Centre for Atmospheric Research

2018 MONOGRAPH OF ATMOSPHERIC RESEARCH

Edited by A.B. Rabiu and O. E. Abiye

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PREFACE

The Centre for Atmospheric Research was established in January 2013 with a compelling mission to improve our understanding of the behaviour of the entire spectrum of the Earth's atmosphere; promote capacity development in relevant atmospheric sciences as a way of facilitating international competitiveness in research being conducted by atmospheric scientists; and disseminate atmospheric data/products to users towards socio-economic development of the Nation. CAR's extant core research focus includes: space weather, tropospheric studies, atmospheric research software and instrumentation development, microgravity and human space technology, and atmospheric chemistry and environmental research.

Pursuant to the above, The *Monograph of Atmospheric Research* published by the Centre for Atmospheric Research (CAR), is a collection of peer-reviewed manuscripts in Atmospheric Sciences and closely related fields. This maiden edition comprises articles presented during two separate workshops; *1st National Workshop on Microgravity and Environmental Research* (26 - 29 November, 2017) and *1st National Workshop on Air Quality* (13 - 16 March, 2018). Such workshops are integral part of CAR's capacity building program and they were primarily aimed at advancing the course of atmospheric research in Nigeria towards sustainable development. The Microgravity workshop was geared towards introducing new research opportunities in space life science by simulating microgravity conditions here at the earth's surface as a means of investigation space biological environment. The Air Quality workshop was organized in collaboration with Ministry of Environment and Nigerian Meteorological Agency (NIMET). The workshop analysed current Air Quality scenario in Nigeria, explored new opportunities for collaborative research and offered novel means of improving the present quality of life of the populace without jeopardizing the chance of the future generation. Cumulatively 196 participants participated in these two workshops and about 52 articles were eventually submitted for publication consideration in this monograph. The twenty-one articles in this very monograph are the articles that eventually made it through the rigorous peer-review process. We remain grateful to the reviewers for doing thorough work on the articles.

Thus, we are very pleased to present the *2018 Monograph of Atmospheric Research* which contains twenty-one articles, including some review papers, to readers in all spheres of interest across Nigeria and beyond. It is our hope that this effort will continue and will serve as a reference to atmospheric researchers in Nigeria.

Prof. A. B. Rabiu and Dr. O. E. Abiye,
Editors

Centre for Atmospheric Research

Green synthesis of silver nanoparticles in a simulated microgravity condition

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ABSTRACT

In this report we synthesized silver nanoparticles in a one axis clinostat using Ocimum gratissimum leaf extract in order to examine the material properties in a simulated microgravity condition. The sample holder was carefully design to accommodate the mixing ratio of the leaf extract and the silver salt ($AgNO3$) used in the green synthesis. The clinostat was allowed to run for 2hrs, 24hrs, 48hrs and 72hrs respectively. A scanning electron microscope (SEM) equipped with an EDX attachment and a transmission electron microscope (TEM) were used to characterize the nanoparticles surface morphology and measure the size. TEM measurements were performed on a JEOL TEM 1010 transmission electron microscope at 200kV. Carl Zeiss ultra plus field emission scanning electron microscope (FESEM) was used at 5 kV accelerating voltage. The crystallinity of the AgNPs of microgravity and gravity was examined using an X-ray diffractometer with monochromatic CuK radiation (= 1.5406 Å) operating at a voltage of 40 kV and a current of 30 mA at room temperature. The intensity data for the AgNPs was collected over a 2θ range of 200 – 900. Our results agree with existing work that in both environments silver nanoparticles can be synthesized but with some modification in the crystallites sizes, shapes, inter planar spacing between the atoms and elemental compositions in the case of MG AgNPs. The particle size, the dislocation density and specific surface area show that the materials are useful in heterogeneous catalysis and adsorptions.

Key words: synthesis; microgravity; material characterization; nanotechnology; gravity

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INTRODUCTION

Nanotechnology has enabled material synthesis of novel behaviour and applications. Nanotechnology can be achieved using physical, chemical and biological approaches. Biological approach using plant extracts are preferred to chemical, physical and microbial routes because elaborated process of culturing and maintaining the cell, using hazardous chemicals, high-energy and wasteful purifications are eliminated (*Awwad et al., 2013; Dare et al., 2015*).

Recently, efforts have been geared toward understanding the physics of crystal formation in a condition of zero or reduced (micro) gravity. Silver nanoparticles have been utilized as antibacterial and antiviral agents in water purification, medical devices, cosmetics and electronics (*Mostafa et al., 2014; Han et al., 2016*). The importance of gravity as experimental parameter was recognized over 46 years ago when first exploratory experiments were conducted on the return flight of Apollo missions from the moon. Many processes used to produce solid materials on earth started from liquid state. Gravity effects on the microstructure of these materials usually influence their physical properties.

In this study silver nanoparticles are biosynthesized using Ocimum gratissimum aqueous leaf extract in a simulated microgravity condition using one axis clinostat.

MATERIAL AND METHOD

Collection And Preparation Of Plant Material

Fresh Ocimum leaves were collected from a small farm yard, at Shendam road, Lafia. Nasarawa State, Nigeria. The leaves were washed thoroughly with double distilled water (DDW) and cut into small pieces. Approximately 5 g of the leaves was then boiled with 100 mL of DDW for 10 minutes in a 250 mL beaker and filtered through a Whatman number 1 filter paper. The filtrate was then stored in a refrigerator further use.

Synthesis Of Silver Nanoparticles

In these syntheses, the leaf extract was added to 1 mM of $AgNO3$ solution, in the ratio 1:4. 100mL of silver nitrate solution was introduced into the sample holder and 25mL of aqueous Ocimum leaf extract

Microgravity procedure

A specialized sample holder suitable to fit on the clinostat was constructed and great effort was made to remove bubble which could create extraneous forces unaccounted for during clinorotation (*Russomano et al., 2008*). This was achieved by a careful measure of the exact silver nitrate solution and aqueous Ocimum gratissimum leaves extract that made up our mixing ratio without bubbles. The clinostat was set to rotate at 50rpm immediately the leaf extract was added to the silver nitrate solution. The reaction mixture was then subjected to clinorotation for 2hrs, 24hrs 48hrs, 72hrs respectively.

Gravity procedure

The reaction mixture for the synthesis of silver nanoparticles was allowed to incubate at 20oC for 2 hr, 24 hr and 72 hr respectively. The solution changed to a reddish-brown color, indicating the formation of AgNPs (*Adesuji et al., 2016*).

Separation and Purification of Silver Nanoparticles (AgNPs)

The AgNPs synthesized under microgravity and gravity conditions respectively using Ocimum gratissimum were repeatedly centrifuged at 15,000rpm for ten minutes. The centrifugation was repeated three times. This was done to obtain silver nanoparticles that are free of any adsorbed substances.

Characterization of AgNPs

The synthesis of AgNPs under microgravity and gravity conditions at 2, 24 and 72 hr was monitored by UV-Vis spectroscopy. The wavelength ranged between 200 and 900 nm using the T60 UV-Visible spectrophotometer at a resolution of 1nm. A scanning electron microscope (SEM) equipped with an EDX attachment and a transmission electron microscope (TEM) were used to characterize the nanoparticles surface morphology and measure the size. TEM measurements were performed on a JEOL TEM 1010 transmission electron microscope at 200kV. Carl Zeiss ultra plus field emission scanning electron microscope (FESEM) was used at 5 kV accelerating voltage.

Samples were placed on aluminum stabs using carbon tape. The crystallinity of the AgNPs of microgravity and gravity was examined using an X-ray diffractometer with monochromatic CuK radiation (= 1.5406 Å) operating at a voltage of 40 kV and a current of 30 mA at room temperature. The intensity (AU) data for the AgNPs was collected over a 2θ range of 20° – 90°. The particle size was obtained using Debye Scherrer's equation.

$$D = \frac{0.94\lambda}{\beta cos\theta} \quad (1)$$

where

$\beta = FWHW$ (Full width at half maximum)

$\lambda = 0.1542nm$

The crystallite size of AgNPs was estimated using Debye Scherrer's formula in equation 1 and the results presented in Table 1 and 2.

Inter planar Spacing between the atoms were determine using Braggs law (equation 2).

$$d(inter\ planar\ spacing) = \frac{n\lambda}{2sin\theta} \quad (2)$$

Specific surface area (SSA)

This is a derived scientific value of the material property that can be used to determine the type and behaviour of a material. Specific surface area is importance in understanding material adsorption, heterogeneous catalysis and reactions on surfaces.

Energy dispersive X-ray spectroscopy

This is used to quantify the chemical composition of nano

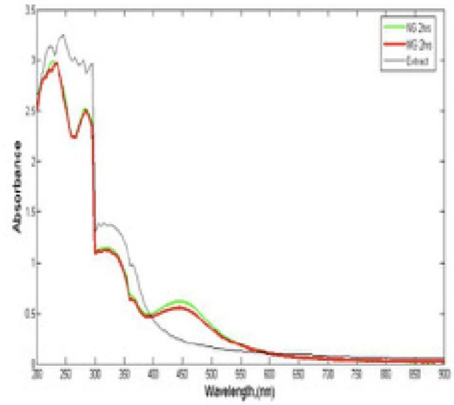

Figure 1: UV-Vis of AgNPs synthesized at normal gravity (NG) and microgravity (MG) conditions at 2 hours

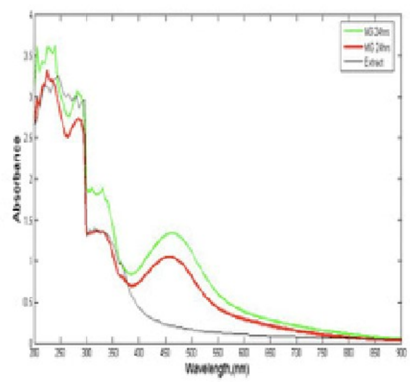

Figure 2: UV-Vis of AgNPs synthesized at normal gravity (NG) and microgravity (MG) conditions at 24 hours

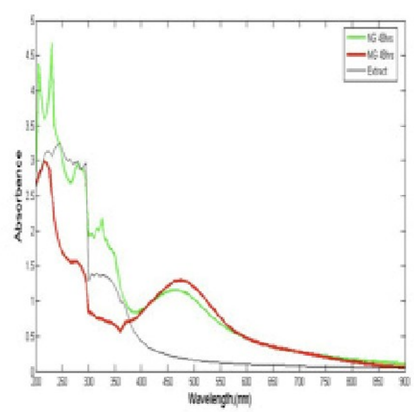

Figure 3: UV-Vis of AgNPs synthesized at normal gravity (NG) and microgravity (MG) conditions at 48 hours

Table 1: NG AgNPs Solid State Properties

2Theta (degree)	FWHW	D (nm)	d (nm)
38.25000	0.00358	42.14	0.235
44.40000	0.00628	23.59	0.204
64.57000	0.00489	46.19	0.114
77.53000	0.00611	49.49	0.123
81.60000	0.00716	20.26	0.118

Average grain size = 36.33nm (NG AgNPs)

Table 2: MG AgNPs Solid State Properties

2Theta (degree)	FWHW	D (nm)	d (nm)
38.51000	0.00445	35.45	0.196
44.67000	0.00689	22.39	0.226
64.83000	0.00550	49.09	0.091
77.73000	0.00663	56.16	0.084
81.82000	0.00724	20.07	1.114

Average grain size = 36.63nm (MG AgNPs)

materials (Dada et al. 2015). The silver nanoparticles synthesized under normal gravity and microgravity conditions respectively were characterized for elemental compositions.

RESULT AND DISCUSSION

UV-VIS

The colour of the solutions changed from pale yellow to reddish brown. This indicate the formation of silver nanoparticles. The surface plasmon resonance (SPR) band for MG AgNPs was observed at 445 nm at 2 hr (Figure 1), 475 nm at 24 and 48 hr (Figure 2 and 3) respectively, while that of NG AgNPs peaked at 465nm. The size, shape, state of aggregation, morphology, composition, and the dielectric medium of the nanoparticles affect the SPR. Collective oscillation of surface electrons in AgNPs is based on the absorption of light at the visible region of the electromagnetic radiation.

This shows that the formation of silver nanoparticles was not determined by gravity at longer reaction time. The observed difference in the intensity of absorption may be ascribed to the increase in the number of AgNPs in the reaction medium (Tagad et al., 2013). It is suggested that the observed difference in the intensity of absorption is a reflection of the number of AgNPs formed under gravity and microgravity conditions, respectively.

XRD

Crystallites Sizes

The results of the crystallites are:

Average grain size = 36.33nm (NG AgNPs)

Average grain size = 36.63nm (MG AgNPs)

As reported in Table 1 and 2.

XRD Dislocation Density

Dislocation is a crystallographic defect or irregularity within a crystal structure. The dislocation density of the nanoparticles were as given below:

NG AgNPs dislocation density = 15.14 X 10 14m-2 and

MG AgNPs dislocation density = 16.03

The result above shows that MG AgNPs has the highest dislocation density of 16.03 while NG AgNPs has 15.14 X 10 14m-2 dislocation density.

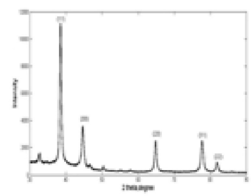

Figure 4: XRD spectrum of MG AgNPs

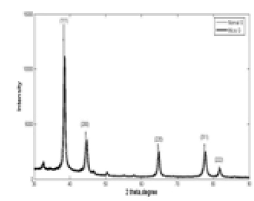

Figure 6: Superimposed XRD of NG and MG AgNPs

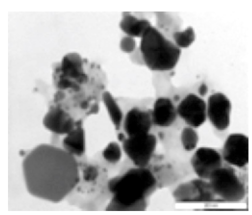

Figure 8: MG AgNPs TEM Micrograph

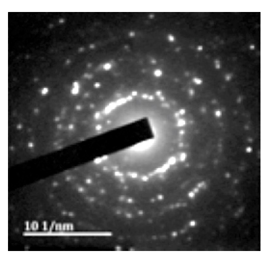

Figure 10: Selected area electron diffraction of NG AgNPs

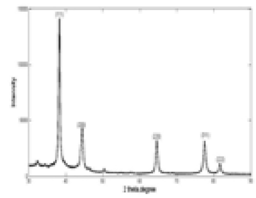

Figure 5: XRD spectrum of NG AgNPs

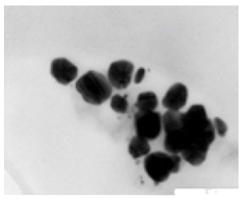

Figure 7: NG AgNPs TEM Micrograph

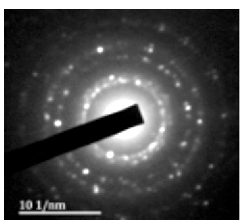

Figure 9: Selected area electron diffraction of MG AgNPs

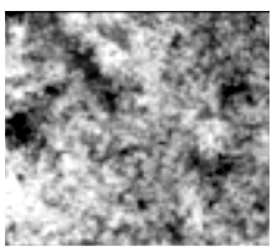

Figure 11: Energy dispersive X-ray of MG AgNPs

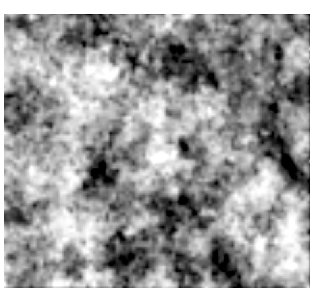

Figure 12: Energy dispersive X-ray of NG AgNPs

Specific Surface area (S)

The Surface area of the two samples *(NG AgNPs and MG AgNPs)* was calculated using Sauter relation below

$$\mathbf{S} = \frac{6 \times 10^3}{grain\ size \times density\ of\ silver} \quad (3)$$

Density of silver = 10.5 g/m^3 *(adopted from Jo Yong-park et al., 2006)*
With the above relation the Specific Surface area of the two syntheses were calculated as given below:
Specific Surface area for NG AgNPs = 15728.84 m2/g
Specific Surface area for MG AgNPs = 15600.02 m2/g
Both samples have a very large surface area which could make them suitable for heterogeneous catalysis and reactions in chemical transformation.

TEM

The TEM images showed that NG AgNPs are mostly spherical and not well distributed while that of MG AgNPs showed shapes such as prism, hexagonal, nanowire and spherical (Figure 8) and sparsely distributed.***Energy dispersive X-ray (EDX)***

The count per seconds at 3 keV confirmed the formation of silver nanoparticles at normal and microgravity conditions respectively.

Table 3: Elemental compositions MG AgNPs

uG_1	Wt%	Wt% Sigma
C	12.51	0.25
N	1.81	0.83
O	2.40	0.27
Cl	0.94	0.04
Ag	82.68	0.76
Total	100.00	

Table 4: Elemental compositions NG AgNPs

uG_1	Wt%	Wt% Sigma
C	5.26	0.31
N	0.01	1.37
O	0.76	0.47
Ag	93.97	1.40
Total	100.00	

The surface plasmon resonance of silver nanoparticles accounts for the typical absorption at 3 keV *(Oluwaniyi et al., 2016)*. The EDX spectrum showed high percentage of silver atom in the nanoparticles biosynthesized both in normal and microgravity conditions. The gravity condition gave silver atom to be 93.97 % while microgravity condition showed silver atom as 82.68 % this show that silver atom are readily populated under normal gravity during nano synthesis than in microgravity condition although in both environment silver nanoparticles is produced. The presence of C, O, N and Cl are from the biomolecules attached to the silver nanoparticles.

CONCLUSION

We have synthesized and characterized AgNPs both in the condition of normal gravity and in a simulated microgravity. Our results agree with existing work that in both environments silver nanoparticles can be synthesized but with some modification in the crystallites sizes, shapes, inter planar spacing between the atoms and elemental compositions in the case of MG AgNPs.

The dislocation density and the specific surface area show that both materials are useful in heterogeneous catalysis and adsorptions.

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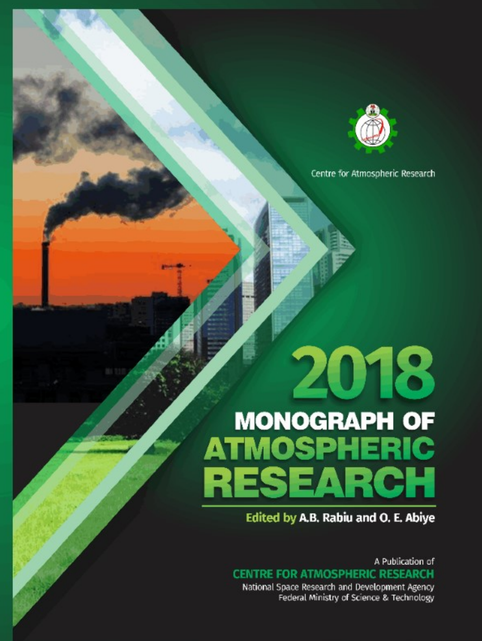

Our Mandates

The Center for Atmospheric Research, CAR, is a research and development center of NASRDA committed to research and capacity building in the atmospheric and related sciences. CAR shall be dedicated to understanding the atmosphere—the air around us—and the interconnected processes that make up the Earth system, from the ocean floor through the ionosphere to the Sun's core. The Center for Atmospheric Research provides research facilities, and services for the atmospheric and Earth sciences community.

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