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Microwave Assisted Synthesis of Nickel NanoparticleUsing Hydrazine Hydrate and Its Antimicrobial Activity.

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Abstract : This study focuses on the synthesis and characterization of nickel nanoparticles. Here, chemical reduction technique is used to synthesize nickel nanoparticles using hydrazine hydrate as the reducing agent, nickel chloride hexahydrate as the precursor and polyvinyl pyrrolidone as the capping agent in water medium. The synthesis is carried out within 5 minutes under microwave irradiation at warm condition. In this process the oxidation state of nickel changes from Ni^{+2} to Ni^{0} and there is a colour change from royal blue to black. UV spectrum of this nanoparticle showed a broad peak at 286 nm. Morphological studies were performed using scanning electron microscopy (SEM) and the elemental composition was determined using energy dispersive X-ray analysis (EDAX). The antimicrobial activity for the synthesized nickel nanoparticles was also examined.

Keywords: nickel nanoparticles, chemical reduction, UV, SEM, EDAX and antimicrobial studies.

Introduction

In recent years, the synthesis of nanoscale materials has gained much importance due to their unusual fascinating properties with various applications. Nanoscale particles are the ultrafine particles with their size ranging from 1-100 nm in at least one dimension. Among the various transition metal nanoparticles, the synthesis of nickel nanoparticles is challenging as they are highly unstable[1].Nickel is a highly useful metal with major uses in stainless steel, metal alloys, plating as well as in electric batteries and chemicals. In recent years nickel nanosized have become one of the interesting materialsin research communities due to the diverse promising application in the field of catalysis[2] and magnetism[3].The nickel nanoparticles are highly useful in detecting the anticancer mechanism[4].Nickel nanoparticles also serve technological applications such as magnetic inks[5], conductive paste[6], high performance electrodes[7], sintering additives[8], magnetic recording[9], fuel cells[10] and antimicrobial activity[11].

Literatures report the synthesis of nickel nanoparticles using microwave assisted synthesis [12], micro-emulsion synthesis [13] and Chemical reduction technique [14], sol-gel technique [15,16], electrochemical deposition[17], polyol synthesis[18], chemical vapour deposition technique[19] and hydrothermal synthesis[20]. In the present work, nickel chloride hexahydrate is used as the precursor, hydrazine hydrate as the reducing agent and water as the solvent. The major problem associated with the chemical reduction technique for the synthesis of nickel nanoparticles synthesized owing to the fact that they have enormous surface energy. When the synthesized nickel nanoparticles are exposed to air, it will lead to the formation of oxides of nickel which will significantly affect the properties of nickel nanoparticles and thus it cannot be utilized for several applications. To overcome this difficulty polyvinyl pyrrolidoneis used as the capping agent[21], the synthesis is done by

simple chemical reduction technique at low temperature ranges under microwave irradiation. The most important advantage of this microwave irradiation technique is that efficient energy heating is possible.

Materials and Methods

All the chemicals used are of L.R grade. Nickel chloride ($Nicl_2.6H_2O$), 98% hydrazine hydrate ($N_2H_4.H_2O$), sodium hydroxide and polyvinyl pyrrolidone from Eswar scientific. All solutions were prepared with DD water.

Synthesis of nickel nanoparticle

Solution Ais prepared by dissolving 0.1g of Nickel Chloride Hexahydrate in 10mL of distilled water. In a separate beaker, solution B was prepared by dissolving different concentrations of NaOH in DD water, PVPin ethanol and Hydrazine Hydrate. Different concentrations of NaOH, PVP dissolved in ethanol and different concentrations of Hydrazine hydrate is then added to solution A. The combined solution turned blue in colour and the solution was kept in microwave oven for 5 minutes and it turns black indicating the formation of nickel nanoparticles. The nanoparticles obtained are washed with ethanol thrice in small portions and dried in hot air oven at 60^0C for one hour to obtain the ultrafine nickel nanoparticles.

Characterization of synthesized nickel nanoparticle

The reaction of nickel chloride hexahydrate solution with hydrazine hydrate and polyvinyl pyrrolidone as the capping agent was optically measured using shimadzu UV-Visible spectrophotometer. The scanning electron microscope is used to study the shape of the synthesized nickel nanoparticles in an effective way. The main element in the synthesized materials was determined using energy dispersive X-ray spectroscopy.

Antimicrobial activity

By disc diffusion method, the antimicrobial activities of the synthesized nickel nanoparticles were studied. Nutrient agar media for bacteria and potato dextrose agar media for fungi were used, sterilized and solidified. Then the bacterial strain (*Escherichia coli*) and the antifungal strain (*Candida albicans*) were swabbed on the plates. The dried Nickel nanoparticle sample were weighed (10mg/10ml) and dissolved in sterile distilled water to prepare appropriate dilution to get required concentrations of about 50µl (50µg), 100µl (100µg) and 150µl (150µg). Control used as de ionized water. They were kept for incubation at 37^0c for 24 hours. Zone of inhibition for control and nickel nanoparticles were measured and the mean values of zone of inhibition were presented.

Results and Discussion

Visual observation

Reduction of Ni^{2+} ions to Ni^0 nanoparticles could be followed by a colour change and UV-Vis spectroscopy. The below Figure(1) shows the photographs of sample solutions containing nickel chloride hexahydrate, sodium hydroxide, polyvinyl pyrrolidone, hydrazine hydrate solutions after completion of the reaction. The appearance of a black color confirms the existence of nickel nanoparticles in the solution.

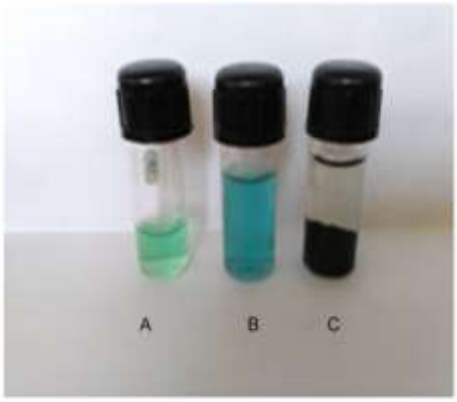

Figure1. (A) Nickel chloride hexahydrate solution (B) combination of NaOH, PVP, Hydrazine hydrate and Nicl2.6H2O (C) Formation of nickel nanoparticles.

UV-Visible Spectroscopy

UV-visible spectroscopy is one of the most widely used techniques for structural characterization of nickel nanoparticles. The absorption of nickel nanoparticles was measured by UV-visible spectroscopy. The absorption band of nickel nanoparticles has been reported be in the range of 250-370 nm[22]. UV - visible absorption spectra of nickel nanoparticles by chemical reduction method is shown in figure 2. This spectrum is recorded immediately after the synthesis of particles. The figure8.shows the UV absorption peaks at 286 nm respectively, which proves the formation of the nickel nanoparticles in the solution. The initial green color of the solution turned into a black precipitate, the shifting in color is due to the surface Plasmon resonance. Metals possess Surface Plasmon resonance band in visible region due to free electrons, which give such intense colors. These properties observed in nickel are due to the presence of free electrons.

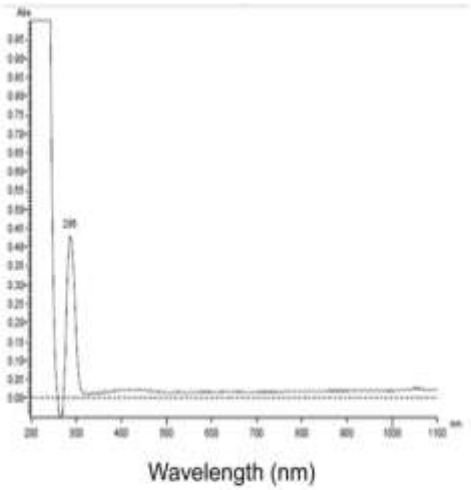

Figure2. UV-VIS Spectroscopy of the synthesized nickel nanoparticles.

Scanning Electron Microscope

Scanning electron microscopy was also used to determine if the nanoparticles were in the nanometer range of size and also to determine its shape. The measurements using the SEM were at the instrument limit of size, however, the image still shows that the nickel nanoparticles are within and morphology study has found out the synthesized nickel nanoparticles are moderately spherical with thorn like structure as result of using nickel chloride hexahydrate as the precursor and polyvinyl pyrrolidone as the capping agent. Moreover, it is supported by other findings, that the nickel nanoparticles formed were uniformly spherical[23].

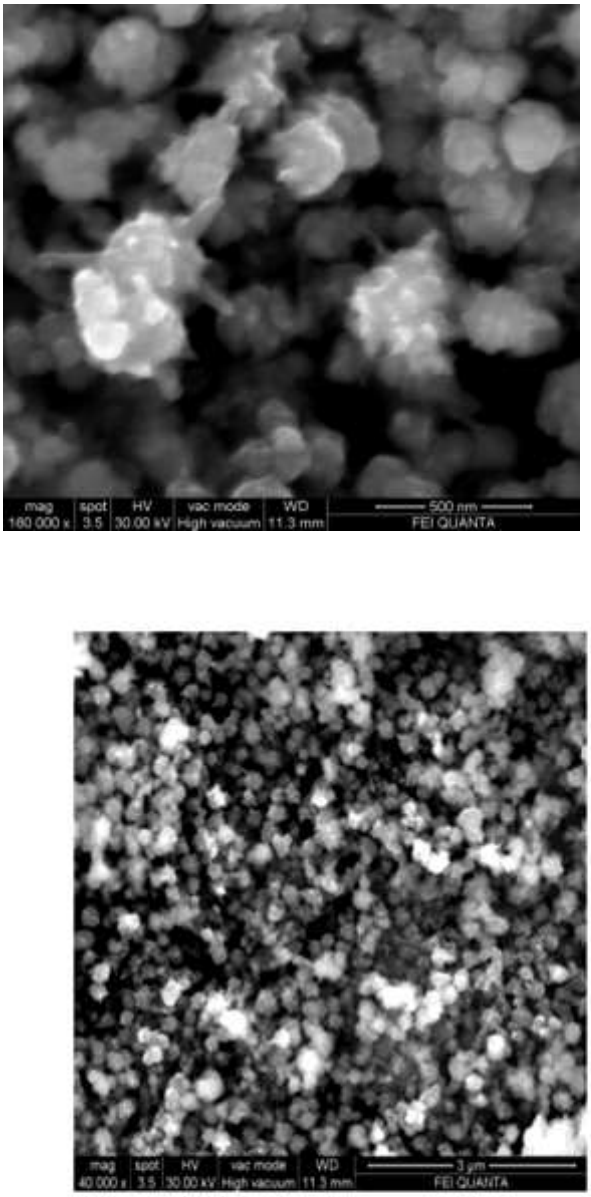

Figure3. SEM images of the synthesized Nickel nanoparticles.

Energy dispersive X-ray analysis

To determine the major elemental composition in the synthesized materials an analysis was done by energy dispersive X-ray spectroscopy. Using this technique the elemental composition of the materials was obtained with high resolution. The EDAX analysis data confirmed that the nickel nanoparticles were present in major composition about 88%.

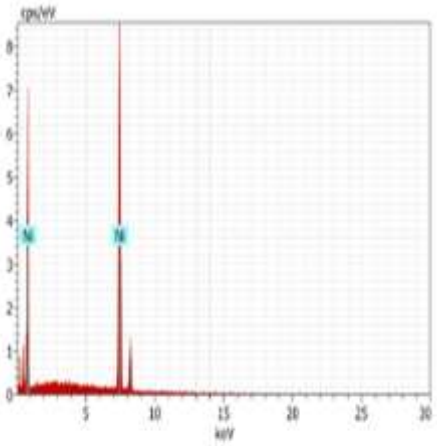

Figure4. EDAX images of the synthesized nickel nanoparticles

Table1. Elemental composition of nickel nanoparticles.

El	AN	Series	Unn.c [wt.%]	Norm.c [wt.%]	Atom.c [At.%]	Error	(1 sigma) [wt.%]
Ni	28	K-series	88.27	100.00	100.00	-	2.41
		Total	88.27	100.00	100.00	-	

Antimicrobial activity of nickel nanoparticles.

The nickel nanoparticles pretence to have an good bactericidal and fungicidal activity[24]. Based on the zone of inhibition produced, the synthesized nickel nanoparticles exhibited good antibacterial activity against gram negative bacteria *Escherichia coli* and the anti-fungicidal activity against *Candida albicans* which is shown in figure 6.

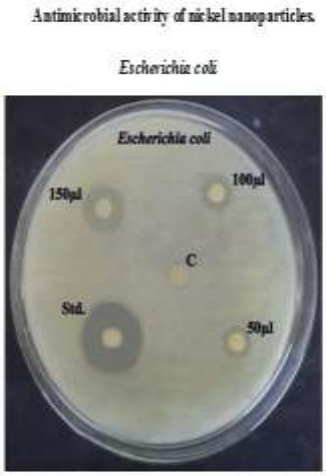

Figure 6. Antibacterial activity for the synthesized Nickel nanoparticles.

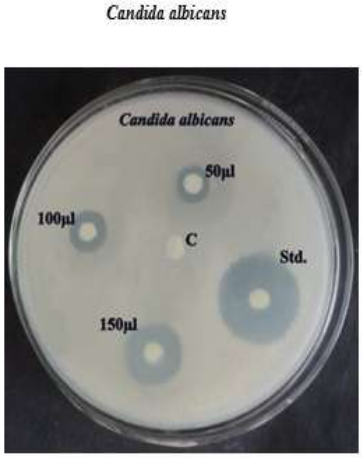

Figure 7. Antifungal activity for the synthesized Nickel nanoparticles.

Table2. Displays various standards used for antimicrobial activity.

Values were expressed as Mean ± SD for triplicates

Antibiotic Bacterial standard	Chloramphenicol
Antibiotic Fungal standard	Fluconazole
Control	Deionized water

Table3. Antibacterial and antifungal activity of synthesized nickel nanoparticles.

Microorganisms	NiNPs			Standard (30µl)	Control (solvent) (30µl)
	(50µl)	(100µl)	(150µl)		
	Bacteria				
Escherichia coli (mm)	1.40±0.09	3.30±0.23	6.80±0.47	10.30±0.71	0
	Fungal				
Candida albicans (mm)	1.50±0.10	3.50±0.24	6.90±0.48	9.20±0.70	0

Conclusion

The below mentioned conclusions were withdrawn based on the work presented in this project work. Nickel nanoparticles were synthesized by simplified chemical reduction technique using hydrazine as the reducing agent and polyvinyl pyrrolidone as the capping agent.The synthesized nickel nanoparticles were characterized using UV-Visible spectroscopy. The optical absorption peak intensity is found at 286 nm.The SEM analysis showed that nickel nanoparticles formed were moderately spherical in shape.The presence of

nickel nanoparticles in major composition was further confirmed from the Energy Dispersive X-ray Analysis. It is further revealed from the X-ray diffraction analysis that the synthesized nickel nanoparticles were present as face-centered cubic in structure.Further the antimicrobial studies showed that the nanoparticles are toxic to *Escherichia coli and Candida albicans.* This finding may also be extended to the field of electronic, industrial and other pharmaceutical applications, making this method potentially exciting for the large scale synthesis of other inorganic Nanomaterials.

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