Synthesis and Characterisation of Nio Nanoparticles by Hydrothermal Method and Electrochemical Sensing Property of Uric Acid

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**Abstract:** Nickel oxide nanoparticles' increased surface area, improved catalytic activity, and superior electrical conductivity have made them promising for use in electrochemical sensing applications. The hydrothermal process provides a regulated and dependable way to create NiO nanoparticles. By adjusting the size, shape, and crystalline structure of the nanoparticles, the hydrothermal synthesis approach makes it possible to modify their characteristics and sensing capabilities. One of our body's essential metabolites, uric acid is a byproduct of purine biosynthesis. NiO nanoparticle production and characterization using the hydrothermal technique and uric acid's electrochemical sensing property. The work's objectives were to produce NiO nanoparticles by a hydrothermal technique, describe them, and ascertain whether or not they could detect uric acid.To synthesize hierarchical NiO nano/microspheres, 6 mmol of nickel nitrate hexahydrate [Ni(NO3)2•6H2O], 18 mmol of urea, and 8 mmol of CTAB were dissolved in 60 mL of deionized water. The resulting mixture was subjected to vigorous stirring for 30 minutes to ensure homogeneity. Afterward, the solution was transferred to a 100 mL Teflon-lined stainless steel autoclave, which was sealed and heated to 180°C for 4 hours.system was allowed to cool to room temperature. The resulting product was separated by centrifugation, then rinsed several times with deionized water and ethanol to remove impurities. Finally, the product was dried at 60°C for 12 hours. The dried material was annealed in air at 600°C for 3 hours, resulting in the formation of hierarchical NiO nano/microspheres.We report the effective hydrothermal production of NiO nanoparticles in this work. The final nanoparticles' composition, shape, and structure were described. Our further investigation into the NiO nanoparticles' electrochemical sensing capabilities for uric acid detection revealed encouraging sensitivity and selectivity, indicating their potential for use in uric acid sensing applications. In biosensing technology, this study demonstrates the potential of NiO nanoparticles for the detection of uric acid, a crucial biomarker linked to a number of medical disorders.We have synthesized the NiO by hydrothermal method and formation of the material was confirmed by various instrumental techniques like XRD analysis for identifying crystalline nature of the sample, FESEM for the morphology analysis it shows nanoparticles formation. Formed nano particle were utilized for the electrochemical sensing of the application

**Key words:** NiO nanoparticles, hydrothermal synthesis, electrochemical sensing, uric acid.

# INTRODUCTION

Nanoparticles are the basic building blocks of nanotechnology. Nanoparticle delivery systems are currently the focus of intensive preclinical research, and a variety of particle-based formulations and technologies have already been employed in the clinic [(Ajay et al., 2023; Chokkattu et al., 2023; Padarthi et al., 2023)](https://paperpile.com/c/O8TQ0u/yV1NS+OdPR9+Usiiv). The Food and Drug delivery (FDA) has authorized oral, local, topical, and systemic delivery of nanoparticles, depending on the intended use or target site[(Dharman et al., 2023; S. Sindhu et al., 2023; Sreenivasagan et al., 2023)](https://paperpile.com/c/O8TQ0u/nfzqB+LmFJ4+emz2M). Administered intravenously nanoparticles are the delivery modality that has received the greatest interest in both preclinical and clinical studies [(Anselmo & Mitragotri, 2016)](https://paperpile.com/c/O8TQ0u/Iu1n). The level of nanoparticles is currently the most developed in terms of scientific understanding and practical applications[(Ramakrishnan et al., 2023; Shenoy & Maiti, 2023; J. S. Sindhu et al., 2023)](https://paperpile.com/c/O8TQ0u/Mo9CA+LqMWF+qoEnZ). Nanoparticles were introduced ten years ago due to their size-dependent physical and chemical features.They are now in the commercial exploring phase. Nano-particles usually form the core of nano-material[(Kasabwala et al., 2021; Rajeshkumar & Lakshmi, 2021; Varghese et al., 2023)](https://paperpile.com/c/O8TQ0u/1aATE+alPxr+52PQU). It can be used as a convenient surface for molecular assembly, and may be composed of inorganic or polymeric materials. The shape is more often spherical but cylindrical, and other shapes are possible. The core particle is often protected by several monolayers of inert material, for example silica [(Salata, 2004)](https://paperpile.com/c/O8TQ0u/qKyL). Silver is the most commonly used nanoparticle [(“An Application of Nanotechnology in Advanced Dental Materials,” 2003)](https://paperpile.com/c/O8TQ0u/kePg). The primary function of the nanoparticles is to maintain or improve the quality of the product by adding various functional groups. As a result, nanotechnology is widely used in a variety of industrial sectors, as well as in the fields of medicine and dentistry [(“Managing Dentin Hypersensitivity,” 2006)](https://paperpile.com/c/O8TQ0u/lNn4).The use of nanomaterials in the treatment of COVID-19 has been made possible by the development of the COVID-19 vaccine [(“Nanoparticles in Clinical Trials of COVID-19: An Update,” 2022)](https://paperpile.com/c/O8TQ0u/QB6u). Nanotechnology offers a tremendous potential to improve drug delivery in the field of veterinary medicine [(Scott, 2007)](https://paperpile.com/c/O8TQ0u/GEE1). Nano pharmaceuticals are a standout amongst the most encouraging and beneficial fields of nanotechnology which has numerous advantages in veterinary medication [(Garg, 2014)](https://paperpile.com/c/O8TQ0u/JSNc). There are various uses for nanoparticles in dentistry, including those that exhibit good biocompatibility, good physico-mechanical properties, strong antibacterial capabilities, etc[(Keerthana & Ramesh, 2021; Murugesan, 2021; Tiwari & Jain, 2021)](https://paperpile.com/c/O8TQ0u/OCAAe+Sdwau+6HqAO)[(Keerthana & Ramesh, 2021; Murugesan, 2021; Subramanian et al., 2021; Tiwari & Jain, 2021)](https://paperpile.com/c/O8TQ0u/OCAAe+Sdwau+6HqAO+OZFbm). Additionally, it emphasized the role that therapeutic and restorative nanoparticles have in dental implants. Dental professionals can utilize a variety of nanocoatings to strengthen teeth structurally[(Pranati et al., 2021; Sakthi et al., 2021)](https://paperpile.com/c/O8TQ0u/RtAsZ+F7SFA). There is currently a lot of interest in research towards developing more biocompatible materials to prevent dental implants from failing [(“Advances of Nanoparticles Employment in Dental Implant Applications,” 2022)](https://paperpile.com/c/O8TQ0u/7K2K).Food and biological samples often contain uric acid, a common antioxidant [(G. & Ganapathy, 2022; Kumar & Ramesh, 2021)](https://paperpile.com/c/O8TQ0u/4gAgn+fMBsU)). Even within the concentration range that is normally present in the human body and foods, differing levels may lead to distinct health problems and changes in the quality of food.Uric acid is a natural waste product produced by our body everyday it is the metabolic breakdown of purines. In blood and urine, normal levels of UA range from 0.14 to 0.4 mmol dm−3 and from 1.5 to 4.5 mmol dm−3 , respectively [(Moghadam et al., 2011)](https://paperpile.com/c/O8TQ0u/4aWf). High amounts of uric acid in the blood plasma causes hyperuricemia which leads to gout and it also increases the risk of cardiovascular diseases [(Sun et al., 2011)](https://paperpile.com/c/O8TQ0u/ArGI).Conversely, low UA levels have been linked to multiple sclerosis, Parkinson's disease, and Alzheimer's disease.NiO nanoparticles have great catalytic effectiveness and neglect the requirement for a second enzyme. NiO nanoparticles solve the poisoning problem that has been observed on the metal electrodes. NiO contains a built-in redox pair that can be used to create an effective urea biosensor without the use of an outside mediator [(“NiO Nanoparticle-Based Urea Biosensor,” 2013)](https://paperpile.com/c/O8TQ0u/BqWq). Due to its lower toxicity, cost-effectiveness, high chemical/thermal stability, availability, and eco-friendliness compared to other metal oxides, nickel oxide (NiO) based electrodes have attracted increasing interest for supercapacitor applications. Additionally, in 0.5 V potential windows, it exhibits large theoretical Cs [(“Epitaxial Growth of Conductive LaNiO3 Thin Films by Pulsed Laser Ablation,” 1996)](https://paperpile.com/c/O8TQ0u/kvv6). NiO is also one of the preferred materials in a variety of research fields, including fuel cell electrode, smart windows, catalysis, electrochromic thin films, and dye-sensitized photo electrodes, because of its special features. NiO can be prepared using a variety of techniques, including hydrothermal, pulsed laser, sputtering, electrochemical, precipitation, sol-gel, chemical vapor deposition (CVD), and chemical bath deposition. The hydrothermal process, among others, has its own benefits [(“A Facile Synthesis Method of Hierarchically Porous NiO Nanosheets,” 2012)](https://paperpile.com/c/O8TQ0u/r6VP). Metal oxide nanoparticles can be produced using the hydrothermal process with a variety of morphologies and architectures that can improve electrochemical characteristics. The current study describes the simple hydrothermal process used to create NiO powder and evaluates its electrochemical characteristics in several electrolytes, including uric acid.

# Materials and methods

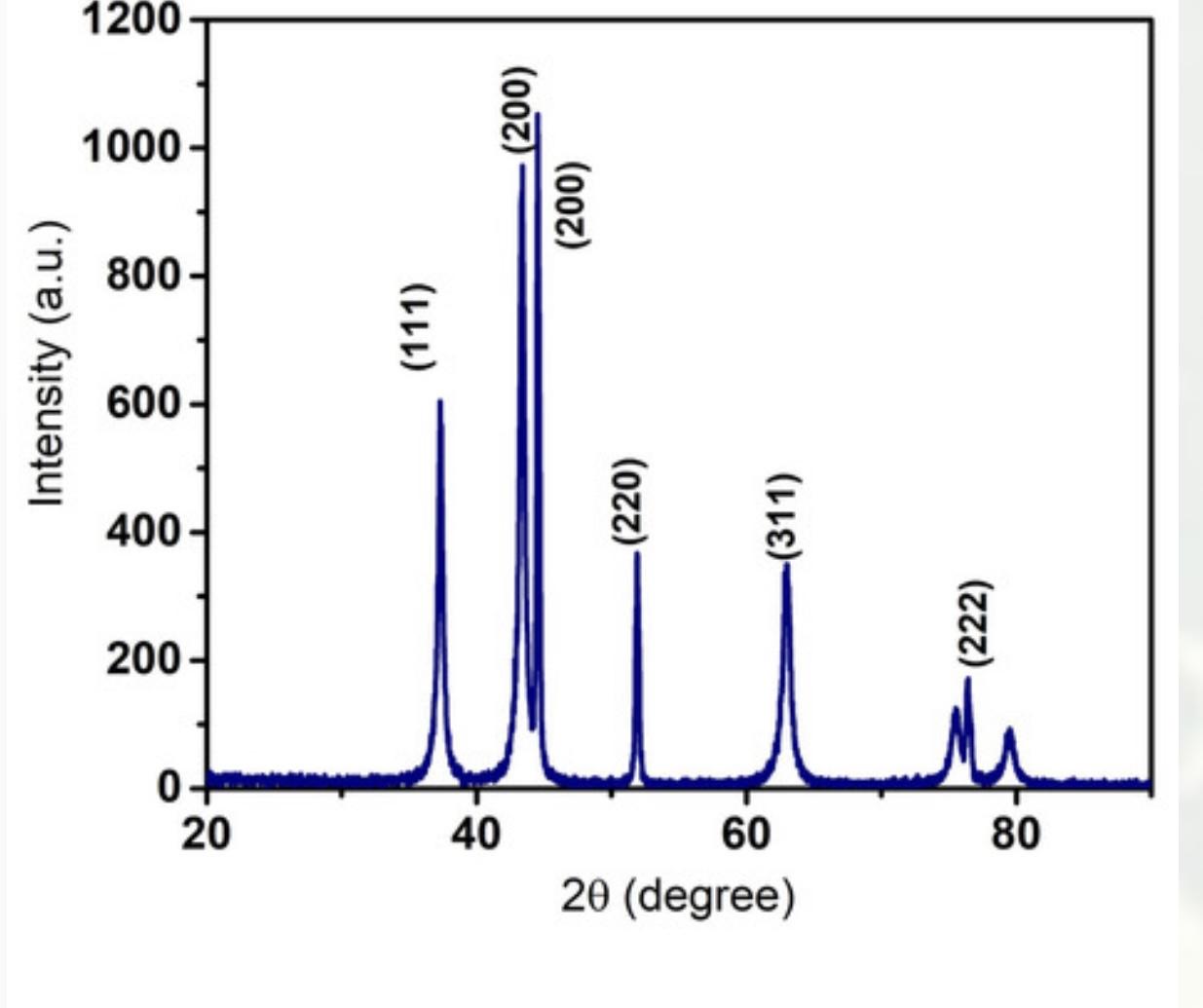
The compounds listed below The following ingredients were mixed together: deionized water, urea, CTAB (a surfactant), and nickel nitrate hexahydrate. The solution was transported and sealed into a Teflon-lined stainless steel autoclave where it was sterilized to kill the dangerous bacteria after rapidly swirling for 30 minutes. The combined solution underwent a four-hour autoclave at 180 °C. Additionally, the mixture was maintained at room temperature before the finished product was centrifuged to separate it from the waste. After centrifuging, the product was rinsed multiple times with ethanol and deionized water before being dried at 60°c for 12 hours. After annealing in the air for three hours at 600°c, hierarchical NiO microspheres were produced.

# RESULT AND DISCUSSION

It is clear that there are many different synthesis processes for a wide range of nanoparticles after several decades of rigorous research. The discovery of synthesis methods was the primary emphasis of nanoparticle research about 20–30 years ago. A fast expanding area of research nowadays is the use of nanoparticles as building blocks, similar to LEGO bricks, and their assembly into bigger structures. Synthesis work is still vital in this regard [(“The Fascinating World of Nanoparticle Research,” 2013)](https://paperpile.com/c/O8TQ0u/gB3T). The so-called quantum size effect is the main driving force behind nanoparticle research. Smaller than a single atom or molecule but larger than bulk materials, metal and semiconducting nanoparticles exhibit strong size- (and also shape-) dependent electrical and optical characteristics [(Henglein, 2002)](https://paperpile.com/c/O8TQ0u/rz8R). If the size and shape of the particles can be rationally adjusted, the observation of such size effects has increased expectations for the improved performance of nanomaterials compared to their bulk counterparts in many applications [(Henglein, 1982)](https://paperpile.com/c/O8TQ0u/L53a).Microorganisms, plants, and other biological structures are involved in the removal of harmful and waste metals from the environment. This is done by oxidizing, reducing, or catalyzing the metals using metallic nanoparticles. The first strategy is known as the "top-down" method and it entails using external force to crush solid materials into little fragments. This method uses a variety of physical, chemical, and thermal approaches to supply the energy required for the production of nanoparticles. The second strategy, referred to as "bottom-up," relies on collecting and fusing gas or liquid atoms or molecules. Both of these strategies have benefits and drawbacks in comparison to one another. The development of nanomaterials is intimately related to the emergence and growth of the solvothermal/hydrothermal process [(“Synthesis and Characterization of FeSe2 Nanoparticles and FeSe2/FeO(OH) Nanocomposites by Hydrothermal Method,” 2015)](https://paperpile.com/c/O8TQ0u/4YDi) Hydrothermal synthesis is a versatile method for producing nanoparticles by leveraging high-pressure, high-temperature aqueous environments(Nikalje et al., 2024) (Chehelgerdi et al., 2023). In this process, a precursor solution containing the desired elements or compounds is sealed in a reactor and heated, simulating natural hydrothermal conditions. The controlled parameters such as temperature, pressure, and reaction time influence the size, shape, and composition of the resulting nanoparticles. The hydrothermal environment allows for precise control over nucleation and growth, yielding nanoparticles with tailored properties for various applications, including in catalysis, medicine, and electronics.

Uric acid, a metabolic product found in the human body, can be detected and measured through electrochemical sensing techniques. Electrochemical sensors designed for uric acid detection typically utilize electrodes, often modified with specific materials or enzymes, to facilitate selective and sensitive detection. The electrochemical reactions involving uric acid are translated into measurable electrical signals, directly proportional to its concentration. This approach provides rapid, real-time, and cost-effective detection of uric acid, making it valuable for diagnosing conditions like gout and hyperuricemia. Additionally, electrochemical sensing offers portability and potential for point-of-care applications, enhancing the monitoring and management of uric acid-related health issues.

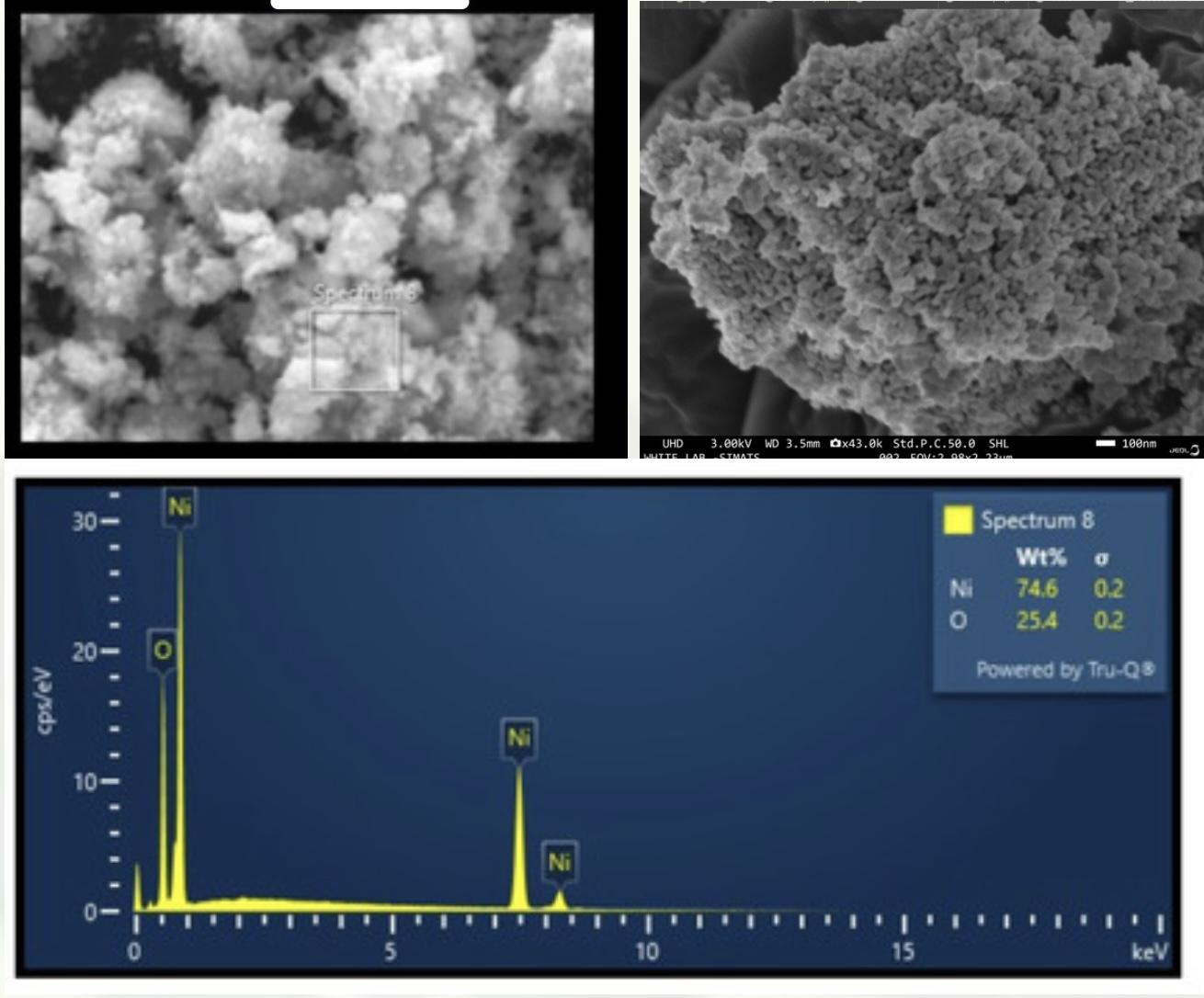
# XRD ANALYSIS



**Figure 1:** An XRD pattern of the electrochemically grown NiO nanoflower after the hydrothermal treatment

Result interpretation of the synthesized nanoparticles were confirmed by XRD analysis; the results show the absence of any other peak; only the peak corresponding to Nio were demonstrated. NiO peaks were compared with standard JCPDS no 040835.

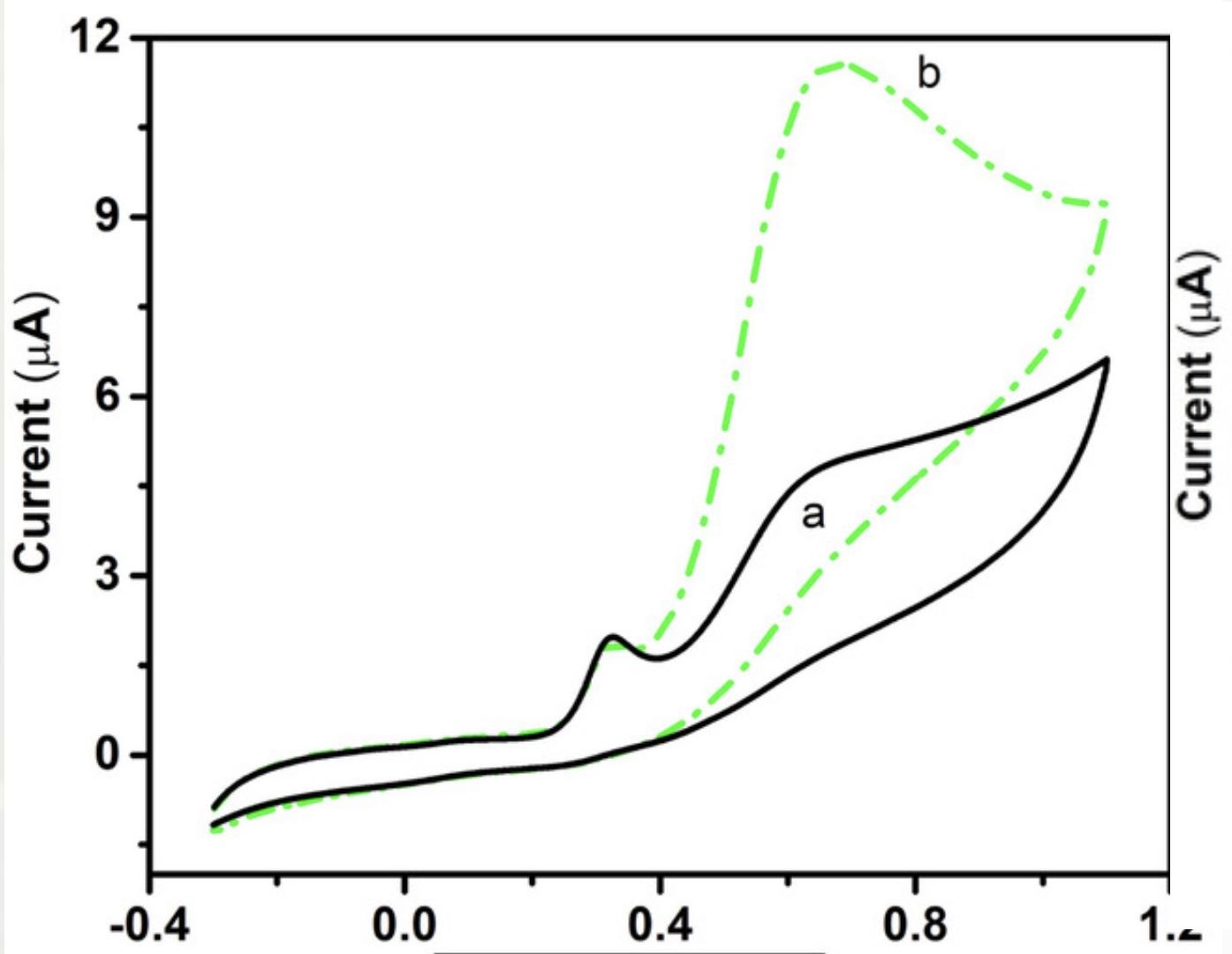
# MOLECULAR ANALYSIS



**Figure 2:** (a) The FE-SEM images of the prepared by NiO hydrothermal treatment. (b) The EDX analysis result of the NiO particles synthesized

EDX: energy-dispersive X-ray; FE-SEM: field emission-scanning electron microscopy; FeS: ferrous sulfideMorphological analysis showed NiO had a particle-like nature of morphology. The size of the particles measured in the range of nanoparticles was in the range of 100 nm.

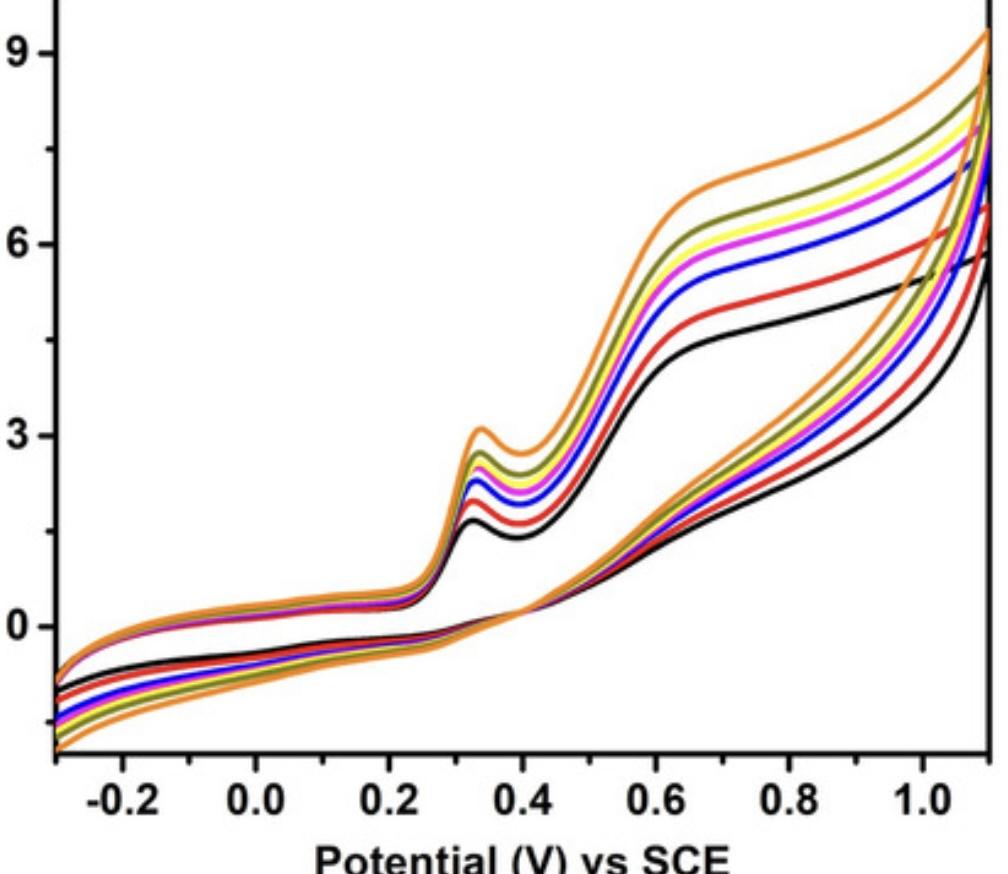
# PURITY TEST



**Figure 3:** Purity test of NiO-modified toward Uric acid.

From the spectrum shows the purity of nickel and oxygen. In the application NiO was coated with a glassy carbon electrode in an electrochemical system we have tested for an uncoated electrode and a coated electrode towards uric acid sensing properties. In fig. 3 black line demonstrates the uncoated electrode which shows the less current response compared to glassy carbon electrodes. The higher enhancement of the current ability of NiO shows the high ability to sense the uric acid cyclic test was done to demonstrate the stability of the nanoparticle.

# STABILITY TEST



**Figure 4:** Stability test of NiO-modified toward uric acid.

In fig 4. Eight cycles were run and all of them showed the same current response. So, it indicated that the sample coated on the surface is highly stable.

# CONCLUSION

Nanoparticles exhibit exceptional electrochemical sensing properties for detecting uric acid due to their unique characteristics at the nanoscale. Their high surface area-to-volume ratio allows for more active sites, enhancing the adsorption of uric acid molecules and improving sensitivity. Additionally, nanoparticles can be tailored and functionalized to optimize the selectivity and efficiency of the electrochemical reaction with uric acid, resulting in highly accurate and specific detection. These properties make nanoparticles a promising choice in the development of advanced electrochemical sensors for uric acid, offering precise and reliable diagnostic tools for various medical applications.

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