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Synthesis, Characterization and Antibacterial Activity of Nickel Chitosan Nanoparticles

Dr.S.Mary Helen, S.Mahil Rani
Department of chemistry, Annai Velankanni College, Tholayavattam-629 157,Kanyakumari District,Tamil Nadu ,India

I. Introduction

Nanoparticles have attracted considerable attraction due to their unusual and fascinating properties, with various applications, over their bulk counter parts. Nanotechnology can be defined as the manipulation of matter through certain chemical and physical processes to create materials with specific properties which can be used in particular applications [1]. Nanotechnology is a field that is burgeoning, making an impact in all spheres of human life. A variety of methods have been reported for the preparation of metallic nanoparticles [2] [3]. Nanomaterials have attracted interest in the past decade and have been studied extensively because of their size and shape dependent physical-chemical and magnetic properties for applications in various useful technologies[4, 5]. After cellulose the chitosan is second most abundantly available polysaccharide. Chitosan used as an excipient in pharmaceuticals is relatively new approach. Chitosan has several desirable qualities for the biomedical field and is found biocompitable[6]. Metallic nanoparticles of Ni, Co and Fe are important due to their magnetic properties and application potential. For such crystallites, the physical and chemical properties depend sensitively on particle size and shape [7-11]. In the last few years, nickel nanomaterial have been synthesized in various forms like nanotubes, nanorods, hollow spheres, nanobelts, nanoprisms, and hexagonal flakes .Magnetic nanoparticles are being widely used in rechargeable batteries [7],optoelectronics [8], chemical catalysts [9], conducting paints [10], magnetic recording media[11] ferro-fluids, magnetic resonance imaging contrast enhancement, drug delivery[12] and magnetic hyperthermia [13, 14]. Several methods have been developed to synthesize particles with controlled size and shape. These methods include photolytic reduction [15], radiolytic reduction [16], sonochemical method [17], solvent extraction reduction [18], microemulsion technique [19], polyol process [20], and chemical route [21]. As an important transition metal, Ni nanoparticles have wide ranging applications in the fields of permanent magnets, magnetic fluids, magnetic recording media, solar energy absorption, fuel cell electrodes, catalysts etc. So the synthesis of Ni nanoparticles has attracted considerable attention. The purpose of this study is to describe a simple way for preparing Ni nanoparticles. Pure Ni nanoparticles have been synthesized and characterized herein.

II. Materials and Methods

Nickel Chloride, Mytilus galloprovincialis(Oyster), Ascorbic Acid,Hydrazine Sodium Hydroxide,Double Distilled Water.

2.1. Synthesis of Chitosan

The oyster was collected from the Colachel beach in Kanyakumari Dist. The exoskeletons of the oyster wastes were removed and was rinsed thrice with tap water and then twice with distilled water. Then they were dried in a hot air oven for about 24 hrs at 55°C. The sample obtained was soaked in boiling 4% sodium hydroxide using 1000 ml beaker for 1 hr. The sample was removed and then allowed to cool at room temperature for 30 minutes.The sample obtained was demineralized using 1% hydrogen chloride with four times its quantity. They were then soaked for 24 hrs to remove minerals. The above samples were treated with 50 ml of 2% sodium hydroxide for 1 hr. The remains of the sample were washed with deionized water and then drained off. The process was then carried out by adding 50% sodium hydroxide to the obtained sample on a hot plate and boiling it for 2 hrs at 100°C. The sample was then allowed to cool at room temperature for 30 minutes. Then they were washed continuously with 50% sodium hydroxide. They sample was filtered and oven-dried for 6 hrs at 110°C. The obtained biomass is treated with hydrogen peroxide to remove colour and other impurities. The pure form of Chitosan is used throughout the study.

2.2. Synthesis of Nickel Chitosan Nanoparticles

Nickel Chitosan nanoparticles were synthesized by taking a green colored aqueous solution of bulk Nickel chloride. This solution was made by taking 0.4 g of Nickel chloride in double distilled water (10 ml). Then the solution was added dropwise to a solution (0.1 M acetic acid) of Chitosan (40 mL), while the colour changed from green to light brown. After stirring and refluxing at 120 °C for about 20 min, 0.05 M ascorbic acid (0.5 ml) was added to the solution, with further stirring for 20 min, 0.6 M NaOH (2 ml) was added to the

solution, and no immediate colour change was observed after the addition. However, after stirring for about 30 min, a light bluish coloration was observed. Hydrazine (0.5 ml) was finally added to give a quick bluish coloration which changed to dark brownish after about 5min of stirring. The solution was stirred for further 30 min for the reaction to complete and the reaction mixture was allowed to cool at room temperature. The solution was centrifuged at 14,000 rpm for 15 min to obtain the Nickel Chitosan Nanoparticles and the supernatant was discarded. The particles were repeatedly washed with hydrogen Peroxide to ensure purity.

2.3. Characterisation of Synthesized Nanoparticles

In the present study the Cobalt Chitosan nanoparticles were synthesized and characterized by SEM, XRD, EDX analysis and UV –Vis spectroscopy. SEM, XRD, EDX and UV spectra confirmed the nanosize of the material. The synthesized nanoparticles were screened for antibacterial activity.

2.4. Antibacterial Activity

Antibacterial activities of the samples were determined by well diffusion method. Four pathogenic bacterial strains namely Escherichia coli, Klebsiella pneumonia, staphylococcus aureus and pseudomonas aeruginosa were used in this investigation. The test bacterial strains were inoculated into nutrient broth and incubated at 37^0C for 24 hrs. After the incubation period, the culture tubes were compared with the turbidity standard. Fresh bacterial culture of 0.1ml having 108 CFU was spread on nutrient agar plate using swab. Wells of 6mm diameter were punched off into medium with sterile cork borer and filled with 50µl of samples using micro pipette in each well at aseptic condition. Plates were then kept in a refrigerator to allow pre-diffusion of extract for 30 minutes .Further the plates were incubated in an incubator at 37^0C for 24 hrs. The antimicrobial activity was evaluated by measuring the zone of incubation.

III. Results and Discussion

3.1. UV-Visible Absorbance Study

UV-Visible spectrum has been widely used to characterize the nanoparticles. UV-Vis spectral analysis was done by using UV-Visible spectrophotometer at the range of 200nm to 800nm. The absorption peak for Nickel Chitosan nanoparticles using Mytilus galloprovincialis at 240nm region due to the excitation of surface Plasmon vibration in the Nickel Chitosan nanoparticles.

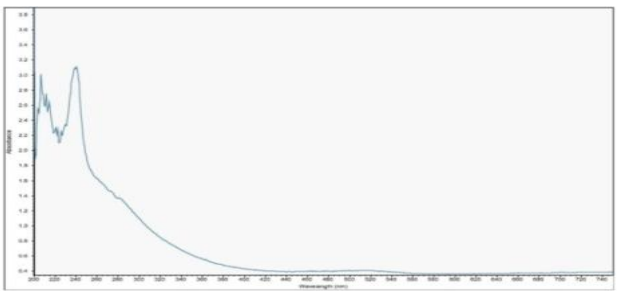

Figure: 1. UV-Vis spectrum of Nickel Chitosan nanoparticles from mytilus galloprovincialis

3.2. XRD Studies

The phase identification and crystalline structures of the nanoparticles was characterized by X-ray diffraction.XRD pattern of synthesized Nickel nanoparticles using Mytilus galloprovincialis shows a high crystallinity of Nickel sample level with diffraction angles of 29.5^0,34.7^0 ,48.6^0which corresponds to the crystal plane of (111),(002),(211) respectively(figure:2).

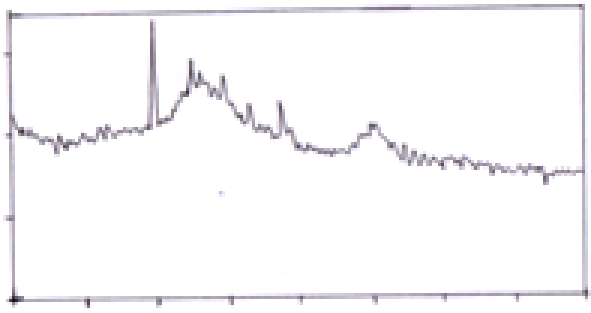

Figure2: XRD pattern of Nickel Chitosan nanoparticles

Debye –Scherrer's formula is used to calculate the crystalline size of the nanoparticles.

D (nm) = K □/ßcos □

d- the average particle size in nm

□ - the wavelength of the X ray (0.15406nm)

ß- the full of the peak at half height in radious

θ -the Bragg diffraction angle in degrees.

3.3. SEM Analysis

Scanning Electron Microscope is one of the powerful tools to identify the shape of the nanoparticles. The surface morphology of the nanoparticles were obtained by SEM. Nickel Chitosan nanoparticles synthesized by using biopolymer of mytilus galloprovincialis is shown in (Figure 3).

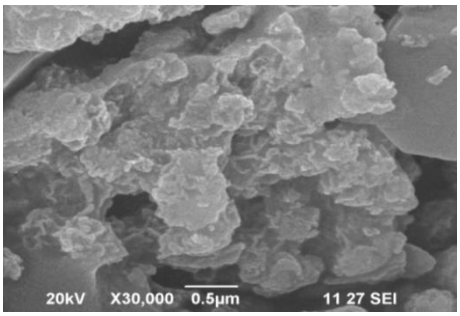

Figure 3: SEM image of Nickel Chitosan nanoparticles from mytilus galloprovincialis

3.4. Energy Dispersive X-ray diffraction spectroscopy (EDX)

The EDX technique detects X-rays emitted from the sample during bombardment by an electron beam to characterize the elemental composition of the materials was analyzed with high resolution. EDX analysis data confirms the main components of the materials. The weight percentage of Nickel Chitosan nanoparticles synthesized using mytilus galloprovincialis is 21.20%.

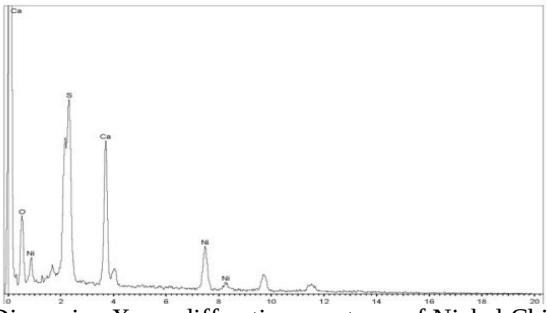

Figure: 4. Energy Dispersive X-ray diffraction spectrum of Nickel Chitosan nanoparticles

3.5. Antibacterial Activity

Antibacterial activities of the Cobalt nanoparticles were examined using the well diffusion method. Antibacterial activities of Cobalt Chitosan nanoparticles were studied against four pathogenic bacteria (Escherichia coli, Klebsiella pneumonia, staphylococcus aureus and pseudomonas aeruginosa). The Nickel Chitosan nanoparticles was found to be active against staphylococcus aureus and Klebsiella pneumonia

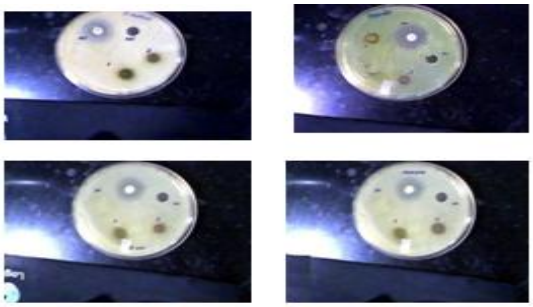

Figure 4: Antibacterial activity against Escherichia coli, Klebsiella pneumonia, staphylococcus aureus and pseudomonas aeruginosa

IV. Conclusion

In this study we have developed an eco friendly and environment safe green method for the synthesis of Nickel Chitosan nanoparticles using mytilus galloprovincialis (oyster). The UV – Vis spectra of Nickel Chitosan nanoparticles fell in the area 240nm , which indicates the particles are nano in size. Scanning Electron Microscope analysis was done for chitosan, Nickel Chitosan nanoparticles, which reveals that the particles consists of nano clusters. XRD study reveals that the Nickel chitosan nanoparticles formed are nanometric size. The diffraction angles of Nickel Chitosan nanoparticles are 29.5^0,34.7^0 ,48.6^0 which corresponds to the crystal plane of (111),(002),(211) respectively. EDX study confirmed the presence of Nickel metal in the sample under studies. The synthesized Nickel nanoparticles showed good antibacterial activity agnist Gram positive (stapylococcus aures) and gram negative (Klebsiella pneumonia) bacteria. In this method variation in presesure and temperature does not affect the reaction. Toxic chemicals were not used throughout the experiments. This method is cheap and ecofriendly. The waste materials(shells) are used to prepare chitosan which is used through out the study.

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