



(RESEARCH ARTICLE)



Synthesis of NiO: BaO nano structure by chemical method with Antibacterial activity to *Escherichia coli*, *Klebsiella spp.*, *Staphylococcus aureus*, *Staphylococcus epidermidis* and *candida spp*

Wahab Basim Mahdi ¹, Sinan Mohammed Hassan ² and Zaid Ali Hussein ^{2,*}

¹ Department of Physics, College of Science, Al Mustansiriyah University, Baghdad, Iraq.

² Al-Nahrain Renewable Energy Research Center, Al-Nahrain University, Baghdad, Iraq.

International Journal of Science and Research Archive, 2024, 13(01), 2446–2454

Publication history: Received on 28 August 2024; revised on 06 October 2024; accepted on 08 October 2024

Article DOI: <https://doi.org/10.30574/ijrsra.2024.13.1.1885>

Abstract

Nanostructured materials have garnered significant attention due to their remarkable chemical and physical characteristics. Because of their exceptional qualities and potential uses in technology. Nanostructured transition metal oxides are one of the many nanomaterials that need particular attention. The goal of this study was to synthesize nickel oxide and barium oxide nanostructures by the reaction of Nano powders: nickel sulphate NiSO₄, barium chloride BaCl₂, and molten sodium hydroxide, potassium hydroxide (NaOH: KOH) and examine their physical and biological properties. According to the results of UV-Vis, the gap band of the nanostructure was calculated with a range of 3.6–3.27 eV. Scanning Electron Microscopy (SEM) pictures appear to create aggregated nanoparticles with particle size of 29 nm according to Transmission electron microscopy (TEM) with a spherical shape. This results proved that NiO: Ba nanostructure an efficient and stable. It was observed that the diameter of the inhibition zone bacteria include *E. coli* and *Klebsiella spp.* (21,18) mm while inhibition zone of bacteria *S. aureus*, *S. epidermidis* gram positive bacteria and yeast *candida spp.* (19,19 and 21)mm. As can be shown, the antibacterial activity of the (NiO: BaO), nanostructure increases with decreasing concentration and is more effective against *E. coli* gram-negative than it is against *S. aureus* and *S. epidermidis* gram-positive. This finding might be explained by the fact that, due to differences in their cell wall composition, gram-negative bacteria are more vulnerable to antibacterial medications than gram-positive ones. The synthesized NiO: Ba nanostructures had the potential for higher inhibition and killing of gram-negative than gram-positive bacteria.

Keywords: NiO: Ba; Antibacterial; UV-Visible; SEM and TEM

1. Introduction

Owing to their remarkable chemical and physical characteristics, nanostructured materials have garnered significant attention. Because of their exceptional qualities and possible uses in technology (Djaja, N.F *et al.*, 2014). nanostructured transition metal oxides are one of the many nanomaterials that require particular attention. Specifically, anomalous electrical and magnetic properties, as well as unique catalytic capabilities, are displayed by nanoscale nickel oxide (Ahmed, K.B. *et al.*, 2018). Another important application of NiO is in battery systems due to its defective structure, a possible gas sensor for H₂ and highly active in phenol degradation, phenolic derivatives, and non-stoichiometric nickel oxide is a strong P-type semiconductor and dyes (Adhikary *et al.*, 2015). Nickel NPs have potential applications in various fields including electronics, magnetism energy technology and biomedicines because their high reactivity, operational simplicity and eco-friendly properties they are used to catalyze various organic reactions including the chemo-selective oxidative coupling of thiols, reduction of aldehydes and ketones, hydrogenation of olefins, synthesis of stability from alcohol through Wittig-type olefination and α -alkylation of methyl ketone, catalyze some inorganic

* Corresponding author: Zaid Ali Hussein

reactions such as ammonia decomposition. Their position in the manufacture of carbon nanotubes is one of their latest applications (CNTs), which have cytotoxicity against malignant cells, as exhibited by the morphological changes these cells are knowledgeable following their exposure to Ni NPs. When Ni NPs are capped with biomolecules like glucose, their biocompatibility is greatly enhanced, and they can be utilized as heat nonmediators and biosensors for cancer. hyper thermal (Imran and Rani,2016) .]. NiO nanoparticle is recognized as an efficient photocatalyst in the removal of dyes and organic contaminants from wastewater due to its capacity to successfully treat a field of bacterial infections (Khawlah Salah Khashan et al ., 2016). It is also good to impart cytotoxic activity on different cells such as human epithelial airway (HEp-2) and human breast cancer. More people are interested in nickel oxide nanoparticles because of their unique characteristics. It is a p-type semiconductor with a wide 3.6–4.0 eV band gap, chemical strength, and excellent electro-optical efficiency for a range of uses (Ezhilarasi et al.,2017). The top of the valence band in NiO consists of O_{2p} bands while the bottom of the conduction band comprises Ni_{3d} states (Bonomo,2018). Nickel oxide nanoparticles are employed in a wide field of applications, such as gas sensors, adsorbents, catalytic agents, solar and fuel cells, magnetic materials, and antimicrobial substances. Small particle sizes—ideally less than (20 nm) must be achieved for NiO nanoparticles to maximize their potential applications' efficacy. The physicochemical properties are influenced by the size, shape, and strong crystallinity of the particles. The nickel oxide (NiO) nanostructure is of particular interest due to its superior chemical and thermal properties (Dcruz,2020). Additionally, NiO shows the least amount of non-stoichiometry in its bunsenite form $Ni_{1-x}O$ ($x < 0.001$). However, the introduction of dopants such as iron (Fe) creates defect formations within the NiO structure (Arfan *et al.*,2018). The composite-hydroxide-mediated (CHM) approach is a relatively new chemical synthesis pathway that has garnered significant attention in research because of its ease of use and versatility in creating a wide variety of nanomaterials. In contrast to other chemical processes, CHM is a very straight forward and eco-friendly method. The CHM technique is simple and relies on the usage of basic source material and molten hydroxides (NaOH: KOH). A range of nanostructures, including ($BaTiO_3$, CdO, ZnO, and NiO), are created chemically (*Shahid et al.*,2018) .Although confirmed oxides, such as $BaNiO_3$, contain the nearby element nickel, similar high oxidation states of the transition metal are observed; however, these oxides have not been thoroughly studied. Chain units ($NiO_6/2$) $\infty\infty$ -oriented parallel to each other are produced by the NiO_6 octahedral sharing faces, which can be used to define the metal/oxygen sublattice of $BaNiO_3$ (Figure 1). Because they frequently stop bacteria from promote a resistance to them, nanoparticles are used as possible antibacterial agents..(Wang L *et al.* , 2017) Currently, silver and gold nanoparticles are widely used as antimicrobial agents.(Beyth N *et al.* ,2015) But moreover research is needed to fully understand the antibacterial characteristic of NiO nanoparticles. (Panyala NR *et al.* ,2009). Thus,this study was aimed to study the antibacterial effects of NiO nanoparticles on the P. aeruginosa, S. aureus, E.coli, K. pneumoniae, S. marcescens, S. epidermidis, M.luteus and B. subtilis. Additionally, the techniques used to synthesize NiO nanoparticles (e.g. hydrothermal,sol-gel, solid-state reaction, electrochemistry, micro emulsions, spray pyrolysis and precipitation methods) are usually complex and expensive.(Shanaj BR and John XR , 2016) Therefore, we also introduced a new simple and cheap method to produce NiO nanoparticles.

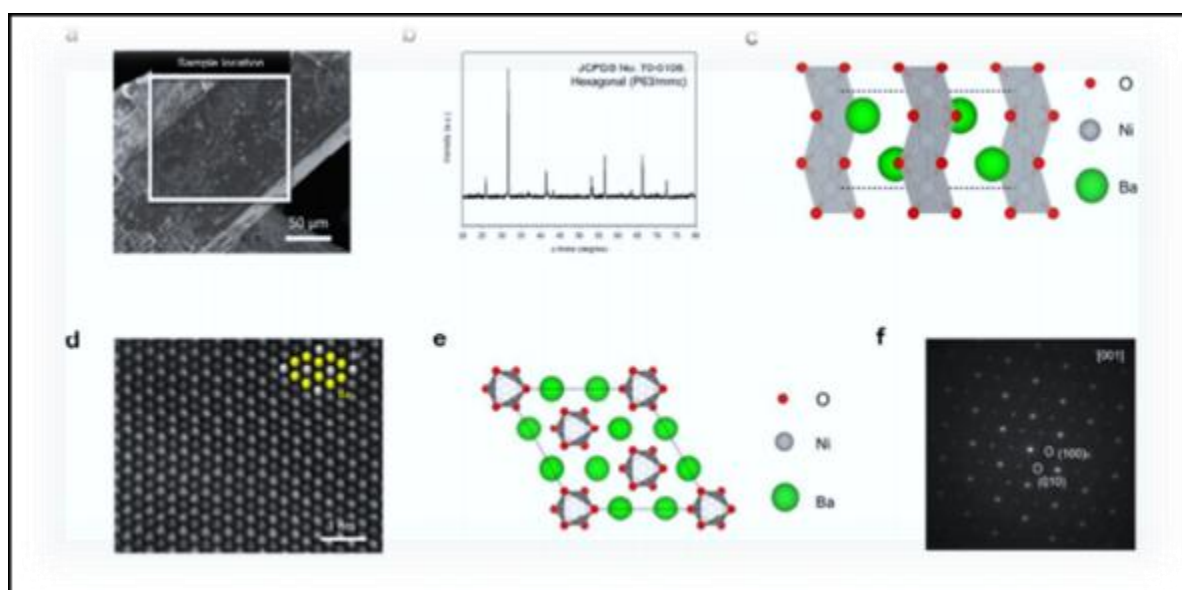


Figure 1 Morphology and crystal structures of the $BaNiO_3$ (Devamani and Alagar,2014)

2. Methods

2.1. Synthesis NiO:Ba nanostructure using chemical method

NiO: BaO Nanostructure was prepared by chemical wet method using nano powders, nickel sulfate NiSO_4 , barium chloride BaCl_3 with molten sodium hydroxide, potassium hydroxide (NaOH: KOH). The analytical grade was of all the chemicals and solvents used. The solution consisting of (NiSO_4 , NaOH: KOH) with salt ratios (2, 0.1: 0.1) g. dissolved in 50 ml deionized water (DDW) and 50 ml ethanol and put the mixture on the magnetic stirrer at room temperature through 10 min. to complete formation NiO. Then, added 2 g. of BaCl_3 the diluted by deionized to solution and the mixture was stirred until was obtained a new Nanostructures (NiO: BaO) Nanostructure at one hour. A specific quantity of byproduct (NiO: BaO) Nanostructure has been dissolved in different volumes of deionized water. (10:5, 7.5: 2.5, 6:1 mg/ml). It was stored in plastic containers and later used to treat various types of cancer cells as shown in fig.(2).

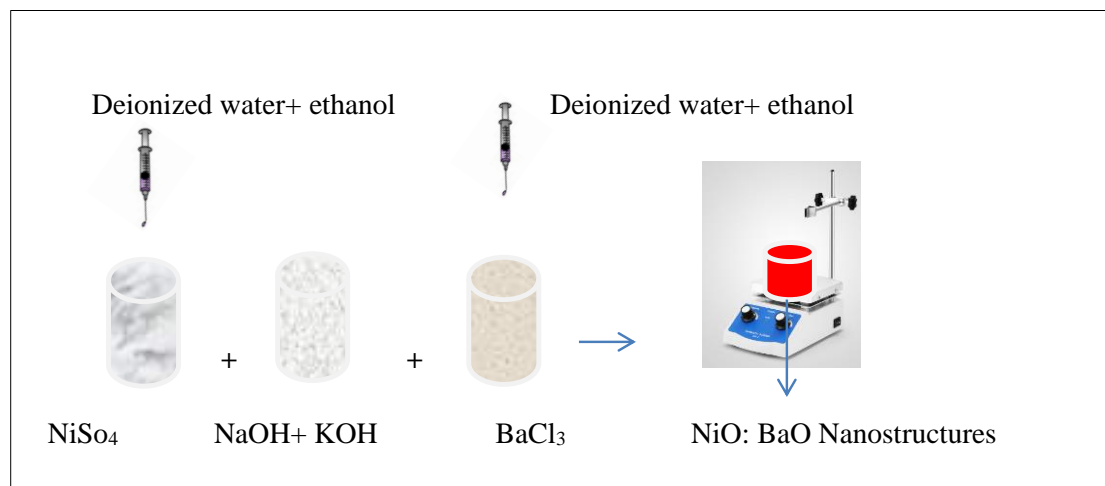


Figure 2 Synthesis NiO:Ba nanostructures using chemical method

3. Results and Discussion

3.1. Analysis UV-Vis characterization for nanostructures NiO: BaO

Properties of NiO: BaO nanostructures optical such as transmittance, absorbance, and energy gap were measured by the UV-Vis absorption spectra as shown in figure (3) that the absorption gradually decreases with increasing wavelength. Thus, absorption edges can be determined for the samples synthesized at different molar ratios (10 mg/ml, 7.5 mg/ml, and 6 mg/ml) in the precursors within the wavelengths (290, 255, and 231 nm) respectively which correspond to the NiO: BaO Nano powders with optical band gap energies of (3.6, 3.41 and 3.27 eV) respectively. Figure (3) shows the relationship between the transmittance spectrum as a function of the wavelength of the (NiO:Ba) nanostructures sample that was measured by the UV absorption spectrum that the transmittance gradually increases at the concentrations (10 mg/ml, 7.5 mg/ml and 6 mg/ml) with the increase in the wavelength (359 nm, 380 nm and 390 nm) respectively, then it takes the plane shape to saturation due to indirect electronic transitions and the occurrence of a change in the values of wavelengths with increasing concentrations (Jaisai, M. *et al* 2013).

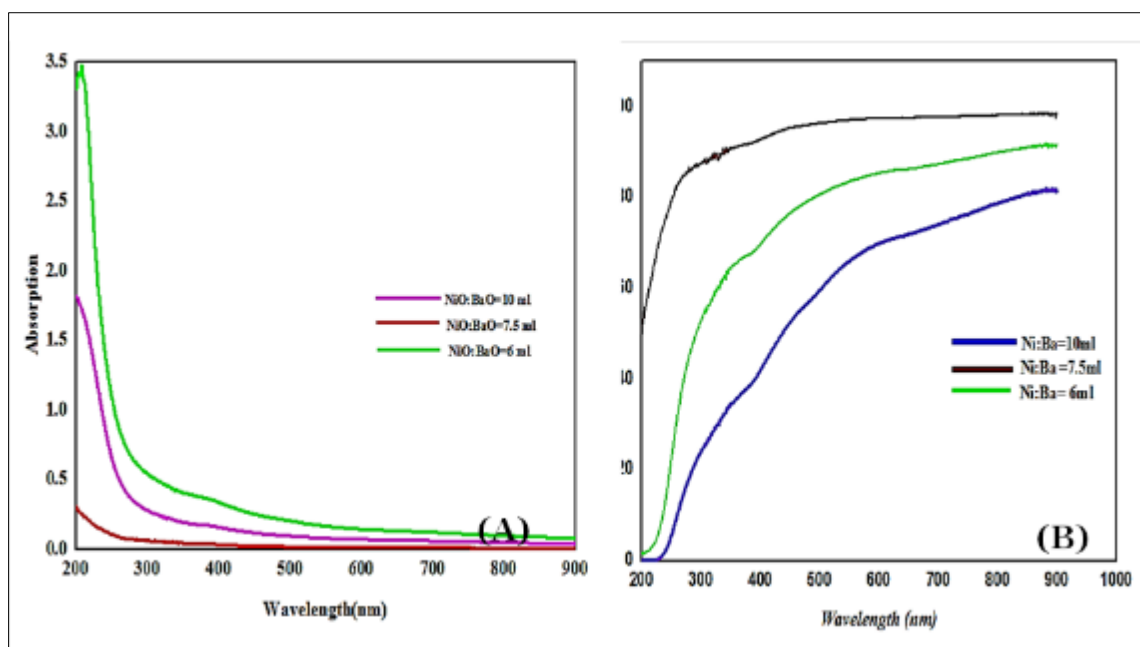


Figure 3 A- Ultraviolet-visible absorption spectra of NiO:Ba nanostructure synthesized at different molar ratios at room temperature and a constant reaction time (10 min) . B- Ultraviolet-visible absorption spectra of (NiO: BaO) Nanostructure synthesized at different molar ratios at room temperature and a constant reaction time (10 min)

3.2. FT-IR spectra analysis of (NiO: BaO) Nanostructure

Results in figure (4) show FT-IR transmission spectra were taken on a JASCO infrared spectrometer in the range of (4000–400 cm^{-1}) at room temp. The peak around (3414 cm^{-1}) on the FT-IR spectrum is related to the O-H bond. The absorption at (1632 cm^{-1}) attributed to hydroxyl groups. The absorption bands at (1420 cm^{-1} and 1117 cm^{-1}) indicate the existence of carbonates. As shown in Fig 4, the absorption bands at (470 and 522 cm^{-1}) are associated with NiO: BaO nanostructure vibration bond, but the absorption band at (619 cm^{-1}) is assigned to Ni-O-H stretching bond. The above information confirmed the formation of NiO: BaO nanostructure. Presence of Carbon impurity in the samples (B. Chinnappa Reddy *et al.*, 2022).

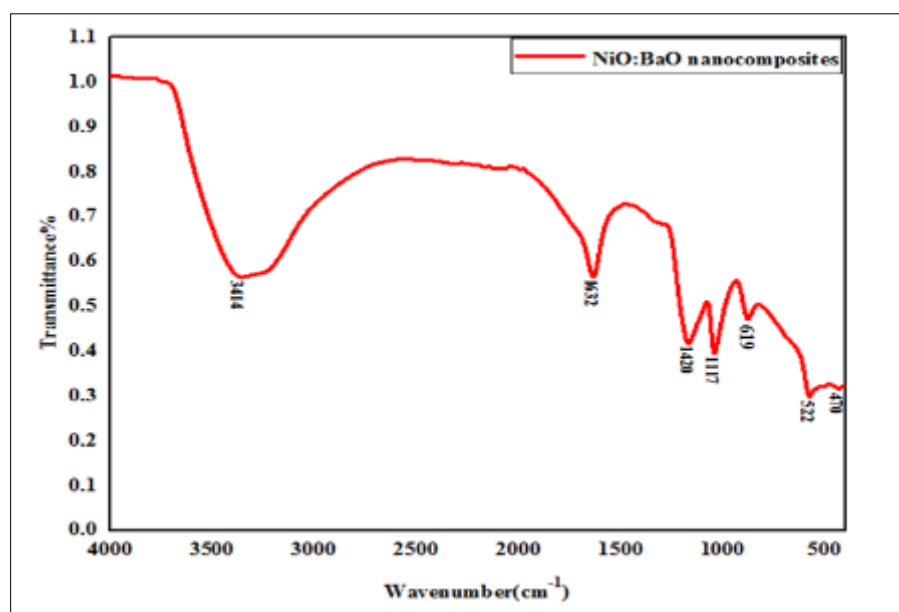


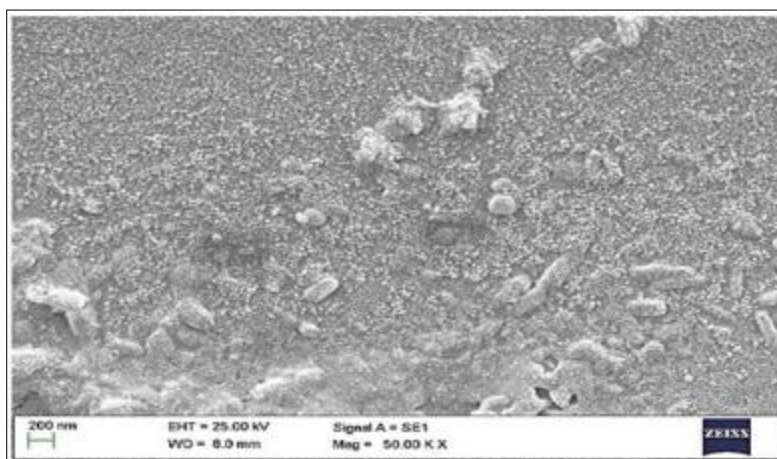
Figure 4 FTIR spectra of (NiO: BaO) Nanostructure synthesized at various concentrations

Table 1 Assignment of characteristic IR absorption bands of (NiO: BaO) Nanostructure.

Wavenumber (cm ⁻¹)	Functional group	Reference
3414	OH stretching vibration	(Q.A. Drmosh <i>et al.</i> , 2010)
1632	C =C stretching vibrations	(B. Chinnappa Reddy <i>et al.</i> , 2022)
1420	C–O–C stretching of epoxy groups	(W. Qin, C <i>et al.</i> ,2011)
1117	C–O stretching of alkoxy group	
619	asymmetric epoxide bands	
522	OH out-of-plane bend	(I. O. Faniyi <i>et al.</i> , 2019)

3.3. Analysis SEM characterization for (NiO: BaO) Nanostructure

Results in figure (5) shown the SEM images of the product NiO: BaO nanostructure synthesized which Nano colloidal samples were investigated morphologically are produce because of the aggregation of small particles that high surface free energy. The generate nanoparticles (NPs) were small size, indicating efficient synthesis. NiO surface is composed of oval nanoparticles which agglomerate and increase along the surface with Ba-doping. At 10% of BaO atoms, the substrate becomes completely covered with NiO particles exhibiting Nano agglomerations continue its formation in becoming micro agglomerations (Jiban Podder ,2015).

**Figure 5** The SEM image of (NiO: BaO) Nanostructure

3.4. Transmission Electron Microscopy (TEM) analyses for NiO: BaO nanostructure

The size and morphology of the nanoparticles were characterized by TEM. Typical images of the NiO :BaO nanostructure are shown in figure (6, 7). The TEM image of the clearly shows a particles with a oval shape having a diameter of about 50nm. However, TEM image of NiO: BaO nanostructure reveal oval particles shape with smooth and uniform particle morphology, results show the particles are irregularly distributed and the average size between 0.29 and 0.55 nm with mean size of the obtained nanoparticles of 0.40 nm, as shown in the histogram. in addition notice that the mean particle size calculated by program(MBF Image) (Pratima R.Solank *et al.*,2015).

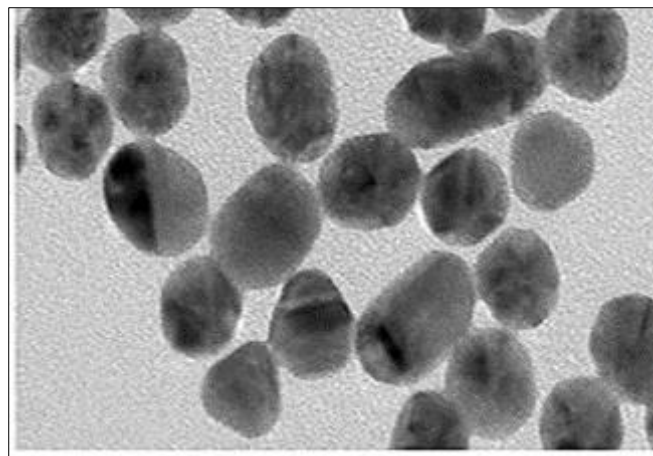


Figure 6 TEM images for (NiO :BaO) Nanostructure

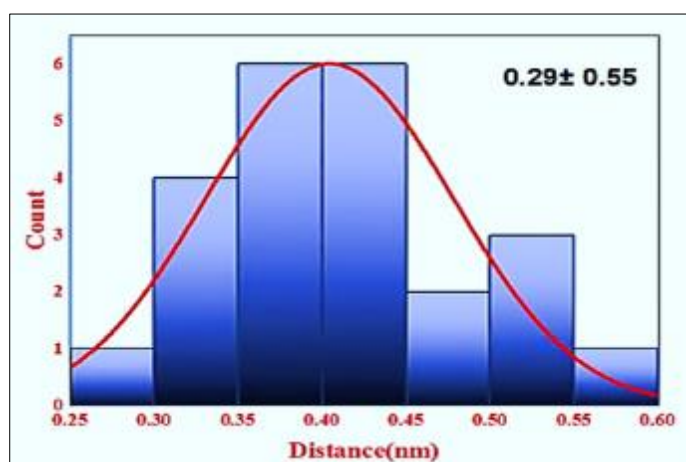


Figure 7 The size distribution histogram with curve of normal-log distribution, mean value

3.5. Antibacterial activity

The inhibitory zone (mm) assay was used to value the generate antibacterial activity (NiO: BaO) nanostructure at different doses. And to determine the extent of the sensitivity of bacteria to these nanocomposites, the diffusion zone was measured for each petridish containing this substance.. It was observed that the diameter of the inhibition zone bacteria include *E. coli* and *Klebsiella spp.*(21,18)mm while inhibition zone of bacteria *S. aureus*,*S. epidermidis*gram positive bacteria and *candida spp.*(19,19 and 21)mm. The final ZOI is displayed in table (1), and figure (8) provides a selection of the experimental petridish plate photographs. As can be shown, the antibacterial activity of the NiO: BaO nanostructure increases with decreasing concentration and is more effective against *E. coli* (gram negative) than *S. aureus* and *S. epidermidis* (gram positive). This finding might be explained by the fact that, due to differences in their cell wall composition, gram negative bacteria are more vulnerable to antibacterial medications than gram positive ones (Chithra MJ *et al.* , 2012). Either direct contact with cells or inferior products corroding microorganisms could be the source of the action. One possible mechanism is a synergistic interaction, involving the nanostructure (NiO:BaO), metal ions, reactive oxygen species, and NP form. Therefore, their capacity to generate dense metal ions that might interact with bacterial cell membranes and introduce them as possible antibacterial agents makes sense as an antibacterial activity of these ions (Shkir M and Abbas HM , 2021).

Table 1 Zone of inhibition (mm) measurements for the five selected groups(Sarmad Ghazi Al-Shawi *et al.* , 2021)

Inhibition zone diameter (mm)				
<i>Staphylococcus aureus</i>	<i>Staphylococcus epidermises</i>	<i>Escherichia coli</i>	<i>Candida albicans</i>	<i>Klebsiella spp.</i>
19	19	21	21	18



Figure 8 Antibacterial activity against, A- *S. aureus*, B- *E. coli*, C- *S. epidermidis*, D- *Candida albicans* and E- *Klebsiella spp*

4. Conclusion

- The size of the prepared NiO:Ba NPs ranged between 20–40 nm with a spherical shape.
- UV-Vis spectroscopy show a sharper slope.
- The synthesized NiO:Ba nanostructures had potential for higher inhibition and killing of gram negative than gram positive bacteria where can be applied in the treatment of intestinal infections, inflammations by stomach and liver. (Khan, W. et al 2019).

Compliance with ethical standards

Disclosure of conflict of interest

No conflict of interest to be disclosed.

References

- [1] Djaja, N.F., Saleh, R., Aditya Warman, D. (2014). J Phys: Conf Ser, 495, 012029.
- [2] Ahmed, K.B. et al. (2018). Micro & Nano Letters, 13(11), 1426-1430.
- [3] Adhikary, J., Chakraborty, P., Das, B., Datta, A., Dash, S. K., Roy, S., ... & Chattopadhyay, T. (2015). Preparation and characterization of ferromagnetic nickel oxide nanoparticles from three different precursors: application in drug delivery. RSC Advances, 5(45), 35917-35928.
- [4] Imran Din, M., & Rani, A. (2016). Recent advances in the synthesis and stabilization of nickel and nickel oxide nanoparticles: a green adeptness. International journal of analytical chemistry, 2016.
- [5] Khawlah Salah Khashan , Ghassan Mohammad Sulaiman , Farah Abdul Kareem Abdul Ameer , Giuliana Napolitano, Synthesis, characterization and antibacterial activity of colloidal NiO nanoparticles, Pak J Pharm Sci. 2016 Mar;29(2):541-6.
- [6] Ezhilarasi, A. A., Vijaya, J. J., Kaviyarasu, K., Kennedy, L. J., Ramalingam, R. J., & Al-Lohedan, H. A. (2018). Green synthesis of NiO nanoparticles using Aegle marmelos leaf extract for the evaluation of in-vitro cytotoxicity, antibacterial and photocatalytic properties. Journal of Photochemistry and Photobiology B: Biology, 180, 39-50.

- [7] Bonomo, M. (2018). Synthesis and characterization of NiO nanostructures: a review. *Journal of Nanoparticle Research*, 20(8), 222.
- [8] Dacruz, D. A. (2020). Synthesis of NiO Nanoparticles by Chemical Precipitation Method. Available at SSRN 3684208..
- [9] Arfan, M., Shahid, T., & Taj, M. K. (2018). A facile composite-hydroxide-mediated route for preparation of composite Fe³⁺-NiO nanostructures. *Nanotechnology Letters*, 2.
- [10] Shahid, T., Arfan, M., Zeb, A., BiBi, T., & Khan, T. M. (2018). Preparation and physical properties of functional barium carbonate nanostructures by a facile composite-hydroxide-mediated route. *Nanomaterials and Nanotechnology*, 8, 1847980418761775.
- [11] Wang L, Hu C, Shao L. The antimicrobial activity of nanoparticles: present situation and prospects for the future. *Int J Nanomedicine*. 2017;12:1227-1249. doi:10.2147/ijn.s121956.
- [12] Beyth N, Hourı-Haddad Y, Domb A, Khan W, Hazan R. Alternative antimicrobial approach: nano-antimicrobial materials. *Evid Based Complement Alternat Med*. 2015;2015:246012. doi:10.1155/2015/246012.
- [13] Panyala NR, Peña-Méndez EM, Havel J. Gold and nanogold in medicine: overview, toxicology and perspectives. *J Appl Biomed*. 2009;7(2):75-91. doi:10.32725/jab.2009.008.
- [14] Shanaj BR, John XR. Effect of calcination time on structural, optical and antimicrobial properties of nickel oxide nanoparticles. *J Theor Comput Sci*. 2016;3(2):149. doi:10.4172/2376-130X.1000149.
- [15] Gottschall, R., Schöllhorn, R., Muhler, M., Jansen, N., Walcher, D., & Gütlich, P. (1998). Electronic state of nickel in barium nickel oxide, BaNiO₃. *Inorganic Chemistry*, 37(7), 1513-1518.
- [16] Lee, J. G., Hwang, J., Hwang, H. J., Jeon, O. S., Jang, J., Kwon, O., ... & Shul, Y. G. 2016. A new family of perovskite catalysts for oxygen-evolution reaction in alkaline media: BaNiO₃ and BaNiO. 8302. 5. *Journal of the American Chemical Society*, 138(10), 3541-3547.
- [17] Beyth N, Hourı-Haddad Y, Domb A, Khan W, Hazan R. Alternative antimicrobial approach: nano-antimicrobial materials. *Evid Based Complement Alternat Med*. 2015;2015:246012. doi:10.1155/2015/246012
- [18] Panyala NR, Peña-Méndez EM, Havel J. Gold and nanogold in medicine: overview, toxicology and perspectives. *J Appl Biomed*. 2009;7(2):75-91. doi:10.32725/jab.2009.008.
- [19] Shanaj BR, John XR. Effect of calcination time on structural, optical and antimicrobial properties of nickel oxide nanoparticles. *J Theor Comput Sci*. 2016;3(2):149. doi:10.4172/2376-130X.1000149
- [20] Jaisai, M. et al. (2013). *Ceramics International*, 39(1), 403–406.
- [21] Pratima R. Solankia*, Manoj Kumar Patelb ,Md. Azahar Alic, B. D. Malhotra, Chitosan Modified Nickel Oxide Platform for Biosensing Applications, *Journal of Materials Chemistry B*,2015.
- [22] B. Chinnappa Reddy a, H.C. Manjunatha b, Y.S. Vidya c, K.N. Sridhar d, L. Seenappa b, S. Manjunatha e, R. Munirathnam a f, P.S. Damodara Gupta b, Madhuri P. Rao , Study on anticancer properties of BaO:Fe₂O₃:NiO,nanocomposites, *Applied Surface Science Advances* ,2022.
- [23] Q.A. Drmsh, M.A. Gondal, Z.H. Yamani, T.A. Saleh, Spectroscopic characterization approach to study surfactants effect on zno₂ nanoparticles synthesis by laser ablation process, *Appl. Surf. Sci.* 256 (14) ,2010, 4661–4666.
- [24] Jiban Podder, Investigations on structural, optical, morphological and electrical properties of nickel oxide nanoparticles, *Int. J. Nanoparticles*, Vol. 8, Nos. 3/4, 2015.
- [25] W. Qin, C. Yang, R. Yi, G. Gao, Hydrothermal synthesis and characterization of single- crystalline [alpha]-fe₂O₃ nanocubes, *J. Nanometer*. 2011.
- [26] I. O. Faniyi¹ • O. Fasakin • B. Olofinjana • A. S. Adekunle • T. V. Oluwasusi • M. A. Eleruja • E. O. B. Ajayi, The comparative analyses of reduced graphene oxide (RGO) prepared via green, mild and chemical approaches, *SN Applied Sciences* 2019, 1:1181.
- [27] Chithra MJ, Beena B, Jawahar KM, Urmila KR, Pradeep B. (2012). *Bulletin of Materials Science*, 35(7), 1121–1126.
- [28] Shkir M, Abbas HM. (2021). *Results in Physics*, 22, 103948.

- [29] Sarmad Ghazi Al-Shawi, Natalia Andreevna Alekhina , Surendar Aravindhhan , Lakshmi Thangavelu , Akulina Elena , Natalia Viktorovna Kartamyshev , and Rafina Rafkatovna Zakieva. Synthesis of NiO Nanoparticles and Sulfur, and Nitrogen co Doped-Graphene Quantum Dots/ NiO Nanocomposites for Antibacterial Application, J Nanostruct 11(1): 181-188, Winter 2021.
- [30] Khan, W. et al. (2019). Journal of Materials Research and Technology, 9(3), 7432-7437