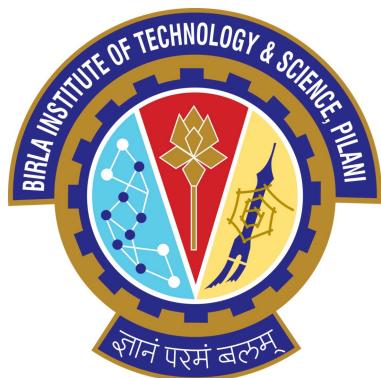


A Project Report  
On  
**Synthesizing shape selective Nickel Nano-crystals**  
BY  
**NAMRATA RAJESH AHUJA**  
**2020B2A81978G**

Under the supervision of  
**Dr Rabi Narayan Panda**  
**Associate Professor, Department of Chemistry**

**SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENTS OF**  
**CHEM F376: DESIGN PROJECT**



**BIRLA INSTITUTE OF TECHNOLOGY AND SCIENCE PILANI**  
**K.K. BIRLA GOA CAMPUS**  
**(DECEMBER 2022)**

## **ACKNOWLEDGMENTS**

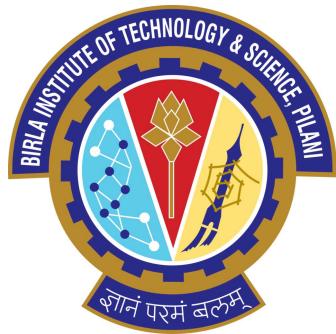
I would like to express my sincere gratitude to Dr Rabi Narayan Panda to let me work under his guidance and for allowing me to explore and learn about Nanoparticles. I would like to thank him for his continuous support, motivation and encouragement and for providing valuable inputs and resources throughout the completion of my research work.

I would also like to express my sincere gratitude towards Ph. D. Scholar, Ms Patil Anagha Baburao for guiding me in the experimentation process.

I would also like to express my sincere gratitude to the University for giving us the opportunity and resources to work on projects.

I would also like to thank our Instructor-in-charge for the project course, Dr Halan Prakash, for giving me a chance to be a part of this project course.

Finally, I owe my warmest gratitude to my parents and friends for their constant moral support.



**Birla Institute of Technology and Science-Pilani**

**K.K. BIRLA GOA CAMPUS**

## **CERTIFICATE**

This is to certify that the project report entitled “**Synthesizing shape selective nickel Nanocrystals**” submitted by **Ms NAMRATA RAJESH AHUJA ( ID No. 2020B2A81978G )** in partial fulfilment of the requirements of the course **CHEM F376**, Design Project Course, embodies the work done by her under my supervision and guidance.

**Date:**

**Dr Rabi Narayan Panda**

**Designation:**

**Associate Professor,  
Department of Chemistry  
BITS- Pilani-K K Birla Goa Campus,  
Goa, India.**

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## **ABSTRACT**

Shape Selective Synthesis of spherically shaped nickel (Ni) nanocrystals have been carried out by reducing Nickel (II) chloride hexahydrate and with sodium borohydride in the presence of PEG 200 as an organic modifier. The organic modifiers play an essential role in deciding the size and structure of the nickel nanoparticles. The as-prepared nanostructured Ni samples have been characterised by powder X-ray diffraction (XRD) and Field emission scanning electron microscopy (FESEM). The modifying and stabilising effects of PEG 200 have been investigated. With the development of nanotechnology, more and more efforts have been directed toward the preparation of novel nanomaterials, with diverse applications. Among the transition metal magnetic particles, Ni nanoparticles have been extensively studied because of their multifarious uses including magnetic sensors, memory devices and biomolecular separation. The purity, structure, size and shape of Ni nanoparticles greatly influence the ultimate performance of the materials and devices made of them. So it is significant to prepare high-quality Ni nanoparticles by using a convenient and low-cost method.

## **WORK OBJECTIVES**

- Finding a method which has good yield as well as is cost-effective for the synthesis of nickel nanocrystals
- Synthesis of Nickel nanocrystals in four batches. Two in the absence of PEG 200 as an organic modifier and two in the presence of the same.
- Heat treatment of the samples synthesised at varying temperatures to study the effect of temperature on the structure of the nanocrystal synthesised.
- Powder X-ray diffraction (XRD) of all the samples synthesised and analysis of the same.

# **PROJECT WORK PLAN**

## **September**

- Week 1 – Initial Project discussion Dr Rabi Narayan Panda to understand the working of the project. Preliminary lab tour and understanding of the various instruments present.
- Week 2 – Researching the various ways to synthesize shape selective nickel nanocrystals and gathering resources for further reading.
- Week 3 – Finalizing the shape of the nanocrystal to be synthesised and understanding the process to synthesize the same (Spherical Nickel nanocrystals).
- Week 4 – Lab orientation and gathering all the necessary apparatus & chemicals required.

## **October**

- Week 5 – Documentation of the synthesis process, the steps to be carried out and the precautions to be taken. Carrying out the necessary calculations for the experiment.
- Week 6 –Trial Synthesis of spherical Nickel Nano-crystals.
- Week 7 – Heat treatment of the nanocrystal synthesised.

## **November**

- Week 8 – Midsemester Presentation and Final Synthesis of Nickel Nano-Crystals.
- Week 9 – Trial Synthesis of Nickel Nano-Crystal with PEG 200 coating.
- Week 10 – Final Synthesis of Nickel nano Crystal with PEG 200 coating.

## **December**

- Week 11 – Analysis of the Nanocrystal synthesized (XRD Scans) and comparing the various parameters with theoretical as well as previously synthesized nanocrystals and documentation of all the results obtained.
- Week 12 – End-Semester Presentation.

# **INTRODUCTION**

## **1.1 Introduction to Nanoparticles**

Nanocrystals are aggregates of atoms that combine into a “cluster” and are less than 1  $\mu\text{m}$  in size. Typical sizes range between 10 and 400 nm. Their physical and chemical properties are observed somewhere between that of bulk solids and molecules. As the size gets reduced its effective surface area increases, which will ultimately increase the solubility and bioavailability [1]

A further characteristic is that drug nanocrystals are composed of 100% drug; there is no carrier material as in polymeric nanoparticles. Dispersion of drug nanocrystals in liquid media leads to so-called “nanosuspensions” (in contrast to “micro-suspension” or “macro-suspensions”). In general, the dispersed particles need to be stabilized, such as by surfactants or polymeric stabilizers. Dispersion media can be water, aqueous solutions or nonaqueous media (eg, liquid polyethylene glycol [PEG], oils).[2]

Depending on the production technology, the processing of drug microcrystals to drug nanoparticles can lead to either crystalline or an amorphous product, especially when applying precipitation. In the strictest sense, such an amorphous drug nanoparticle should not be called a nanocrystal. However, often one refers to “nanocrystals in the amorphous state”.[2]

## **1.2 Properties of Nickel**

Nickel (Ni), a chemical element, ferromagnetic metal of Group 10 (VIIIB) of the periodic table, markedly resistant to oxidation and corrosion. Silvery white, tough, and harder than iron, nickel is widely familiar because of its use in coinage but is more important either as a pure metal or in the form of alloys for its many domestic and industrial applications. Nickel (atomic number 28) resembles iron (atomic number 26) in strength and toughness but is more like copper (atomic number 29) in resistance to oxidation and corrosion, a combination accounting for many of its applications. [3]

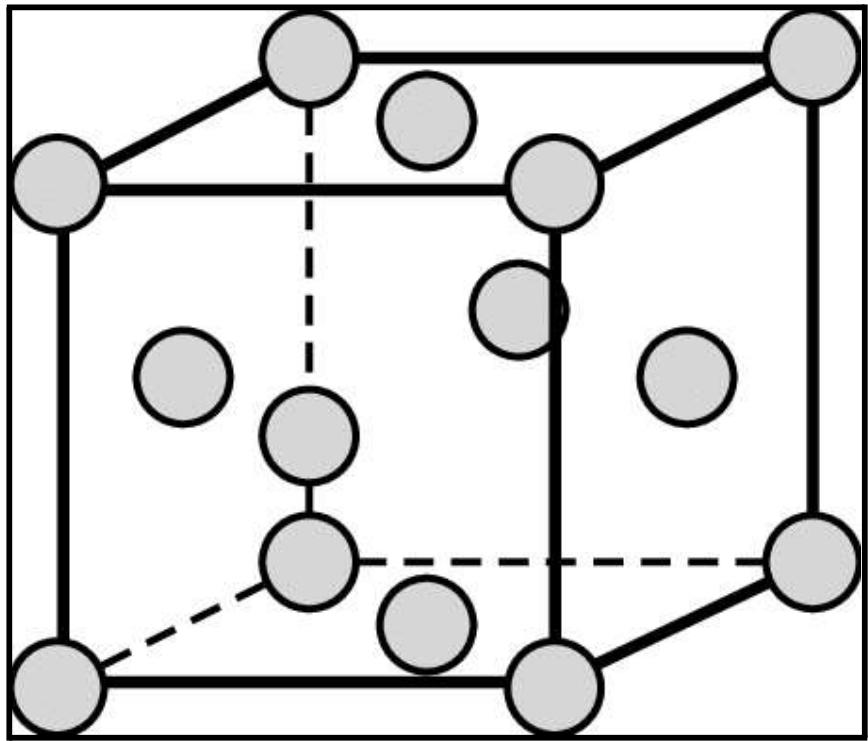


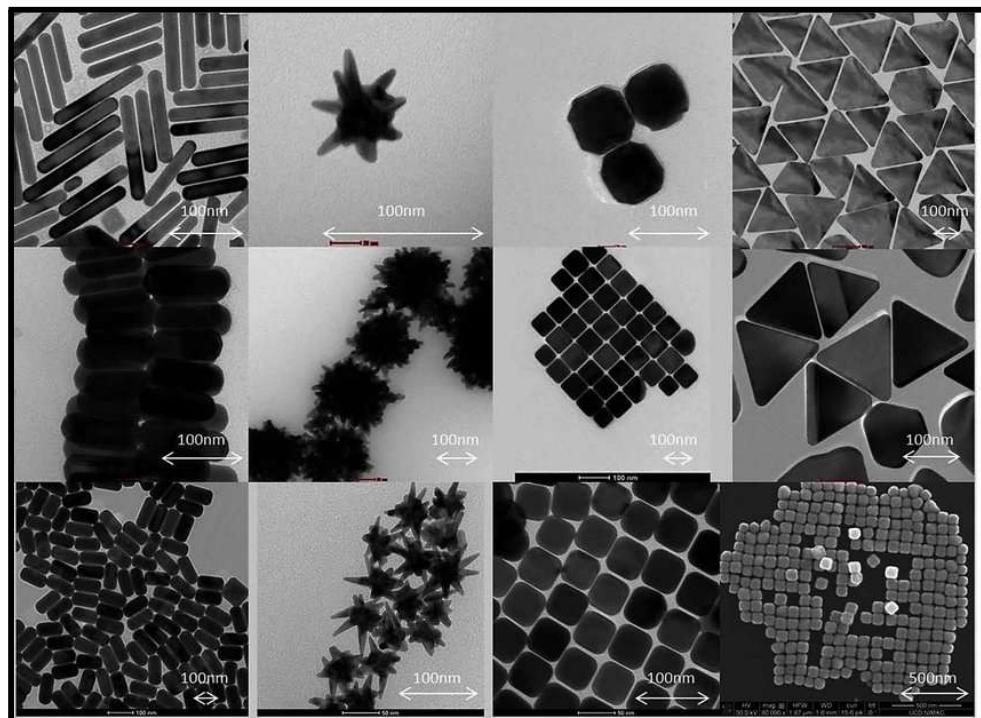
Fig 1:FCC lattice of Nickel

atomic number	28
atomic weight	58.69
melting point	1,453 °C (2,647 °F)
boiling point	2,732 °C (4,950 °F)
density	8.902 (25 °C)
oxidation states	0, +1, +2, +3
electron configuration	[Ar]3d <sup>8</sup> 4s <sup>2</sup>

Table 1: Physical properties of Nickel

### 1.3 Shape Selective Synthesis

The properties of nanomaterials sensitively depend on their compositions and morphologies. Recently a variety of novel shapes such as quantum dots, nanorods, nanoribbons, nanowires, nanotubes, and hollow spheres have been synthesized through varied synthetic reactions at room or slightly elevated temperatures.[4]



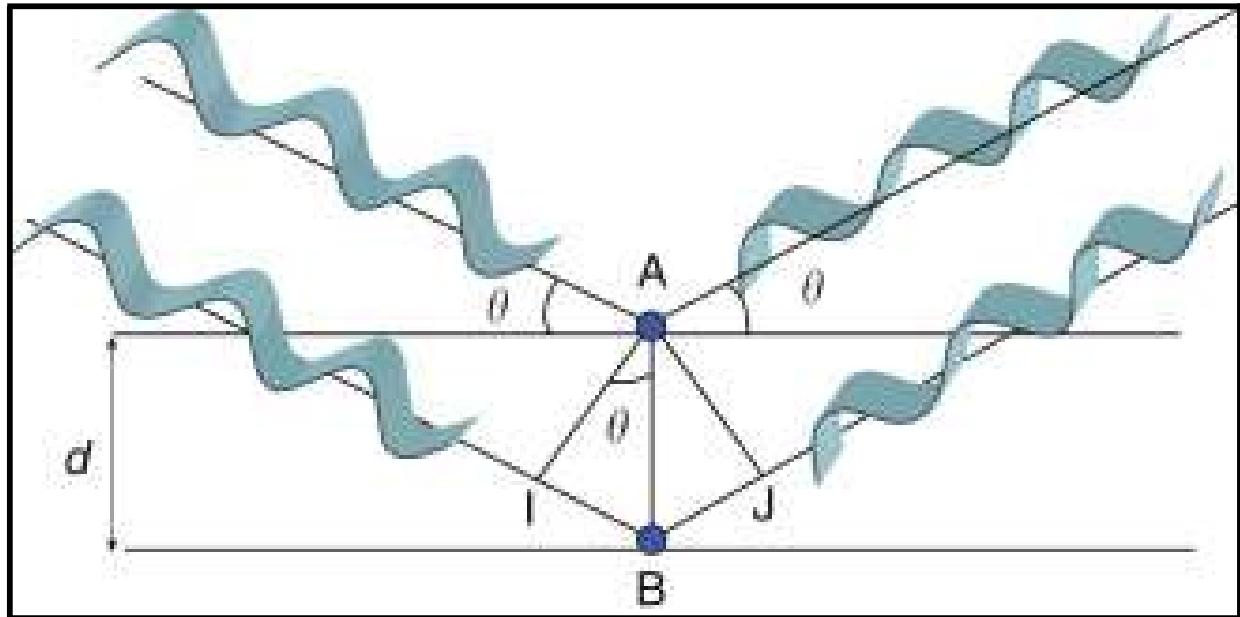
**Fig 2:Different shapes of Nanoparticles**

### 1.4 X-Ray Diffraction

X-ray diffraction (XRD) is a widely used technique to assess the crystallinity and structure of solid samples. In summary, the crystal X-ray diffraction phenomenon results from a scattering process in which X-rays are scattered by the electrons of atoms present in the sample without changing the wavelength. Since X-rays have wavelengths (between 0.2 and 10 nm) comparable to the interatomic spacing of a crystalline solid, the incident X-ray beam diffracts in specific directions predicted by Bragg's law. The resulting diffraction pattern, given by the positions and intensities of the diffraction effects, is a fundamental physical property of the material, providing not only the identification but also the complete elucidation of its structure.[5]

## 1.5 Bragg's Law

Bragg's law gives the simple condition under which a diffracted beam can be observed. Figure 3 shows a beam of parallel X-rays penetrating a set of parallel lattice planes with indices  $h, k, l$  of spacing  $d$  and at an angle of incidence  $\theta$ . The lattice planes are represented as behaving as a mirror.



**Fig 3: Braggs Law Condition**

The path difference between the waves scattered in A and B is equal to:

$$IB + BJ = 2d \sin \theta \quad \text{equation [1]}$$

The ‘reflected’ waves will combine to form a diffracted beam (maximum constructive interference, commonly called ‘reflection’) if the path difference is a multiple of the wavelength  $\lambda$ :

$$2d h k l \sin \theta = n \lambda \quad \text{equation [2]}$$

Equation [2] is the Bragg's equation. ‘Reflected’ waves that do not obey this rule will interfere destructively. In equation [2], the value  $n$  gives the ‘order’ of the diffraction. [6]

## MAGNETIC PROPERTIES OF NICKEL

Ni nanoparticles are ferromagnetic and show ferromagnetic–paramagnetic transitions at their Curie points. The saturation magnetization  $M_s$  is size-dependent, with a maximum value of 52.01 and 82.31 emu/g at room temperature.[8] With the increase of particle size, the saturation magnetization increases monotonically, and the coercivity decreases at first and then increases.

According to the size-dependent cohesive energy model[9], a simplified model can be developed to describe the relationship between the Curie temperature  $T_c(D)$  of ferromagnetic nanomaterials and the average size  $D$  of nanomaterials:

$$\frac{T_c(D)}{T_{c0}} = \exp\left(-\frac{2S_{vib}}{3R} \frac{1}{D/6h-1}\right) \quad \text{equation [3]}$$

where  $T_{c0}$  is the Curie temperature of bulk materials,  $S_{vib}$  denotes the vibrational part of the overall melting entropy  $Sm$ ,  $R$  is the ideal gas constant, and  $h$  denotes the atomic diameter.

Considering our samples, which are the ferromagnetic Ni nanoparticles, the relevant parameters are  $T_{c0} = 631$  K,  $S_{vib} \approx Sm = 10.12$  J/(mol K), and  $h = 0.2492$  nm [10]. Substituting  $R = 8.314$  J/(mol K) into Eq.[3], the  $T_c$  of Ni nanoparticles with size  $D$  can be expressed as

$$T_c = 631 \exp\left(\frac{1.2133}{1.4952-D}\right) \quad \text{equation [4]}$$

Sample	C	D (nm)	$T_c$ (K)	$M_s$ (emu/g)		$H_c$ (Oe)	
				300 K	5 K	300 K	5 K
M1	1/200	24	593	25.25	50.19	120	287
M2	1/100	50	612	32.30	53.49	79	250
M3	1/50	96	622	40.59	57.03	18	230
M4	1/20	165	626	46.66	64.77	146	379
M5	1/10	200	627	52.01	82.31	158	403

**Table 2: Particle size ( $D$ ), Curie temperature ( $T_c$ ), saturation magnetization ( $M_s$ ) and coercivity ( $H_c$ ) for the Ni samples synthesized at different precursor concentrations (C).[11]**

## EXPERIMENTAL

### 3.1 Materials

Nickel (II) chloride hexahydrate and sodium borohydride were analytical reagent grade. PEG 200 were chemical reagent grade. All of them were used as received. Water was used as the solvent.

### 3.2 Calculations

**NiCl<sub>2</sub>.6H<sub>2</sub>O (0.1M, 250ml soln )**

Element	Atomic Weight (g)
Ni	58.693
Cl	35.453
H	1.008
O	15.999

**NaBH<sub>4</sub> ( 1M, 100ml soln freshly prepared )**

Element	Atomic Weight (g)
Na	22.990
B	10.811
H	1.008

$$\text{NiCl}_2 \cdot 6\text{H}_2\text{O} = 237.689\text{g}$$

$$1\text{ M} = 1000\text{ ml} = 237.689\text{ g of NiCl}_2 \cdot 6\text{H}_2\text{O}$$

$$0.1\text{ M} = 250\text{ ml} = 'a'\text{ g of NiCl}_2 \cdot 6\text{H}_2\text{O}$$

$$a * 1 * 1000 = 0.1 * 250 * 237.689$$

$$'a' = 5.9422\text{ g}$$

$$\text{NaBH}_4 = 37.833\text{g}$$

$$1\text{ M} = 1000\text{ ml} = 37.833\text{ g of NaBH}_4$$

$$0.1\text{ M} = 250\text{ ml} = 'b'\text{ g of NaBH}_4$$

$$b * 1 * 1000 = 1 * 100 * 37.833$$

$$'b' = 3.783\text{g}$$

### 3.3 Synthesis of Spherically Shaped Ni nanocrystals

In a typical experiment 80 ml, 0.1 M  $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$  solution is taken in a round bottom flask .1M freshly prepared  $\text{NaBH}_4$  soln was added dropwise (with constant stirring ) in the  $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$  up till the precipitate appears[7]. This should be performed in a water bath maintaining a temperature < 25 °C. After the precipitate is formed filtration should be carried out. Wash the residue several times with water followed by acetone. The solid prepared is dried for 4-6 hrs at 55°C. Heat under  $\text{N}_2$  gas for 3-4 h. The resultant nanostructured Ni was then characterised using X-ray diffraction.



**Fig 4:Filtration process in the synthesis of Ni nanoparticles**

### 3.4 Drying the sample

After washing several times with acetone and water, the solid prepared is dried for 4-6 hrs at 55°C. This is done to remove the water after which heat treatment would be carried out.

### 3.5 Yield Calculations

Weight of empty bottle	=9.3515g
Weight of Bottle with sample	=9.9910g
Weight of Sample	=9.9910g-9.3515g =0.6395g
$\text{Ni}^{+2} \rightleftharpoons \text{Ni}^0$ $n_1 \qquad n_1$	
$n_1$	=moles of $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ =given wt/molecular wt =5.9427/237.689 =0.025 moles
Weight of $\text{Ni}(0)$ {theoretical} to be formed	= $n_1 \cdot$ molecular weight of Ni =0.025*58.293 =1.467g
Observed weight of $\text{Ni}(0)$ {synthesised}	=0.6395g
%yield	=synthesised wt*100/theoretical wt =0.6395*100/1.467 =43.592%
<b><math>\therefore \text{The Yield of the nanoparticle synthesised is } 43.592\%</math></b>	

### 3.6 Heat Treatment

Before the Heat treatment is started, nitrogen gas is purged at high pressure. This is done to create an atmosphere of Nitrogen and purge out all the air present. If not done properly oxidation might take place and we would not be able to get pure nickel nanoparticles. Heat treatment is done in an atmosphere of nitrogen since it creates an inert environment. Heat under the flow of N<sub>2</sub> gas for 3-4 h.

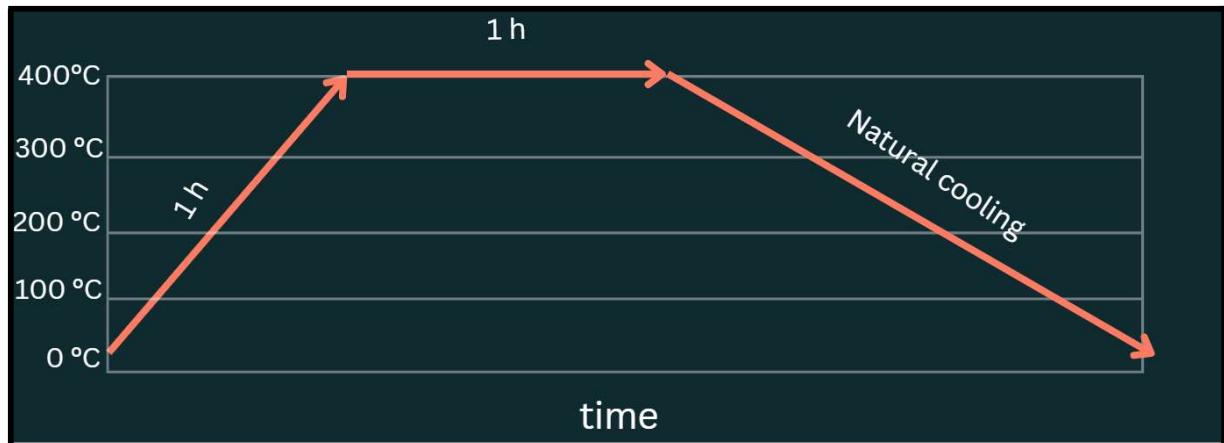


Fig 5: Graph depicting the heat treatment carried out in N<sub>2</sub> atmosphere at 400 °C

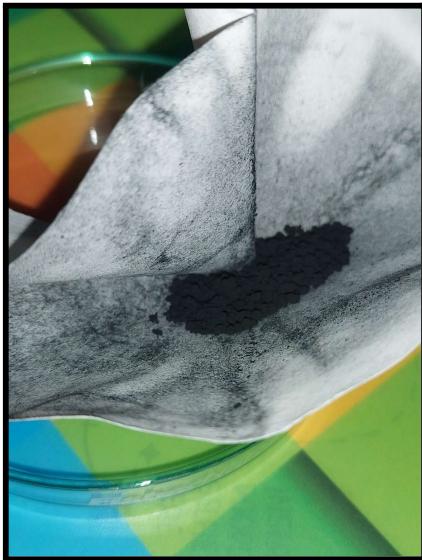


Fig 6: Sample after drying to processed for Heat Treatment



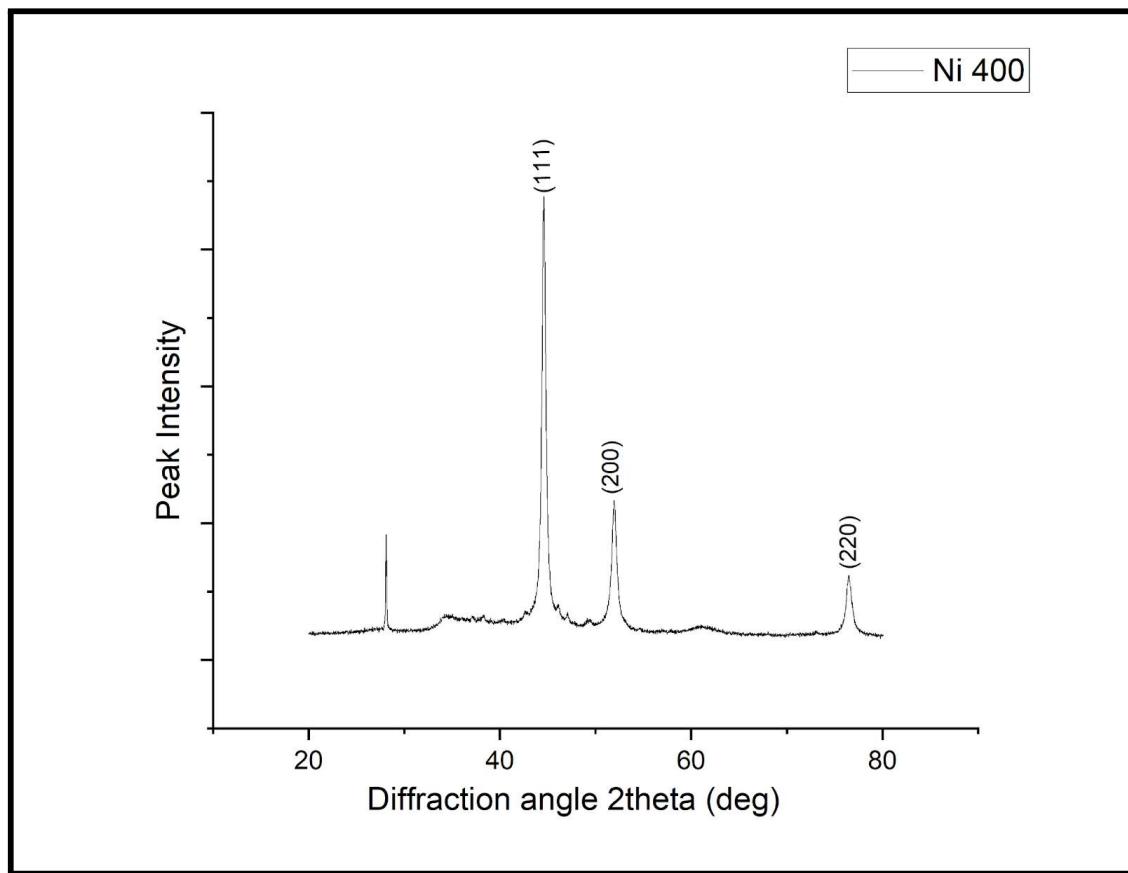
Fig 7: Sample labelled and stored after heat treatment at 400°C

## ANALYSIS

The nanoparticles synthesised were characterised by X-Ray Diffraction and FESEM.

### 4.1 X-Ray Diffraction (XRD)

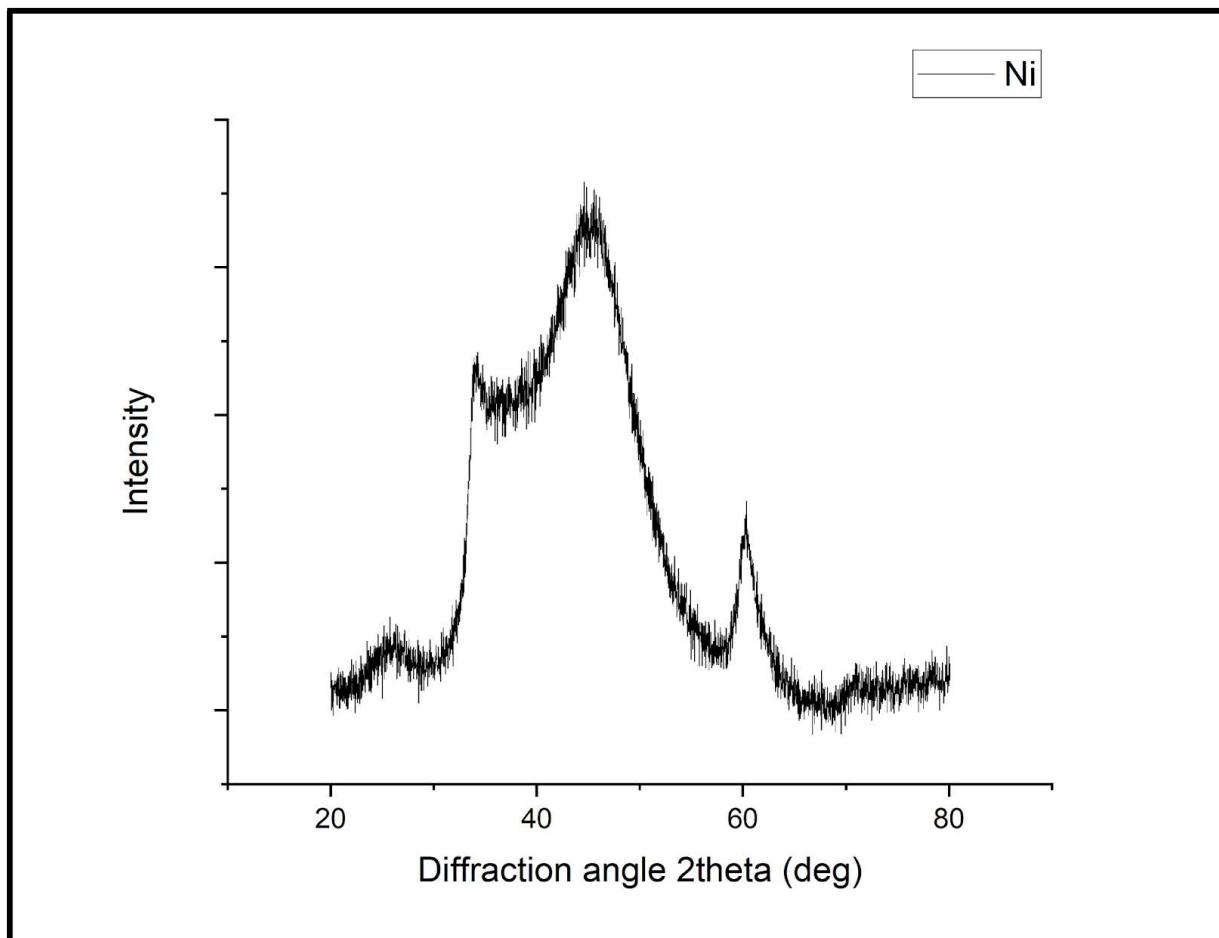
Figure 8 shows the X-ray diffraction pattern of the nanoparticles of Ni samples. From Fig. 8 four characteristic lines of nickel [ $2\Theta = 44.5, 51.9, 76.5$ ] corresponding to Miller indices (1 1 1), (2 0 0), (2 2 0) are observed which exactly coincide with the (JCPD file no. 03-1051 and 04-0850) values. The micron sample has sharp constructive diffraction lines in its XRD patterns variety in the peak intensities broadening in peaks indicates the formation of Ni nanoparticles. Furthermore, the deducted pattern showed no oxides or hydroxide content. It showed a peak at  $2\Theta =$  which on analysis is due to some amount of sodium (from reducing agent sodium borohydride) which might not have been removed from the residue.



**Fig 8:XRD of Ni nanoparticles after heat treatment at 400°C**

**Table 3:Calculation of d from  $2\Theta$  using Braggs Law**

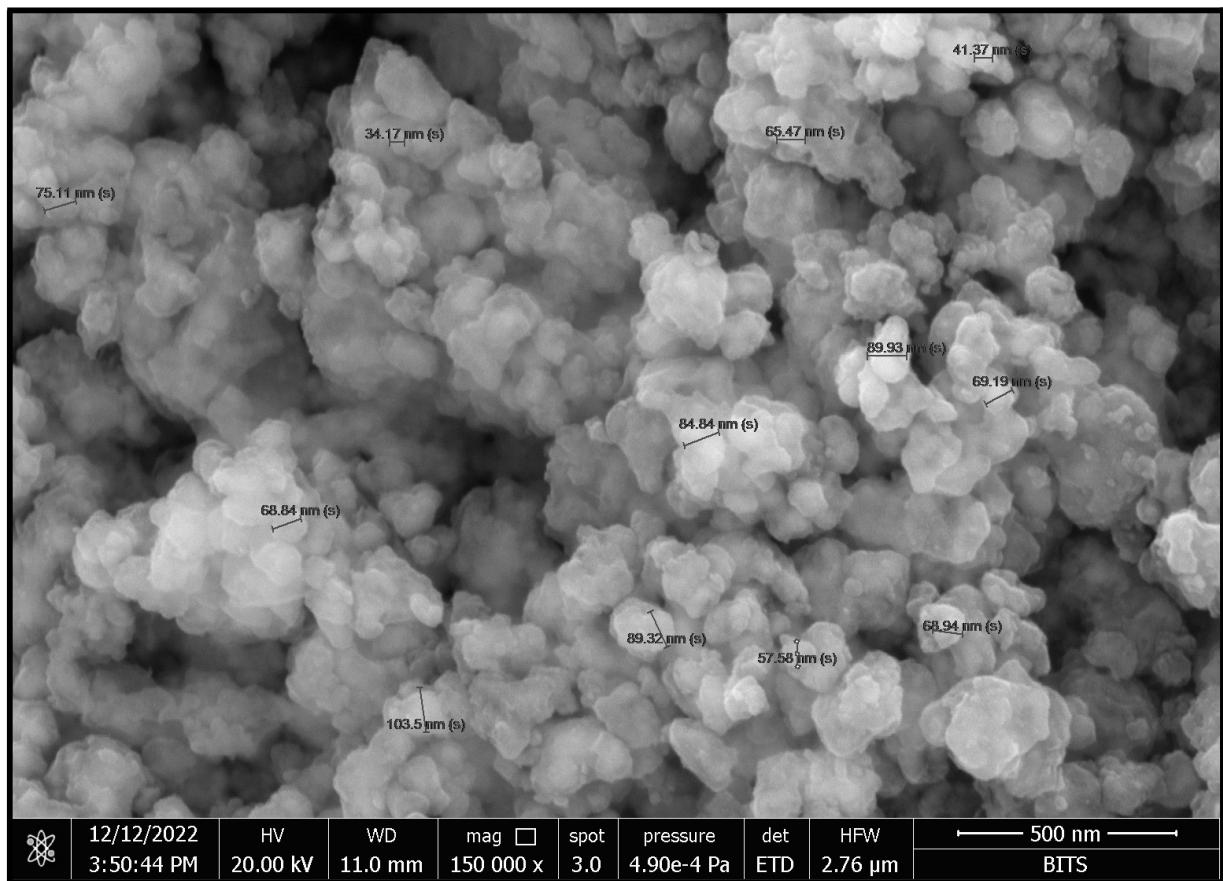
Peak No	$2\Theta$ (deg)	$\Theta$ (deg)	$\Theta$ (radian)	$\sin\Theta$	$2\sin\Theta$	$n\lambda$ (Å)	$d=n\lambda/2\sin\Theta$
111	44.5	22.25	0.39	0.38	0.76	1.54056	2.03
200	51.9	25.95	0.45	0.43	0.86	1.54056	1.79
220	76.5	38.25	0.67	0.62	1.24	1.54056	1.24



**Fig 9:XRD of Ni nanoparticles before heat treatment**

## 4.2 Field emission scanning electron microscopy (FESEM)

SEM image of the obtained nickel nanoparticles is shown in Fig. 5. The shape of the particles is nearly spherical with the homogeneous size distribution. Nickel nanoparticles are in the range of 70nm. The average size calculated from SEM analysis has agreed with that of the size calculated from XRD analysis.



**Fig 10:FESEM image of synthesized Nickel nanoparticles**

## **APPLICATIONS OF NICKEL NANOPARTICLES**

Generally, nanostructures such as nanoparticles, nanotubes, nanowires, nano-springs, nanobelts, and nanorings are utilised in the development of flat-panel display, nonlinear optical devices, light-emitting diodes, transistors, and logic gates. Ni-based materials were also applied in turbines, automobile moulds, and aerospace. The application of Ni nanoparticles includes medical applications, as a catalyst, applications in sensor development, materials enhancement, and dye adsorption. These are discussed in the following sections.[12]

### **5.1 Biomedical applications of Ni nanoparticles**

The use of Ni nanoparticles in biomedical applications and as an antibacterial agent has been reported in the literature. These include drug and gene delivery, magnetic resonance imaging, cell separation, biomedical detection, and diagnostics [13]. Guo et al. reported that functionally charged NiNPs could increase cell membrane permeability and promote cellular absorption into cancer cells of the outer target molecules [14]. These results show that NiNPs may have a possible mechanism for targeting the cytotoxicity of leukaemia cancer cells and that NiNPs may be implemented in related biomedical and clinical fields.

### **5.2 Application of Ni nanoparticles in superconductors and enhancement of materials**

Zhang et al. reported the fabrication of electrodes employing Ni nanocomposite materials. The electrodes showed low charge-transfer resistance, outstanding cycle stability, high specific capacitance, and good rate performance. The high capacitation of the electrode nanocomposite was due to enhanced conductivity, regularly scattered nanoparticles of Ni(OH)<sub>2</sub>, low interfacial resistance, and synergetic effects of each portion. They suggested the composite as a promising material for high-energy supercapacitor applications with improved electrochemical performance [15]. Similarly, Li et al. developed a high-performance electrochemical supercapacitor from amorphous Ni (OH)<sub>2</sub> nanospheres. The developed electrode was found to exhibit high capacitance, high energy density, and long life. They suggested that the electrode can be used in advanced electrochemical pseudocapacitor material [16]. Agegnehu et al. reported the use of Ni and NiO nanoparticles for the enhancement of photocatalytic hydrogen evolution from aqueous methanol. They suggested that the Ni/graphene oxide exhibits high activity attributed to the ease of trapping photogenerated electrons by Ni and NiO nanoparticles [17].

### **5.3 Application of Ni nanoparticles in dye-sensitized solar cells and sensors**

Krishnapriya et al. described the fabrication of dye-sensitized solar cells with various nanostructured TiO<sub>2</sub>. It was incorporated with Ni nanocomposites with morphologies like interconnected bead-like, spindle shape-like, square platelets-like, and porous sphere-like to yield dye-sensitized solar cells. The nanostructures were synthesized in different solvents, namely ethanol, a mixture of ethanol and water, as well as HF in conjunction with shape- and size-tuned Ni nanocomposites of mixed triangular and hexagonal morphological crystals. These had sizes ranging from 15 to 62 nm. They reported that the incorporation of Ni nanocomposites effectively traps incident light and successively improves the rate of electron-hole pair formation and short-circuit current. The fabricated dye-sensitized solar cells were found to exhibit excellent stability in conventional electrolytes over a month [18].

## **CONCLUSION**

Spherical fine Ni nanoparticles were successfully carried out by reducing Nickel (II) chloride hexahydrate and sodium borohydride. The XRD patterns revealed that a homogeneous structure of Ni particles was obtained. In addition, The FESEM image ensures the formation of spherical fine Ni particles which were well structured. The magnetic properties of the nanosized Ni depend strongly on its size. This method is a relatively cheap process and environmentally friendly, and could be used in an efficient way to produce low-size distribution, high purity and nearly spherical shape Ni nanoparticles.

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