ADVANCES IN SYNTHESIS; CHARACTERIZATION AND APPLICATION OF *Sorghum bicolor* STRAW NANOPARTICLES

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CERTIFICATION

This is to certify that this project work was carried out by **Aranmoletieso Falilat Eniola, Arowolo Esther Oluwatosin, Akinola Alake Rachael and Oke Favour Sonia** with Matriculation Numbers **17/06/0098, 17/06/0099, 17/06/0100, 17/06/0101,** respectively in the Department of Science Laboratory Technology, School of Science, Abraham Adesanya Polytechnic, Ijebu-Igbo, under my supervision.

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DEDICATION

This project is dedicated to all prolific scientist in the world that are working relentlessly in order to make planet Earth a safer and a better place for habitation.

ACKNOWLEDGEMENT

We acknowledge the Almighty God who has been our Comforter since the very first minute of our lives to this present moment, with this, we say Glory be to God in the highest.

Also, our sincere appreciation goes to our parents for giving us the best education in life and for their supports financially, morally and spiritually. May they live long to reap the fruit of their labour.

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May God bless you all.

ABSTRACT

Nanotechnology is making an impact in every field of life. Researchers are expanding their interests towards synthesise of silver nanoparticles as they provide superior properties for different types of applications. Conventionally nanoparticles have been synthesised by various physical and chemical methods, having negative impact on environment. The production of nanoparticles using plant extract is alternative the conventional methods. The photosynthesis is a green and eco-friendly technology used for production of large scale nanoparticles. Plant extracts may act both as reducing agents and stabilizing agents in the synthesis of nanoparticles. The various phytochemicals present in plant extract are used for the reduction and stabilisation of nanoparticles. This research work is concentrated on the advances in synthesis, characterization and application of nanoparticles using sorghum bicolor plant extract.

Keywords: *Characterization Sorghm bicolor, Nanoparticles, UV spectrophotometer, FTIR (Fourier Transform Infra-red).*

TABLE OF CONTENTS

TABLES AND FIGURES

TABLES:

FIGURES:

CHAPTER ONE

1.0 INTRODUCTION

1.1 Background of the Study

Due to swift industrialization and urbanization, our environment has undergone huge smash up and a large amount of perilous and superfluous chemical, gases or substances are released, and so now it is our need to learn about the secrets that are present in the Nature and its products which leads to the growth of advancements in the synthesis processes of nanoparticles. Nanotechnology applications are highly suitable for biological molecules, because of their exclusive properties. The biological molecules undergo highly controlled assembly for making them suitable for the metal nanoparticle synthesis which was found to be reliable and eco-friendly (Harekrisna et al., 2009).

The synthesis of metal and semiconductor nanoparticles is a vast area of research due to its potential applications which was implemented in the development of novel technologies (Cassandra *et al.*, 2007). The field of nanotechnology is one of the upcoming areas of research in the modern field of material science. Nanoparticles show completely new or improved properties, such as size, distribution and morphology of the particles etc.

Novel applications of nanoparticles and nanomaterials are emerging rapidly on various fields (Kaviya,and Viswanathan 2011).

Metal nanoparticles have a high specific surface area and a high fraction of surface atoms. Because of the unique physicochemical characteristics of nanoparticles, including catalytic activity, optical properties, electronic properties, antibacterial properties, and magnetic properties (Catauro *et al.*,2005; Krolikowska *et al.*,2003; Zhao *et al.*, 2005).

They are gaining the interest of scientist for their novel methods of synthesis. Over the past few years, the synthesis of metal nanoparticles is an important topic of research in modern material science. Nano-crystalline silver particles have been found tremendous applications in the fields of high sensitivity biomolecular detection, diagnostics, antimicrobials, therapeutics, catalysis and micro-electronics. However, there is still need for economic commercially viable as well as environmentally clean synthesis route to synthesize the silver nanoparticles. Silver is well known for possessing an inhibitory effect toward many bacterial strains and microorganisms commonly present in medical and industrial processes (Jiang *et al.*, 2004).

In medicines, silver and silver nanoparticles have a ample application including skin ointments and creams containing silver to prevent infection of burns and open wounds (Duran *et al.*, 2005) medical devices and implants prepared with silver-impregnated polymers. In textile industry, silver-embedded fabrics are now used in sporting equipment (Klaus *et al.*, 1999).

Nanoparticles can be synthesized using various approaches including chemical, physical, and biological. Although chemical method of synthesis requires short period of time for synthesis of large quantity of nanoparticles, this method requires capping agents for size stabilization of the nanoparticles. Chemicals used for nanoparticles synthesis and stabilization are toxic and lead to non-ecofriendly byproducts. The need for environmental non-toxic synthetic protocols for nanoparticles synthesis leads to the developing interest in biological approaches which are free from the use of toxic chemicals as byproducts. Thus, there is an increasing demand for green nanotechnology (Garima *et al.*, 2011).

Many biological approaches for both extracellular and intracellular nanoparticles synthesis have been reported till date using microorganisms including bacteria, fungi and plants (Spring *et al.*, 1995). Sometimes the synthesis of nanoparticles using various plants and their extracts can be advantageous over other biological synthesis processes which involve the very complex procedures of maintaining microbial cultures (Sastry *et al.*, 2003).

Many such experiments have already been started such as the synthesis of various metal nanoparticles using fungi like *Fusarium oxysporum* (Nelson *et al.*, 2005; Hemanth *et al.*, 2010) *Penicillium sp.* and using some bacteria such as *Bacillus subtilis* etc. (Nataranja and Elumalai *et al.*, 2010). But, synthesis of nanoparticles using plant extracts is the most adopted method of green, eco-friendly production of nanoparticles and also has a special advantage that the plants are widely distributed, easily available, much safer to handle and act as a source of several metabolites (Ankamwar *et al.*, 2005).

There has also been several experiments performed on the synthesis of silver nanoparticles using medicinal plants such as *Oryza sativa, Helianthus annus, Saccharum officinarum, Sorghum bicolour, Zea mays, Basella alba, Aloe vera Capsicum annuum, Magnolia kobus, Medicago sativa (Alfalfa), Cinamomum camphora* and *Geranium sp.* in the field of pharmaceutical applications and biological industries.

The use of plant extracts has been extensively examined for metallic nanoparticle synthesis as they improve the mono-dispersity of nanoparticles. The biomolecules like phenolics, polysaccharides, flavones, terpenoids, alkaloids, proteins, amino acids, enzymes and alcoholic compounds present in plants act as both reducing agents as well as capping agents that stabilize and govern the morphology of NPs (Ravindran *et al.*, 2003; Albrecht *et al.*, 2006).

Green synthesis of metallic NPs by using plant extracts has extensively been employed in recent research (Ashok *et al.*, 2015; Nakkala *et al.*, 2011). Nanoparticles possess completely novel physical (optical, magnetic and electronic) and chemical properties. Size range, self-assembly and high antimicrobial activity of silver nanoparticles (AgNPs) regarded as important for their potential role in medical devices, optical devices, electronics biotechnologies and catalysis (Tolaymat *et al.*, 2010; Alvarez-Puebla *et al.*, 2009). Small size, spherical shape and high surface-area-to-volume ratio of AgNPs facilitate them to interact with the cell walls of pathogens, which gives them better antimicrobial activity (Paralikar, 2014).

Plants provide a better platform for nanoparticles synthesis as they are free from toxic chemicals as well as provide natural capping agents. Moreover, use of plant extracts also reduces the cost of microorganisms isolation and culture media enhancing the cost competitive feasibility over nanoparticles synthesis by microorganisms (Garima *et al.*, 2011).

1.2 PROBLEM STATEMENT AND SIGNIFICANCE OF THE STUDY

The use of plant and plant extract in nanoparticle synthesis is considered advantageous over microbial based system because it reduces the elaborate process of maintaining cell cultures. These studies suggested that presence of phytochemicals in plant extracts are the key component in reduction and stabilization of silver ions (Parashar *et al.*, 2011).

1.3 AIM AND OBJECTIVES OF THE STUDY

This research aims to explore more about sorghum bicolor and the advances made towards the development of the plant in nanotechnology. Considering the facts above, the present study was framed with the broad objective as to Extract nanoparticles from sorghum, synthesize and characterize the nanoparticles. Exhibit its application in the field nanotechnology.

1.4 SCOPE OF THE STUDY

To date, several microorganisms, including bacteria, fungi and yeast, as well as plants have been explored for the synthesis of metal nanoparticles, while the synthesis of nanoparticles have been reviewed elsewhere (Pereira *et al.*, 2015; Baker *et al.*, 2013; Thakkar *et al.*, 2010). This research study is limited to the recent advances in the synthesis, characterization and application of *sorghum bicolor* nanoparticles.

CHAPTER TWO

2.0 LITERATURE REVIEW

2.1 Plant of Study- Sorghum Bicolor

Sorghum (Sorghum bicolor (L.) Moench) popularly called as *Jowar*, is the "King of millets" and is the fifth in importance among the world's cereals, after wheat, rice, maize and barley. It is a staple food grain in many Indian states. It is grown especially in the arid and semi-arid regions (Vikas, 2003).

Sorghum (Milo) was introduced to the United States from Africa in the early part of the seventeenth century. It was not grown extensively in this country until the 1850s, when the forage variety Black Amber (also called "Chinese sugarcane") was introduced by way of France. Since then many other varieties have been introduced from other countries and developed domestically. Sorghum was grown primarily as a source of sugar for syrup until the settlement of the semiarid west created a demand for drought-resistant forage crops. By the 1950s, about 90% of the acreage of sweet sorghum in the United States was grown for forage.

Sorghum is a tropical grass grown primarily in semi-arid parts of the world. In Africa, a major growing area runs across West Africa south of the Sahara, through Sudan, Ethiopia and Somalia. It is grown in Upper Egypt and Uganda, Kenya, Tanzania, Burundi and Zambia. It is an important crop in India, Pakistan, Thailand in central and northern China, Australia, in the drier areas of Argentina and Brazil, Venezuela, USA, France and Italy. The crop has spread over the drier areas of the world; it does better when it is dry and cool, whereas pearl millet is better adapted to dry hot conditions. Sorghum is a staple food for about 300 million people worldwide (www.jn.nutrition.org, 2009).

Sorghum originated in Eastern Africa and first diverged from the wild varieties in Ethiopia 5000 years ago (Anderson and Martin, 1949). Hence, indigenous knowledge based sorghum classification and naming has a long tradition. Farmers have been growing sorghum for at least 500 years (Mekbib, 2007) Sorghum and millets have been important staples in the semi-arid tropics and principal sources of energy, protein, vitamins and minerals for millions of people in these regions (FAO, 1995).

Sorghum was first described by Linnaeus in 1753 under the name of Holcus. Moench later separated the genus sorghum from the Holcus and made the combination of Sorghum bicolor. The current formal taxonomic concept of the sorghum genus and species agrees with the one established by Moench.

All the different names given by the various taxonomists and are hence taken as synonym to S. bicolor (L.) Moench (Snowden, 1998). The many subspecies are divided into four groups - grain sorghums, grass Sorghums (for pasture and hay), sweet sorghums formerly called Guinea corn, (used to produce sorghum syrups) and broom corn (for brooms and brushes) (Anderson and Martin, 1949).

Sorghum bicolor and its other species are grown in the Northern states of Nigeria which is one of the five largest producers of the cereal. They are grains, which hold a dominant position among arable crops in Africa and other parts of the world (FAO, 1995). Sorghum is a genus of numerous species of grasses, some of which are raised for grain and many of which are used as fodder plants either cultivated or as part of pasture. The plants are cultivated in warmer climates worldwide. Species are native to tropical and subtropical regions of all continents in addition to the South West Pacific and Australia (Watson and Andrew, 1983).

Wild varieties of sorghum are attested as early as 8000 BP in the Nilotic regions of Southern Egypt and the Sudan, the location of its true domestication within East Africa are still speculative. It is widely held that genetic separation of domesticated S. bicolor from its progenitor did not occur much before the B.C./A.D., changeover somewhere in East Africa, possibly the Ethiopian highlands, but more likely further West.

The presence of true domesticated S. bicolor is claimed much earlier than this (2900-1700 B.C.) in India, Oman and Yemen, although the identity of the remains as full domesticates is still disputed (www.mdidea.com, 2008).

According to Wikipedia contributors (2006), Sorghum plant has been cultivated in Southern Africa for over 3000 years. Sorghum bicolor is an African crop, which is widely distributed throughout the world. Different cultivars are found in different regions depending on the climate. It is adapted to a wider range of ecological conditions. It is mostly a plant of hot, dry regions, still survives in a cool weather as well as waterlogged habitat.

2.1.1 Ecology

Sorghums exhibit different heights and maturity dates depending on whether they are grain sorghums (Sorghum bicolor ssp. bicolor), forage sorghums (Sorghum bicolor), Sudan grass (*Sorghum bicolor sp. drummondii*), or sorghum-Sudan grass hybrids (Sorghum bicolor x Sorghum bicolor var. sudanense). Growth characteristics also vary depending on the location grown, inputs, and agronomic practices. In general, forage sorghums are taller plants with later maturity dates and more vegetative growth than grain sorghums. Sudangrass and sorghum Sudangrass hybrids fall in between grain sorghums and forage sorghums in height (Undersander, 2003).

It is an upright, short-day, summer annual that is a member of the Poaceae family. The grass blades are flat, stems are rigid, and there are no creeping rhizomes. Sorghum has a loose, open panicle of short, few-flowered racemes. As seed matures, the panicle may drop. Glumes vary in color from red or reddish brown to yellowish and are at least three quarters as long as the elliptical grain. The grain is predominately red or reddish brown (Kearney and Peebles, 1969; Barkworth, 2003).

Sorghum is planted from seed, usually in rows and in spring. As it is a little more frost hardy than maize it can be planted up until mid-summer if the rains are late. The seeds are planted 15 cm apart in rows and 3-5 cm deep in nature and watered if no rain falls to germinate the seed. After harvesting the stalks can be used for cattle food or fuel. It is best to practice crop rotation and grow sorghum on the same land every 4 years (www.mdidea.com, 2008)

2.1.2 Scientific Classification of Sorghum Bicolor

Kingdom	*Plantae* (plants)
Subkingdom	*Tracheaobionta*(vascular plants)
Superdivision	*Spermatophyta*(seed plants)
Division	*Magnoliophyta* (flowering plants)
Class	*Liliopsida* (monocotyledons)
Order	*Cyperales*
Family	*Poaceae* (grass family)
Subfamily	*Panicoideae*
Tribe	*Andropogoneae*
Genus	*Sorghum Moench* (Sorghum)
Species	*Sorghum bicolor* (L) Moench sorghum

2.1.3 Alternate Names of Sorghum bicolor

Alternate Common Names: sweet sorghum, sorgo forrajero (Spanish), durra (Africa), guinea corn, black amber, chicken corn, shattercane, wild cane, broomcorn, grain sorghum, forage sorghum, Sudangrass

The genus Sorghum is a genetically diverse with both wild (Sorghum halepense and Sorghum propinquum) and cultivated (Sorghum bicolor) species (Kimber, 2000). Select varieties of sorghum have considerably high concentrations of phenolic compounds and antioxidant capacities that are located primarily in the bran fraction of the grain. Flavonoids, phenolic acids and tannins are three phenolic categories found in sorghum. The flavonoids consist of anthocyanins, flavanols, flavones, and flavanones and the phenolic acids are benzoic and cinnamic derivatives (Awika *et al.*, 2005).

One type of anthocyanin that is unique to sorghum is 3-deoxyanthocyanin (Awika *et al.*, 2004). Sorghum contains only non-hydrolysable tannins as proanthocyanidins. Of the total anthocyanin content, nearly all black and brown varieties of sorghum contain 36-50% of apigeninidin & luteolinidin, two types of 3-deoxyanthocyanins (Dykes and Rooney, 2006). The proanthocyanidins are found in highest concentrations in brown varieties of sorghum. The phenolic, flavonoid and tannin contents vary greatly among the different varieties of sorghum.

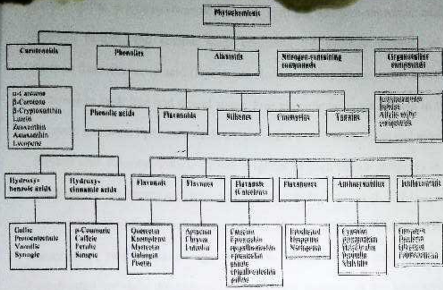

Figure 1.2: Classification of dietary phytochemicals. Adapted from Liu, R.H. J. Nutr. 2004 134:3479S-3485S.

2.1.4 Phytochemicals

Phytochemicals are defined as bioactive non-nutrient plant compounds in fruits, vegetables, grains and other plant foods that are linked to reducing the risk of many chronic diseases. More than 5000 phytochemicals have been identified but a large percentage still remains undocumented (Liu, 2004). Phenolic compounds are one of the most studied phytochemicals. Sorghum brans can contain large concentrations of phenolic compounds (Awika and Rooney, 2004).

Phenolic Acids: Phenolic acids are further divided into hydroxylbenzoic acids and hydroxycinnamic acids and are present in all types of cereal grains. Hydroxybenzoic acids are derived from benzoic acid and include gallic, p-hydroxybenzoic, vanillic, syringic, and protocatechuic acids. Caffeic, ferulic, and sinapic acids constitute the hydroxycinnamic acids derived from cinnamic acid (Dykes and Rooney, 2004). Phenolic acids occur in cereal in both free and bound states. Free phenolic acids are located in the pericarp and bound phenolic acids are esterified to cell walls in the endosperm (Dykes and Rooney, 2007). Brown sorghum (a high-tannin category) is a particularly rich source of ferulic and coumaric acids (Hahn et al., 1984).

Flavonoids: Flavonoids are compounds with a C6-C3-C6 skeleton that consists of two aromatic rings joined by a three carbon link. They are subdivided by their differences in the generic structure of the heterocycle carbon ring as flavonols, flavones, flavanols (catechins), flavanones, anthocyanidins, and isoflavonoids

Some of the common types of flavonoids found in fruits and vegetables include flavonols (quercetin, kaempferol, and myricetin), flavones (luteolin and apigenin), flavanols (catechin, epicatechin, epigallocatechin, epicatechin gallate, and epigallocatechin gallate), flavanones (naringenin), anthocyanidins, and isoflavonoids (genistein) (Liu, 2004). Flavonoids contribute the blue, purple and red colors in plants. Flavonoids possess various biological activities including anticancer, antimicrobial, antiviral, anti-inflammatory, immunomodulatory and antithrombotic effects (Kim et al., 2004).

There is not a vast amount of literature concerning identification of flavonoids in Sorghum. It is known, however, that different varieties of sorghum have significant differences in both their type and content of flavonoids. For example, black sorghum contains the highest amount of 3-deoxyanthocyanins, an anthocyanin lacking a C-3 hydroxylation in the C ring, when compared to red and brown varieties.

Additionally, 3-deoxyanthocanins apigeninidin and lutelinidin account for nearly 50% of the total anthocyanin content. The 3-deoxyanthocyanins are synthesized within the sorghum plant to help protect it from bacterial and fungal infections (Lo *et al.*, 1999).

These compounds may prove useful as natural food colorants as they have increased stability in acidic solutions compared to other common anthocyanins found in fruits and vegetables that are easily converted to their anthocyanidin counterpart (Dukes and Rooney, 2004). Evidence that anthocyanins in black sorghum are responsible for its high antioxidant activity was confirmed by a positive correlation between total anthocyanin content and antioxidant activity (Awika *et al.*, 2005).

Black sorghum bran was reported to contain the highest amount of flavan-4-ols compared to other varieties of sorghum with luteoforol and apiforol predominating (Dykes *et al.*, 2005).

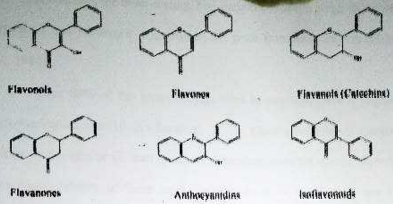

Figure 1.4: Structure of flavonoids. Adapted from Adapted from Liu; R.H. J. Nutr. 2004:134:3479S-3485S.

Tannins: Tannins convert animal skin to leather during the tanning process, hence their name. The two types are condensed tannins or proanthocyanidins and hydrolysable tannins. The proanthocyanidins in sorghum are mainly polymerized flavan-3-ol and/or flavan-3,4-diol units, consisting largely of catechin (88%) and epicatechin (12%) subunits Sumac bran (sorghum variety) contains nearly 4% tannins (Awika et al 2003); this stands in contrast to other varieties which may be relatively free of such molecules.

As a sorghum kernel matures, the flavonoid monomer units are condensed, forming oligomeric proanthocyanidin polymers. Due to the bitter taste of tannins in foods, many types of high-tannin sorghum may prove useful in astringent tasting foods such as dark chocolate.

In addition, the bitter-tasting, high-tannin content in sorghum imparts a degree of bird resistance and protects the grain from mold growth (Hahn *et al.*, 1984).

Over 60% of the proanthocyanindins in sorghum have a degree of polymerization over 10 (Awika *et al.*, 2003). Since absorption of molecules either by the skin or GI tract is largely dependent upon the size of molecules, the bioavailability of these large compounds is in question. It has been reported that proanthocyanindins with degrees of polymerization measuring less than 7 were readily "absorbed" in the intestinal epithelium cell monolayer.

Deprez *et al.*, (1999) showed that large polymers are degraded by the colonic microflora into lower molecular weight phenolic acid compounds that can be easily absorbed. The significance of these metabolites that are bioavailability renews interest in the biological effects of high molecular weight tannins. Condensed tannins have many beneficial pharmacological and biological effects; among these are significant anti-cancer, anti-inflammatory and antioxidant properties. Tannins have potent oxygen radical scavenging capabilities (Xu *et al.*, 2007; Claus *et al.*, 2006) and can effectively inhibit oxidation of lipids contained in LDLs (Sanchez-Moreno *et al.*, 2000).

Tannins have also been reported to inhibit platelet aggregation in vitro (Beecher, 1999) and are efficient iron and copper metal chelators, reducing oxidative damage to the myocardium (Beecher, 1999).

Phytosterols: Cholesterol-like compounds that are natural components of plant cell membranes are called phytosterols. Phytosterols mimic and compete with cholesterol absorption in the GI tract and can produce a cholesterol-lowering effect. These compounds are primarily found in the wax and bran fractions of cereal grains. Sitosterol, campesterol and stigmasterol have been identified in sorghum. Awika and Rooney, (2004) reported nearly 0.5 mg/g of phytosterols in sorghum while corn contains 0.9 mg/g.

2.1.5 Applications and Uses of Sorghum Bicolor

Sorghum bicolor has extensive applications and uses in health and medical aspect for example, in Folk Medicine it is reported to be antiabortive, cyanogenetic, demulcent, diuretic, emollient, intoxicant, and poison, sorghum is a folk remedy for cancer, epilepsy, flux, and stomachache (Duke and Wain, 1981).

The root is used for malaria in southern Rhodesia; the seed has been used for breast disease and diarrhea; the stem for tubercular swellings. In India, the plant is considered anthelminthic and insecticidal, and in South Africa, in combination with *Erigeron canadense L.*, it is used for eczema (Watt and Breyer-Brandwijk, 1962).

calcium, significant nitrogen, amino acids, fats in addition to various other constituents, vitamins and minerals, fiber and as well as carbohydrates. The whole plant may be used as medicine.

The seeds or grains are edible and they are a basic staple in Africa, the Middle East, West Indies as well as Latin America. The seeds are grounded into a meal, and it can also be grounded into flour to make bread. This can also be used to make porridge and other staple meals and they can be popped just like popcorn and eaten in the same manner. Noted as a forage, the grain is also used to feed animals. It is cultivated in the United States mainly for the feeding of animals and is seen as having almost the same nutritional values as corn.

2.2 NANOTECHNOLOGY

Nanotechnology is an important field of modern research dealing with design, synthesis, and manipulation of particles structure ranging from approximately 1-100 nm in one dimension. Remarkable growth in this up-and-coming technology has opened novel fundamental and applied frontiers, including the synthesis of nanoscale materials and exploration or utilization of their exotic physicochemical and optoelectronic properties.

Nanotechnology is rapidly gaining importance in a number of areas such as health care, cosmetics, food and feed, environmental health, mechanics, optics, biomedical sciences, chemical industries, electronics, space industries, drug-gene delivery, energy science, optoelectronics, catalysis, reorography, single electron transistors, light emitters, nonlinear optical devices, and photoelectrochemical applications (Colvin *et al.*, 1994; Wang, 1991).

Nanomaterials are seen as solution to many technological and environmental challenges in the field of solar energy conversion, catalysis, medicine, and water treatment. In the context of global efforts to reduce hazardous waste, the continuously increasing demand of nanomaterials must be accompanied by green synthesis methods.

Nanotechnology is fundamentally changing the way in which materials are synthesized and devices are fabricated. Incorporation of nanoscale building blocks into functional assemblies and further into multifunctional devices can be achieved through a "bottom-up approach". Research on the synthesis of nanosized material is of great interest because of their unique properties like optoelectronic, magnetic, and mechanical, which differs from bulk (Atul *et al.*, 2010).

2.2.1 Nanoparticles

The term "nanoparticles" is used to describe a particle with size in the range of 1nm-100nm, at least in one of the three possible dimensions. In this size range, the physical, chemical and biological properties of the nanoparticles changes in fundamental ways from the properties of both individual atoms/molecules and of the corresponding bulk materials.

Nanoparticles can be made of materials of diverse chemical nature, the most common being metals, metal oxides, silicates, non-oxide ceramics, polymers, organics, carbon and biomolecules.

Nanoparticles exist in several different morphologies such as spheres, cylinders, platelets, tubes etc. Generally the nanoparticles are designed with surface modifications tailored to meet the needs of specific applications they are going to be used for. The enormous diversity of the nanoparticles arising from their wide chemical nature, shape and morphologies, the medium in which the particles are present, the state of dispersion of the particles and most importantly, the numerous possible surface modifications the nanoparticles can be subjected to make this an important active field of science now-a-days.

Nanoparticles have been in use in pottery and medicine since ancient times. There are different ideal methods for nanoparticle to get synthesized. The following aspects involved for synthesizing nanoparticle are neutral pH, low cost and environmental friendly fashion.

Nanoparticles get produced by plants are more stable and the rate of synthesis is faster than that in other case of organism (Siavash Iravani and Behzad Zolfaghari, 2013). Mainly these methods for synthesizing nanoparticle have been developed in different methods in upcoming days because of the cost efficient and require little or no maintenance (Naheed Ahmad and Seema Sharma, 2012).

2.2.2 Types of nanoparticles:

Nanoparticles can be broadly grouped into two, namely, organic nanoparticles which include carbon nanoparticles (fullerenes) while, some of the inorganic nanoparticles include magnetic nanoparticles, noble metal nanoparticles (like gold and silver) and semi conductor nanoparticles (like titanium oxide and zinc oxide). There is a growing interest in inorganic nanoparticles i.e. of noble metal nanoparticles (Gold and silver) as they provide superior material properties with functional versatility.

Due to their size features and advantages over available chemical imaging drug agents and drugs, inorganic particles have been examined as potential tools for medical imaging as well as for treating diseases. Inorganic nonmaterial have been widely used for cellular delivery due to their versatile features like wide availability, rich functionality, good compatibility, and capability of targeted drug delivery and controlled release of drugs (Xu and Yu, 2006).

2.2.3 Optical Properties of Nanoparticles

One particular subset of nanoparticles is the quantum dot. These are generally particles with diameters of less than 10 nm, although in some cases the particle size may be as large as 50 nm. They have perhaps the most distinctive size-related properties of all nanoparticles. Quantum dots are semiconducting nanoparticles where their dimensions are so small the size of the particle affects the intrinsic band gap of the semiconductor.

A simple way to understand this is to consider a semiconductor as consisting of a valence and conduction band which are the result of the bonding and antibonding configuration of the crystal lattice. A simple diatomic system will have a single bonding and a single antibonding orbital. As more atoms are added to the lattice the new bonds will have slightly differentbonding and antibonding energies.

For a large lattice these orbitals will begin to form a continuum which is considered as the band in the final bulk material. It is therefore clear that at some point the number of bonds in the semiconductor particle will not be a good approximation to an infinite lattice and the band structure will begin to change.

This results in a widening of the bandgap of the semiconductor. Most quantum dots exhibit photoluminescent properties. A photon of incident light can excite an electron from the valence band of the semiconductor to the conduction band leaving a hole in the valence band. This photon then has various possible fates: recombination of the electron and hole with the emission of light, trapping of the electron/hole in a defect in the crystal, reaction with the capping agent resulting in the formation of a radical, reaction with the solvent to form a radical.

Many of these processes will also occur in particles which do not exhibit quantum confinement; however the minimum energy required to form the excited state will increase as the particle size decreases in a quantum-confined system. This minimum energy is the bandgap of the particle. This is a small but important area of nanotechnology, and the authors would direct the reader to some excellent reviews on the preparation and properties of quantum dots if more detail is required (Trindade *et al.*, 2001).

2.2.4 Phytofabrication of Nanoparticles

The term phytofabrication indicates the synthesis of the nanoparticles with the help of the plant constituents. Plant constituents include the enzymes and protein contents such as reductases which are involved in the biological reduction of the substrates, such as silver nitrate, aurum chloride, and titanium chloride, into their corresponding nanoparticles such as silver, gold, and titanium. These nanoparticles have a wide range of the applications in the fields of physical, chemical, material, and biological sciences.

2.2.5 Synthesis of Nanoparticles

Nanoparticles can be synthesized physically, chemically or biologically. Many adverse effects have been associated with chemical synthesis methods due to the presence of some toxic chemical absorbed on the surface. Eco friendly alternatives to Chemical and physical methods are Biological ways of nanoparticles synthesis using microorganisms(Klaus *et al.*, 1999; Konishi *et al.*, (2007), enzymes (Wilner and Wilner, 2006), fungus (Vigneshwaran *et al.*, 2007) and plants or plant extracts (Shanker, 2004; Ahmad *et al.*, 2011).

The development of these eco friendly methods for the synthesis of nanoparticles is evolving into an important branch of nanotechnology especially silver nanoparticles, which have many applications (Armendariz *et al.*, 2002; Kim *et al.*, 2010; Kyriacou *et al.*, 2004).

2.2.6 Biosynthesis of Nanoparticles

For the biosynthesis of nanoparticle - Preparation of botanical extracts, Bioreduction depends on reaction mixture and incubation time, Nanoparticles formation analysed by UV-Visible spectroscopy, Characterization of nanoparticles by SEM, TEM, XRD, FTIR, EXD, Purification and its application. There are basic steps involved in the biosynthesis of nanoparticles (Kavitha *et al.*, 2013; Jitendra *et al.*, 2014).

2.2.7 Characterization of Nanoparticles

Nanoparticles can be characterized by their shape, size, surface area and dispersity (Jiang *et al*., 2009). For their characterization some common techniques are used like UV-visible spectrophotometer, dynamic light scattering (DLS), scanning electron microscopy (SEM), transmission electron microscopy (TEM), Fourier transforms infrared spectroscopy (FTIR), X-ray diffraction (XRD) & energy dispersive spectroscopy (EDS) (Feldheim and Foss, 2002; Sepeur, 2008; Shaverdi *et al*., 2011), auger electron spectroscopy (AES), X-ray photoelectron spectroscopy (XPS), time of flight secondary ion mass spectrometry (TOF-SIMS), low energy ion scattering (LEIS), scanning tunneling microscopy (STM) and atomic force microscopy (AFM), scanning probe electron microscopy (SPM) etc.

UV-Vis spectrophotometer allows identification, characterization and analysis of metalic nanoparticles. Generally 300-800nm light wavelength is used for the characterization of size range 2 to 100nm (Feldheim and Foss, 2002). The dynamic light scattering (DLS) is used to characterize the surface charge & the size distribution of nanoparticles (Jiang *et al*., 2009).

Electron microscopy is a common method for surface and morphological characterization. Scanning electron microscopy (SEM) & transmission electron microscopy (TEM) are used for the morphological characterization at the nanometer to micrometer scale (Schaffer, 2009). SEM can provide morphological information on the submicron scale & elemental information at the micron scale, but TEM has a 1000 fold higher resolution compared with the SEM.

Characterization of nanoparticle using FTIR is very useful for the surface chemistry because the organic functional groups can be determined which are attached to the surface of nanoparticles (Chitrani *et al.*, 2006). XRD is used to examine the overall oxidation state of the particles as a function of time, i.e. phase identification & characterization of the crystal structure of the nanoparticles (Sun *et al.*, 2010).

To know the elemental composition of metal nanoparticles EDS is used (Strasser *et al.*, 2010). AES & XPS, TOFSIMS, scanning probe microscopy (SPM) techniques are important for the primary surface analysis of nanoparticles. AES and XPS are used to determine the presence, composition & thickness of coating on nanoparticles, surface enrichment and depletion at particle surfaces.

Sometimes XPS is used to determine particle sizes when conditions are not appropriate for analysis by other methods. TOF-SIMS is useful for obtaining molecular information about surface layers, functional groups which are added to the surface. In LEIS process the amount of energy lost by the ion during this scattering process due to a low energy ion beam can be determined.

This scattering process is used to determine the identity of the elements present in the outermost surface of the material under analysis. Recently it is found that it is useful due to its high sensitivity to the outermost atomic layers of a sample (Brongersma *et al.*, 2007). AFM and STM provide surface characterization at the atomic scale.

CHAPTER THREE

3.0 MATERIALS AND METHODS

3.1 MATERIALS

3.1.1 Plant Material

Sorghum bicolor **straw**

Dried straw of *sorghum bicolor* were the utilized biomass for this research. The plant was gotten from local market, Ijebu-Igbo, Ogun State, Nigeria. The area has two distinct seasons; rainy (March/April to October) and dry season (October/November to March/April) (Community portal of Nigeria, 2003). The plant material was identified and authenticated by a taxonomist in the department of Science Laboratory Technology of the Polytechnic.

3.1.2 Apparatus, Chemicals and Equipment

The following apparatus were used in this work: Whatman No.1 filter paper, funnel, pipette, wash glasses, measuring cylinders, standard volumetric flasks, refrigerator, and beakers.

The chemicals and reagents used in the experiments and solvent used for extraction of plant material, $AgNO_3$ and distilled-deionized water. All the chemicals used were of analytical grade. They were used as obtained.

The following pieces of equipment were used: Analytical balance, Water bath, UV-scanning spectrophotometer (Perkin Elmer, Lamda 25), Fourier Transform Infra-Red (FTIR).

3.2 METHODS

3.2.1 Preparation of Plant material

The dried straw of *sorghum bicolor* plant were carefully selected for the nanoparticles synthesis. The plant material was cleaned and air dried in the laboratory for 7 days, and stored in dry place for further analysis.

3.2.2 Extraction of Plant Sample Material

Approximately, 5 g of plant sample material was weighed using analytical balance. 100 cm^3 of distilled water was added to the plant material in a 250cm^3 beaker. The mixture was left for a period of 1hr for proper extraction to be done at room temperature. The mixture was then filtered with

Whatman No. 1 filter paper. The filtrate was collected and kept for nanoparticles - silver synthesis according to Ahmad and Sharma (2012).

Extraction Process

3.2.3 Synthesis of Silver Nanoparticles (Ag-NPs) using the Plant Extracts at 70°C

5 ml of the plant extract was added to 50 ml of the aqueous silver nitrate solution (2 mM). The resulting mixture was continuously stirred and gradually heated until reaction solution changed in colour. The bio-reduction of Ag^+ ions to AgO was monitored by taking samples (5ml) at 5, 10, 15, 20, 25 minutes intervals of reaction time. The plant extract acted as the reducing and stabilizing agents (Ahmad & Sharma, 2012) in this study.

Sample bottles containing synthesized silver nanoparticles

3.2.4 Characterization of Silver Nanoparticles

A Perkin Elmer UV-scanning spectrophotometer was used to determine the optical measurements, carried out at 450 nm wavelength range. Maximum absorption wavelength was determined by placing each aliquot sample taken at time intervals in quartz cuvette operated at a resolution of 1 nm, distilled-deionized water was used as reference solvent.

The photoluminescence of the biosynthesized nanoparticles was measured by Perkin-Elmer Lamda 25, spectrophotometer. The samples were placed in 1 x 1 cm quartz cell, operated at a resolution of 1 nm and maximum wavelength of 450 nm so as to determine the absorbance peak.

The purified and dried nanopowders were subjected to FT-IR analysis to identify possible functional groups which may support existence of capping and stabilization by the plant extracts.

CHAPTER FOUR

4.0 RESULTS AND DISCUSSION

4.1 RESULTS

4.1.1 UV Spectrometer absorbance result on the plant extract

Table 4.1: Absorbance of the extract at different time

TIME (mins)	ABSORBANCE
5	0.1611
10	0.155
15	0.08
20	0.131
25	0.19

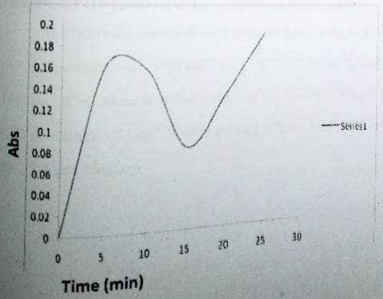

Figure 4.1: Absorbance of the plant extract against time

4.1.2 FTIR Result on the synthesized nanoparticles

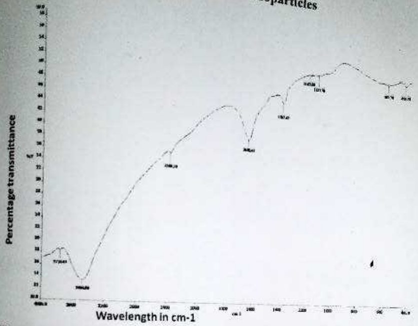

Figure 4.2: FTIR spectrum of Ag- plant extract after 5 minutes

The following absorption peaks were observed in the FTIR spectrum of $AgNO_3$ nanoparticles: 3752 cm^{-1}, 3464 cm^{-1} (O-H stretching), 2368 cm^{-1}, 1640 cm^{-1} (C=C alkene stretching), 1387 cm^{-1} (C=C-H stretching), 1167 cm^{-1} (C-O stretching), 1121 cm^{-1} (C-O stretching), 540 cm^{-1} (C-Br stretching), 453 cm^{-1} (C-I stretching).

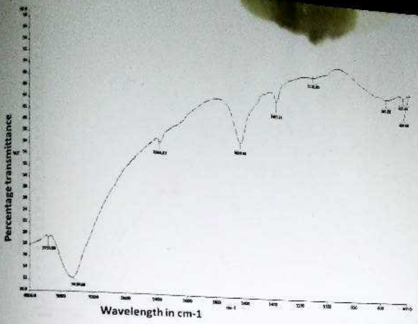

Figure 4.3: FTIR spectrum of Ag- plant extract after 10 minutes

The following absorption peaks were observed in the FTIR spectrum of $AgNO_3$ nanoparticles: 3758 cm^{-1}, 3459 cm^{-1} (O-H stretching), 2368 cm^{-1}, 1639 cm^{-1} (C=C alkene stretching), 1387 cm^{-1} (C=C-H stretching), 1121 cm^{-1} (C-O stretching), 581 cm^{-1} (C-Br stretching), 457 cm^{-1} (C-I stretching), 409 cm^{-1} (C-I).

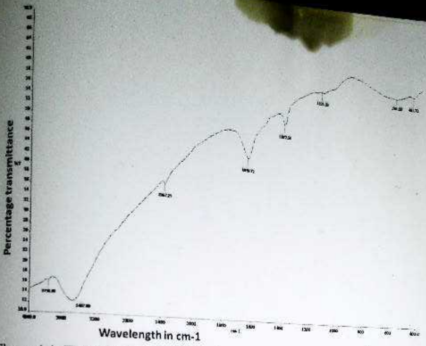

Figure 4.4: FTIR spectrum of Ag- plant extract after 15 minutes

The following absorption peaks were observed in the FTIR spectrum of $AgNO_3$ nanoparticles: 3758 cm^{-1}, 3467 cm^{-1} (O-H stretching), 2367 cm^{-1}, 1639 cm^{-1} (C=C alkene stretching), 1387 cm^{-1} (C=C-H stretching), 1129 cm^{-1} (C-O stretching), 583 cm^{-1} (C-Br stretching), 461 cm^{-1} (C-I stretching).

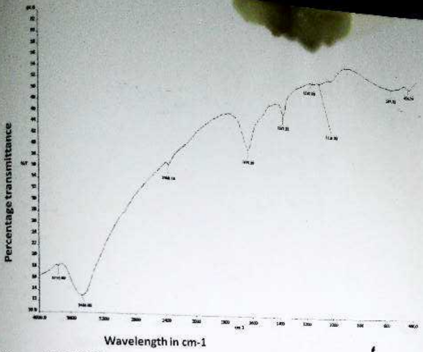

Figure 4.5: FTIR spectrum of Ag- plant extract after 20 minutes

The following absorption peaks were observed in the FTIR spectrum of $AgNO_3$ nanoparticles: 3755 cm^{-1}, 3469 cm^{-1} (O-H stretching), 2368 cm^{-1}, 1639 cm^{-1} (C=C alkene stretching), 1387 cm^{-1} (C=C-H stretching), 1167 cm^{-1} (C-O stretching), 1126 cm^{-1} (C-O stretching), 587 cm^{-1} (C-Br stretching), 456 cm^{-1} (C-I stretching).

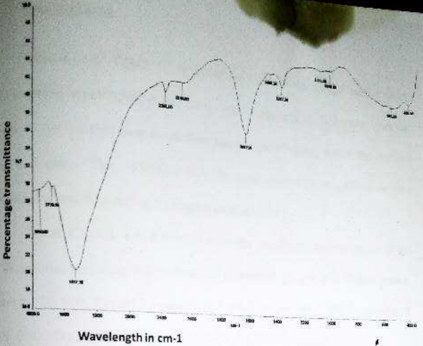

Figure 4.6: FTIR spectrum of Ag- plant extract after 25 minutes

The following absorption peaks were observed in the FTIR spectrum of $AgNO_3$ nanoparticles: 3910 cm^{-1}, 3750 cm^{-1}, 3457 cm^{-1} (O-H stretching), 2361 cm^{-1}, 2146cm^{-1} (C≡C alkyne stretching), 1637 cm^{-1} (C=C alkene stretching), 1468 cm^{-1} (C=C-H stretching), 1387 cm^{-1} (C=C-H stretching), 1111 cm^{-1} (C-O stretching), 1098 cm^{-1} (C-O stretching), 585 cm^{-1} (C-Br), 460 cm^{-1} (C-I).