PREPARATION, CHARACTERIZATION AND DETERMINATION OF ANTIMICROBIAL ACTIVITIES OF ($MgNiO_2$) NANOPARTICLES USING OXALIC ACID

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ABSTRACT - The present research deals with a study on the synthesis and characterization of $MgNiO_2$ nanoparticles. The nanoparticles was synthesized by sol-gel method using oxalic acid. Magnesium nitrate and nickel nitrate were used to obtain homogeneous solution. The gel powder was dried in an oven at temperature of 105˚C to obtain constant weight. The resulting sample was thermally heated in muffle furnace at temperature of 500˚C for four hours to get required mixed metals oxide nanoparticles. The prepared nanoparticle was characterized by Energy Dispersive X-ray Fluorescence (EDXRF) and X-ray powder diffraction (XRD). Then determination of antimicrobial activities of ($MgNiO_2$) nanoparticles was carried out by Agar Well Diffusion Method. According to the XRD analyzed data, the average particle sizes of $MgNiO_2$ nanoparticles was 14.70 nm.

Key words : **nanoparticles, sol-gel method, XRD, EDXRF and Agar Well Diffusion Method**

1. INTRODUCTION

In current years, researchers from the developed countries have focused to research various types of nanoparticles. Many questions may be asked about the nanoparticles. Among them, two questions should be asked and could be solved about nanoparticles. Firstly, what is nanoparticle? Secondly, why are researchers from many countries focusing on nanoparticle? Nanoparticle is a small particle that ranges between 1 to 100 nanometers in size. Undetectable by humans' eye, nanoparticles can exhibit significantly different physical and chemical properties to their large material counterparts[Gunter Schmid (Aug 25,2010)]. The material properties change as their size approaches the atomic scale. This is due to surface area to volume ratio increasing, resulting in material surface atoms dominating the material performance[Gunter Schmid (Aug 25,2010)]. Owing to their very small size, nanoparticles have a very large surface area to volume ratio when compared to bulk material, such as powders, plate and sheet [Gunter Schmid(Aug 25,2010)]. This feature enables nanoparticles to possess unexpected optical, physical and chemical properties, as they are small enough to confine their electrons and produce quantum effects[Gunter Schmid(Aug 25,2010)]. For example, copper is considered a soft material, with bulk copper bending when its atoms cluster at the 50nm scale. Consequently, copper nanoparticles smaller than 50nm are considered a very hard material, with drastically different malleability and ductility performance when compared to bulk copper[Gunter Schmid(Aug 25,2010)]. The change in size can also affect the melting characteristics; gold nanoparticles melt at much lower temperatures (300^0C for 2.5nm size) than bulk gold (1064^0C). Moreover, absorption of solar radiation is much higher in materials composed of nanoparticles than in thin films of continuous sheets of material[Gunter Schmid (Aug 25,2010)].

The use of nanomaterials spans across a wide variety of industries, from healthcare and cosmetics to environmental preservation and air purification[Gunter Schmid(Aug 25,2010)]. The healthcare field, for example, utilizes nanomaterials in a variety of ways, with one major use being drug delivery[Gunter Schmid(Aug 25,2010)]. One example of this process is whereby nanoparticles are being developed to assist the transportion of chemotherapy drugs directly to cancerous growths as well as to deliver drugs to areas of arteries that are damaged in order to fight cardiovascular disease[Gunter Schmid(Aug 25,2010)]. In aerospace, carbon nanotubes can be used in the morphing of aircraft wings. The nanotubes are used in a composite form to bend in response to the application of an electric voltage. Elsewhere, environmental preservation processes make use of nanomaterials too, such as nanowires. Applications of nanowires are being developed in flexible solar cells as well as to play a role in the treatment of polluted water. Absorption of solar radiation is much higher in materials composed of nanoparticles than in thin film of continuous sheets of material[Gunter Schmid(Aug 25,2010)]. In the cosmetic industry, mineral nanoparticles, such as titanium oxide are used in sunscreen for UV protection. The sports industry has been producing baseball bats that have been made with carbon nanotubes, making the bats lighter and therefore improving their performance. Furthermore, nanomaterials can be used in antimicrobial nanotechnology. Towels and mats used by sport peoples are made of nanomaterials in order to prevent illnesses caused by bacteria [Gunter Schmid(Aug 25,2010)]. Nanomaterials have also been developed for use in the military. One example is the use of mobile pigment nanoparticles being used to produce a better form of camouflage, through injection of the particles into the materials of soldiers' uniforms[Gunter Schmid(Aug 25,2010)].

Gradually, primary resources such as fossil fuel, coal and natural gas are depleting, while the global energy consumption is increasing. Solar energy, wind energy, biomass, tidal and geothermal resources is emerging as an answer to our energy starved planet. These renewable energy sources are freely available in

nature, non-polluting and help in reducing global CO_2 emissions. Out of the mix of available sources of energy, solar energy is the cleanest and the most abundant. We focused on different solar technologies and materials that can be used to make an efficient photovoltaic cell to solve the energy problem. Available pv cells can be broadly classified into first, second and third generation solar cells. First generation cells are basically silicon based crystalline cells while second generation cells are thin film based and third generation cells comprise new emerging technologies. Solar cells used for power generation must possess certain characteristic like high efficiency, low cost materials , simple fabrication technique , ease of solar panel installation and long term stability.

In the recent years, researchers have focused on the development of cost-effective and feasible non-silicon solar cell technologies. Magnesium-ion batteries are considered as a new rechargeable battery system comparable to lithium-ion batteries. Magnesium that would be used as an active material for an anode is possible to provide a considerably high energy density due to its high electrochemical equivalence and considerably negatively electrode potential. Therefore, if an excellent cathode material can be sought somehow, rechargeable magnesium-ion batteries can be realized in future . In this research work, we focused on a synthesis and characterization of ($MgNiO_2$) nanoparticles.

II. MATERIALS AND METHODS

All chemicals were analytical grade. Magnesium nitrate [$Mg(NO_3)_2.6H_2O$], Nickel nitrate [$Ni(NO_3)_2.6H_2O$] and oxalic acid ($C_2H_2O_4$) were Merck product with a purity of 99.99%. Ethylene glycol was product from Applichem, Germany. All solutions were prepared using distilled water during preparation procedures. Various conventional and modern instrumental techniques were used throughout the experimental procedure. These include X-ray Diffraction (XRD) and Energy Dispersive X-ray Fluorescence (EDXRF).

Preparation of $MgNiO_2$ nanoparticles using oxalic acid

5.12 g of magnesium nitrate $Mg(NO_3)_2.6H_2O$ was dissolved in 50 mL of distilled water to obtain solution A. Solution B was prepared by dissolving 5.8 gm of $[Ni(NO_3)_2.6H_2O]$ in 50 mL of distilled water. The solution A and B were mixed to obtain solution(I). 2.5 g of oxalic acid was dissolved in 100 mL of distilled water to get solution (II). Solution(I) and (II) were mixed together with 10mLof ethylene glycol to obtain a homogeneous mixture solution. The mixture solution was stirred and heated on a magnetic stirrer hot plate at 70-80˚C for 2 hours. The gel-powder was obtained. The gel-powder was dried in an oven at 105˚C until constant weight was obtained. The dried as prepared powder was calcined in a muffle furnace at 500˚C for 4 hours to obtain $MgNiO_2$ nanoparticles (MN-1).

Characterization of ($MgNiO_2$) nanoparticles

Crystal structure and phase analysis were performed by X-ray diffraction (XRD) using Rigaku, D-Max 2200, Japan in Department of Chemistry, Yangon University. The elemental compositions of the prepared sample was confirmed by using EDXRF 700 spectrometer in Department of Chemistry, Monywa University.

Determination of Crystallite Size and Interatomic Spacing

The crystallite sizes of ($MgNiO_2$) can be calculated by using Debye-Scherrer' formula,

$D = 0.9\lambda/\beta\cos\theta$ and interatomic spacing by Bragg's equation $d = \lambda/2\sin\theta$

Where, λ = the wave length of X-rays (1.540560A for Cu/K-alpha 1)

`θ = the diffraction angle

β = full width at half maximum in radian

D =average crystal size

d = interatomic spacing

Determination of Antimicrobial Activities of ($MgNiO_2$) Nanoparticles

Table(1) Types of Microorganisms and Infection Diseases

No	Test Organism	Infection Diseases
1	*Bacillus pumilus* IFO 12102	Fever
2	*Bacillus subtilus* IFO 90571	Fever
3	*Candida albicans* NITE 09542	Candidasis, Skin disease
4	*Escherichia coli* AUH 5436	diarrhoea
5	*Staphylococcus aureus* AUH 8465	Food poisoning
6	*Salmonella typhi* AUH 7943	Typhoid fever

Chemicals and reagents

Glucose, yeast extract, peptone, Agar, Distilled water.

Apparatus and equipments

Outoclave, An incubator, hot plate, Petri-dishes, measuring cylinder, micropipette and clipper.

Screening of Antimicrobial Activity of Different Crude Extract by Using Agar Well Diffusion Method (Collin, 1965)

Glucose 0.5g, Yeast extract 0.3g, Peptone 0.3g, Agar 1.7g, 100mL of distilled water were added in a 250mL sterile conical flask and heated on hot plate until boil medium. Then, the mouth of the flask was plugged with a piece of cotton wool. This medium was sterilized in an autoclave at 121^0C for 45 minutes. After 45 minutes, a 0.1 mL test organisms were inoculated in to 20mL of medium agar at about 40^0C and were poured into the sterile petri- dishes at aseptic condition. After the agar become solid, cock borer was used to make the wells (8mm in diameter). Then extract samples (20µL) were introduced into the well and they were incubated at room temperature for 24-48 hours. After 24-48 hours of incubation, the clear zones were measured. Clear zone surrounding the wells indicated the presence of antimicrobial active compound in the extracts which inhibit the growth of the test organisms.

Reference:

Colin, C.H. 1965. Microbiallogical Methods. Butterworth and Co, Publishers Ltd, Landon.

III. RESULTS AND DISCUSSION

Characterization of ($MgNiO_2$) Nanoparticles

EDXRF Analysis of ($MgNiO_2$) Nanoparticels

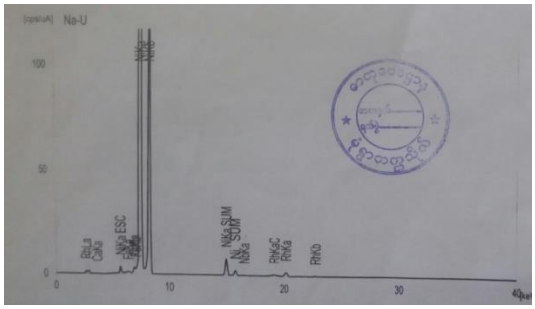

Fig : 1 EDXRF spectrum of $MgNiO_2$ nanoparticles

Table (2) Quantitative Results of $MgNiO_2$ Nanoparticles by EDXRF

No	Analytes	Results (%)
1	Cu	99.647
2	Ni	0.223
3	Ca	0.075
4	Fe	0.033
5	Nb	0.022

From the EDXRF data, amount of Cu, Ni, Ca, Fe, and Nb in prepared nanoparticles $MgNiO_2$ were found to be 99.647% , 0.223%,0.075%, 0.033% and 0.022% respectively.

XRD Analysis of ($MgNiO_2$) Nanoparticles

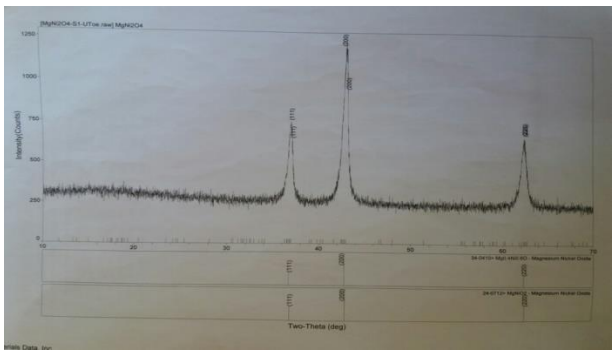

Figure : 2 XRD Diffractogram of $MgNiO_2$ nanoparticles

Table (3) The Particles Size of $MgNiO_2$ Nanoparticles

No	Bragg angle (2θ)	Miller indices (h k I)	(β) radiation	Inter planar spacing d (nm)	Particle size D (nm)
1	36.690	111	0.0095	0.24473	15.4
2	36.779	111	0.0091	0.24416	16.12
3	42.701	200	0.0100	0.21158	14.91
4	42.750	200	0.0097	0.21134	15.41
5	62.180	220	0.0128	0.14917	12.61
6	62.270	220	0.0118	0.14897	13.73

Range of crystalline size = 12.61 – 16.12 nm
Average value = 14.70 nm

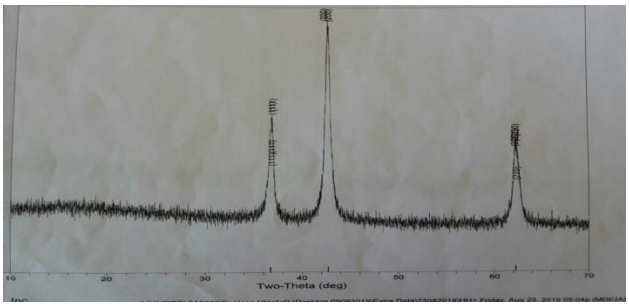

Figure : 3 XRD Diffractogram of $MgNiO_2$ nanoparticles

Table (4) The phase ID Report of $MgNiO_2$ Nanoparticles

No	Bragg angle (2θ)	Miller indices (h k I)	Area (%)	Inter planar spacing d (nm)	Phase ID
1	36.690	111	48.1	0.24473	MgO.4NiO.6O
2	36.779	111	48.4	0.24416	$MgNiO_2$
3	42.701	200	100.0	0.21158	$MgNiO_2$
4	42.750	200	99.9	0.21134	MgO.4NiO.6O
5	62.180	220	45.9	0.14917	$MgNiO_2$
6	62.270	220	50.4	0.14897	MgO.4NiO.6O

Prepared nanoparticles were MgO 4NiO.6O, and $MgNiO_2$.

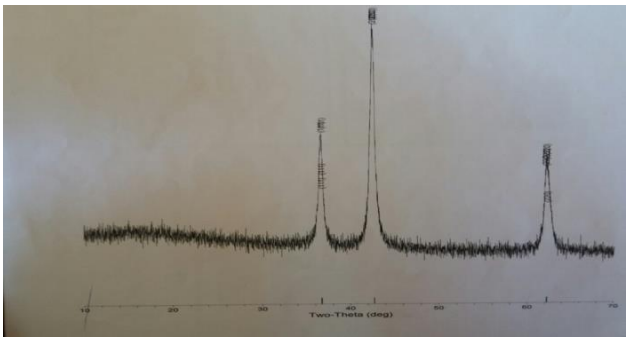

Figure : 4 XRD Diffractogram of $MgNiO_2$ nanoparticles

Table (5) Lattice Constants from Peak Locations and Miller Indices for $MgNiO_2$

No	Bragg angle (2θ)	Miller indices (h k I)	Inter planar spacing d (nm)	a-axis (nm)	b-axis (nm)	c-axis (nm)
1	36.690	111	0.24473	0.42389	0.42389	0.42389
2	36.779	111	0.24416	0.42290	0.42290	0.42290
3	42.701	200	0.21158	0.42315	0.42315	0.42315
5	62.180	220	0.14917	0.42191	0.42191	0.42191
6	62.270	220	0.14897	0.42136	0.42136	0.42136

Average lattice constant = 0.42264 nm
Average lattice constants calculated from XRD pattern for $MgNiO_2$ nanoparticles were a = b = c = 0.42264 nm. The crystal structure is cubic.

Determination of Antimicrobial Activities of ($MgNiO_2$) Nanoparticles

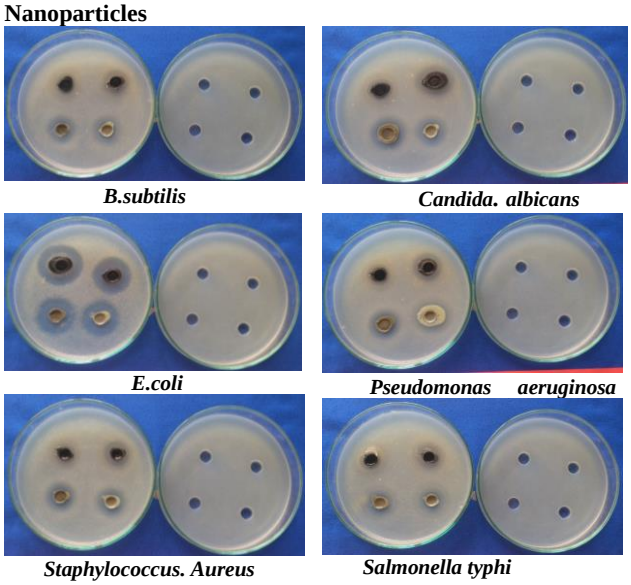

Figure: 5 Screening of antimicrobial activities of ($MgNiO_2$) nanoparticles

In the figure, right dish of each pair shows activity of blank solution(only distilled water). The left holes of lower pairs in left dishes show activity of $MgNiO_2$.

Table (6) Results of Screening of Antimicrobial Activities of ($MgNiO_2$) Nanoparticles

No	Microorganisms	Inhibition zone diameter of samples ($MgNiO_2$) (mm)
1	*Bacillus subtilis* IFO 90571	-
2	*Candida albicans* NITE09542	16.48(++)
3	*Escherichia coli* AUH 5436	24.22(+++)
4	*Pseudomonas aeruginosa*	14.68(+)
5	*Staphylococcus aureus* AUH 8465	14.06(+)
6	*Salmonella typhi* AUH 7943	13.52(+)

(-) No activity
(+) 9-14mm, Low activity
(++) 15-20mm, Medium activity
(+++) 21mm-above, High activity

IV. CONCLUSION

In this research, $MgNiO_2$ nanoparticles was synthesized from magnesium nitrate and nickel nitrate by sol gel method using oxalic acid. The prepared nanoparticles was characterized by modern sophisticated methods such as EDXRF and XRD. From the EDXRF data, The amount of nickel in $MgNiO_2$ was found to be 99.647%.From the XRD results, the average crystalline size of prepared nanoparticles for $MgNiO_2$ was found to be 14.70 nm. The crystal structures of $MgNiO_2$ nanoparticles was cubic.

The $MgNiO_2$ nanoparticle was also detected in antimicrobial activities by Agar Well Diffusion Method (Collin,1965). According to the screening of antimicrobial activities of crude extract by using Agar Well Diffusion Method, $MgNiO_2$ showed no activity in *Bacillus subtilisas*, but medium activity in *Candida albicans* as well as high activity in *Escherchia coli* and low activity in *Pseudomonas aeruginosa*, *Staphylococcus aureus* and *Salmonella typhi*.

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