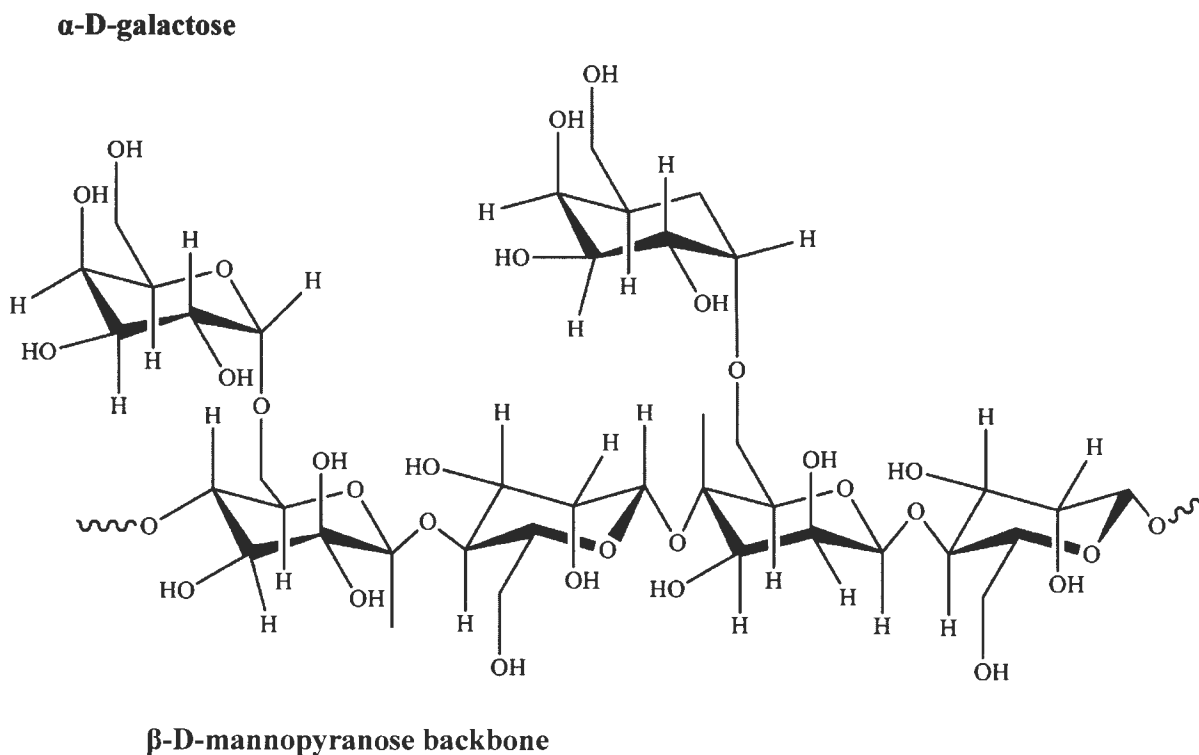


Figure 2.5: Structure of β -D-mannopyranosyl units with (1 $\rightarrow$ 6)-linked α -D-galactopyranosyl residues [55].

2.4.3 Health benefits of the plant

Cluster beans (*Cyamopsis tetragonoloba*) are a rich source of soluble fibre content and are recognized for their cholesterol reducing effect [57]. Guar gum – a galactomannan has been shown to be a potent hypocholesterolaemic agent in both normal and hypercholesterolaemic animals [58]. These cluster or guar beans are also helpful in building bones, teeth, maintaining blood pressure and resolving anemia. The beans have antidiabetic effect, antiulcer, cytoprotective and anticholinergic effect, hypoglycemic and hypolipidemic effect, antiasthmatic activity and antiinflammatory activity on human body [59].

2.5 Silver and silver containing composites

Silver nanoparticles and composites containing silver NPs have been applied in photocatalytic degradation of dyes in water. In all cases, enhanced activity was observed in the degradation of the dye molecules to non-harmful compounds.

2.5.1 Photocatalytic activities using silver nanoparticles from plants extracts

Silver NPs obtained from phytochemicals remarkably display good activity towards photo induced catalytic degradation of dyes. Unripe fruit extracts of *Solanum nigrum* have been used to prepare silver NPs which showed high efficiency in the degradation of industrial effluent containing methyl orange [60]. The aqueous extract from the leaves of *Lagerstroemia speciosa* has been utilised for the preparation of silver NPs which showed photocatalytic activity for methyl orange and methylene blue [61]. Silver NPs obtained using grape seed extract have been used for direct reductive decomposition of Orange 26 [62]. Dried leaf of *Parkia roxburghii* has been utilized as both reducing agent and stabilizer in the synthesis of (Ag) NPs which showed pronounced effect on degradation of methylene blue (MB) and rhodamine b (RhB) under solar light illumination [63].

Roy *et al.* [64] reported some biogenic silver NPs prepared using potato (*Solanum tuberosum*) infusion which showed good photocatalytic activity against methyl orange. Silver nanocatalyst synthesized by one pot approach using edible coconut oil displayed the ability to degrade different dye molecules [65]. The aqueous extract of the flower of *Cassia auriculata* L. was used to prepare silver NPs which showed photocatalytic activity in the degradation of 4-nitrophenol and methyl orange [66]. Spherical Ag NPs stabilized with polyphenols obtained from palm shell extract were evaluated for photocatalytic activity. The results showed remarkable degradation of phthalocyanine based dye, xanthene dye and also a substantial reduction in Total Organic Carbon (TOC) [67].

2.5.2 Photocatalytic activities of composites containing silver NPs

The nanocomposite which showed enhanced photocatalytic activity in the degradation of phenol has been obtained by incorporating silver into titanate nanotubes [68]. Silver and Ag– ZnO composite synthesized by dispersing ZnO into silver nitrate and using *Ocimum tenuiflorum* leaf extract as a reducing agents showed photocatalytic degradation. The result showed that small loading of silver on zinc oxide largely enhances its photocatalytic degradation of Rh B [69]. ZnO microstructures

decorated with Ag NPs were found to enhance the photocatalytic degradation of methylene blue dye [70]. Zinc tetraamino phthalocyanine (ZnTAPc) conjugated to Ag NPs (ZnTAPc–AgNPs) has been reported to show photocatalytic degradation of Rhodamine 6G [71]. Silver NPs have been incorporated into star-shaped zinc oxide particles with enhanced photodegradation of methylene blue solution under UV- irradiation [72]. Zhou and Srinivasan [73] achieved a complete degradation of methylene blue, methyl orange and rhodamine chloride by using silver NPs immobilized on the surface of a quartz bed.

2.5.3 Antimicrobial application of silver NPs from plants extracts

The antibacterial properties of plants extracts could be used to improve antimicrobial potential of silver NPs that could be applied in the removal of bacteria from contaminated water [74]. The antibacterial effects of green synthesized silver NPs from extract of *Solanum nigrum* were tested on different gram-positive and gram-negative bacteria. The inhibition in the growth of the bacteria indicates an antimicrobial activity [60]. Aqueous leaf extract of an aquatic fern, *Salvinia molesta* and *Allium cepa* were effective as eductants in the preparation of silver NPs which showed good efficiency as effective antibacterial agents against both gram-positive and gram-negative bacteria [75, 76]. Spherically shaped silver NPs mediated by banana peels extract were effective against representative pathogens [77]. Size dependent antimicrobial activities of silver NPs obtained using *Asiatic Pennywort* and *Bryophyllum* leaf extracts were studied against both gram negative bacteria *Pseudomonas Fluorescens* and gram positive bacteria *Staphylococcus epidermidis*. Based on the size difference and composition of the plant extracts, the results showed that NPs obtained from *Asiatic pennywort* were more effective on gram positive bacteria, while silver NPs obtained from *Bryophyllum* showed more activity against gram negative bacteria [78].

Priya *et al.* [79] has reported silver NPs using *Euphorbia hirta* and *Nerium indicum* and also examined the antimicrobial activity of the NPs against various bacteria such as *S. pyrogens*, *S. aureus*, *B. Subtilis*, *S. typhi* and *Citrobacter sp.* which showed highly effective results [89].

2.5.4 Antimicrobial application of composites containing silver NPs

The antimicrobial potency of silver NPs is also manifested even when encapsulated in other materials to form composites. Different reports abound in which nanocomposites exhibit antimicrobial activity

by virtue of the presence of silver. Mesoporous Ag-MnO₂ nanocomposites prepared using anon-ionic surfactant and reduced by the leaf extract of

Alternanthera bettzickiana displayed antibacterial activity against gram-positive (*Staphylococcus aureus* and *Streptococcus mutans*) and gram-negative (*Salmonella typhi*, *Escherichia coli* and *Pseudomonas aeruginosa*) bacteria. The results showed that only the biogenic AgNPs and AgNPs@meso-MnO₂ exhibited effective inhibition zones against all pathogens while no activity was observed for control and bulk meso-MnO₂ [80].

CHAPTER THREE

METHOD OF INVESTIGATION

3.1 Preparation of leaf extract

The three plants were sourced as follows: Umbrella and Cluster beans plant were collected from Johannesburg and Mafikeng, South Africa respectively, while Welsh Onion plant was collected from Surat, Gujarat, India. All the plants were satisfactorily identified by botanist in the North West University, South Africa. The leaves were washed with distilled water, air dried and then blended to powdered form. In the preparation of plant extract, 2 gm of the dried powder leaves were boiled with 1000 mL distilled water (hot percolation method) and the resulting infusion filtered thoroughly until no insoluble material appeared in the broth [15].

3.2 Preparation of silver nanoparticles (AgNPs) using aqueous bioextract of the different plants

In separate preparations, different quantities of the broth were added to different concentrations of AgNO_3 solution (1 mM, 2 mM, 3 mM) giving different ratios of 1:5 and 1:10. About 75 mL of each of the leaf aqueous broth was introduced to 350 mL (1 mM) aqueous solution of AgNO_3 . Afterwards, the solution was stirred and heated up to approximately 90 °C, resulting into a gradual colour change from light yellow to reddish brown [81, 82]. Thereafter the solution was centrifuged at 4000- 6000 rpm for 1 h. The solution was removed and the NPs re-dispersed and dried in an oven at 50 °C for characterization. [83].

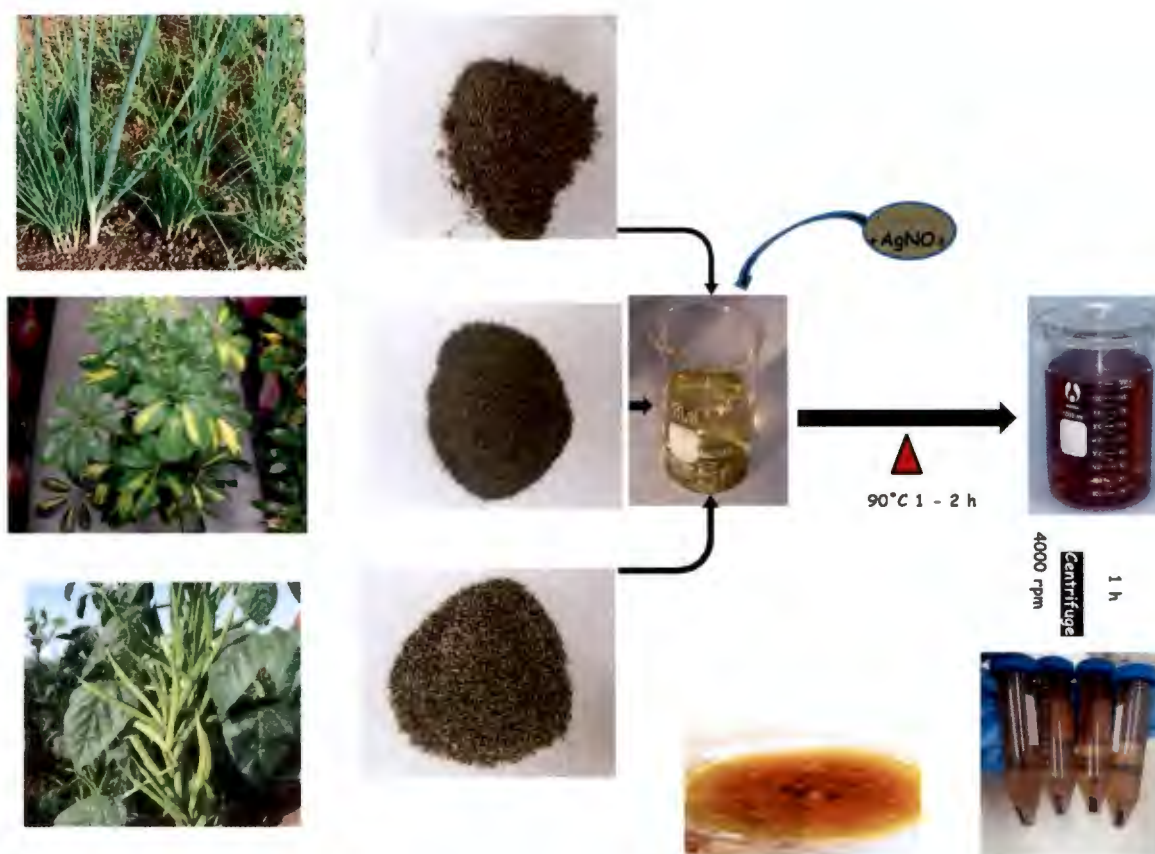


Figure 3.1: Schematic representation of AgNPs preparation with dark brown color signifying that silver NPs have been formed [84].

3.3 Characterization of silver NPs

Varian Cary 300 Bio UV–visible spectrophotometer was used, with a resolution of 1 nm, and distilled water was used as a reference to obtain the absorption spectra of the sample in the range of 200–800 nm. [85]. Image of instrument shown in Figure 3.2.



Figure 3.2: Cary 300 UV-vis spectrophotometer.

Bruker alpha-P FTIR spectrophotometer with wavenumber in the range of 400 to 4000 cm^{-1} was used for FTIR analysis of the functional groups present in plant extracts and the synthesized silver NPs [85].



Figure 3.3: Bruker alpha- P FTIR spectrophotometer.

To check the sizes of the NPs and study the surface morphology, Transmission electron microscope (TEM) model JEOL2100 instrument was used [86].



Figure 3.4: JEOL 2100 Transmission electron microscopy.

The synthesized AgNPs were sonicated for an hour prior to TEM analysis and a drop of the AgNPs solution was placed on carbon coated copper grids. The solution was dried at room temperature and analyzed using an accelerating voltage of 50 kV [85].

Powdered X-ray diffraction (pXRD) analysis was conducted by XPERT-PRO using monochromatic Cu α radiation ($\lambda = 1.5406 \text{ \AA}$) operated at 40 kV and 30 mA at a 2θ angle pattern. The scanning was done in the region of 20° – 80° [87].



Figure 3.5: X-ray diffractometer.

3.4 Photocatalytic studies

Photocatalytic activity of the biosynthesized AgNPs was studied for degradation of methyl red (MR) in presence of UV light irradiation and as a light source 15 watt mercury lamp was used. Before this, about 10 mg of biosynthesized AgNPs was mixed with the MR solution (100 mL, 20 mg/L) and stirred for 30 min to ensure MR equilibrium reaction. After the collaboration, illumination of the UV lamp was exposed for 3 h with continuous stirring in the photocatalytic reactor [88]. According to the Lambert-Beer Law, [89] the absorbance (A) of MR solution was proportional to its concentration (c), which generally followed the following Equation

$$A = \epsilon bc \quad (3.1)$$

Where ϵ is the molar absorption coefficient, b is the thickness of the absorption cell and c is the concentration.

The percentage efficiency of photo degradation of dyes was determined using following equation [90].

$$\% \text{ Dye degradation} = \frac{C_0 - C_t}{C_0} \times 100 \quad (3.2)$$

Where, C_0 and C_t are the solution concentration or absorbance before and after degradation respectively.



Figure 3.6: Photocatalytic reactor.

UV-vis spectrophotometer was used to measure the degradation efficiency of MR at different time intervals. Peaks were observed in the range of 600 to 700 nm which was ascribed to the absorption of the π -system.

3.5 Antibacterial analysis of the synthesized silver NPs.

The antibacterial studies of the silver NPs were investigated utilizing agar well diffusion assay with little modification [86, 92, and 93]. Clinical isolates of gram-negative *Escherichia coli*, *Klebsiella pneumonia* and gram-positive *Staphylococcus aureus* and *Bacillus cereus* were used as the test organisms. The petri plates were prepared using sterile Muller–Hinton agar (MHA). A pure culture (106CFU/mL) was streaked on the sterilized petri plates with a cotton swab and allowed to dry for 15 min, five wells were dug at the surface of the plates with a 6 mm diameter cork borer. The first well contained the positive control (Ciprofloxacin) while the other wells were loaded with 0.05, 0.1, 0.25, 0.5 mg/mL concentration of the AgNPs dispersed in distilled water. The plates were incubated for 24 h at 37 °C for the bacteria organisms. About 10 μ L of 1.25mg/mL 3-(4, 5dimethylthiazol-2-yl)-

2, 5-diphenyltetrazolium bromide (MTT) was introduced to individual plate. So a purple color change was observed. After incubation at 37 °C for 30 min, which showed microbial growth. The sensitivities of the microorganism to the NPs samples were determined by evaluating the sizes of the zones which were inhibited (including the diameter of disk) on the agar surface around the disks, and values < 6 mm were considered as inactive against microorganisms. The experiment was done twice and zones of inhibition were recorded in millimeters. The results of the experiment were given as mean $\pm$ S.D. of the two parallel measurements.

CHAPTER FOUR

RESULTS AND DISCUSSION

4.1 Characterisation of the silver NPs obtained using the aqueous extract of umbrella plant (UmAgNPs)

4.1.1 UV-visible analysis/ surface plasmon bands (SPBs) formation

The UV-vis analysis is fundamental for the confirmation of NPs formation in a typical synthesis involving a reducing agent. Appearance of surface plasmon bands between 400 and

499 nm is characteristic of silver NPs. Three different concentrations of the precursors

(AgNO₃): 1 mM, 2 mM and 5 mM, and two substrates to precursor volume ratios (1:5 and 1:10) were used for the nano-synthesis. The surface plasmon bands gave interesting peaks for each synthesis, and the results are shown in Figures 4.1- 4.6.

The SPBs in the reaction involving 1 mM AgNO₃ (1:5) are narrower than that of the 1 mM AgNO₃ (1:10) ratio with the peaks of the absorption maintained at 410 nm. In the absorption spectra of silver NPs, higher intensity signifies generation of more particles. In the reaction involving the 1:10 ratio, appearance of surface plasmon bands occurred at 433 nm after 30 min and blue shifted to 424 nm which indicates reduction in size of the NPs.

In the synthesis involving 2 mM AgNO₃, the SPBs in both 1:5 and 1:10 ratios showed absorption peaks at 423 nm. The UV-vis results indicate that the same type of reaction seems to occur at the two different substrates to precursor ratios. At the 1:10 ratio, the absorption peak recorded was almost the same as that of the 2 mM (1:10) AgNO₃.

However, using the 5 mM AgNO₃, a delay in the appearance of surface plasmon bands was observed for the 1:5 volume ratio. The SPBs started to appear after 60 min, with the peak of absorption at 430 min which also showed narrowing of peak as the reaction continued. In the reaction involving 1:10 volume ratio, the surface plasmon bands appeared earlier and continued to increase with similar absorption peaks as observed in the 1:5 volume ratio of the same concentration.

The formation of SPBs in all the concentrations used in this study differs from earlier studies by Li *et al.* [93] and Elemike *et al.* [94]. They reported a shift to higher wavelength of the SPBs as the

concentration of the substrate/reducing agent increased shifts. Nevertheless, instances whereby both 1:5 and 1:10 concentration ratios, resulted in the formation of SPBs at the same absorption peak. This shows that different plant substrates behave differently towards the formation of NPs. There are other observations made when this work is compared with some reported studies. For different substrates used, the absorption peak differs. It is obvious that with blue shift in the SPBs formed, a size decrease is expected. However, this phenomenon is only applicable to the substrate under investigation.

For example, studies by Tippawwayat *et al.* [95] showed that at absorption peak around 420 nm for silver NPs mediated by extract of aloe vera plant, the sizes of the NPs ranges from 70 to 192 nm. This does not imply that under the same conditions of synthesis and using another plant extract, as the synthesis mediator, the particle sizes would be in the same size range. There are many factors that affect the particle sizes of the NPs and since different plants contain varying biocomponents and in different proportions which are immobilized in different ways, SPBs, particle sizes with different substrates.

Generally, from the absorption bands obtained in the three different concentrations, the 1 mM preparation of the UmAgNPs seems to give lower particle sizes compared to the other concentrations.

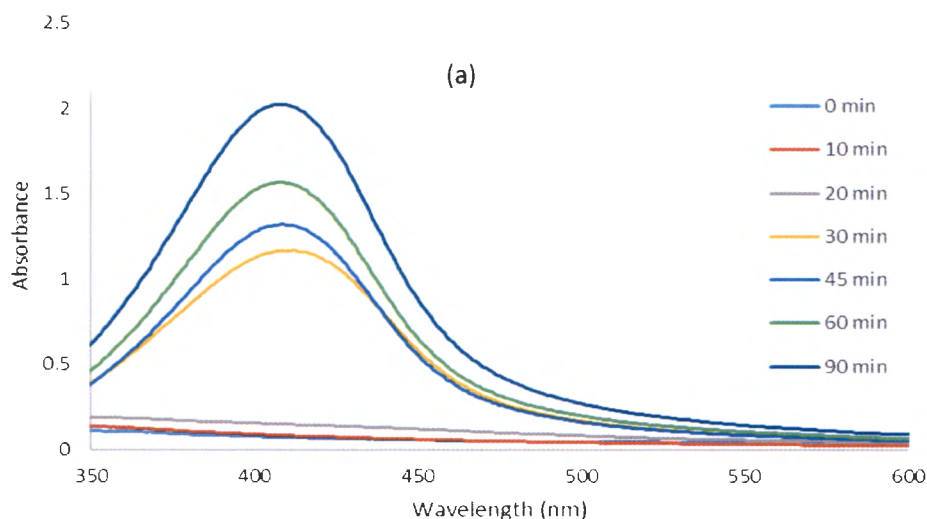


Figure 4.1: UV-visible spectra of the UmAgNps synthesized using 1 mM AgNO_3 and 1:5 substrate to precursor ratio.

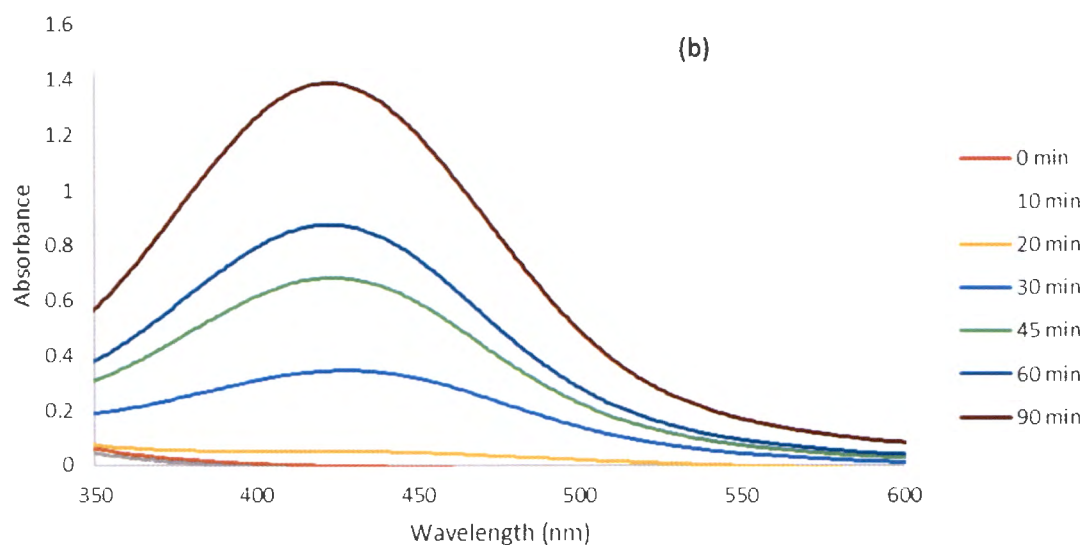


Figure 4.2: UV-visible spectra of the UmAgNps synthesized using 1 mM AgNO_3 and 1:10 substrate to precursor ratio.

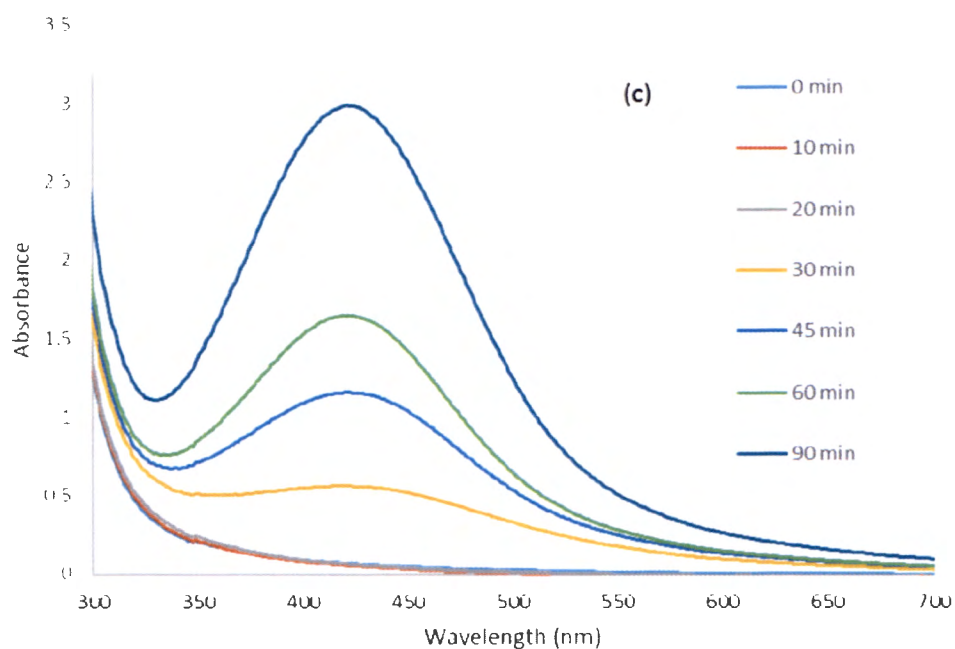
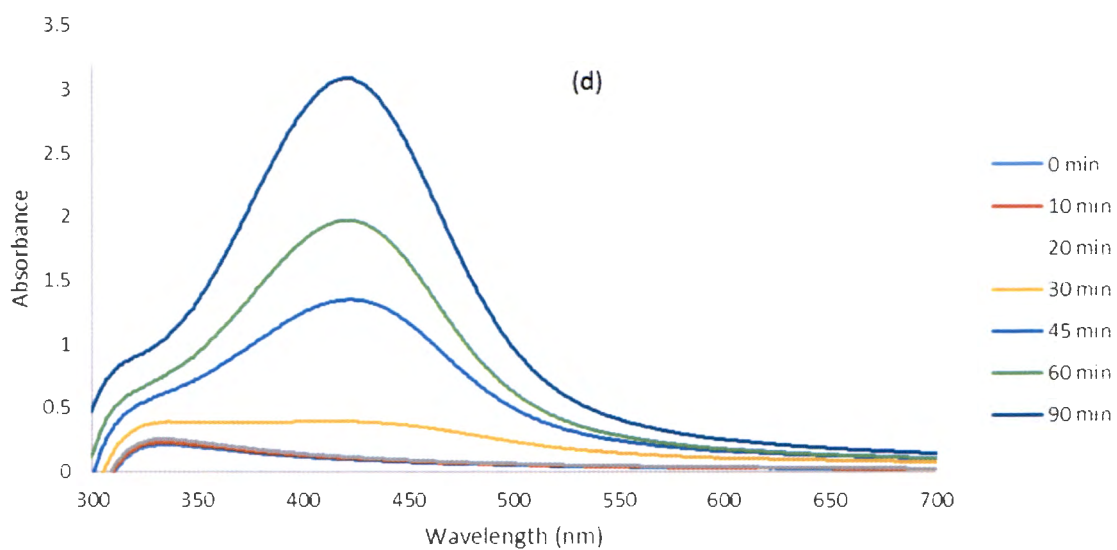


Figure 4.3: UV-visible spectra of the UmAgNps synthesized using 2 mM AgNO_3 and 1:5 substrate to precursor ratio.



to precursor ratio.

Figure 4.4: UV-visible spectra of the UmAgNps synthesized using 2 mM AgNO_3 and 1:10 substrate to precursor ratio.

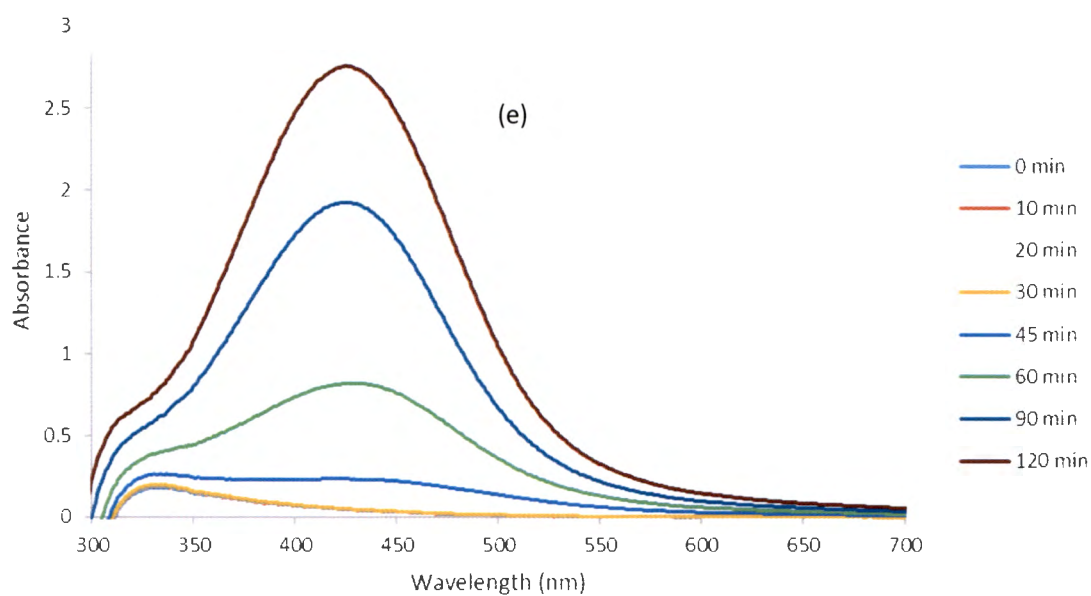


Figure 4.5: UV-visible spectra of the UmAgNps synthesized using 5 mM AgNO_3 and 1:5 substrate to precursor ratio.

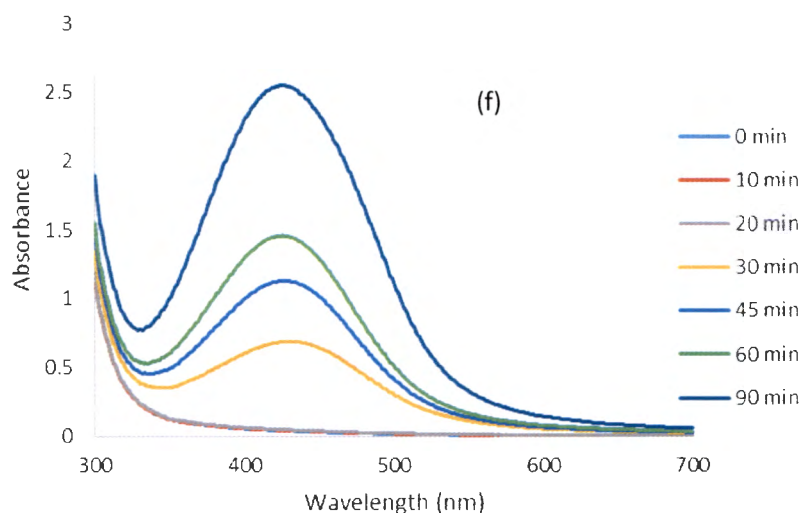


Figure 4.6: UV-visible spectra of the UmAgNps synthesized using 5 mM AgNO_3 and 1:10 substrate to precursor ratio.

4.1.2 Fourier transform infrared (FTIR) analysis of the Umbrella plant and UmAgNPs

FTIR analysis was conducted to probe the surface composition of the NPs and gain insight into the possible plant molecules which are responsible for the reduction and the stability of the NPs. Some molecules can act as reducing, stabilizing and capping agents [96]. All these depend on the constituents of the plant extracts and with such multi properties, efficient and multifunctional NPs could be obtained.

Figures 4.7 and 4.8 show the FTIR spectra of the plant and UmAgNPs respectively. The spectrum of the leaf showed a broad peak around 3271 cm^{-1} which was an indication of the presence of alcohols or amine functional groups. The groups may probably be due to glucose, proteins, flavonoids or tannins metabolites. The intense peaks around 2915 and 2848 may be ascribed to the symmetric and asymmetric C-H stretching vibrations [97]. Peaks around 1730, 1602 cm^{-1} revealed the characteristic C=O vibration which could be due to acids, esters or amides. The band at 1025 cm^{-1} could be due to the vibrations resulting from the C-N or C-O groups. The major peaks recorded are shown in Table 4.1, and these biomolecules indicate the possible components of the plant responsible for the reduction of Ag ions to Ag.

There was no clear distinction between the spectrum of the synthesized UmAgNPs and that of the plant because the plant materials were used as both reducing agent and capping molecules for AgNPs. This is a confirmation that there was an effective capping of the synthesized silver nanoparticles by the extracts of the plant material. The only major difference noticed is that the bands of the functional groups in the spectrum of the plant are more intense and pronounced than that of the UmAgNPs. The vibrations of the UmAgNPs were also noticed at a slightly higher wavelength than the plant material. A reduction in the degree of the hydrogen bonding responsible for the broad nature of the OH/N-H band is an indication of the coordination between the plant molecules and the silver NPs through the coordinating atoms (O or N).

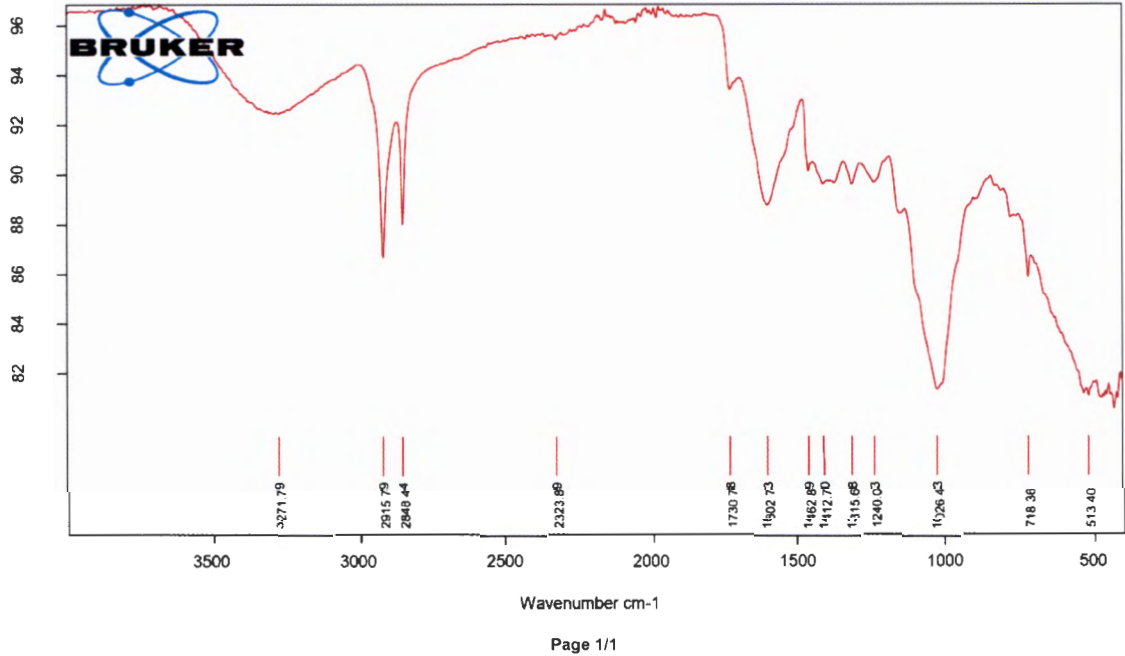


Figure 4.7: FTIR spectrum of the Umbrella leaf material.

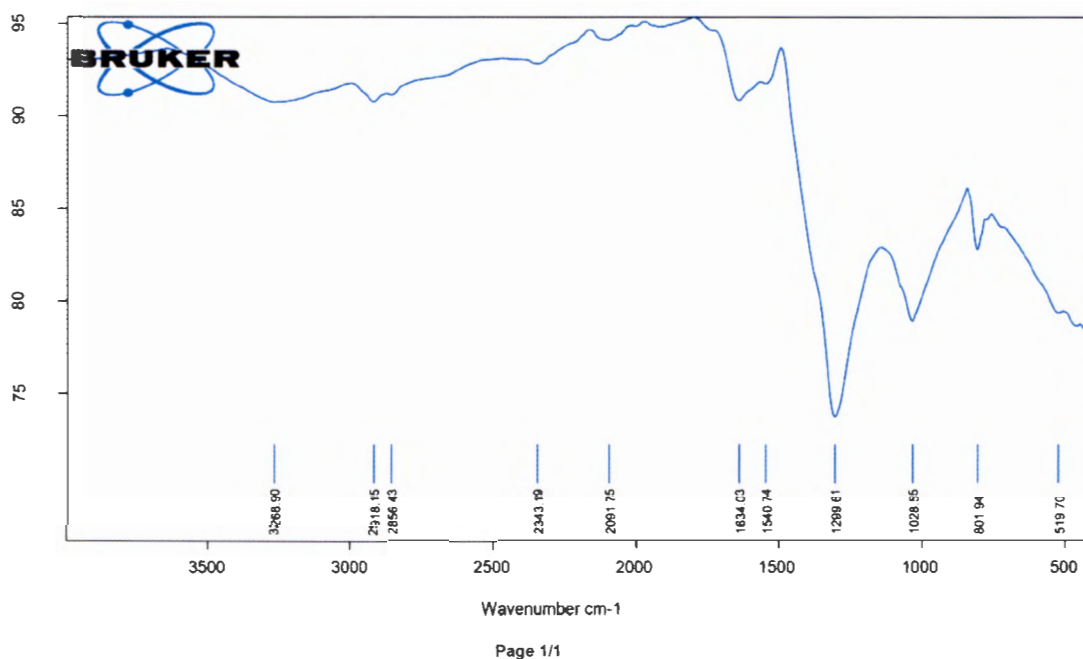


Figure 4.8: FTIR spectrum of the UmAgNPs.

Table 4.1: Possible functional groups present in the umbrella plant and NPs

Functional group	Wavenumber (cm ⁻¹)		Intensity
	Plant material	UmAgNPs	
O-H	3271	3288	strong
C-H	2915	2918	strong
=C-H	2848	2856	medium
C=O	1730,1602	1634	strong
C-O C-N	1026	1028	strong
C-Cl stretch	718	801	Strong

4.1.3 Transmission electron microscopic (TEM) analysis

Transmission electron microscopy (TEM) gave clear information on the various sizes and morphologies of the NPs. It also provides supporting and confirmatory results on the data obtained from the UV-visible spectroscopy. Image J software was used to determine the average sizes of the NPs and the corresponding particle size distributions are displayed in a histogram. The TEM images,

presented in Figures 4.9- 4.14, show particles with predominantly spherical morphology and size variations recorded due to difference in concentrations of the precursor. In the TEM images of the 1 mM of AgNO₃ using 1:5 ratio, the sizes are in the range 3.01 to 14.75 nm with the mean size of about 6.88 nm, whereas the same concentration of AgNO₃ and at 1:10 ratio gave average size of 5.16 nm and size range of 1.65-8.41 nm. Figure 4.11 presents the TEM micrograph for 2 mM AgNO₃ at 1:5 ratio. The figure evidenced some degree of polydispersity, with an increase in the formation of larger NPs, a situation which may be due to Oswald ripening process. For the 2 mM AgNO₃, both the 1:5 and 1:10 volume ratio gave comparable average nanoparticle sizes of 8.13 and 8.01 nm respectively. The TEM images also show more concentrations (with high intensity of smaller particles) of NPs in the 2 mM AgNO₃ than in the 1 mM AgNO₃ (Figure 4.9 and 4.10). In the 5 mM concentration, the average size recorded in the 1:5 volume ratio is 15.48 nm, whereas an average particle size of 4.13 nm was recorded in the 1:10 ratio which reflects a very huge difference in particle size. There was a remarkable observation in the 1:5 ratio whereby a polygonal-shaped nanoparticle was obtained. This indicates an aggregation of the smaller particles to form the big particle, a mechanism of possible Oswald ripening as also observed in the 2 mM AgNO₃ with 1:5 volume ratio. Therefore, it could be inferred that at high substrate concentration (1:5) and increased concentration of the precursor (2 mM to 5 mM), an Oswald ripening process of nanoparticulate formation system tends to set in. The least particle size obtained using this plant extract is in the (1:10 ratio) for 5 mM concentration which gave interestingly monodispersed NPs.

In general, increasing the AgNO₃ concentration from 1 to 5 mM for the 1:5 ratio, results in a gradual increase in size which means that the rate of nuclei formation decreases considerably compared to the rate of growth of silver NPs [98]. In the case of 1:10 ratio, similar situation was observed from 1 to 2 mM but from 2 to 5 mM, there was an observed surface activation of the NPs which somewhat supports digestive ripening of larger particles to give smaller particles.

Table 4.2: Size distribution at different precursor and substrate concentrations of the synthesized UmAgNPs

Concentration of precursor: AgNO ₃ (mM)	Volume ratio of substrate to precursor	Average size of AgNPs (nm)	Size Range (nm)
1	1:5	6.88	3.01-14.75

	1:10	5.16	1.65-8.41
2	1:5	8.13	4.38-11.92
	1:10	8.01	5.17-14.68
5	1:5	15.48	5.11-92.61
	1:10	4.13	1.89 -7.62

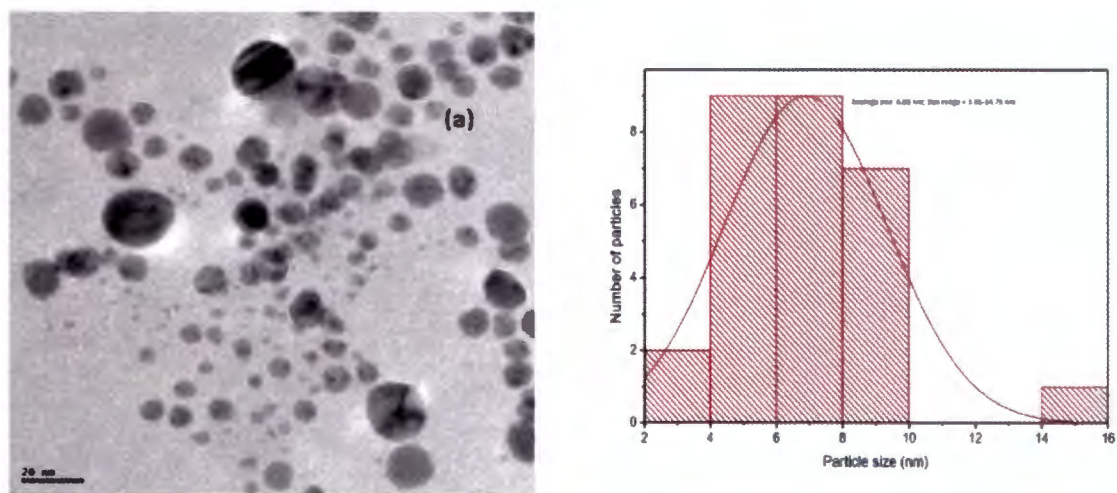


Figure 4.9: TEM image and particle size distribution histogram of the NPs obtained using 1 mM AgNO₃ and 1:5 volume ratio of aqueous plant extract: AgNO₃

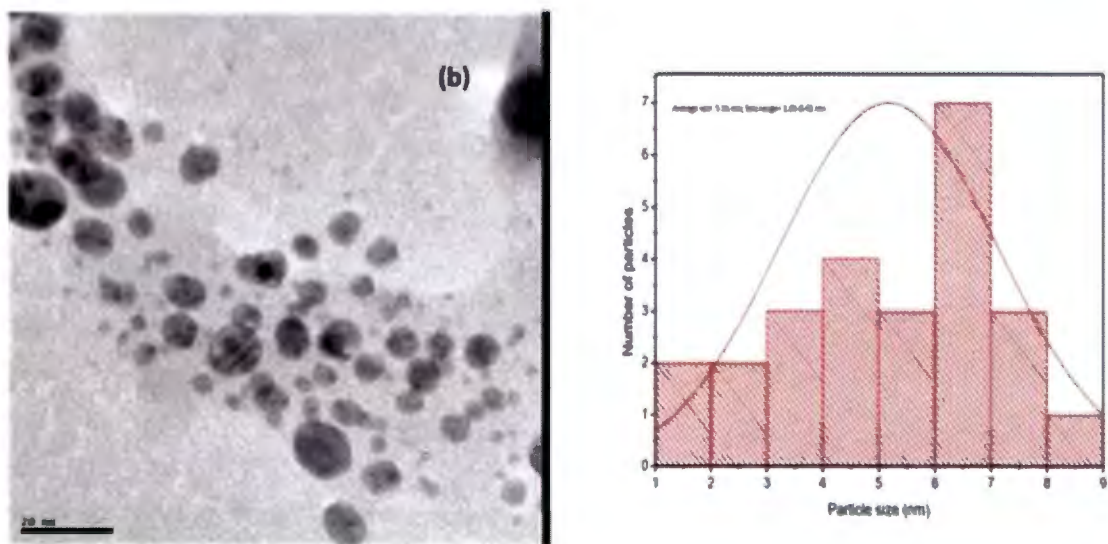


Figure 4.10: TEM image and particle size distribution histogram of the NPs obtained using 1 mM AgNO_3 and 1:10 volume ratio of aqueous plant extract: AgNO_3 .

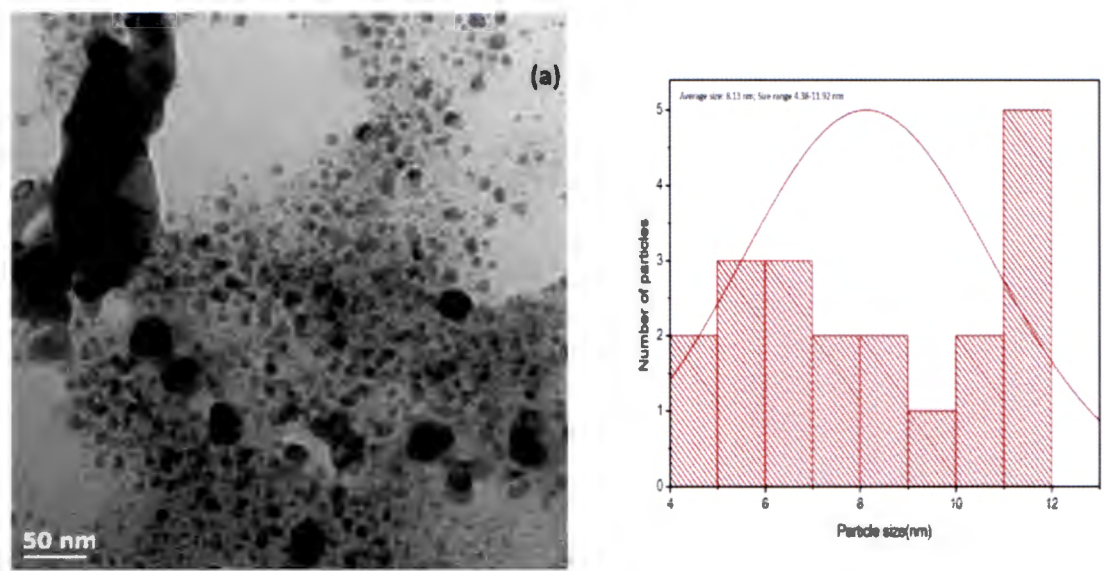


Figure 4.11: TEM image and particle size distribution histogram of the NPs obtained using 2 mM AgNO_3 and 1:5 volume ratio of aqueous plant extract: AgNO_3

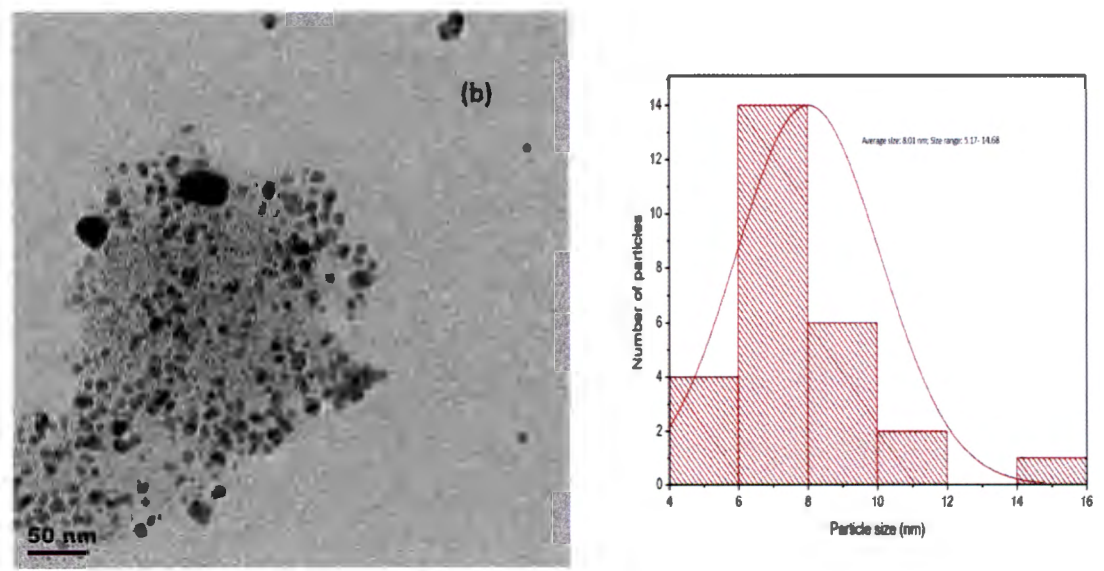


Figure 4.12: TEM image and particle size distribution histogram of the NPs obtained using 2 mM AgNO₃ and 1:10 volume ratio of aqueous plant extract: AgNO₃

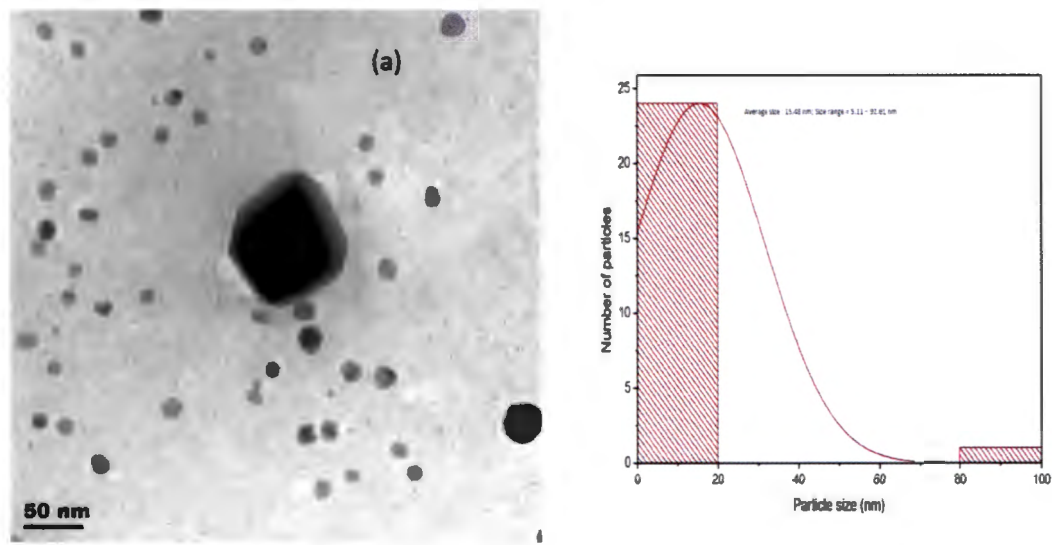


Figure 4.13: TEM image and particle size distribution histogram of the NPs obtained using 5 mM AgNO₃ and 1:5 volume ratio of aqueous plant extract: AgNO₃

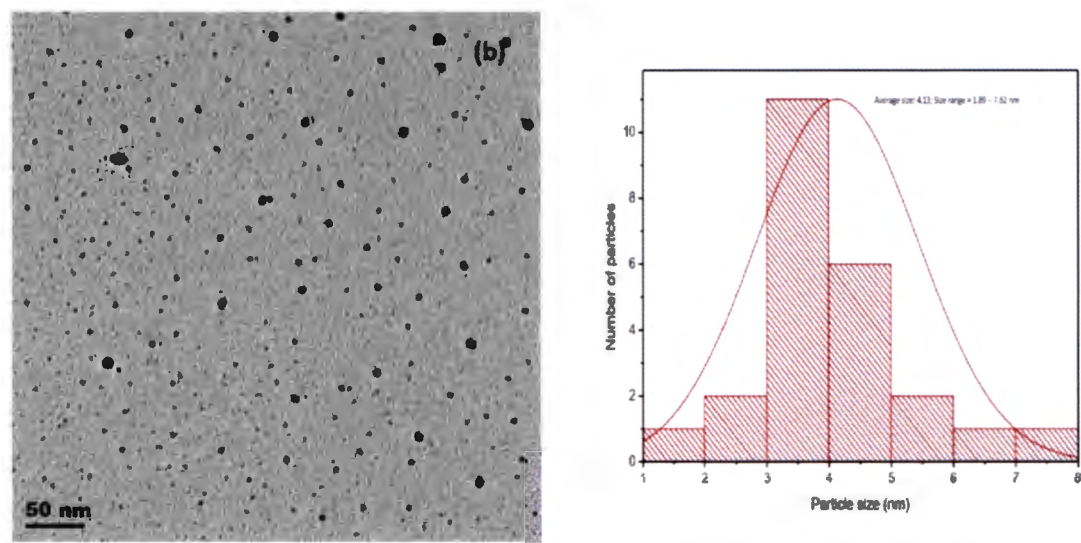


Figure 4.14: TEM image and particle size distribution histogram of the NPs obtained using 5 mM AgNO₃ and 1:10 volume ratio of aqueous plant extract: AgNO₃

4.1.4 Powder X-ray diffraction analysis

The powder XRD measurement was used to determine the crystalline size. The diffraction pattern presented in Figure 4.15 showed some reflections at 2θ values of 37.62, 44.63, and 54.25 which correspond to the (111), (200) and (220) planes respectively, and indicate Ag with face centered cubic structure. There are other peaks that appeared in the diffractogram which may be due to little traces of Ag_2O , AgCl and some biomolecular compounds involved in the synthesis. However, the stronger planes, as highlighted earlier, indicate silver as the predominant constituent in the biosynthesis [99].

Other elements such as oxygen, chlorine that may have led to formation of the AgCl , Ag_2O NPs could be from the proteins/enzymes or other constituents in the plant extract. These elements attest to the fact that the substrate act as reducing agents in addition to its role as stabilizer for the NPs [100].

The average crystalline size calculated using the most intense peaks of representative XRD for 1 mM AgNO_3 for both 1:5 and 1:10 volume ratio by applying Debye–Scherrer equation ($D =$

$K\lambda/\beta\cos\theta$). Where D is the average crystalline value, λ = wavelength of the radiation (0.154nm), $k=0.9$, $\beta=3.14 \times \text{FWHM}/180$, FWHM is full width of half maximum); gave similar values to the results obtained using TEM: x nm and y nm.

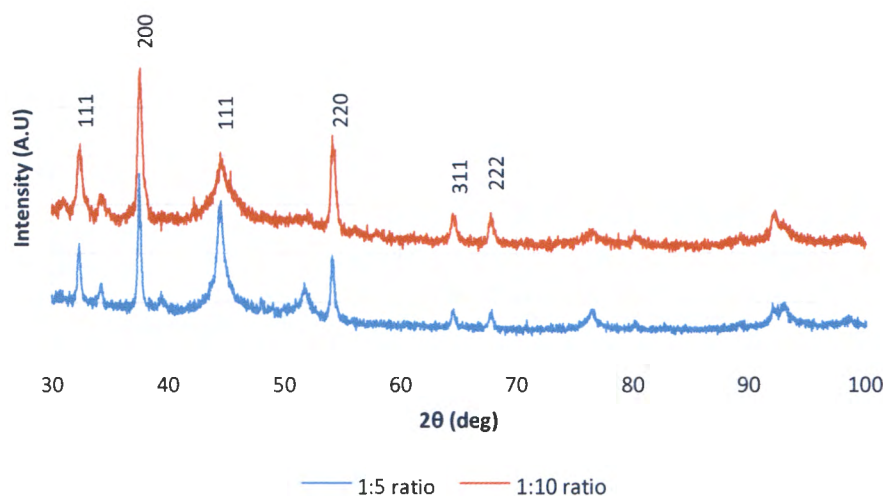


Figure 4.15: pXRD pattern of the NPs (UmAgNPs) obtained using 1 mM AgNO_3 .

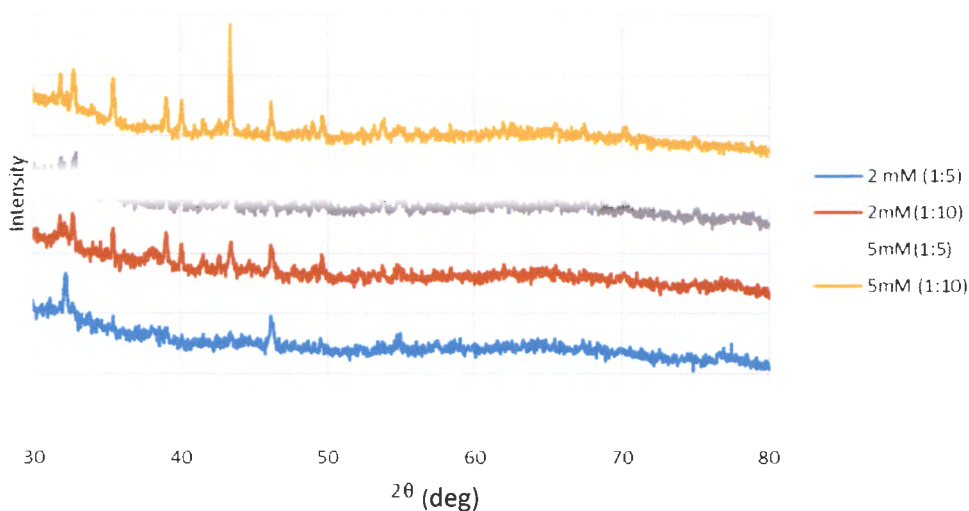


Figure 4.16: pXRD pattern of the NPs (UmAgNPs) obtained using 2 and 5 mM AgNO_3 .

4.1.5 Photocatalytic degradation studies of methyl red solution using the UmAgNPs

The degradation of dyes using biosynthesized NPs has attracted research interest in recent times because the photocatalysts are environmentally friendly.

Dyes such as methyl-red are used in the industries, and they contribute to environmental pollution when discharged as effluents. The synthesized silver NPs using umbrella plant were investigated for their photocatalytic properties in the degradation of methyl red. A photocatalytic reactor fabricated at North-West University was used for the study. The degradation reaction was monitored for 2 h using the NPs obtained from 1 and 5 mM AgNO_3 (1:5) and the reduction in peak intensity (435 nm) was recorded. Figures 4.17 and 4.18 show the degradation reaction with time while Table 4.3 gives the percentage degradation at each time of sampling. The results show that the UmAgNPs obtained from 1 mM AgNO_3 recorded better efficiency than the particles obtained from 5 mM AgNO_3 . This could be due to the relatively smaller particle size.

Generally, the degradation efficiency was poor as the maximum recorded at 120 min was 29.73%.

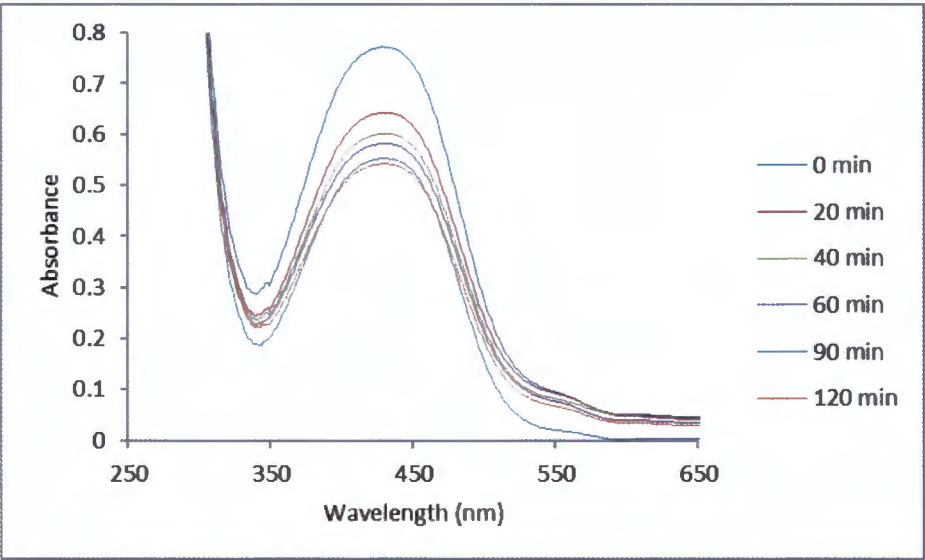


Figure: 4.17 Degradation of methyl red using UmAgNPs obtained from 1 mM AgNO_3 with 1:5 volume ratio of aqueous leaf extract: AgNO_3 .

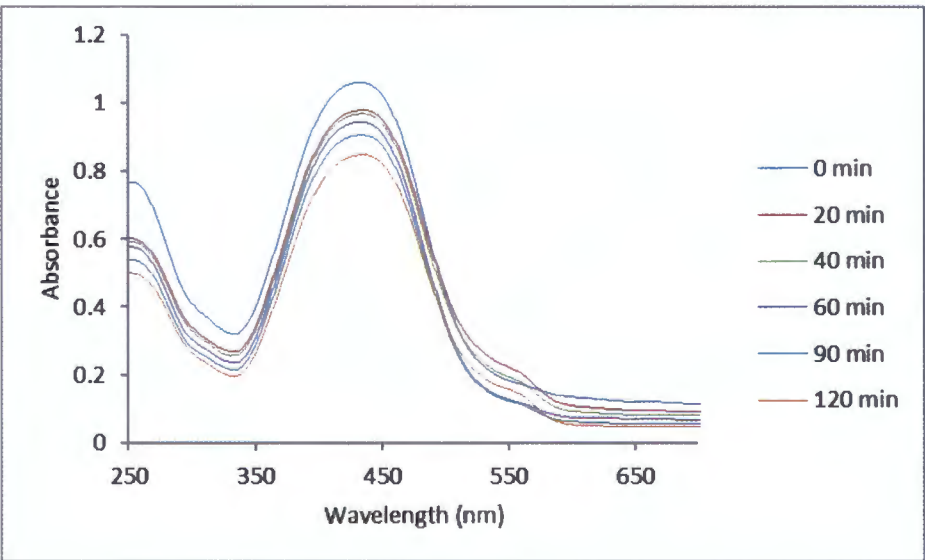


Figure 4.18: Degradation of methyl red using UmAgNPs obtained from 5 mM AgNO_3 with 1:5 volume ratio of aqueous leaf extract: AgNO_3

Table 4.3: Percentage degradation of methyl red using UmAgNPs

Time (min)	1 mM AgNO ₃ (1:5)		5 mM AgNO ₃ (1:5)	
	Absorbance	% degradation	Absorbance	% degradation
0	0.7686	-	1.0591	-
20	0.6407	16.64	0.9681	8.59
40	0.5977	22.23	0.9667	8.72
60	0.5783	24.76	0.9437	10.89
90	0.5410	29.61	0.9049	14.56
120	0.5401	29.73	0.8496	19.78

4.2 Characterisation of the silver NPs obtained using the aqueous extract of Welsh onion plant (WoAgNPs)

4.2.1 UV-vis spectroscopy

The bioreduction of silver nitrate to silver NPs has been made possible by the reducing agents contained in the plant extracts. The initial colour change followed by the appearance of surface plasmon bands suggested the formation of Ag NPs. In the appearance of surface plasmon bands (SPBs), different characteristics were recorded for the different concentrations (1 mM, 2 mM, and 5 mM) of the precursor used. In this synthesis, the concentrations of the metal salt (AgNO₃) were varied and the temperature of reaction was maintained in order to obtain varying sizes of AgNPs as was described in the synthesis of UmAgNPs. Using the solution from 1 mM AgNO₃ (1:5 ratio), the appearance of SPBs occurred after 60 min (figure 4.19) of the reaction. In the 1:10 precursor to plant extract solution volume ratio, SPBs was recorded after the 90th min as shown in figure 4.20. In figure 4.21 and 4.22, which are for 2 mM at 1:5 and 1:10 respectively, formation of AgNPs were evidenced by the SPBs after 60 min and 45 min with corresponding wavelengths of 418 and 412 nm. For the 5 mM concentration of AgNO₃, both ratios of 1:5 and 1:10 had their initial SPBs after 45 min, with wavelengths of 423 and 427 nm respectively. In all the three different concentrations, the 2

mM sample showed more uniform SPBs with narrower peaks that suggest more spherical NPs without aggregation. The 1:5 ratio from the 5 mM

AgNO_3 has the highest intensity of AgNPs at 90 min of 1.62 compared to the others that ranged between 0.29-1.33. Among the three concentrations, the highest wavelength of 427 nm was observed at the 1:10 (5 mM) which suggests larger NPs. Moreover the highest intensity observed in the 1:5 (5 mM) may be due to the availability of more silver ions that were reduced to NPs (NPs). The nature of SPBs observed in the 2 mM shows monodispersity of the NPs. In all the concentrations studied, the particles obtained from 2 mM AgNO_3 and at 1:10 ratio seems to have the smallest particle size. Generally, the samples from 1:10 ratios of the different molar concentrations tend to have lower wavelengths than the samples from 1:5 ratios. The UV-vis spectra of the NPs obtained using 2 mM AgNO_3 seem to be more uniform and sharper than of the particles obtained using 1 and 5 mM AgNO_3 . This observation is contrary to some earlier studies which report that as absorption peaks get sharper and tends to be blue-shifted as the concentration of the AgNO_3 is increased [101].

Common in the peak of all the SPBs is the fact that more NPs are formed when 1:5 ratios were used in all the precursor molar concentrations. This implies that there were insufficient biomolecules to act as reducing agents when 1:10 ratios was used.

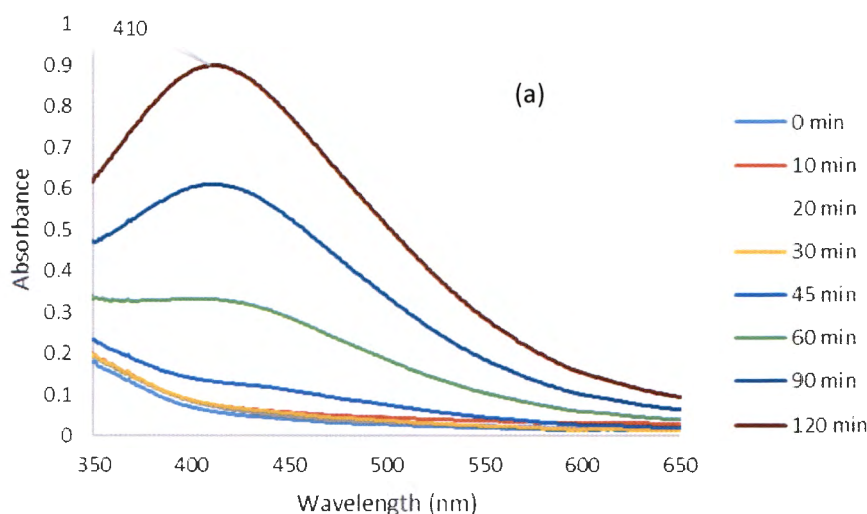


Figure 4.19: UV-visible spectra of the WoAgNps synthesized using 1 mM AgNO_3 and 1:5 substrate to precursor ratio.

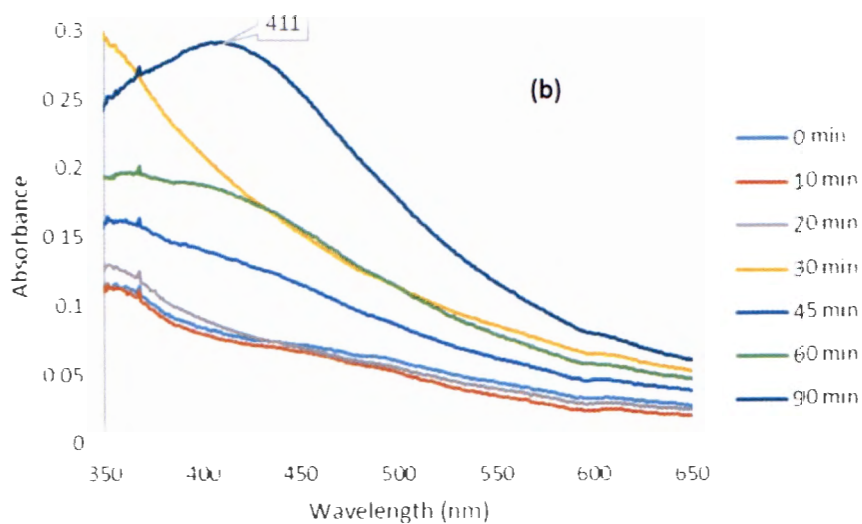


Figure 4.20: UV-visible spectra of the WoAgNps synthesized using 1 mM AgNO_3 and 1:10 substrate to precursor ratio.

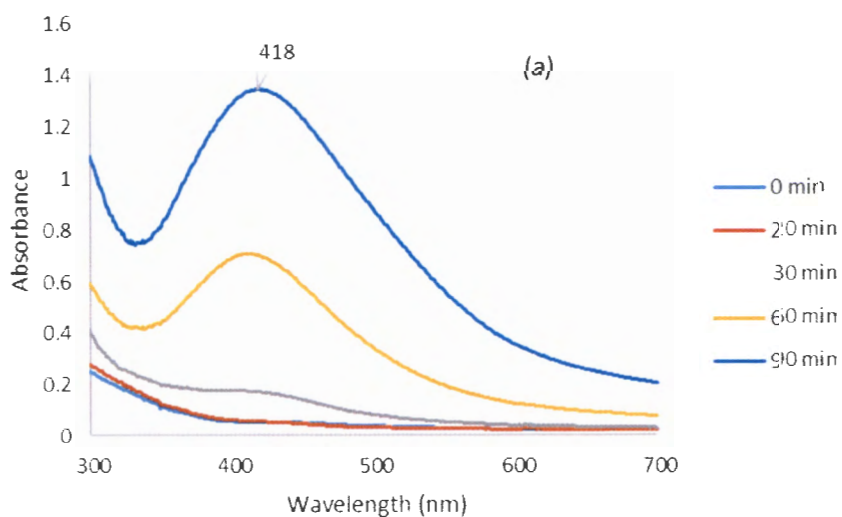


Figure 4.21: UV-visible spectra of the WoAgNps synthesized using 2 mM AgNO_3 and 1:5 substrate to precursor ratio.

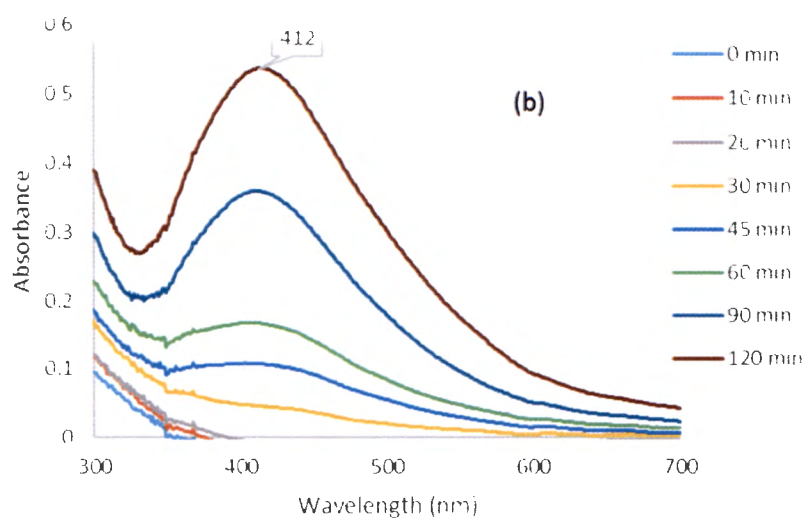


Figure 4.22: UV-visible spectra of the WoAgNps synthesized using 2 mM AgNO_3 and 1:10 substrate to precursor ratio.

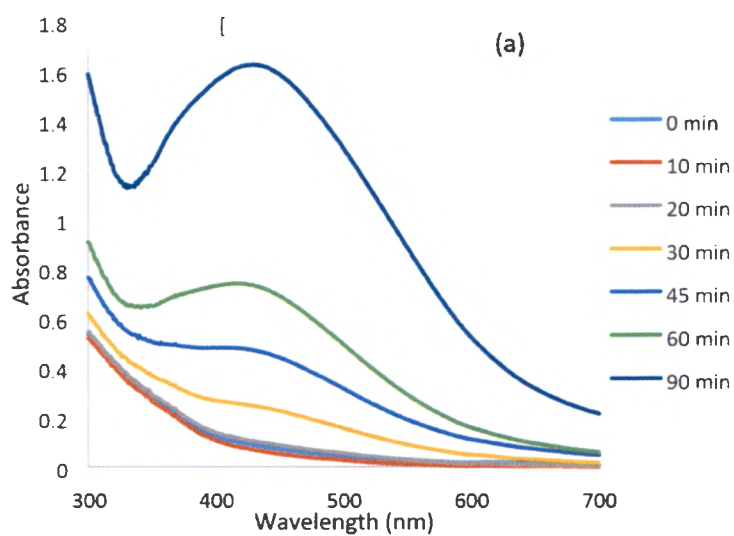


Figure 4.23: UV-visible spectra of the WoAgNps synthesized using 5 mM AgNO_3 and 1:5 substrate to precursor ratio.

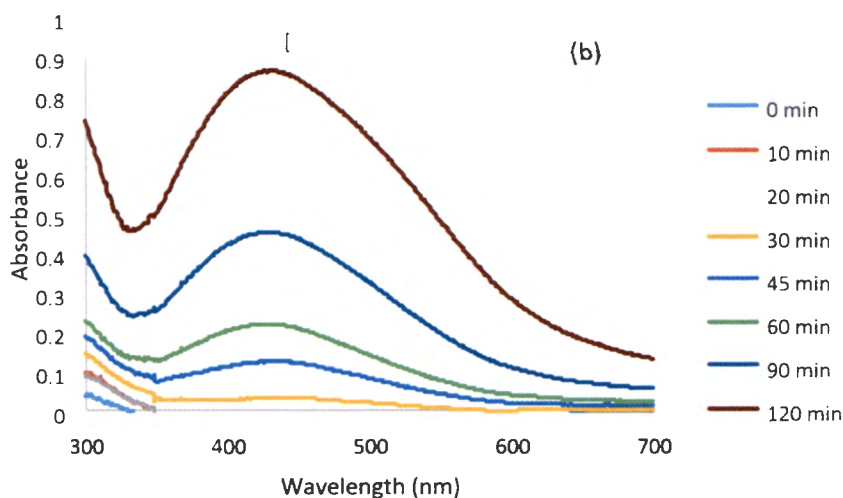


Figure 4.24: UV-visible spectra of the WoAgNPs synthesized using 5 mM AgNO₃ and 1:10 substrate to precursor ratio.

4.2.2 Fourier transform infrared (FTIR) analysis of the Welsh onion plant and WoAgNPs

The fourier transform infrared spectrum of the AgNPs from *Allium fistulosum* plant extract showed some prominent peaks, and the vibrational frequency responsible for these peaks were assigned based on literature reports. The broad peak around 3268 cm⁻¹ corresponds to the OH stretching of alcohol or phenol groups. The sharp peaks at 2918 cm⁻¹ and 2853cm⁻¹ may be attributed to the C-H stretching of alkanes [102]. The peak at 2101 cm⁻¹ indicates the presence of strong C≡C bonds while peaks at 1729 cm⁻¹ could be due to the presence of C=O characteristic of ketone group. Sharp peak around 1603 cm⁻¹ is indicative of carbonyl N-H stretch of primary amines due to vibrations in amide linkages or carbonyl stretch of protein [97], while peaks found in the range 1238-1292 cm⁻¹ could be due to the CO bond of carboxylic acid group [103]. The strong peaks at 1013 cm⁻¹ shows bending vibration of C-O or C-N group, while peaks at 803, 519 cm⁻¹ indicate the presence of C-Cl or C-Br group [104].

The spectrum of the WoAgNPs also displayed peaks similar to the values observed in the plant. The similarities between both spectra with some marginal shifts in peak position clearly indicate the presence of the plant extract in the sample as a capping agent to the silver NPs [105]. Table 4.4 presents a summary of the functional groups present in the welsh onion plant as well as in the NPs.

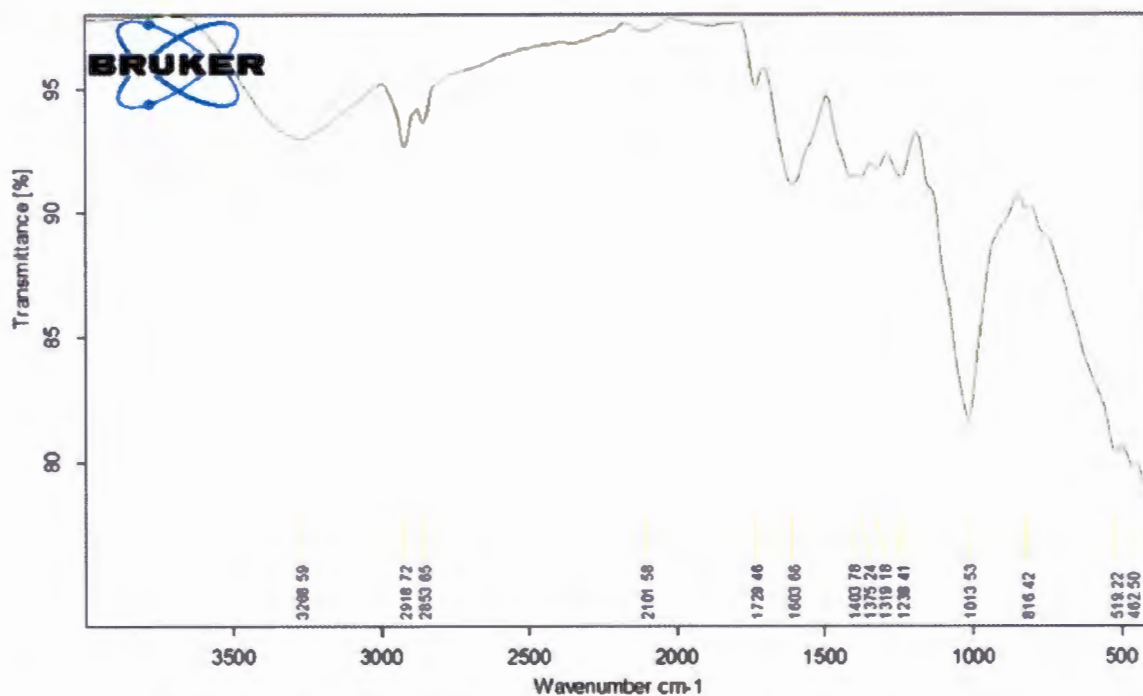


Figure 4.25: FTIR spectrum of the Welsh onion leaf material.

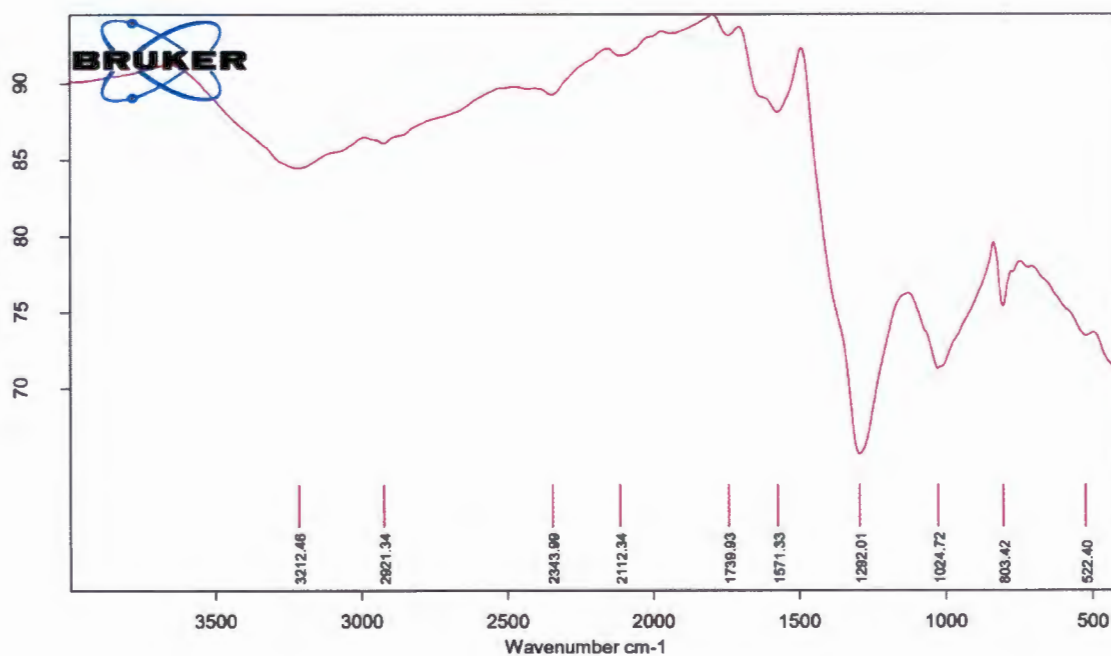


Figure 4.26: FTIR spectrum of the WoAgNPs.

Table 4.4: Functional groups present in the welsh onion plant and NPs

Functional groups	Frequency cm^{-1}		Intensity
	Welsh onion plant	WoAgNPs	
OH	3212	3268	strong, broad
C-H	2918	2921	strong
C $\equiv$ C	2101	2112	variable
C=O	1729	1739	strong
C=O	1571	1603	strong
C-O	1238	1292	strong
C-O,C-N	1013	1024	strong
C-Cl	803	816	strong

4.2.3 Transmission electron microscopic analysis of WoAgNPs

The TEM images revealed larger sizes of NPs for the synthesis involving 1:10 ratio for both the 1 and 5 mM AgNO₃ except for the 2 mM precursor concentration. The 2 mM AgNO₃ concentrations gave the lowest particle sizes of 8.03 and 3.74 nm for the synthesis involving 1:5 and 1:10 volume ratios respectively. In the reaction involving 1 mM AgNO₃ (1:10), a wide range of size between 1.26- 78.39 nm appeared. The largest size NPs observed here appear as a nanoblock and smaller NPs tend to grow on the existing larger particles. This could be as a result of weak surface interaction which initiated growth aggregation of smaller particles to form larger ones [106]. The 1 mM AgNO₃ concentration displayed quite a different behaviour from the other concentrations by the fact that agglomeration was greatly noticed in both 1:5 and 1:10 ratios. In general, there are observed agglomeration in the particles synthesized using 1 and 5 mM but monodispersed NPs in the 2 mM AgNO₃ concentration. The mean sizes of the different concentrations are shown in Table 4.5. Although it is difficult to obtain uniformly sized NPs using plant extracts, the images obtained using 2 mM concentration show nearly uniform one. This thus, suggest that 2mm concentration is very promising towards the synthesis of same size NPs.

Table 4.5: Size distribution at different precursor and substrate concentrations of the synthesized WoAgNPs

Concentration of Precursor(AgNO_3) (mM)	Ratio of Substrate to precursor	Average size of AgNPs (nm)	Size Range (nm)
1	1:5	15.72	6.20-41.97
	1:10	13.39	1.26-78.39
2	1:5	8.03	3.05-12.59
	1:10	3.74	1.89-9.47
5	1:5	10.29	5.42-18.81
	1:10	14.22	6.32 -24.96

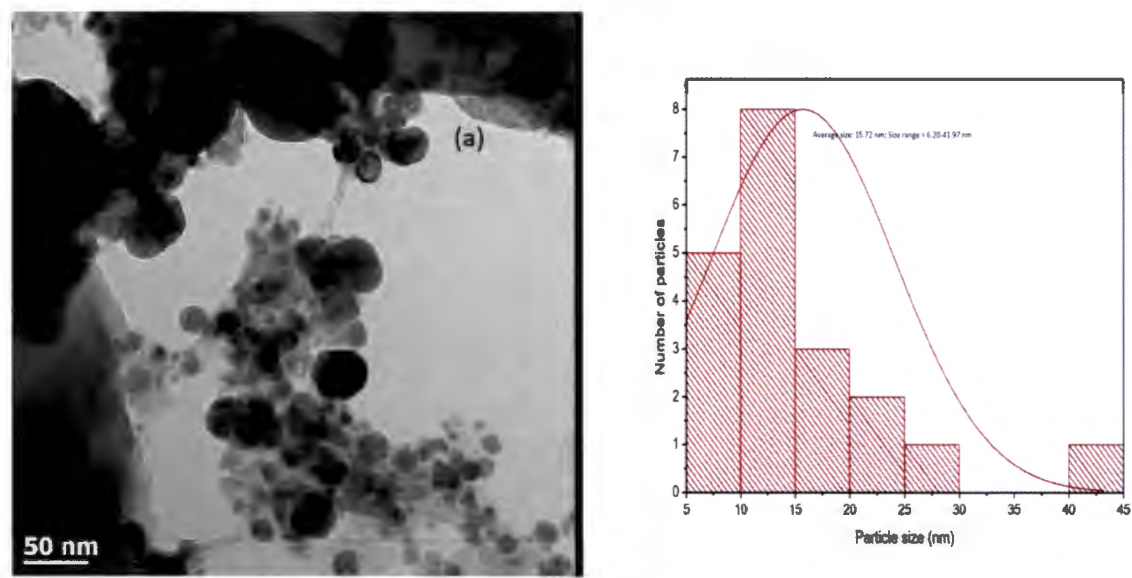


Figure 4.27: TEM image and particle size distribution histogram of the NPs obtained using 1 mM AgNO_3 and 1:5 volume ratio of aqueous plant extract: AgNO_3

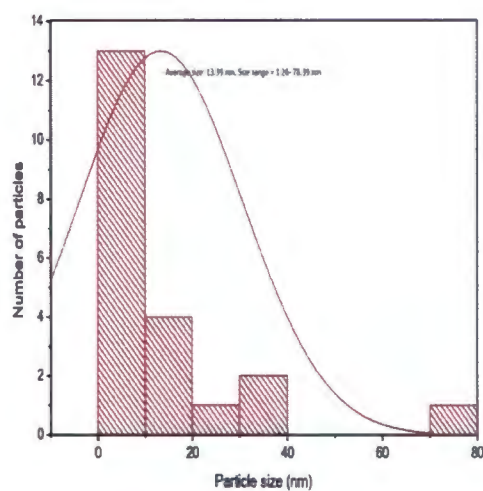
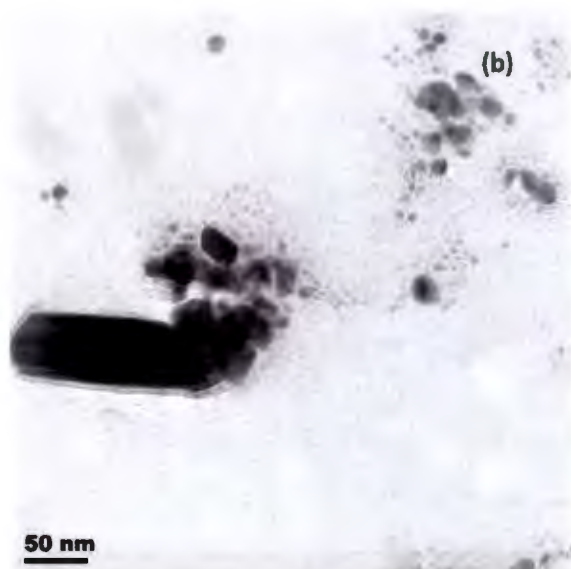


Figure 4.28: TEM image and particle size distribution histogram of the NPs obtained using 1 mM AgNO_3 and 1:10 volume ratio of aqueous plant extract: AgNO_3

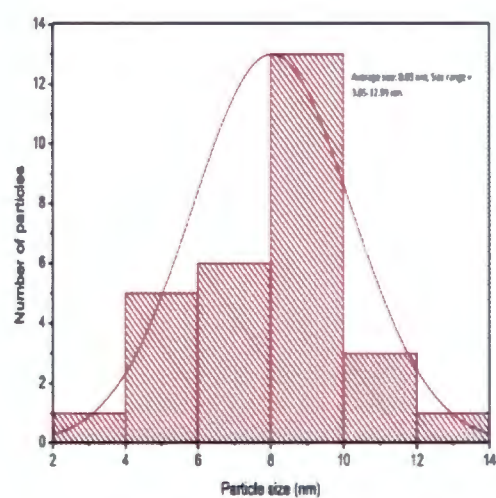
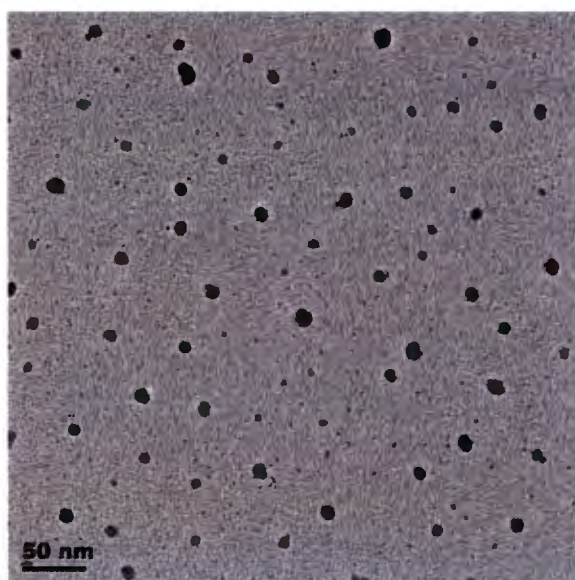


Figure 4.29: TEM image and particle size distribution histogram of the NPs obtained using 2 mM AgNO_3 and 1:5 volume ratio of aqueous plant extract: AgNO_3

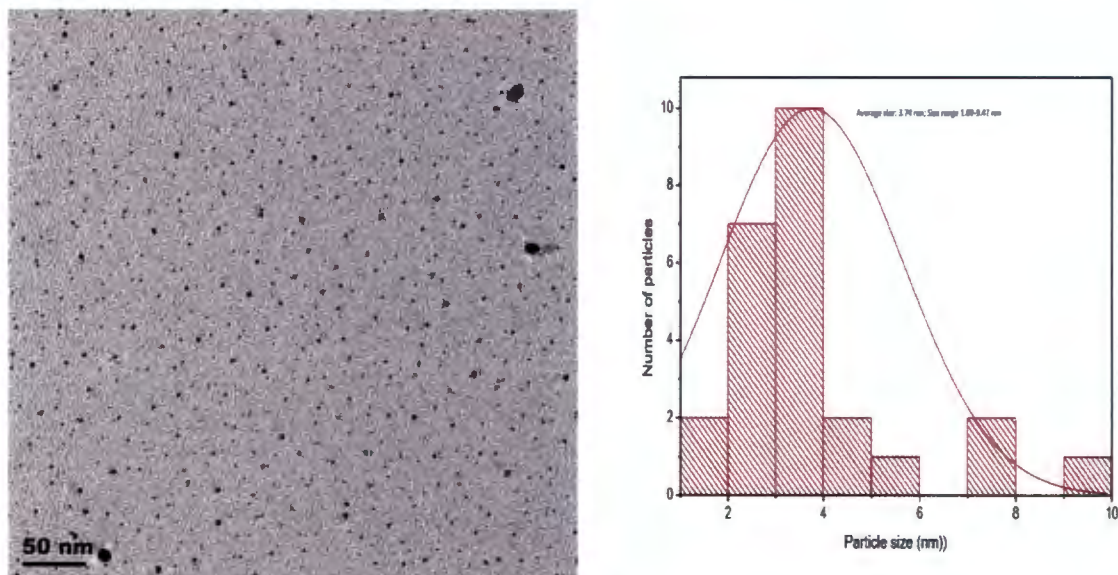


Figure 4.30: TEM image and particle size distribution histogram of the NPs obtained using 2 mM AgNO_3 and 1:10 volume ratio of aqueous plant extract: AgNO_3 .

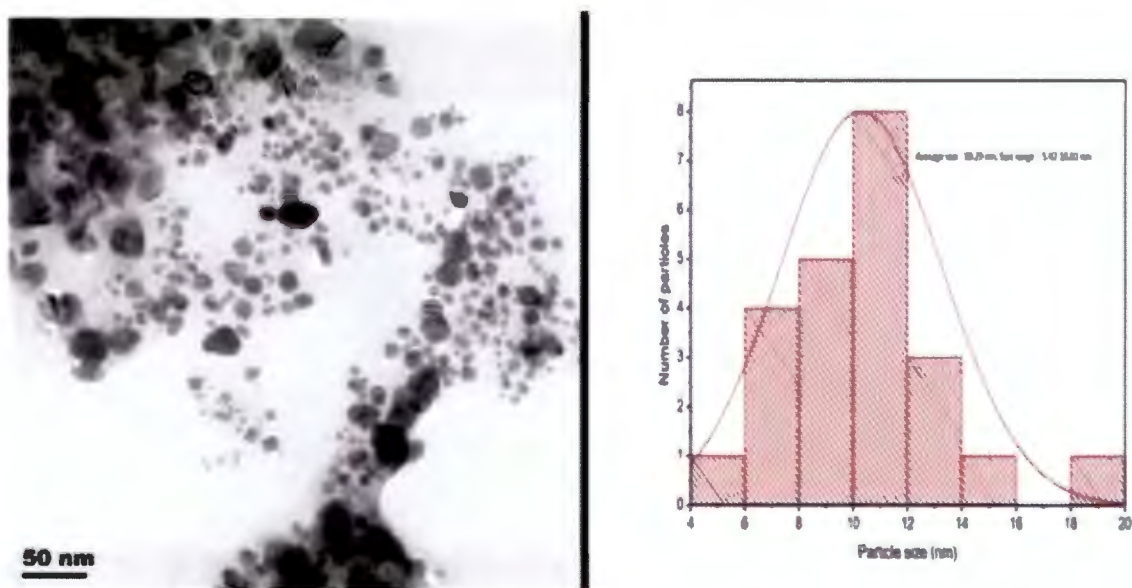


Figure 4.31: TEM image and particle size distribution histogram of the NPs obtained using 5 mM AgNO_3 and 1:5 volume ratio of aqueous plant extract: AgNO_3

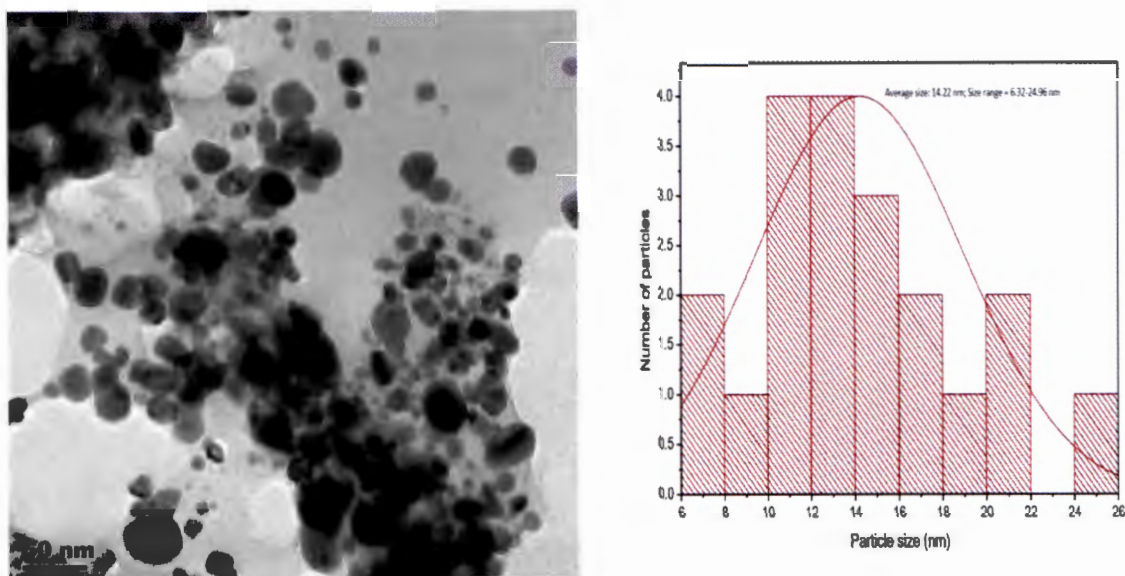


Figure 4.32: TEM image and particle size distribution histogram of the NPs obtained using 5 mM AgNO₃ and 1:10 volume ratio of AgNO₃: aqueous plant extract.

4.2.4 Powder X-ray diffraction (pXRD) studies

The PXRD patterns revealed the crystalline nature of silver and silver chloride NPs mediated by Welsh onions and the pattern is shown in Figure 4.33.

The diffraction pattern showed peaks which correspond to both pure silver and traces of silver chloride. The diffraction features for pure silver which occurred at 39.08, 64.39 and 76.50 can be assigned to the 111, 220 and 311 reflections characteristic of face centred cubic structures. However, the peaks at 32.73, 46.14, 53.86, 57.50 and 67.61 represents Bragg reflections of 200, 220, 311, 222 and 400 for cubic structured AgCl which can be compared to the published standard silver chloride peaks [107].

The width of peaks clearly shows that the Ag/AgCl crystallites are of very small sizes. There are some other peaks in the XRD pattern which could be as a result of precipitation of some bio-materials which appeared as organic phase.

In addition, with increasing concentration of the precursor compounds, more peaks seems to appear; some could be assigned to Ag₂O while others may be due to incomplete reduction of the nitrate compounds. In most reports, silver NPs have been reported instead of

Ag/AgCl NPs. The reaction conditions, substrate type and other factors immensely affect the biotransformation of Ag^+ ions to AgCl. Generally, the biological extracts contain different compounds and ions which upon reaction with precursor compounds may give rise to different NPs with diverse shapes. In this report, the Welsh onion produced a mixture of spherical Ag/AgCl NPs with interesting smaller sizes. There are other reports of AgCl NPs with different shapes which result from various reaction conditions.

Kumar *et al.* [108] reported spherical AgCl NPs of diameter between 10-30 nm which were determined by the Beta vulgaris peel extract used as stabilizing and capping agent.

AgCl NPs have been prepared and the reported with size was around 18–42 nm which are quite larger compared to the particles reported here [109].

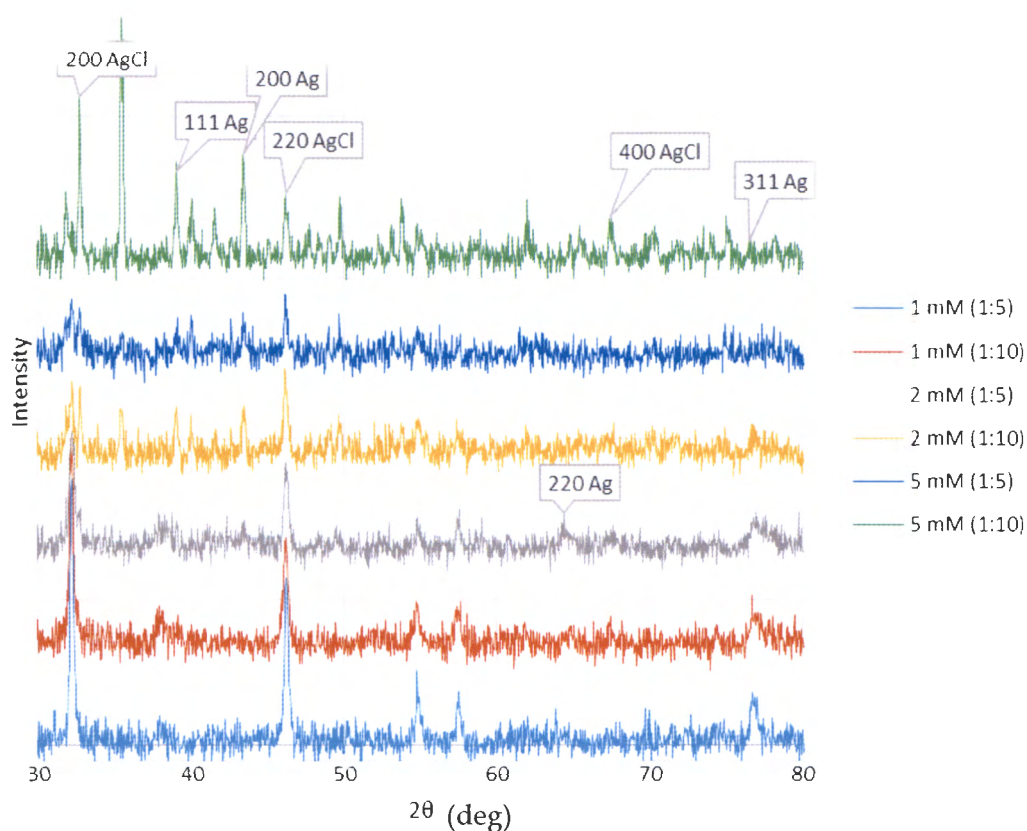


Figure 4.33: pXRD patterns of the WoAgNPs.

4.2.5: Photocatalytic activity of the synthesized WoAgNPs

Photocatalytic, as observed in Ag NPs, is due to the migration of charges when the surface is irradiated leading to interfacial transfer between the metal and dielectric medium [110]. The photocatalytic behaviour of the NPs was monitored at different intervals using a photocatalytic reactor. The degradation of the organic dye (methyl red) was followed at 438 nm, where differences in absorbance were obtained. The results revealed that the WoAgNPs did not perform excellently as photocatalysts; the adsorption of the NPs on the methyl red was considerably low throughout the period of the reaction. In the first 20 minutes of the reaction, only 3.26% degradation was recorded for the particles obtained from 1mM AgNO₃ and 1.00% for the particles obtained from 5 mM AgNO₃ solution. For the overall time of the evaluation, the highest degradation obtained was 6.07% for the particles obtained from 1 mM concentration and 21.25% for the ones derived from 5 mM WoAgNPs.

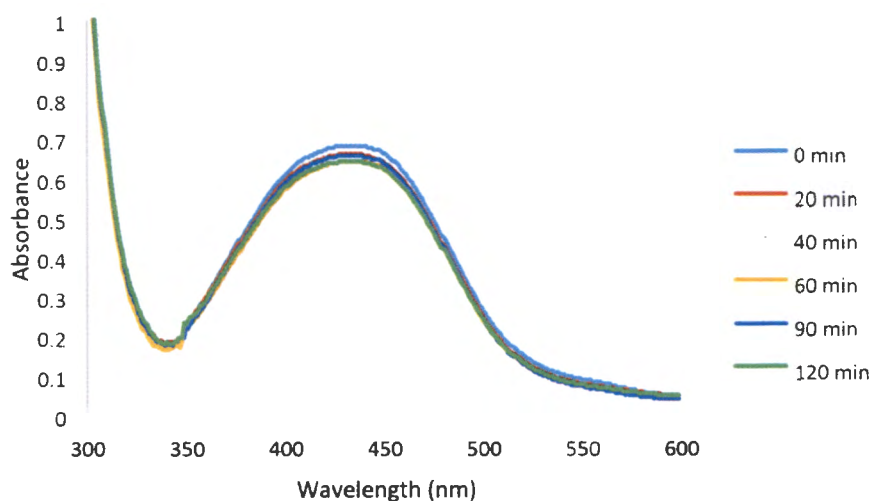


Figure 4.34: Degradation of methyl red using WoAgNPs obtained from 1 mM AgNO₃ with 1:5 volume ratio of aqueous leaf extract to AgNO₃.

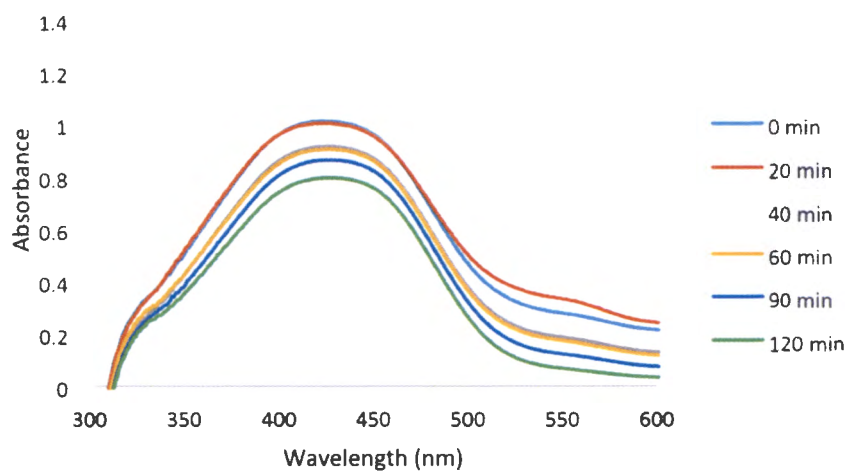


Figure 4.35: Degradation of methyl red using WAgNPs obtained from 5 mM AgNO₃ with 1:5 volume ratio of aqueous leaf extract: AgNO₃

Table 4.6: Percentage degradation of methyl red using WAgNPs

Time(min)	1mm(1:5)		5mm(1:5)	
	Absorbance	% degradation	Absorbance	% degradation
0	0.6824	-	1.0082	-
20	0.6601	3.26	0.9983	1.00
40	0.6423	5.87	0.9023	10.50
60	0.6423	5.87	0.8610	14.60
90	0.6413	6.02	0.8610	14.60
120	0.6410	6.07	0.7939	21.25

4.3 Characterisation of the silver NPs obtained using the aqueous extract of Guar plant (GuAgNPs)

4.3.1 UV-visible spectroscopy

Figures 4.36 – 4.41 show the absorption spectra of the GuAgNPs obtained at different concentrations. In all the spectra, the evidence of formation of AgNPs was noticed after 45 min of the reaction. The peaks were all broad showing polydispersity of the AgNPs except in the particles prepared from 2 mM AgNO₃ using 1:10 ratio. In all the formulations, the GuAgNPs from 1 mM AgNO₃ using 1:5 ratio has the highest concentration of NPs at 434 nm. The same absorption peak was also observed in the GuAgNPs from 1:10 ratio. In the particles prepared from 2 mM AgNO₃ using 1:5, a higher peak of 449 nm was obtained compared to using 1:10 ratio (which has peak of absorption around 440 nm). The lower absorption peak of the samples from 1:10 ratio suggests smaller particle size. In the GuAgNPs from 5 mM concentration, the 1:5 ratio has lower absorption peak of 447 nm compared to the 449 nm exhibited by the 1:10 ratio. This clearly shows that the lower the wavelength, the smaller the nanoparticle size. There is no specific order observed based on the different concentrations used in this study. Ideally, at higher concentration of the precursor (AgNO₃), one expects an increase in the formation of NPs as more silver ions would be reduced; but in this work GuAgNPs from 1 mM (1:5) showed maximum intensity and also larger number of smallest particle size judging from the wavelength of 434 nm. The nature of the SPBs displayed by the samples from 1 mM using 1:5 also revealed high level of polydispersity with varying sizes of NPs. This indicated the availability of the reducing components in the plant extract which are always ready to convert the silver ions to NPs.

In the work of Amin *et al.* [111], in which silver NPs were formed by using *solanum xanthocarpum* berry extract, it was observed that as the concentration of the extract increased, the SPBs became sharper and the number of NPs also increased. They also observed that there is a limit to which the biomolecules ceases to act as capping agent and in that case, there is an increase in bandwidth which leads to anisotropic or larger size particles.

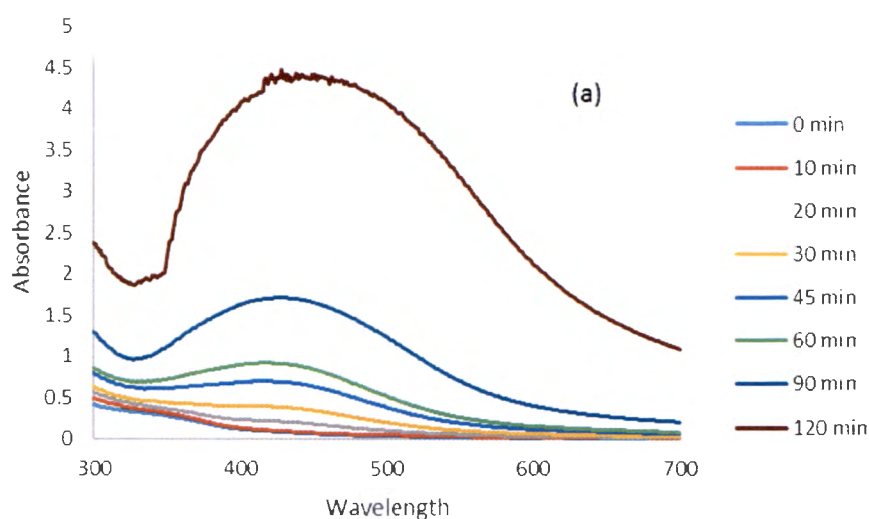


Figure 4.36: UV-visible spectra of the GuAgNps synthesized using 1 mM AgNO_3 and 1:5 substrate to precursor ratio.

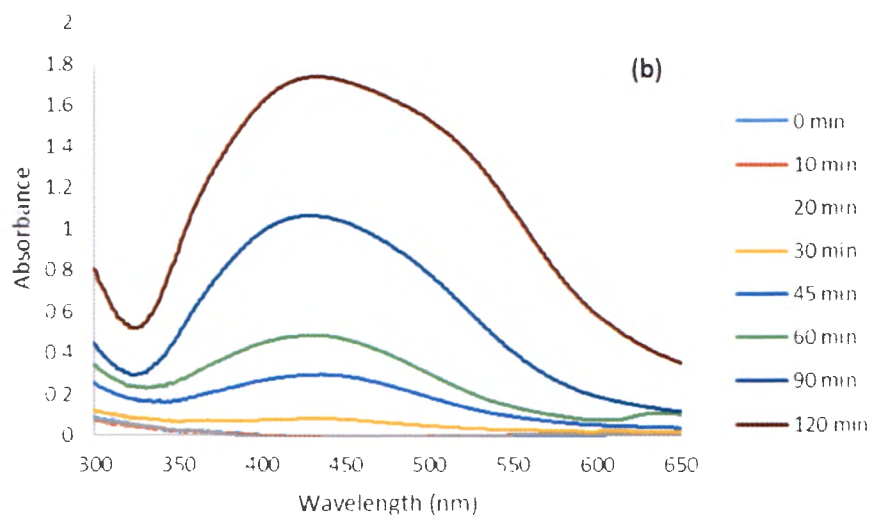


Figure 4.37: UV-visible spectra of the GuAgNps synthesized using 1 mM AgNO_3 and 1:10 substrate to precursor ratio.

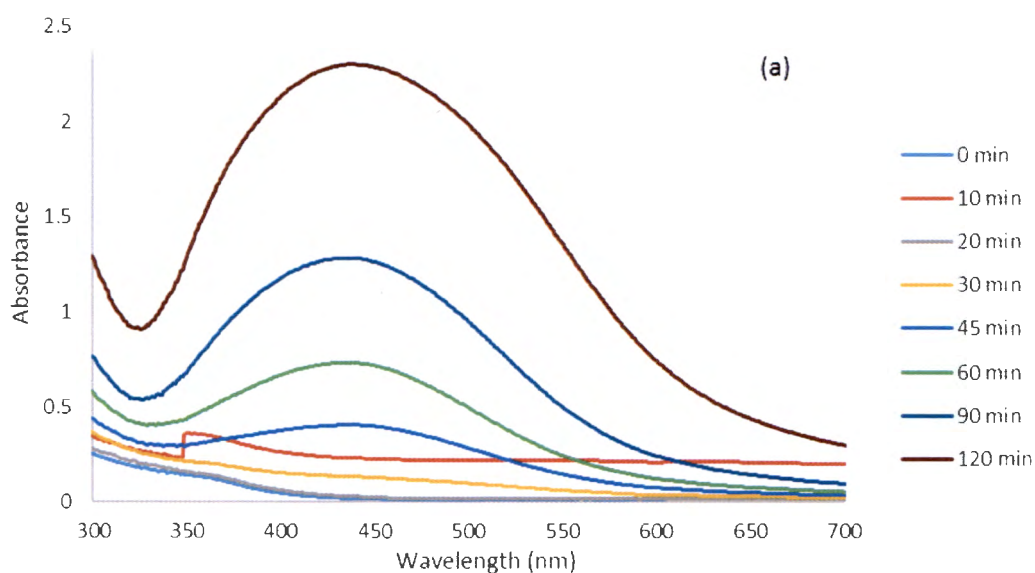


Figure 4.38: UV-visible spectra of the GuAgNps synthesized using 2 mM AgNO_3 and 1:5 substrate to precursor ratio.

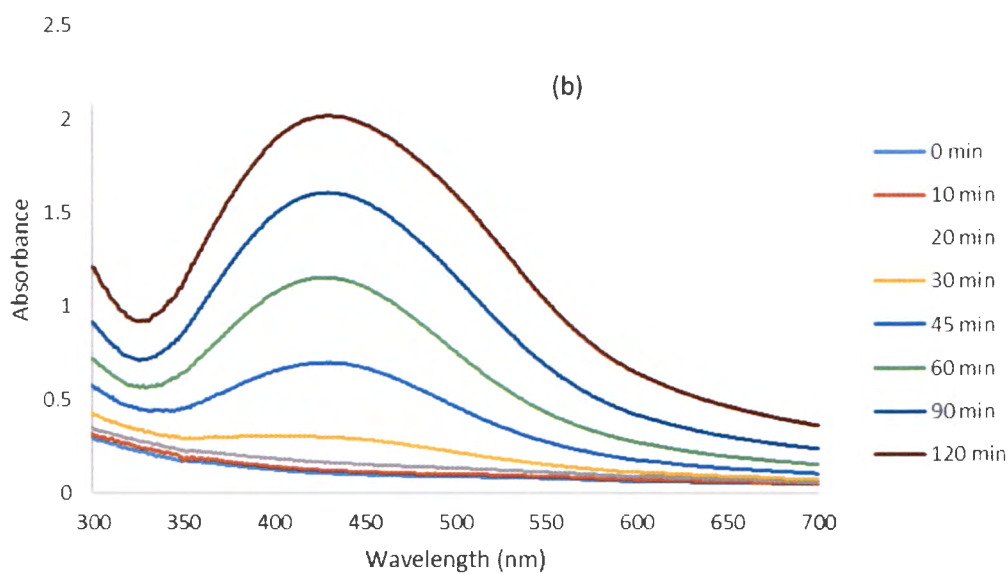


Figure 4.39: UV-visible spectra of the GuAgNps synthesized using 2 mM AgNO_3 and 1:10 substrate to precursor ratio.

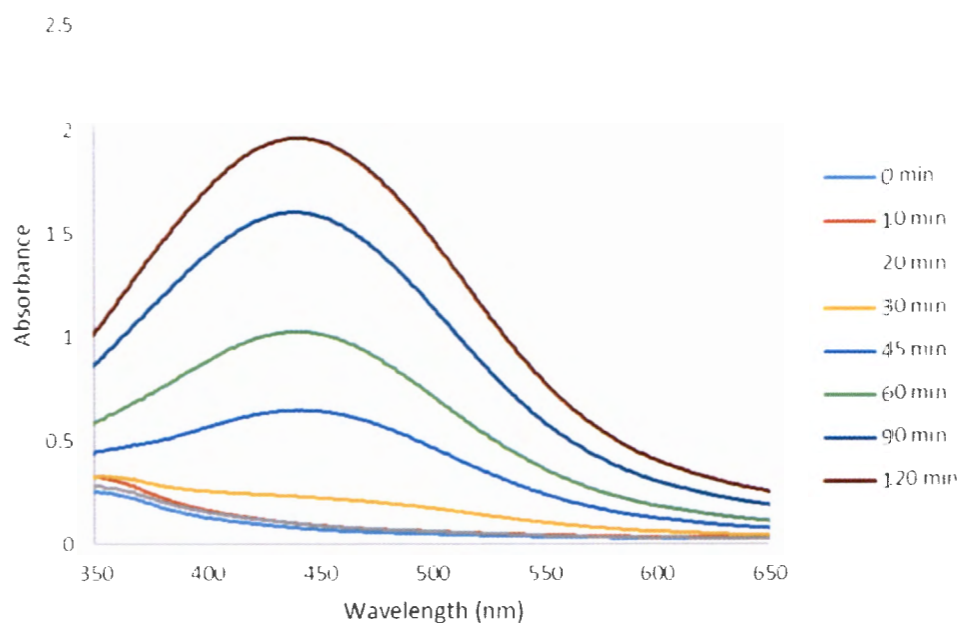


Figure 4.40: UV-visible spectra of the GuAgNps synthesized using 5 mM AgNO_3 and 1:5 substrate to precursor ratio.

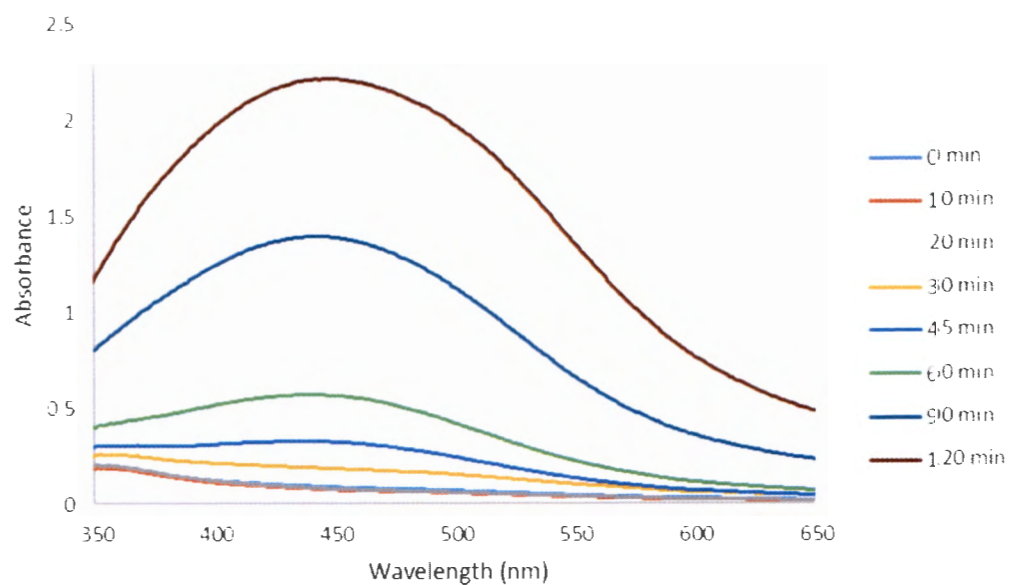


Figure 4.41: UV-visible spectra of the GuAgNps synthesized using 5 mM AgNO_3 and 1:10 substrate to precursor ratio.

4.3.2 FTIR Spectroscopic analysis of the GuAgNPs

Similar to other substrates used, the FTIR measurement of the plant and the silver NPs obtained using the plant extract was carried out to identify the biomolecules that serve as reducing, capping and stabilizing agents in the synthesized silver NPs from the *Cyamopsis tetragonoloba*. The spectrum of the Guar plant (Figure 4.42) showed peaks at 3271, 2918, 2854, 2206, 2089, 1726, 1614, 1540, 1417, 1240, 1018 and 522 cm⁻¹. The broad peak in the higher energy region of 3272 cm⁻¹ indicates the presence of the OH group in the extract of *cyamopsis tertagonoloba* [112], while the sharp peaks at 2854 cm⁻¹ and 2919 cm⁻¹ could be due to the stretching vibration of C-H of the alkanes [106]. The weak peaks around 2206, 1462, 1571 cm⁻¹ could be ascribed to the unsaturated aromatic C=C bonds [105], while peaks around 1726 cm⁻¹ corresponds to C=O of esters, ketone or acids. The 1018 cm⁻¹ peak could be due to the stretching vibration of C-O from ester group [104] and the peaks in the range 522 - 507 cm⁻¹ indicates C-Cl stretching of halide groups [113]. In the spectrum of GuAgNPs (Figure 4.43), the peak positions are similar to that of the plant but of lower intensity which signifies stabilization of synthesized NPs by the plant extracts.

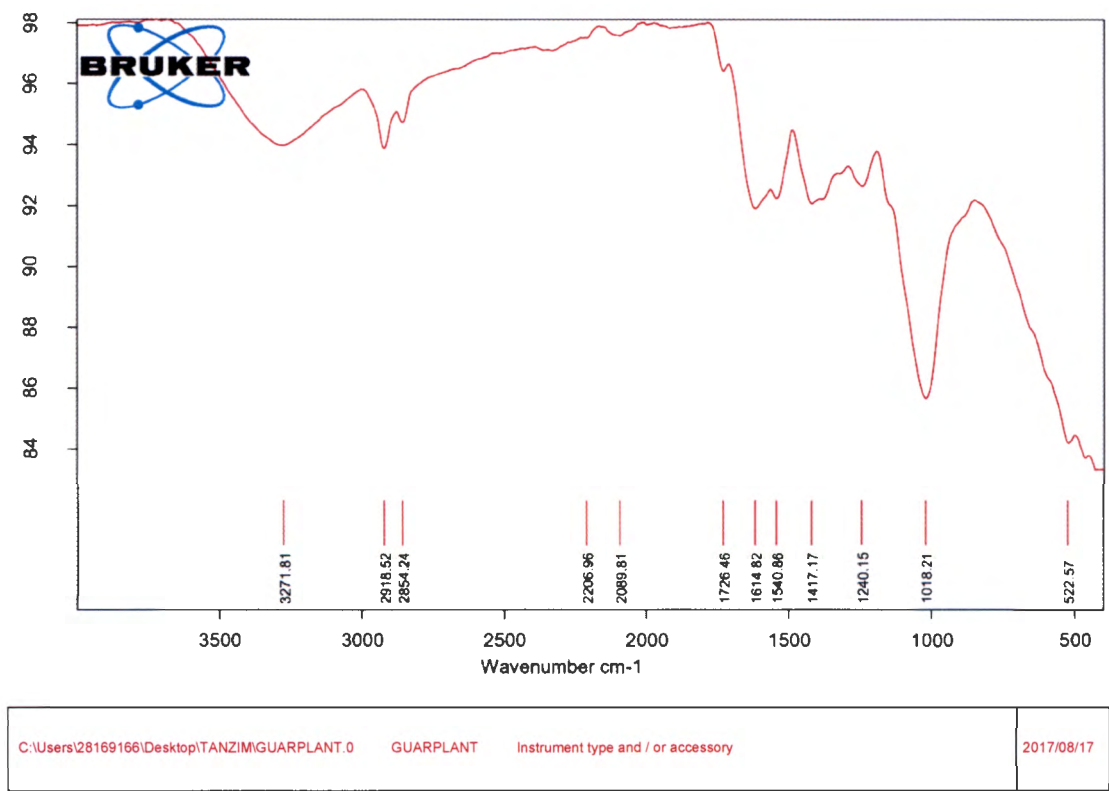


Figure 4.42: FTIR spectrum of the Guar Plant

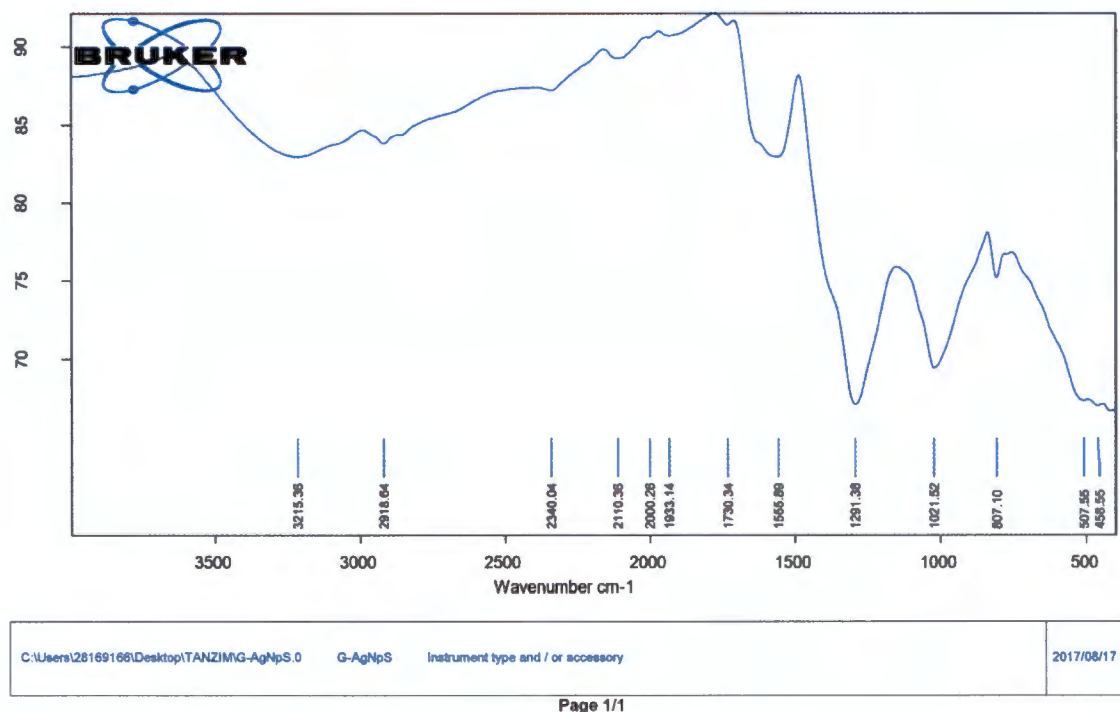


Figure 4.43: FTIR spectrum of the GuAgNPs.

Table 4.7: Functional groups present in the guar plant and NPs

Functional group	Frequency (cm^{-1})		Intensity
	Guar plant	GuAgNPs	
OH	3272	3215	Strong, broad
Alkane -C-H	2854, 2919		strong
-C $\equiv$ C-	2206	2110	Variable
C=C	1571	1540	Medium-weak multiple bands
C=C	1462	1417	Strong
C=O	1739	1726	Strong
C-O	1018		Two bands or more
C-Cl	522	507	Strong

4.3.3 TEM Analysis of the GuAgNPs

There were variations in the TEM micrographs of the three different concentrations of the precursor used for nanoparticle synthesis as shown in Figures 4.44 – 4.49. However, in all,

average spherical shape NPs were obtained. The samples from 2 mM using 1:10 ratio gave the least size of NPs, whereas the samples from 5 mM using 1:10 gave the largest size. The lower size range of NPs were mostly recorded with both samples obtained from the 1 mM AgNO₃ using 1:10 and 2 mM AgNO₃ using 1:10 with monodispersed particles. However, there was a striking appearance of an agglomerated shape in the particles from 2 mM when 1:10 ratio was used which looked like truncated nanorods. The images obtained in all the concentrations corroborate the results of the UV-vis spectra which showed broad peaks, an indication of polydispersity, except for the particles from 2 mM AgNO₃ using 1:10. The clustered peaks shown by the SPBs of the samples from 1mM (1:5) illustrate the varying particle sizes of the NPs which were also confirmed by the TEM image. Ordinarily, the samples obtained from 1 mM using 1:5 ratio have the highest number of lower sized NPs which was also shown by the wavelength of absorption compared to other concentrations. However, on the average, the samples from 2 mM (1:10) were recorded to have lowest size of NPs. The mechanism was such that the rate of nuclei formation was higher than the nanoparticle growth rate thereby giving rise to a large number of smaller particles [98]. In the growth system observed in the particles from 1mM (1:5), oriented attachment seem to occur as against secondary growth due to Ostwald ripening observed in the particles from 5 mM using 1:10.

In the use of guar plant for nanoparticle synthesis, the samples obtained from 5 mM using 1:10 ratio have the largest particle size compared to other concentrations used. Different plants behave differently and it could be observed that, in this synthesis, the large size could be due to the low concentration of the substrate (1:10) and the high concentration of the precursor (5 mM). This is comparable to the work of Shukla *et al.* [98], which reports that as the concentration of the silver nitrate increases, the size of the NPs increases. The amount of active biocomponents that transform the precursor to their NPs, determines the formation rate of the Ag/AgCl nuclei, the collision rate and general reaction mechanism.

Table 4.8: Size distribution at different precursor and substrate concentrations of the synthesized GuAgNPs

Concentration of Precursor(AgNO_3) (mM)	Ratio of Substrate to precursor	Average size of AgNPs (nm)	Size Range (nm)
1	1:5	5.21	2.03-9.36
	1:10	8.67	3.71-13.66
2	1:5	11.00	6.98-15.27
	1:10	3.61	1.85-7.39
5	1:5	10.02	4.23-21.19
	1:10	12.92	6.83-23.43

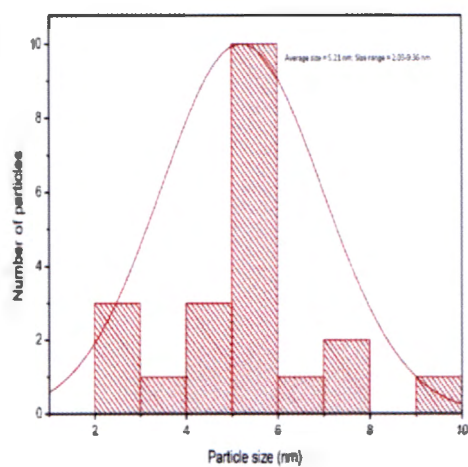
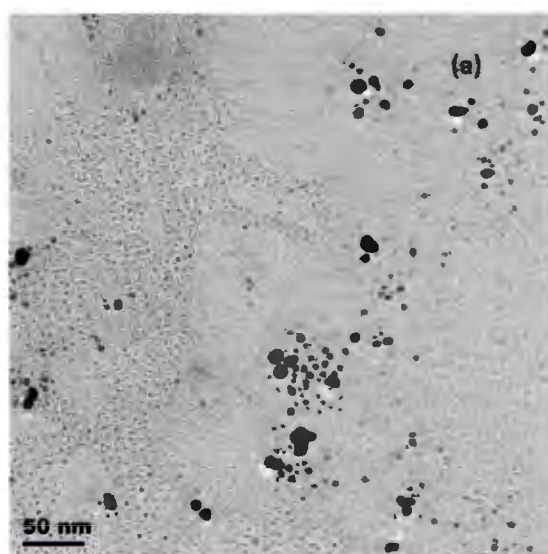


Figure 4.44: TEM image and particle size distribution histogram of the NPs obtained using 1 mM AgNO_3 and 1:5 volume ratio of aqueous plant extract: AgNO_3

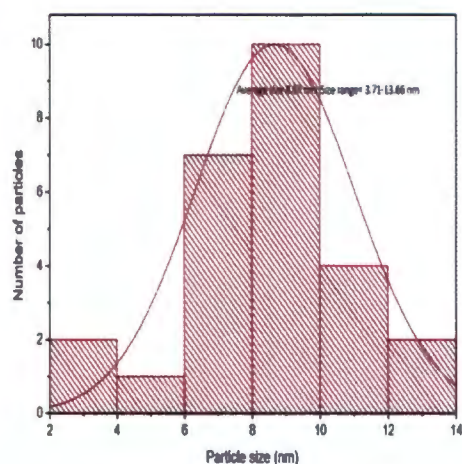
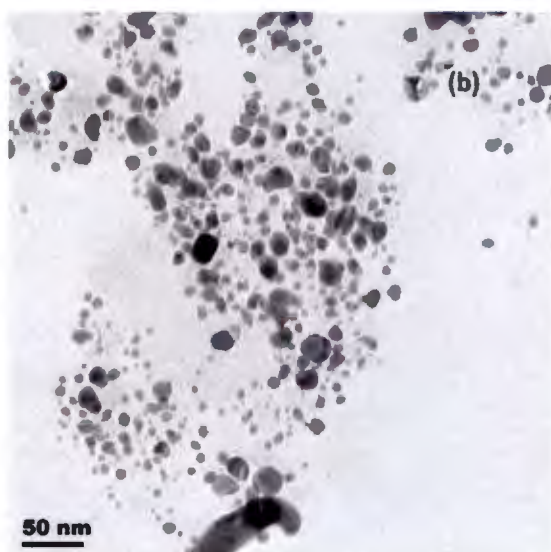


Figure 4.45: TEM image and particle size distribution histogram of the NPs obtained using 1 mM AgNO_3 and 1:10 volume ratio of aqueous plant extract: AgNO_3

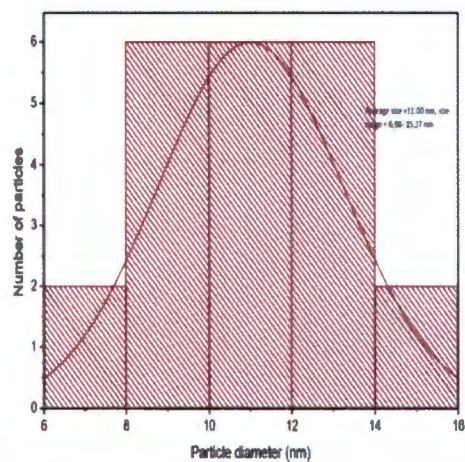
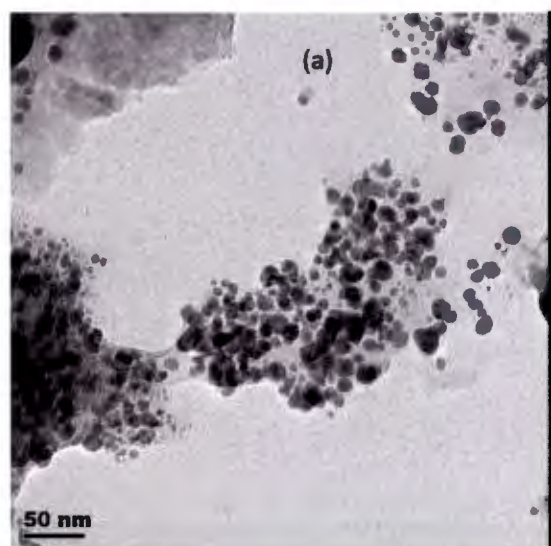


Figure 4.46: TEM image and particle size distribution histogram of the NPs obtained using 2 mM AgNO_3 and 1:5 volume ratio of aqueous plant extract: AgNO_3

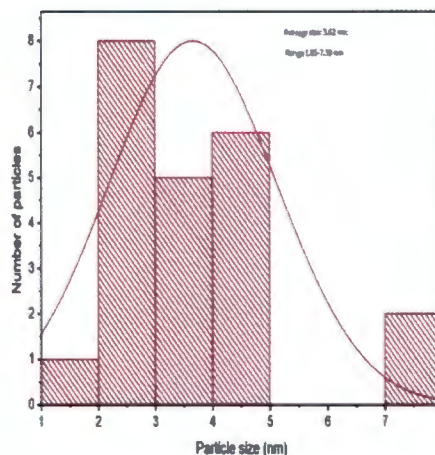
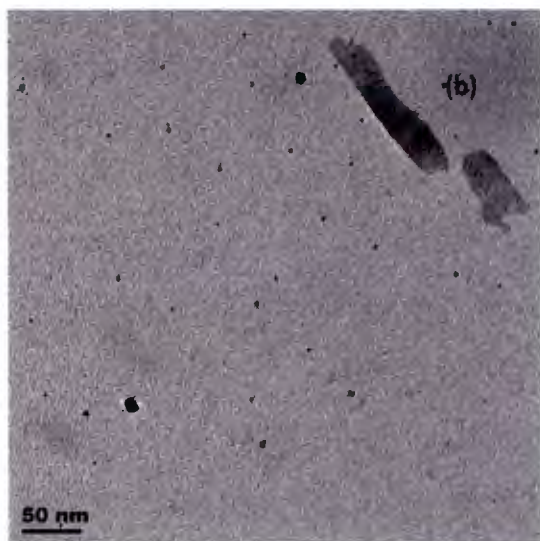


Figure 4.47: TEM image and particle size distribution histogram of the NPs obtained using 2 mM AgNO_3 and 1:10 volume ratio of aqueous plant extract: AgNO_3

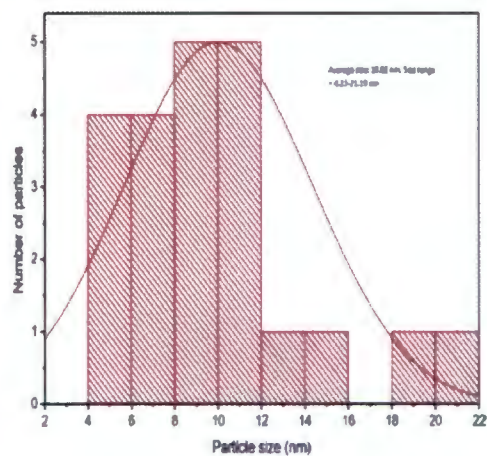
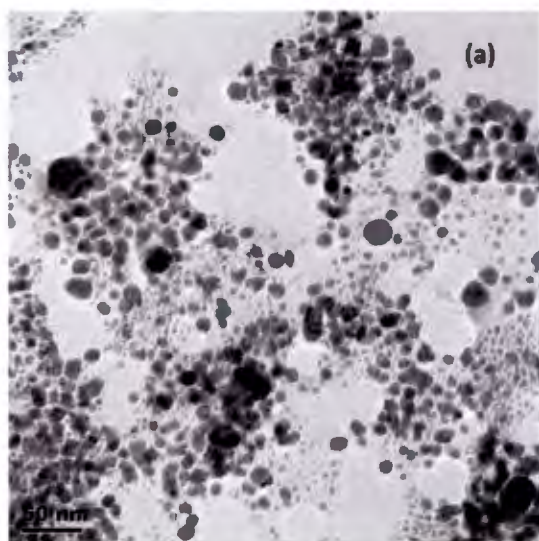


Figure 4.48: TEM image and particle size distribution histogram of the the NPs obtained using 5 mM AgNO_3 and 1:5 volume ratio of aqueous plant extract : AgNO_3

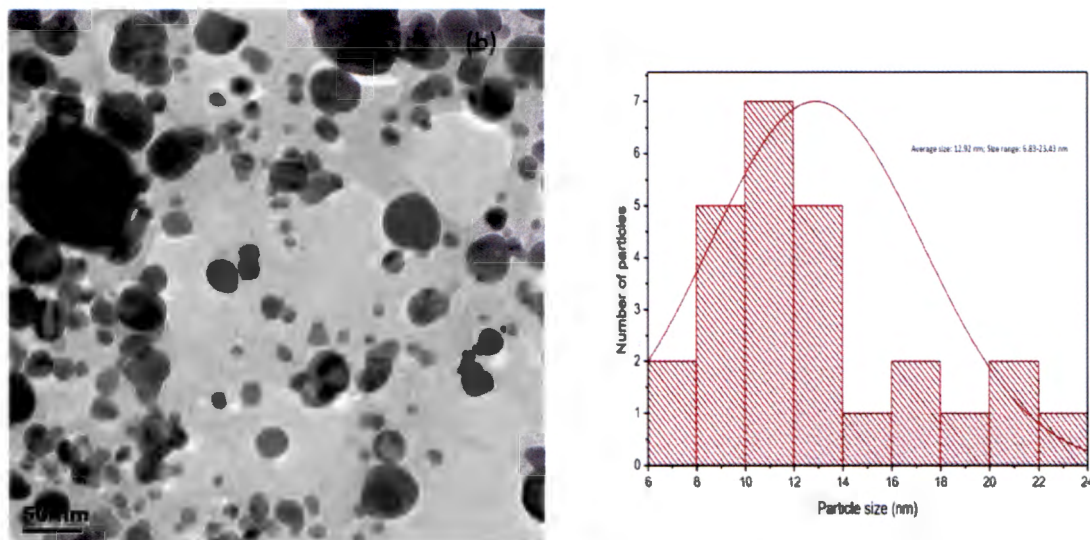


Figure 4.49: TEM image and particle size distribution histogram of the the NPs obtained using 5 mM AgNO_3 and 1:10 volume ratio of aqueous plant extract: AgNO_3 .

4.3.4 Powder X-Ray diffraction (pXRD) Studies

The X-ray diffraction pattern of GuAgNPs, obtained from the aqueous extract of Guar plant, is presented in Figure 4.50. The peaks reveal crystalline structure and Bragg's reflections angles at 39.32, 43.47, 64.56 corresponding to 111, 200, 220 lattice planes of Ag while the peaks at 32.69 and 46.28 reflect 200 and 202 planes of AgCl. Other peaks could be due to the phytocompounds present in the extracts.

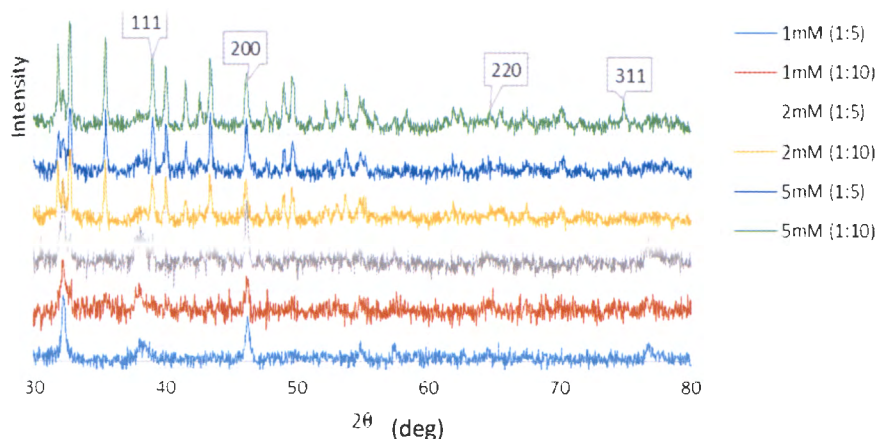


Figure 4.50: pXRD patterns of the GuAgNPs.

4.3.5 Photocatalytic behavior of the GuAgNPs

The degradation of environmental pollutants especially dyes has been a great concern among industrialists and environmentalists (deg) and the use of green synthesized silver NPs has been offering some hope.

In this research, silver NPs were used to verify the degradation of methyl red [114]. The photocatalytic process was monitored at specific intervals, and the decrease in intensity of absorption at 435 nm was recorded. The percentage degradation was calculated using the equation,

$$\% \text{ Dye degradation} = \frac{C_o - C_t}{C_o} \times 100 \quad (4.1)$$

Where C_o and C_t are the concentration of the dye at initial and time t , respectively. Since absorbance is proportional to concentration, the value of the concentrations of the methyl red at the different times were derived from the absorbance peak value at 435 nm. Methyl red ($C_{15}H_{15}N_3O_2$) is an azo dye, that is carcinogenic and harmful to living organisms. Within the aquatic system, they pose great danger to all living system organisms.

The mechanism of the photocatalytic reaction could be explained thus: The nanoparticles absorb ultraviolet radiation, causing the surface to be activated and electrons are excited;

these photogenerated electrons interact with the dissolved oxygen molecules to yield O^2 radicals that can degrade the organic dye.

At 438 nm, the % degradation recorded for the samples obtained from 1mM AgNPs is 17% while for the samples from 5 mM, it is 14% after 90 min reaction. The values show low percentage degradation compared to earlier reports. Perhaps, there was no effective activation of the surfaces of the silver NPs and therefore, intermediate molecules such as the dissolved oxygen radicals which could lead to efficient degradation were not sufficiently produced. The results recorded here reiterate the fact that photocatalytic behaviour of NPs is prominently due to their band structure [115].

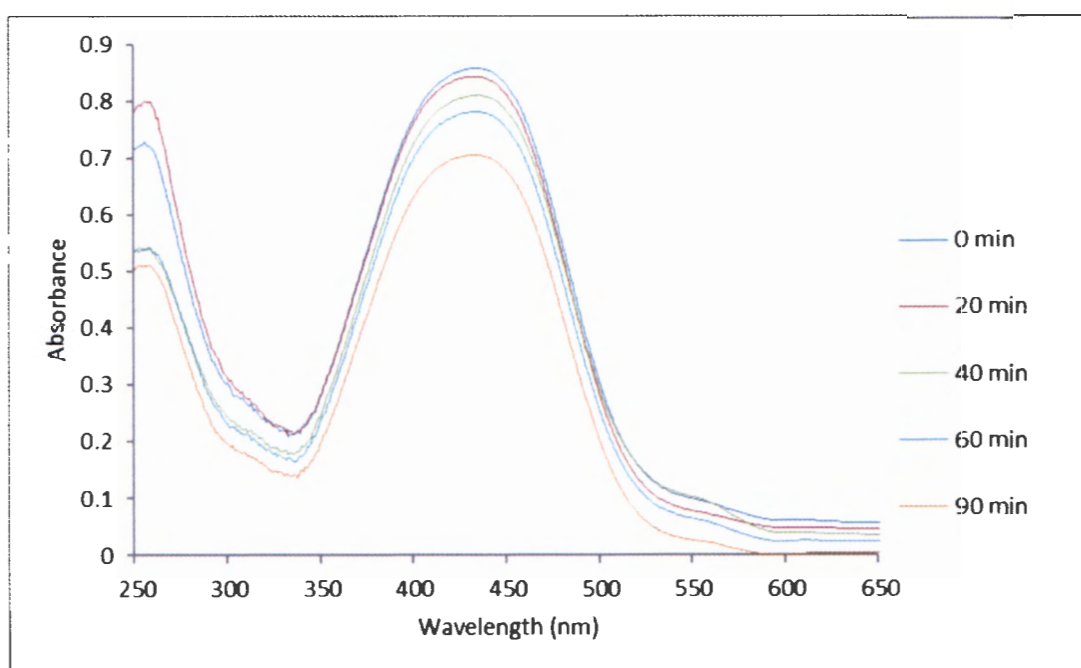


Figure 4.51: Degradation of methyl red using GuAgNPs obtained from 1 mM $AgNO_3$ with 1:5 volume ratio of aqueous leaf extract: $AgNO_3$.

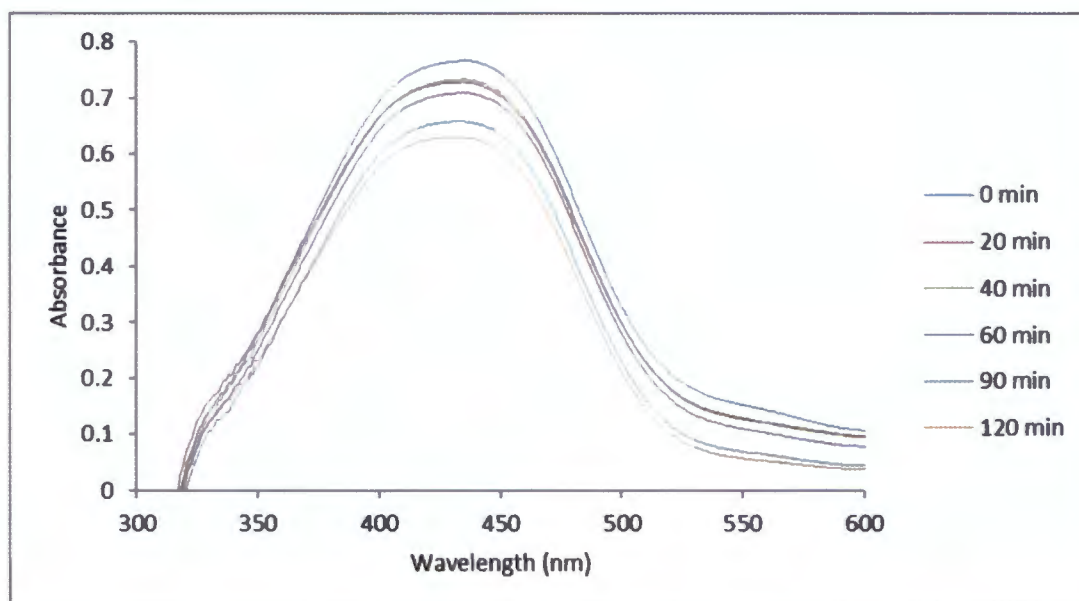


Figure 4.52: Degradation of methyl red using GuAgNPs obtained from 1 mM AgNO₃ with 1:5 volume ratio of aqueous leaf extract: AgNO₃.

Table 4.9: Percentage photocatalytic degradation recorded at 438 nm at different time intervals

Time (min)	1 mm (1:5)		5 mm (1:5)	
	Absorbance	% degradation	Absorbance	% degradation
0	0.8556	-	0.7651	
20	0.8421	1.58	0.7307	4.50
40	0.8088	5.47	0.7305	4.52
60	0.7804	8.79	0.7076	7.52
90	0.7049	17.61	0.6562	14.40
120	0.7050	17.62	0.6285	17.85

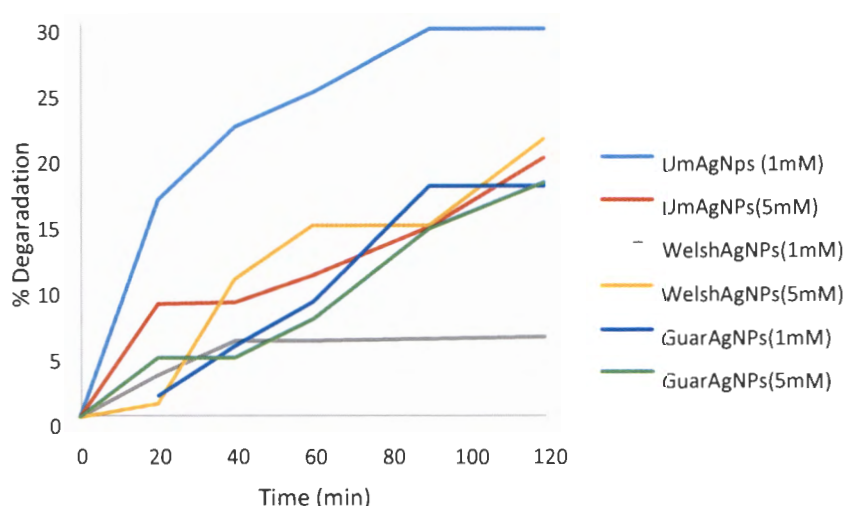


Figure 4.53: Comparative photocatalytic degradation of the three plants used in this work.

4.4 Comparative antibacterial analysis

The antimicrobial activity of the NPs was studied using some bacteria and selected bacteria which are responsible for some water borne diseases and the results are presented in Table 4.10. The AgNPs do not show antibacterial activity at the concentrations of 5 - 15 $\mu\text{g/mL}$ with no clear zone of inhibition obtained. However, at 25 $\mu\text{g/mL}$ (which also serves as their MIC), the bacteria strains were susceptible to the AgNPS and the antibacterial results were reported.

From the table, the gram-negative organisms were more susceptible to the biosynthesized AgNPs compared to the gram-positive bacteria organisms. Lipophilicity could be a major factor in the activity of NPs to bacterial strains, and may be attributed to the presence of the robust lipid cell wall membrane of the gram-positive bacteria organisms. This majorly prevents easy penetration of antibiotics [116,117].

In addition, *E.coli* was more susceptible to the AgNPs compared to *Kleb. Pneumonia*. This could be attributed to the sophisticated defence mechanism in *Kleb pneumonia* which involves its ability to produce extended spectrum beta-lactamases (ESBL) that inactivates the compounds and makes them non-toxic [118].

The antibacterial properties of leaf extracts of Umbrella, Welsh and Guar plants have been reported and the extracts were found to be active against bacteria organisms but not as active as the AgNPs with the isolates absorbed on its surfaces. The AgNPs obtained from the 1:10 ratio were more active towards the test organisms compared to the ones from 1:5 ratio. This could be attributed to the effect of the increase in the components of the leaf extract present on the NPs surface which might have increased its antibacterial potency.

The minimum inhibitory concentrations of the samples are presented in Table 4.11. The MIC results of NPs against *Staphylococcus aureus* and *E.coli* are lower than 0.05 mg/mL and this corresponds to that of ciprofloxacin against the bacteria strains. Hence, the NPs are more active against *E.coli* than the other bacteria strains.

Table 4.10: Antibacterial results of AgNPs (Zone of inhibition in mm)

Samples	<i>Staphylococcus aureus</i>	<i>Bacillus cerues</i>	<i>Kleb. pneumonia</i>	<i>E. Coli</i>
GAgNPs	10.5±0.0	08.2±0.7	11.2±0.3	14.7±1.2
UmAgNPs	12.7±0.7	10.9±0.7	13.1±0.7	16.0±0.0
WoAgNPs	17.3±0.7	11.1±1.2	15.4±0.3	19.8±1.2
Ciprofloxacin	20.0±1.4	25.8±0.0	23.0±0.0	25.0±0.7
Water	R	R	R	R

Values (in mm) represent the mean of double replications and their standard deviation; standard drug (Ciprofloxacin) was used as positive control for antibacterial studies, water was used as negative control and showed no activity, R = resistant. The well diameter was 6 mm, and any activity below 6 mm was considered resistant. Table 4.11: Minimum inhibitory concentration (MIC)

Samples	<i>Staphylococcus aureus</i>	<i>Bacillus cerues</i>	<i>Kleb. pneumonia</i>	<i>E. Coli</i>
AgNPs	<0.05	0.1	0.1	<0.05
Ciprofloxacin	<0.05	<0.05	<0.05	<0.05

CHAPTER FIVE

CONCLUSION AND RECOMMENDATION

In this research work, three sets of plants have been used for the synthesis of silver NPs. The plants are Umbrella plant, Welsh onion plant and Guar plant. The aqueous extract from each of the plants, acts as the substrate for the NPs synthesis. The aqueous solutions act as both the capping and reducing agent without the use of non-eco-friendly solvents, consequently satisfying the objective of green chemistry. The precursor compound was silver nitrate and three different concentrations were made to evaluate their effect towards the properties of the NPs. The concentrations prepared were 1 mM, 2 mM and 5 mM AgNO_3 . In each of the concentrations of the precursors prepared, 1:5 and 1:10 volume ratios of AgNO_3 to aqueous solution of plant extract were used for the synthesis. The formation of silver NPs was monitored at different time intervals using absorption spectroscopy.

The UV results gave different SPBs thereby indicating that concentrations play a critical role in the formation of NPs and eventual application. In all the different concentrations of the three plant substrates used, the earliest formation of NPs as revealed by the surface plasmon bands was after 30 min when the aqueous extract of umbrella plant was used at the two ratios (1:5 and 1:10). The silver nanoparticle obtained by using the aqueous extract from plants of welsh onion and guar plants represented as WoAgNPs and GuAgNPs respectively formed after 60 and 45 mins reaction time respectively. The UmAgNPs gave the narrowest peaks compared to the others, while the GuAgNPs gave broad peaks with higher wavelength of absorption. The 2 mM of the UmAgNPs gave the lowest wavelength peaks compared to the other concentrations and seems to have the best SPBs among all.

The FTIR results of the three plants gave similar peaks around $3300\text{-}3200\text{ cm}^{-1}$ (broad), $2950\text{--}2850\text{ cm}^{-1}$ (twin peaks), $1620\text{-}1600\text{ cm}^{-1}$ (C=O) and $1080\text{-}1000\text{ cm}^{-1}$ (strong peak) which reflect some degree of similarities in the constituents of the plants. From the vibrational peaks they could be primary metabolites such as proteins, carbohydrates and secondary metabolites such as flavonoids, tannins, terpenoids etc. clearly, these biomolecules have served as reducing, stabilizing, capping agents and also contain important nutritive phytochemicals which promote their acceptability for other applications. The spectral bands are more intense in the plant than in the corresponding NPs.

Further characterization using TEM revealed interesting size ranges of the NPs. The different concentrations and volume ratios gave varying sizes with the 2 mM UmAgNPs having the most

stable and monodispersed particle sizes with average particle size of 8.15 nm (for both 1:5 and 1:10 ratios). The largest mean particle sizes were observed in all the 5 mM samples except for the UmAgNPs (1:10). The 5 mM (1:5) UmAgNPs also gave monodispersed lower particles but the appearance of an extraordinary polygon-shaped particle which possibly formed through particle aggregation by a suspected Ostwald ripening process. The particles from 1 mM (1:5, 1:10) WoAgNPs gave the largest mean particle size among the three plants used for the synthesis.

The pXRD patterns mostly revealed purity of silver with traces of AgCl/Ag₂O. The anions which most likely originated from plants constituents could also indicate the different types of anions present in the plants components. Peaks that show Bragg reflections of silver were obtained and compared to XRD patterns of published standards. Thus, from XRD the crystalline nature of the nanoparticles was also confirmed.

The photocatalytic activities of the synthesized NPs were verified using methyl red, to determine their potentials in the organic dye degradation and possible remediation of waste water polluted by dyes. Generally, all the NPs showed some degree of photodegradation of the methyl red although the activity varies with plants and concentrations. Among all, UmAgNPs (1 mM) showed the best photocatalytic property with 29.73% and WoAgNPs (1 mM) showed the least at 6.02% after 120 min reaction time.

The antimicrobial analysis revealed that the NPs are averagely good antibacterial agents with the WoAgNPs showing the greatest inhibition potency against some disease causing bacteria. This project has explored the different potentials of some nutritive and ornamental plants beyond their common usage towards the synthesis of silver NPs.

Although, different challenges such as obtaining large quantities of NPs for characterization and applications in addition to the inefficient photocatalytic degradation reactions, were encountered during the course of this research. Nevertheless, the project was able to present a facile route to making of silver NPs which could be used to tackle some of the environmental menace. This work still continues as emphasis would be on the optimization of the reaction conditions to achieve multiple degradation of pollutants and excellent antimicrobial activities for clean water technology. The government needs to strengthen this field of science in order to get the best out of it.

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