ISSN: 0974-5823 Vol. 7 No. 1 January, 2022

International Journal of Mechanical Engineering

Synthesis of nickel oxide Nanoparticles by hydrothermal method

Sura sabri hasson ,AbdulkareemM.A.Alsammarraie

Department of Chemistry/College of Science/University of Baghdad/Baghdad/Iraq

***Abstract*:**

Nickel Oxide NPs are well-known as excellent photo catalyst because of their unique optical properties such as band gap energy, retention time, the high light absorption, electron-hole recombination time, and high negative reduction-oxidation potential of excited electrons. Nanoparticles of (NiO) were synthesized successfully from the reaction .of a mixture of nickel hexahydrate precursor, ethanol, and distilled water was effectively used to make nanoparticles of NiO in a hydrothermal Teflon-lined stainless-steel bomb at temperature of 120 °C . Particle size and surface morphology of the prepared NiO nanoparticles were calculated using Scanning Electron Microscopy (SEM), they show a NiO spherical particle and rod patical with diameters around 60 nm. X-Ray diffraction (XRD) revealed a NiO cubical crystal structure. The absorption spectra of NiO NPs in the ultraviolet and visible ranges show a blue at λ max of 341 nm. . Fourier transforms infrared (FTIR) spectroscopic analysis show the nickel oxide band at 627 cm−1.

***Keywords*:** Nickel Oxide nanoparticle; hydrothermal method; UV-visiblespectroscopy; X-ray diffraction (XRD); scanning electron microscopy (SEM).

1.Introduction:

NiO is a semiconductor material with an inorganic component necessary for its function. It is a material with a wide bandgap ranging from 3.6 to 4.0 eV, and nickel oxide is a P-type semiconductor, and it has good electron transfer efficiency. Its conduction type is related to the internal oxygen vacancies [1]. . Nanoparticles have attracted a lot of attention and aroused the interest of scientists due to their potential applications and their outstanding physical and chemical properties. The structural property of the particles is directly related to the preparation procedures (particle size, distribution, and morphology) [2]. and the low cost and high sensitivity make them more attractive although there are limitations, such as the lack of selectivity and the strong interference effect of water vapor that can limit its use as measuring instruments [3], But its electrical and magnetic properties in addition to its surface chemistry have increased its importance which made it get a lot of attention recently. Because it produces good homogeneity, crystallization, low reaction temperatures, low energy consumption, easy sample preparation, high purity, and stable nanoparticles, a process was chosen as Hydrothermal synthesis [4, 5] . This type of material properties aroused the authors' interest in synthesis, structural and optical properties [6]. The high-pressure and high-temperature hydrothermal method can be utilized to produce highly crystalline materials in a single step that does not require any post-annealing treatment and enhances the production rate at lower reaction temperatures, making it the most promising method for heterogeneous structure synthesis [7].

2.Experimental Part

The hydrothermal process took place in an autoclave, a steel cylinder with a thick wall that was hermetically sealed to endure high pressures and temperatures for extended periods. A corrosion-resistant Teflon vessel was introduced into the autoclave's internal cavity. The vessel had a 30 mm inner diameter and a capacity of 120 mL. Many of the chemicals were analytical and had not undergone any further purification. At 120 degrees Celsius[8], the trials were carried out with various composition concentrations. The following were the details of the experiment: Nickel hexahydrate (C4H6NiO4 6H2O), ethanol, and distilled water to maintain the pH of the process, 0.1 M (1.294 g) of nickel precursor was dissolved in 20 mL of deionized water-ethanol (volume 1: 1 ratio). The NaOH solution was gradually added to the solution.Continuous stirring is required for a patch nickel solution to work.The pH level was kept constant at 11. These homogenous solutions were then transferred to a 50 mL autoclave with a Teflon liner and heated at 120 °C[9] for 12 hours daily. These reaction mixtures were heated for 5 minutes at 5 degrees Celsius. After the reaction was finished, it was cooled to room temperature and the powder sample was thoroughly washed with deionized water until it achieved a neutral pH of 7. After drying the samples for 12 hours at 80 ° C, a green powder was obtained[10].

X-ray diffraction was used to investigate the structure of the NiO samples using a Siemens model D500, SEM ZEISS model: sigma VPEDS and mapping: oxford instruments, UK.the Fourier transform infrared spectroscopy (FTIR) Shimadzu FT-IR8400S, Japan was applied.Shimadzu UV 1800 twin beam, Japan, area (200-1100) nm, was used to get the absorption spectra, and to analyze the composition and quality of the compound in the rang (4000-400 cm-1)

3.Result and discussion

3.1XRD analysis

XRD Pattern of [NiO] nanostructure, the peaks are contributed to NiO, which has diffraction peaks at the surface 2θ = 19.4°, 38.6°, 62.8° which are matching to crystal planes of (001), (111),(220) respectively, (JCPDS No. 10-0325).The size of the crystallite can be calculated using the Debye-Scherrer formula[11]:

$$D = k\lambda / \beta COS\theta$$

The average crystallite sizes were estimated to be (30.2 nm).The diffraction peaks correspond to pure cubic[12], shown in figure (1).

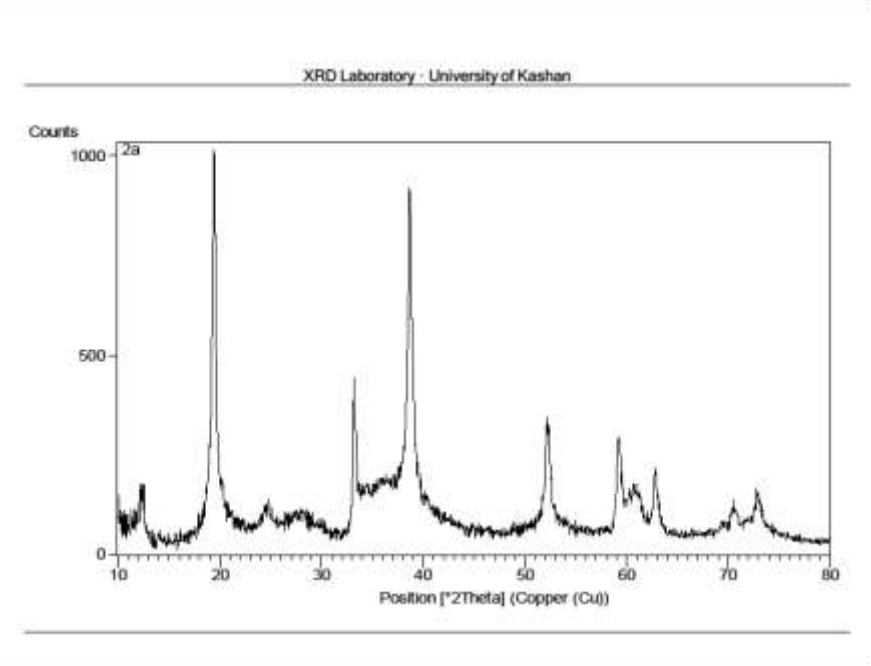
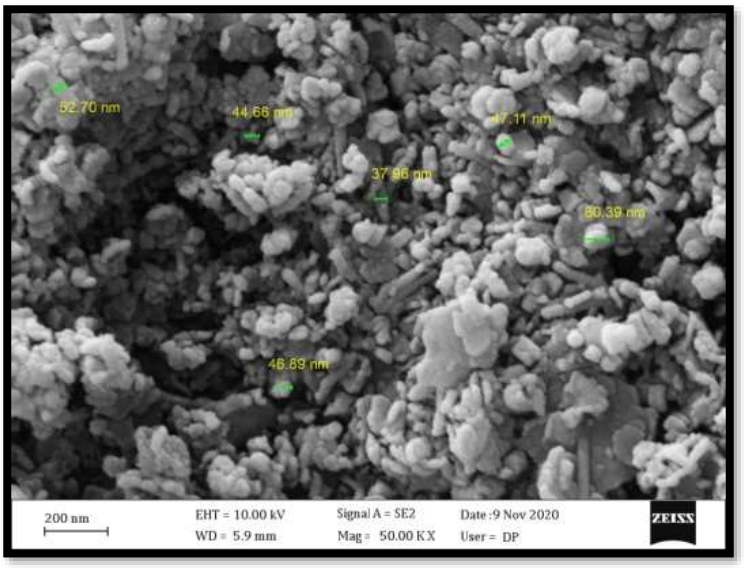


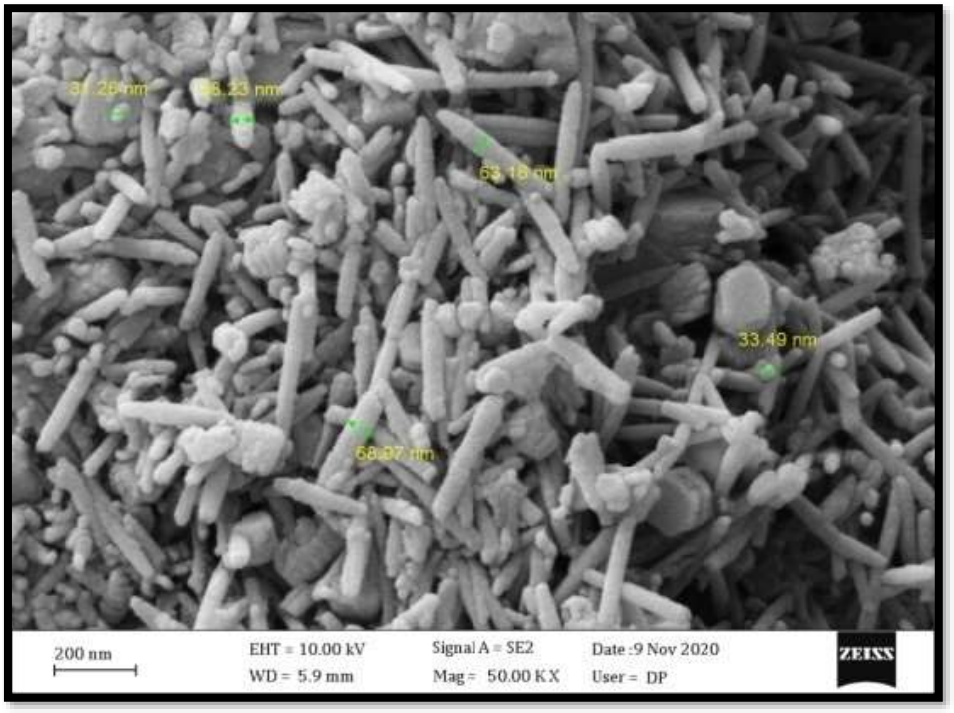
Figure (1): XRD Pattern of NiO nanostructure by hydrothermal method.

3.2.SEM images

The diagnostics of the nanocomposites showed different shapes and sizes of the nanoparticles of nickel oxide prepared by the hydrothermal method Images were recorded by SEM of the nanocomposites of nickel oxide at different magnifications[13] and all indicated the compounds prepared It contains nanoparticles with wires and a small rod of size around (31.29nm,53.28nm,63.16nm), as well as discovered spherical aggregate compounds of size around (37.36,47.11nm,52.70nm).Showfigure(2,3)



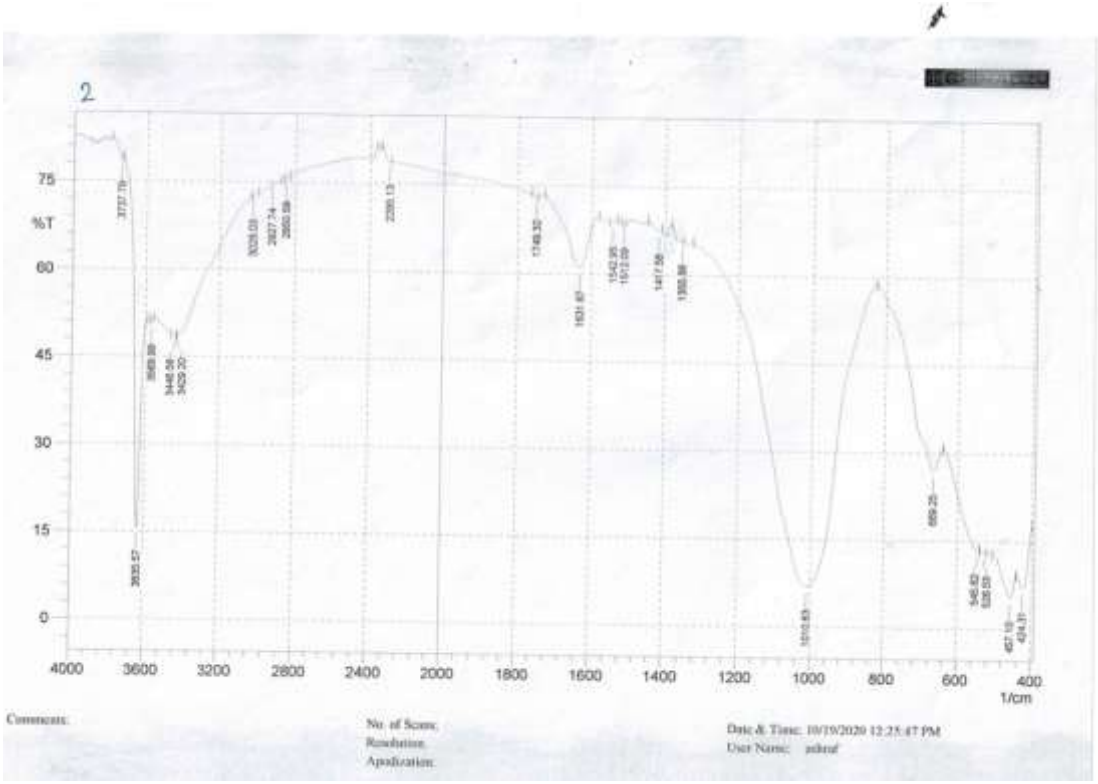
Figure(2):SEM NiO nano prepared by the hydrothermal method spherical shape



Figure(3): SEM NiO nano prepared by the hydrothermal method,wires and a small rod shape

3.3.FTIR spectra

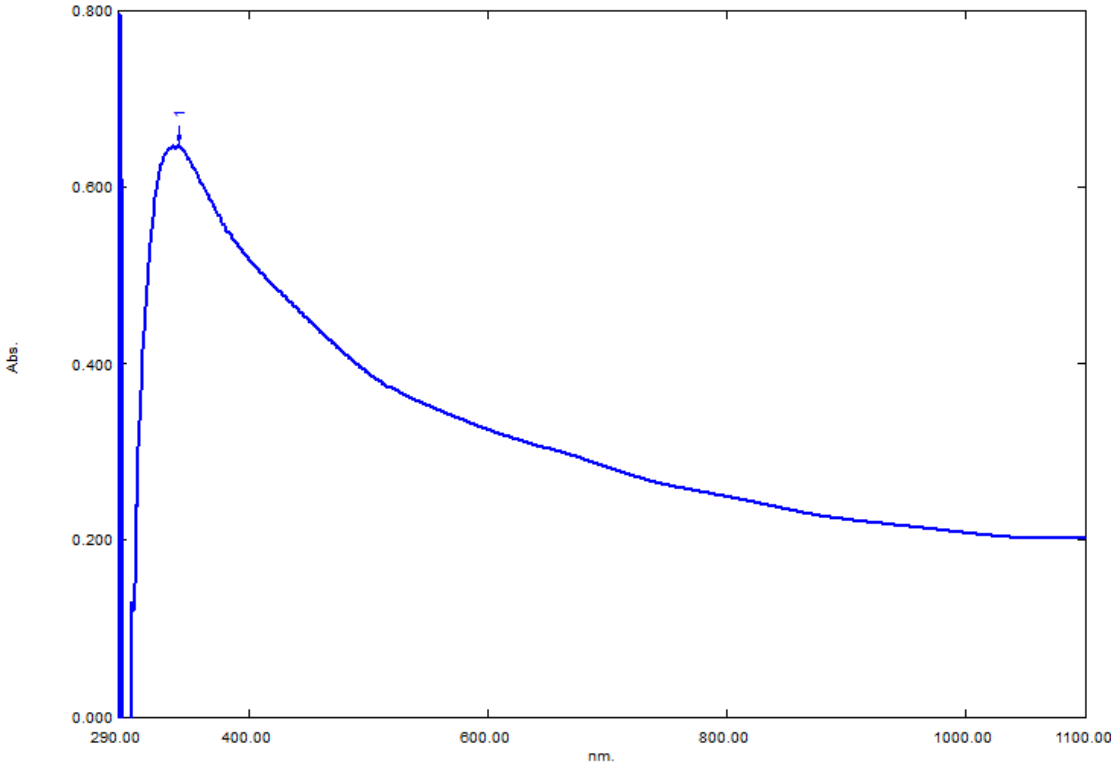
The FTIR spectra of the sample were investigated in the 600–4000 cm 1 spectral range. The FTIR map for NiO has been organized. The peak, which belonged to the FTIR results, was discovered in the 4000-400 cm1 region,belonged to the FTIR performance, was discovered to be in the 4000e400 cm1 region. Furthermore, the peaks have been discovered at 457, .1010,1355, and 3559 cm1, The stretching vibration of Ni-O has been assigned to the observed wide peak in the 450-855 cm1 region. However as shown in Moisture absorbed/adsorbed on the surface of theThe broad absorption peak at n=1020 cm-1corresponds to the stretching band of C-O bond, which is due to the presence of the acetate,and shown in moisture absorbed on the surface of the nanoparticles and the (OH) hydroxyl groups[5], respectively, may explain the weak bands at 1645 and 3468 cm1.shown Figure**(4).**



Figure(4): The Analysis of (FT-IR) of NiO nanoparticles by hydrothermal

3.4.UV-VIS spectra

The UV-VIS spectra of NiO samples are shown in Figure(5). It shows a strong absorption peak by NiOaround 341 nm corresponding to the optical band gap[14, 15].



Figure(5): shows the UV-Vis absorption spectra of the synthesized NiO by hydrothermal method.

Conclusion

A hydrothermal technique was used to successfully produce nanocrystalline NiO at 120 °C for 12 hours at various NaOH concentrations. The average crystallite size of NiOnanoparticles was determined by X-ray diffraction tests to be 40 nm, 61 nm, 39 nm, and 45 nm for NaOH 5M concentrations.In the ultraviolet and visible ranges, the absorption spectra of NiO NPs display a blue peak at 341nm.

References

1. Mironova-Ulmane, N., et al. *Structural and magnetic properties of nickel oxide nanopowders*. in *Solid State Phenomena*. 2011. Trans Tech Publ.
2. Al-Hajry, A., et al., *Low-temperature growth and properties of flower-shaped β-Ni (OH) 2 and NiO structurescomposed of thin nanosheets networks.* Superlattices and Microstructures, 2008. **44**(2): p. 216-222.
3. Noukelag, S., et al., *Structural and optical investigations of biosynthesized bunsenite NiO nanoparticles (NPs) via an aqueous extract of Rosmarinus officinalis (rosemary) leaves.* Materials Today: Proceedings, 2021. **36**: p. 245-250.
4. Agrawal, S., A. Parveen, and A. Azam, *Microwave assisted synthesis of Co doped NiO nanoparticles and its fluorescence properties.* Journal of Luminescence, 2017. **184**: p. 250-2.55
5. Bashir, A., et al., *Biosynthesis of NiO nanoparticles for photodegradation of free cyanide solutions under ultraviolet light.* Journal of Physics and Chemistry of Solids, 2019. **134**: p. 133-140.
6. Ramasami, A.K., M. Reddy, and G.R. Balakrishna, *Combustion synthesis and characterization of NiO nanoparticles.* Materials Science in Semiconductor Processing, 2015. **40**: p. 194-202.
7. Virgin Jeba, S., et al., *Synthesis, growth mechanism and photocatalytic properties of nickel oxide (NiO) nanoflower: a hydrothermal process.* Inorganic and Nano-Metal Chemistry, 2020: p. 1-11.
8. Byrappa, K. and M. Yoshimura, *Noyes Publications.* New Jersey, USA," Handbook of Hydrothermal Technology, 2001.

9. Jain, G., L. Patil, and V. Gaikwad, *Studies on gas sensing performance of (Ba0. 8Sr0. 2)(Sn0. 8Ti0. 2) O3 thick film resistors.* Sensors and Actuators B: Chemical, 2007. **122**(2): p. 605-612.
10. Ahire, D., et al. *Synthesis of nanostructured NiO by hydrothermal route and its gas sensing properties*. in *2012 Sixth InternationalConference on Sensing Technology (ICST)*. 2012. IEEE.
11. Krishnaveni, T., et al., *Exploration of Catalytic Activity of Quercetin Mediated Hydrothermally Synthesized NiO Nanoparticles Towards C–N Coupling of Nitrogen Heterocycles.* Catalysis Letters, 2020 :(6)150 .p. 1628-1640.
12. Yi, C., et al., *A facile hydrothermal synthesis of graphene/RuO 2/Co 3 O 4 nanocomposites with high pseudocapacity.* New Journal of Chemistry, 2018. **42**(9): p. 7066-7072.
13. Dhas, S.D., et al., *Synthesis of NiO nanoparticles forsupercapacitor application as an efficient electrode material.* Vacuum, 2020. **181**: p. 109646.
14. Suyver, J., et al., *Synthesis and photoluminescence of nanocrystalline ZnS: Mn2+*. Nano Letters, 2001. **1**(8): p. 429-433.
15. Santhoshkumar, A., H.P. Kavitha, and R. Suresh, *Hydrothermal synthesis, characterization and antibacterial activity of NiO nanoparticles.* Journal of Advanced Chemical Sciences, 2016: p. 230-232.