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SYNTHESIS CHARACTERIZATION AND APPLICATION OF NICKLE OXIDE NANOPARTICLES IN SURFACTANT **ASSISTED MEDIA**

Nidhi Gupta **Research Scholar Dr. B.R Ambedkar University**

Dr. R. K. Shukla Associate Professor Department Of Chemistry R.B.S Colleg, Agra **Dr. B.R Ambedkar University**

ABSTRACT

Nickel oxide (NiO) has garnered a significant amount of attention over the course of the last few decades as a result of its remarkable electrical, magnetic, and catalytic capabilities. For the purpose of this investigation, NiO nanoparticles were manufactured using the sol-gel method, which is among the most straightforward and economical approaches. In order to successfully complete the synthesis, the surfactant that was utilized was Poly(alkylene oxide) block copolymer, and the inorganic precursor that was utilized was Ni(NO3)2•60. During the course of this study, the creation of NiO nanoparticles was examined in relation to the influence of experimental parameters such as calcination temperatures and the content of H2O. For the purpose of determining the microstructure and specific surface area of the samples, techniques such as TGA, XRD, SEM, TEM, and N2 adsorption-desorption isotherms were utilized respectively. Both TGA and FTIR tests revealed that the expulsion of copolymers occurred at a temperature of 573 K. It was shown that the calcination temperature had a significant impact on the creation of NiO nanoparticles as well as the structural characteristics of these particles. As demonstrated by XRD, the sample that was calcined at a temperature of 923 K was made up of pure NiO nanoparticles. The reoxidation process of metallic nickel to generate nickel oxide would decrease as the concentration of hydrogen peroxide (H2O) increased; however, this would not have any impact on the structural type of nickel oxide nanoparticles. The addition of water would, in general, reduce the effects of oxidation and prevent them from occurring. All the way up to 823 degrees Kelvin, the temperature of the stable metallic *Ni was raised. It was determined through the N2 adsorption–desorption experiment that the samples were* composed of non-porous NiO nanoparticles based on the specific surface area that was assessed. There was a correlation between the addition of more water and an increase in the specific surface area of the nanocrystalline NiO powder.

Keywords: Nickel oxide; Nanoparticle.

INTRODUCTION

Nanotechnology is a rapidly emerging area that involves the design and synthesis of nanoparticles with diameters ranging from one one hundred nanometers to one hundred nanometers. This growing field has a wide range of applications in a variety of economic sectors, such as the cosmetics industry, the food industry, the pharmaceutical industry, agriculture, optics, cancer theranostics, catalysis, and many more. Researchers have recently paid a large amount of attention to nanoparticles metals due to the fact that they possess a wide range of unique characteristics in comparison to bulk materials. These characteristics have been utilized in the fields of diagnosis, cell tagging, antibacterial agents, medications, and cancer therapy. When it comes to the treatment of oncology, nanotechnology is one of the most important topics. It offers options to the development of controlled release systems for the treatment of various diseases and lowers the adverse effects of drugs. There are a number of nano-systems that may be categorized according to their composition. These nano-systems can be inorganic, lipid, or polymeric, and they will allow for the reformulation and improvement of cancer treatments that are already in existence. The field of nanobiotechnology focuses on the production and application of nanoparticles, and it plays a significant role in the expansion of a variety of industrial fields, including food, cosmetics, agriculture, the distribution of medications, and carcinogenic theranosis. The production of nickel oxide nanoparticles (NiONPs) has been accomplished through the utilization of a wide range of physical and chemical techniques, including sol-gel chemistry, electrodeposition, combustion, thermal route, and so on. However, these techniques have a number of potentially harmful side effects, which restrict the range of biological applications they can be used for. It is normal practice to generate harmful chemicals that contribute to environmental toxicity and materials that are not biodegradable through the process of chemical synthesis. On the other hand, physical methods require significant amounts of energy inputs. Methods that are efficient, environmentally friendly, and green have been developed by scientists in order to address these issues. The synthesis of desirable metal nanoparticles that are clean, cost-effective, renewable, and environmentally acceptable can be accomplished through the utilization of a variety of biological sources, such as trees, algae, and microbes. Because plant extracts include a high concentration of phytochemicals (alkaloids, flavonoids, and polyphenols), which are effective agents for stabilizing and chelating biogenic nanoparticles, green synthesis has become increasingly prevalent in plant extracts. The use of plant extract as an effective agent for capping and decreasing, as well as for controlling the size and shape of NPs, is also the possibility. The manufacture of metallic nanoparticles through the use of plants has also been a primary focus of research among scientists.

Nickel oxide, also known as NiO, is a chemical that is known to be non-toxic to people, as well as affordable, abundant, photo-stable, and short-term. In addition, nickel oxide is a P-type semiconductor of biomolecules that has a large bandgap of 3.6–4.0 eV. It has the potential to be used in a variety of applications, including catalysis, batteries, super capacitors, antiferromagnetic components, lithium-ion battery electrode materials, electrochemical supercapacitors, gas sensors adsorbents, optical amplifiers and tunable lasers, photovoltaic systems, electrochromic materials, and applications for fuel cells. In the past, these particles have been utilized in the adsorption of dye rates as well as dangerous contaminants. They also have the ability to be utilized in biomedicines due to the anti-inflammatory qualities that they possess. In previous investigations on NiONP, cytotoxic effects were also revealed by the release of reactive oxygen species (ROS) and nickel ions (Ni2+) due to oxidative damage.

OBJECTIVES

- 1. To study Synthesis of nionps.
- 2. To study Characterisation of nionps.

Synthesis of nionps

In this experiment, fifty milliliters of the precursor solution of nickel (II) chloride hexahydrate (NiCl2•6H2O) with a concentration of 0.1 M was combined with fifty milliliters of grape aqueous extract in a ratio of one to one. In order to accomplish this process, 10 milliliters of the aqueous extract were gradually added to the precursor solution at intervals of five minutes until a noticeable change in color was detected. While the heat was kept at 65 degrees Celsius, the final mixture was agitated with a magnetic stirrer for a period of two and a half hours. Consequently, the color changed from a bright green to a dark green, which is evidence that NiONPs were formed. As can be seen in figure 1, the resultant solution was separated twice using centrifugation at a speed of 3500 revolutions per minute after it had been allowed to return to room temperature.



Fig. 1. Synthesis of NiO nanoparticles.

Characterisation of nionps

SEM and TEM were utilized in order to investigate the morphological characteristics of the NiONPs, which included their dimensions and shapes. Using an atomic force microscope (AA 3000 scan sample microscope, Taiwan), NiONPs were quantified. There was a diffraction meter that was electronic that was used. FT-IR was utilized in order to ascertain the functional groups that were present in the NiONPs solution. In addition, an ultraviolet visible spectrophotometer was utilized in order to investigate the optical characteristics of the NiONPs that were recently synthesized.

Materials and Method

In this study, the synthesis and characterisation of nickel oxide nanoparticles that were generated by thermochemical processes have been the primary focuses of the research. With vigorous stirring (550-700 revolutions per minute), 20 milliliters of ammonia was added to 2 grams of anhydrous NiCl2 powder (Merk, 99.7%) over the course of approximately thirty minutes in order to form the solution that included Ni+2. This was done in order to synthesis nickel oxide nanoparticles. Ammonia should be added to the

precursor in a slow and steady manner because of the exothermic reaction that occurs when NiCl2 reacts with ammonia. Additionally, the pH of the solution was set in the range of 7.5 to 9, while the temperature of the solution was set at RT, 33, 55, and 75 degrees Celsius. Subsequently, distilled water was added as a precipitant agent, and the solution was vigorously stirred until the formation of green precipitate occurred. The precipitate that was produced at the end of the process was filtered and washed twice with ethanol and deionized water. Green precipitates were dried in an oven at 105 degrees Celsius for ninety minutes, and then they were heated in a tube furnace at 410 degrees Celsius for one hour in order to make black NiO nanoparticles.

The nanoparticles were characterized using a variety of techniques, including X-ray diffractometry (Siemens D-500, Germany), transmission electron microscopy (Philips CM-200, Netherlands), scanning electron microscopy (Hitachi S4160, Japan), BET surface area analysis (Micromeritics Gemini, United States of America), thermogravimetric analysis, and UVvisible spectrophotometry (Perkin Elmer Lambda 25, United States of America).

Results and Discussion

The X-ray diffraction (Cu K α radiation) spectra of the nanoparticle samples that were produced by the thermochemical approach has been depicted in Figure 2. These samples were produced at different solution temperatures. Illustrations of the XRD patterns of the dried precipitates and the calcined product at room temperature are shown in Figure 2 (a and b). When the precipitate is dried, there is no discernible peak that can be seen. It can be deduced from this that the precipitate is an amorphous phase that contains a variety of nickel salts or precipitation agents. Calcined at 410 degrees Celsius, a cubic crystal of nickel oxide is formed. It is possible to compare the experimental values obtained for the lattice parameter, d, of the particles with the values provided by the American Society for Testing and Materials (card no. 4-0835). When the data acquired from the diffraction peaks are applied to the Scherrer formula, which is d=0.9 λ /B(cos θ), the result is the average crystal size of the NiO nanoparticles at various temperatures, which is approximately 13-15 nm. Based on the samples depicted in Figure 2 (c, d, and e), it can be observed that the processing temperature did not have any impact on the crystal structure of nanoparticles. Furthermore, the crystal structure remained unchanged even when the processing temperature was increased.



Figure 2. The diffractogram of nanoparticle produced by thermochemical processing at (a) RT and dried at 105 °C for 24 hrs, (b) RT and calcined at 410 °C for 1 hr, (c) 33 °C, (d) 55 °C and (e) 75 °C



Figure 3. SEM micrograph of agglomerated nanocrystalline NiO particles produced at (a) RT, (b) 33 °C, (c) 55 °C and (d) 75 °C

During thermochemical processes, nanoparticles tend to cluster together and form walls with a thickness of nanometers. The scanning electron microscope (SEM) used to depict the NiO products that were produced using the thermochemical technique at various temperatures is shown in Figure 3. Flake-walls with a nanothickness have been shown to develop at lower temperatures (RT). As the temperature rises, the shape of the flake walls undergoes a substantial transformation, becoming more characteristic of sharp walls. It would appear that the nature of the thermochemical processing has a considerable impact on the morphology and thickness of the products. Figure 3 (a and b) demonstrates that flake walls are predominant when processing takes place at room temperature and 33 degrees Celsius. The fraction of flake components, on the other hand, decreases as the processing temperature rises. The particles tend to form sharp walls when the temperature is between 55 and 75 degrees Celsius, as seen in Figure 3 (c and d). All temperatures are characterized by the presence of partial agglomeration of particles. Through the utilization of an

ultrasonic vibrator, the wall-shaped agglomerated product that was produced by the thermochemical synthesis was dispersed. The TEM picture of individual nanoparticles that were synthesized at room temperature is shown in Figure 4. Particles that had agglomerated were subjected to gentle sonication for twenty seconds in order to achieve a more accurate measurement of the size distribution of nanoparticles. Nanoparticles, with an average diameter of approximately 20 nanometers, are the constituents of the powder. Table 1 contains information regarding the BET surface area of the ultrasonicated NiO particles at a variety of temperatures. The specific surface area of nanoproducts is reduced when the processing temperature leads to an increase in particle size, which in turn leads to a decrease in the specific surface area of particles. This could be explained by the fact that the growth rate of particles increases with processing temperature.



Figure 4. TEM image of NiO nanoparticle produced by thermochemical processing at RT (agglomerated nanoparticles was dispersed under ultrasonication)



Figure 5. TG and DTG curves of precipitates produced at RT

As illustrated in Figure 5, the TG and DTG curves of the nanoparticles that were synthesized by the thermochemical procedure at room temperature are displayed. The TG curve demonstrates that there are three stages from which the weight loss happens. Within the temperature range of 40 to 230 degrees Celsius, the initial stage encounters a weight loss of approximately 8%. Possible explanations for the curves

include the presence of physisorbed water, crystalline water, and humidity in pores that have been freed during this temperature range. During the second stage, a weight loss of approximately 10.6% takes place as a result of the breakdown of Ni(OH)2 from 230 to 385 degrees Celsius. In the end, the third stage is associated with the creation of NiO, which may occur at temperatures higher than 385 degrees Celsius.

The UV-visible absorption spectra of the NiO particles that were obtained at a variety of temperatures and dispersed in ethanol is shown in Figure 6. Table 1 is a listing of the optical band gap energy of NiO nanoparticles that were synthesized at room temperature, 33 degrees Celsius, 55 degrees Celsius, and 75 degrees Celsius.



Figure 6. UV-visible absorbance spectrum of NiO nanoparticles produced at (a) RT, (b) 33 $^\circ$ C, (c) 55 $^\circ$ C and (d) 75 $^\circ$ C

Processing	BET surface	Related band gap
temperature (°C)	area $(\mathbf{m}^2/\mathbf{g})$	energy (eV)
RT	80	3.85
33	81	3.83
55	77	3.63
75	70	3.45

Table 1. Nickel oxide nanoparticles specification

By raising the temperature at which the processing is carried out, there is a discernible movement toward a higher wavenumber for the absorption edges. According to this shift, there has been a reduction in the band gap, which can be attributed to an increase in the size of the particles. The value of the absorption edge of NiO particles that were synthesized at room temperature, 33 degrees Celsius, 55 degrees Celsius, and 75 degrees Celsius was 307, 308, 322, and 341 nanometers, respectively. In light of the information

included in the absorption spectra, it is possible to estimate the optical band gaps (Eg) of NiO nanoparticles by employing the equation that is presented below:

 $(\alpha h\nu)^n = A(h\nu - E_g)$

In the equation, the variable hv represents the photo energy, α represents the absorption coefficient, A represents a constant that is relative to the material, and n is either 2 for a material with a direct band gap or 1/2 for a material with an indirect band gap. The optical band gap for the absorption peak can be derived by extrapolating the linear section of the $(\alpha hv)n - hv$ curve so that it is equal to zero, as stated in the equation. A comparison may be made between the band gap energies published by Boschloo, Anandan, Davar, and Wang and the values presented in table 1, which contains the equivalent band gap energies of nanoparticles that were synthesized at various temperatures. It is likely that the flaws or vacancies that are present in the intergranular regions are responsible for the growing trends of the band gap energy upon the decreasing particle size. These defects or vacancies generate new energy levels, which contribute to a reduction in the band gap energy. Since there was no linear relation discovered for n = 1/2, it may be inferred that the NiO nanostructures that were produced in their current state are semiconducting and undergo direct transition at this energy.

CONCLUSION

The synthesis of nickel oxide nanoparticles was accomplished through the chemical treatment of an aqueous solution that contained nickel. There is a proportionate number of both nano sharp-walls and nano flake-walls in the clusters of nickel oxide particles, and this proportion varies depending on the temperature that is currently being utilized. By increasing the processing temperature from room temperature to 75 degrees Celsius, the morphology of the clusters changed from flake wall to sharp wall. This was demonstrated by the micrographs obtained from the scanning electron microscope (SEM). The processing temperature was found to be equivalent with the band gap energy of NiO, which was reported by researchers in the past. This resulted in a decrease in the optical absorption band gap of nickel oxide nanoparticles. Furthermore, by adjusting the processing temperature up to 75 degrees Celsius, it was possible to create a broad range of band gap.

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