

Detection of Nickel Ions in Industrial Water by Fabricating Carbon Paste Electrode modified with Nanoparticles

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ABSTRACT

Nickel must be carefully monitored due to its severe health and environmental impacts. Many innovative nickel ion detection (Ni^{+2}) methods have been developed. These methods vary from fluorescent and nanoscale sensors to biosensors, optical electronics, and larger devices. In this research, a new detector electrode sensor was developed to detect and estimate nickel in industrial and well-water. Nickel oxide II (NiO) nanoparticles were synthesized using an extract of Capparis Spinosa leaves, washed, and dried. Then, the material from fresh green leaves has been extracted. The synthesized NiONPs were incorporated into a carbon paste matrix, forming a modified electrode for the selective detection of Ni^{+2} ions in aqueous samples. NiONPs and their external characteristics have been studied by various techniques. The particle size measured was approximately (15.57) nm. Scanning Electron Microscope SEM analysis revealed that the nanoparticles exhibited both spherical and irregular shapes. In the UV-VIS spectrometry, the peak was at the wavelength (366) nm; this confirms that nickel particles have been obtained. The electrode demonstrated selectivity and good sensitivity with linear response (10^{-1} - 10^{-9}), correlation coefficient (0.9987), electrode lifespan (130) days, and the ideal temperatures for the electrode were measured and determined (15-40) °C. The best degree of acidity (pH) to work (5-8), with a slope value at (25) °C, was (28.5) mV/decade. Limit of detection 8.335×10^{-9} mol/L. The recovery percentage is 100.8%. This work successfully estimated nickel ions in wells and industrial waters.

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1. INTRODUCTION

Chemical sensors are useful for monitoring heavy transitional metals due to their simple installation, excellent sensitivity, and high selectivity. In this study, we developed a new chemical sensor to detect nickel ion II in water[1], [2]. The electrochemical methods are one of the fastest, most accurate, and reliable technologies for detecting heavy metals such as Co^{+2} and Ni^{+2} ions, which have been widely developed. Measuring concentrations against voltage methodology successfully identified and detected cobalt (Co) and nickel (Ni) ions. Co and Ni levels are studied because they can cause water pollution and can be toxic and non-biodegradable, affecting the health of organisms[3], [4]. Heavy metals such as mercury, lead, and cadmium pose significant health risks. The permissible levels of heavy metals in drinking water, according to published studies, are: nickel – 0.07 mg/L, chromium – 0.05 mg/L, zinc – 0.05 mg/L, cadmium – 0.003 mg/L, lead – 0.01 mg/L, iron – 0.3 mg/L, and copper – 2 mg/L. These values are based on international drinking water standards and are essential for evaluating water quality and the potential health risks associated with exposure to heavy metals[5]. Heavy metals, naturally occurring contaminants, cause toxic effects on human health through bioaccumulation, interfere with antioxidant defense mechanisms, and disrupt cellular processes. This can lead to various diseases, including cancer, neurological disorders, and cardiovascular complications[6], [7].

Nickel exposure affects different body organs, causing effects of blood toxicity, immunotoxicity, neurotoxicity, genetic poisoning, and hepatotoxicity. It is also known to cause genital toxicity, affects both the male and female reproductive system, and can lead to toxic effects during pregnancy[8], [9], necessitating effective detection methods. Technologies such as Atomic Absorption Spectrometer (AAS) and Inductively Coupled Plasma Mass Spectrometer (ICP-MS) are highlighted for their sensitivity and multi-element capability, while sensitive ion electrodes provide detection and sensing of elements in the simplest ways and less potential and costs[10], while traditional laboratory methods provide high accuracy, developing portable and cost-effective sensors is critical for widespread surveillance, especially in limited resource places. These innovations can detect in real-time and on-site detection to close the gap between laboratory accuracy and field applicability[11], [12], [13]. Nano-based sensors benefit from nanotechnology by improving sensitivity and selectivity. It can detect minimal levels of heavy metals in environmental samples, although challenges persist regarding the toxicity of nanoparticles. Concentrations can be converted into signals that can be measured, read, and recorded[14]. Ion sensors, known as ion-selective electrodes (ISEs), are specialized sensors designed to detect specific ions in a highly sensitive and selective solution. These electrodes are essential in many applications, including environmental monitoring, clinical diagnosis, and control of industrial processes. The development of ISEs involves integrating materials and techniques that enhance their performance regarding selectivity, sensitivity, and stability. This research explores key aspects of selective and sensitive ion electrodes based on insights from modern research[15], [16]. ISEs provide significant advantages in ion detection. Recent studies aim to solve challenges of overlap, stability, and characterization. The development of new materials and designs, such as hybrid compounds and conductive polymers, continues to enhance the performance of ISEs, expanding their applicability in various fields. With technological advances, integrating ISEs into mobile and wearable devices is expected to revolutionize ion detection and control[17], [18].

In this research, we used (Capparis Spinosa), a shrub plant from the “Capparaceae” family, Figure 1. It features small shrubs, flower buds, and edible fruit[18]. It is known for its anti-inflammatory, antioxidant, anti-microbial effects and cure of cancer, diabetes, kidney, and liver. Promotes the plant's high content of bioactive compounds, like phenols and flavonoids, and its ability as a good reducing agent. It can be used to prepare nanomaterials, detect efficient substances and biologically active compounds at the cellular and molecular level, or integrate with nanomaterials to manufacture electrodes; that's why it was selected in this research[19], [20].



Figure1: Flower, leaves, and the Fruit of Capparis Spinosa Plant

2. APPARATUS AND MATERIALS:

2.1 Apparatus Used:

- pH meter (Jenway 3310, England).
- Calomel Electrode (Me-SC900, England).
- Magnetic stirrer and Hot plate (FTHPM-10, Korea).
- Balance (KERN ABS, Germany).
- PVC tubing or syringe tube.
- Whatman filter paper (No.1).
- Syringe filter 0.45 μm
- Agate mortar (TOB-Agate Mortar-80, China).
- Oven (KAROL, Korea).
- FTIR Instrument (Shimadzu, Japan).
- UV spectrophotometer (SHIMADZU, Japan).

2.2 Chemical Substances and Solutions:

- Anhydrous nickel nitrate solution ~0.1 M is prepared by accurately weighing 1.827 gm of anhydrous nickel nitrate $\text{Ni}(\text{NO}_3)_2$ using a balance. Then, transfer the compound into a clean 100 mL volumetric flask, add about 50 mL of distilled water, stir or shake until the solid dissolves completely, and gradually add more distilled or deionized water to the flask until the solution reaches the 100 mL mark.
- A sodium hydroxide solution of approximately 0.1 M was prepared by dissolving 0.400 gm of NaOH in a 100 ml volumetric flask and completing the volume to the mark with deionized water.
- A hydrochloric acid solution of approximately 0.1 M was prepared by transferring 0.82 ml of hydrochloric acid solution 12.06 M (Specific density 1.19 g/cm³) (solution 37%) to a 100 ml volumetric flask and completing the volume to the mark with deionized water.
- Graphite powder.
- Paraffin oil.

2.3 Preparation of Capparis Spinosa Leaves Extract Solution and NiONPs Formation:

- A 10 gm of dried and crushed Capparis Spinosa leaves are placed in a tea bag. Then, add 150 milliliters of deionized water to a large beaker. Heat to 70-80 °C for 50 minutes, leaving the extract to cool at room temperature. Filter the extract with Whatman No.1 filter paper and then with a syringe filter of 0.45 μm to remove the impurities.
- A 100 milliliters of the pre-prepared Capparis Spinosa extract was taken to a beaker with a capacity of 500 milliliter beaker, and add 100 milliliters of 0.1 M nickel nitrate solution with a concentration of 0.1 molar. It is best to use a burette to gradually add nickel nitrate in small quantities with continuous stirring and heating to 40-50 °C. The color change was observed from light yellow to brown or dark grey. This observation confirmed the reduction of Ni^{2+} ions. Typically, a precipitate does not appear clearly, so the reaction medium should be adjusted from acidic to neutral or basic (7-8) pH by adding a few drops of NaOH 0.1 M. It will be observed precipitation of colloidal particles of nickel hydroxide $\text{Ni}(\text{OH})_2$. Leave the mixture to cool at room temperature. Filter the mixture with Whatman No.1 filter paper, then dry the formed precipitate. Burn the precipitate at 300 °C in an inert atmosphere for 4 hours to turn nickel hydroxide into nano-nickel oxide [21]. See figure 2.

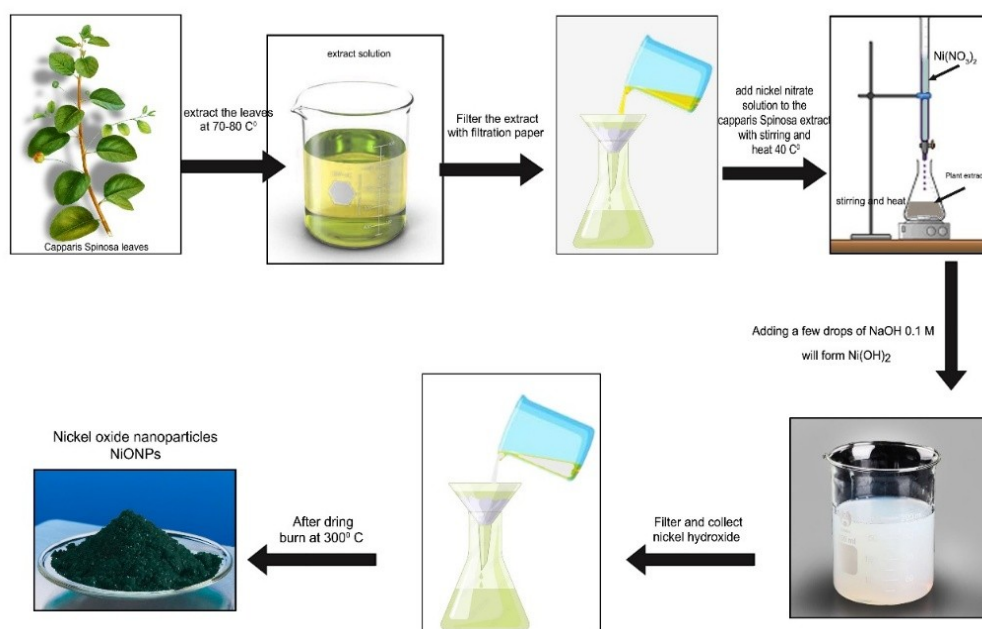


Figure 2: Steps to Prepare Plant Extract and Nickel Oxide Nanoparticles NiONPs

2.4 Prepare Carbon-NiONPs Paste:

In an agate mortar, grind the carbon powder well, then add a small amount of prepared NiONPs to the powdered carbon with a ratio (40:60% nickel oxide: carbon) and mix it for 30 minutes to become homogeneous well, then add paraffin oil slowly, until it reaches a homogeneous sticky texture. Leave the paste for 24 hours for excess paraffin oil to volatilize. Figure 3

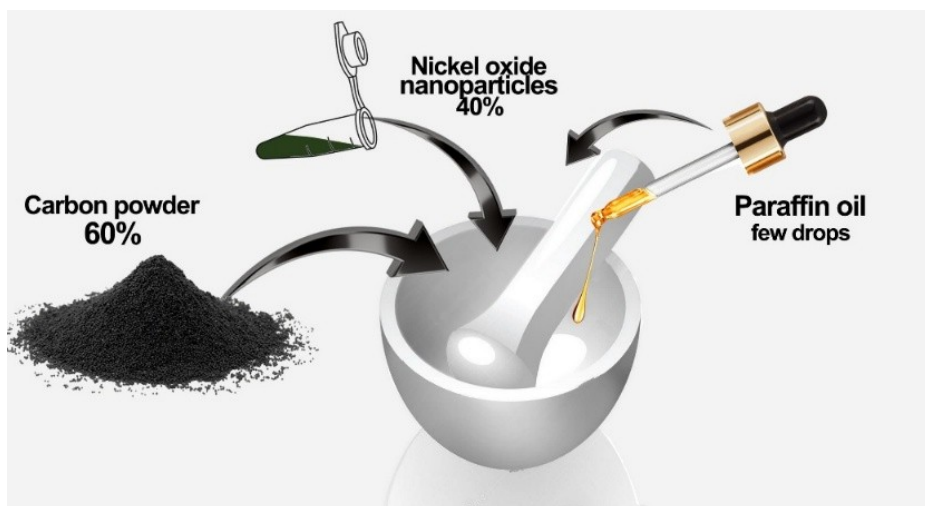


Figure 3: Steps to Prepare Electrode Paste

2.5 Preparation of Modified Carbon-Nickel Oxide Electrode:

A 5cc injection tube was used to create the electrode (a glass or Polyvinyl Chloride PVC plastic tube can be used), and copper wire was inserted at the first end. This end is filled with a few amounts of pre-prepared electrode paste. Direct contact between the copper wire and the electrode paste is essential to ensure efficient electrical conductivity; this process is repeated several times until it reaches an appropriate quantity. The second end is pressed to hold the paste well, and then the electrode is left to dry and dispose of excess paraffin oil, then adjust the electrode surface with filtration paper. It is better to immerse the electrode in 0.1 Molar of nickel nitrate solution for 4 hours to activate the electrode before using. Figure 4.

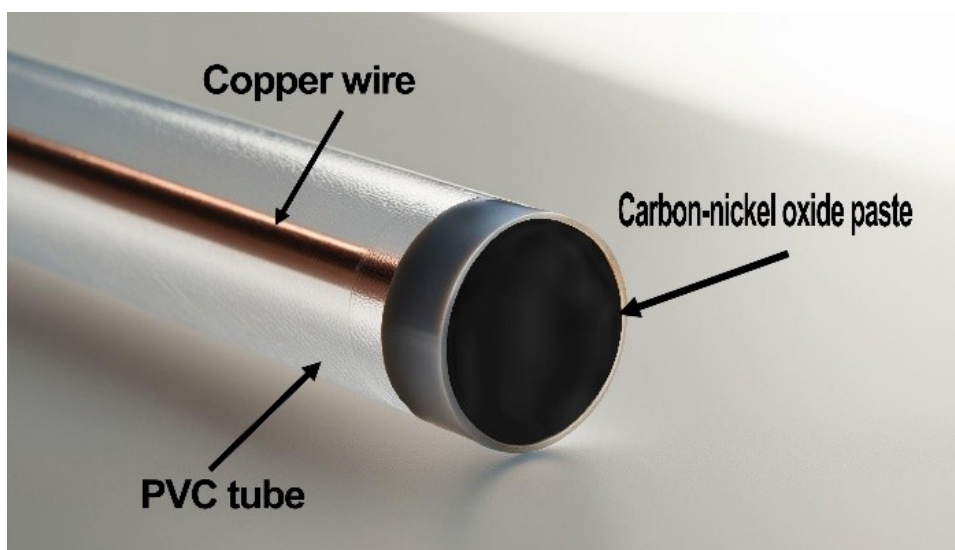


Figure 4: Steps to Prepare Carbon Nickel Oxide Electrode

3. CHARACTERIZATION OF NiONPs:

Different methods were employed to characterize the NiONPs. The crystalline nature, phase purity, and crystallite size of the synthesized NiONPs were determined using an X-ray diffractometer instrument (XRD, Shimadzu 6000, Japan) utilizing the radiation of $\text{CuK}\alpha$ ($1\frac{1}{4}$ 1.5406 Å). The functional groups of NiONPs were identified by Fourier transform infrared (FTIR) spectrophotometer (SHIMADZU Japan). A scanning electron microscope (SEM), type (MIRA3 TESCAN), was utilized to investigate the morphology of the produced nanomaterial surface. The optical absorbance for NPs was carried out via a spectrophotometer (Shimadzu, UV-1800) at room temperature.

3.1 X-ray Diffraction (XRD) Spectrum of NiONPs Prepared by Plant Extracts:

As shown in Figure 5. XRD patterns indicated diffraction peaks for the prepared compound. The size of nickel oxide nanoparticles NiONPs were calculated using the Shearer equation and the results are shown in the table below.

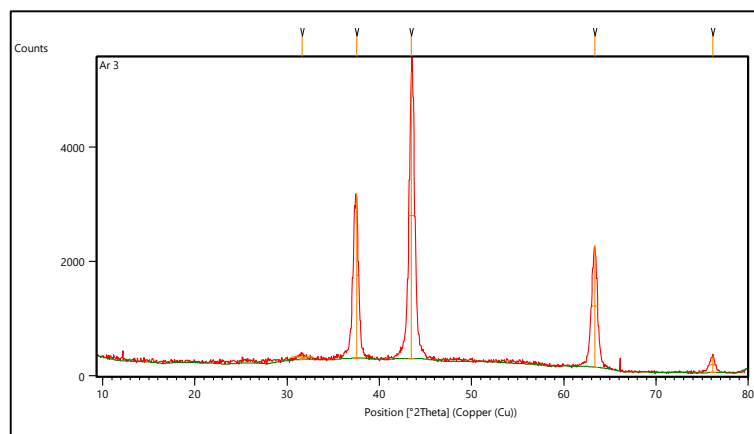


Figure 5: XRD Patterns of Prepared Nickel Oxide Nanoparticles (NiONPs)

Table 1. The XRD results of nickel oxide nanoparticles (NiONPs).

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]	Tip Width	Average D nm
31.6526	91.99	2.3616	2.82682	1.84	2.8339	14.4060
37.5401	2896.90	0.2460	2.39592	57.79	0.2952	14.6385
43.4745	5012.56	0.5412	2.08164	100.00	0.6494	14.9210
63.3863	2140.78	0.6396	1.46742	42.71	0.7675	16.2891
76.1462	281.50	0.5904	1.25017	5.62	0.7085	17.6061
Average particle size (nm)						17.01945

3.2 Scanning electronic Microscopy (SEM) of NiONPs Prepared by Capparis Spinosa Leaves Extracts

SEM is the most preferred method of analysis of microstructures. In this method, electrons accelerate in high voltage (0-30 kV) on the sample. An X-ray captured via detectors is formed during the fall of electrons on the surface of the material's atoms interacting with them. These incoming references to the detector are digital signals and turn into a computer screen[22]. The picture taken gives us the screen information on the exact structure of the sample. Depending on the devices that produce lens electron packages and different vacuum systems, it can scan the surface with a zoom of 1 μm to 1 nm[23]. The produced samples were analyzed using a scanning electron microscope SEM. Figure 6 shows electronic scanning images of nickel oxide particles by several measures; it showed that the particles had spherical and irregular shapes.

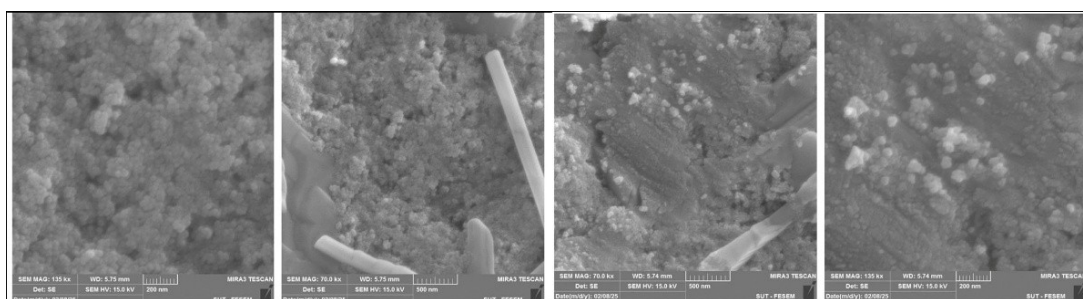


Figure 6: SEM Image of Nickel Oxide Nanoparticles

3.3 Energy Dispersive X-ray Spectroscopy (EDX)

EDX, (usually referred to as EDS or EDXS) is a reliable and widely employed technique for chemical characterization and imaging[24]. Using EDX technology, various strong signals were detected, verifying the formation of nickel oxide particles (NiONPs). The peaks are seen at optical absorption values of 0.9 and 7.5 keV for nickel particles. A strong signature optical absorption peak was observed at 0.6 keV for oxygen particles. This confirms the presence of nickel nanoparticles.

Table 2 indicates a nickel amount of 71.6% and an oxygen content of 22.3%. In addition, lower levels of calcium, sulfur, potassium, and sodium, produced from the Capparis Spinosa plant extract rich in organic acids and bioactive molecules such as phenols and flavonoids, have effectively contributed to the reduction of nickel and the synthesis of nickel nanoparticles. Figure 7.

Table 2. Percentage of Elements in the sample

Element	Line Type	Wt.%	Atomic %
O	K series	22.25	49.63
Ni	K series	71.60	43.52
Ca	K series	1.72	1.53
S	K series	1.21	1.35
K	K series	1.03	0.94
Na	K series	0.87	1.36
P	K series	0.80	0.92
Mg	K series	0.52	0.75
Total:		100.00	100.00

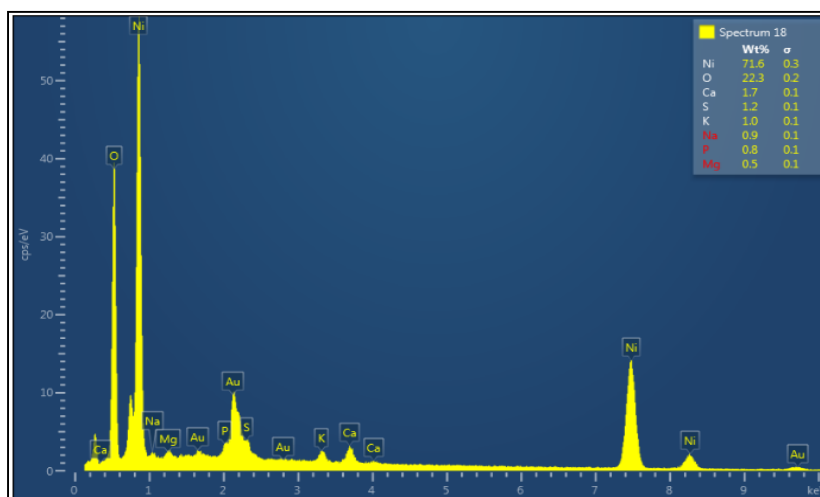


Figure 7: EDS View Showing the Elements Signal and the Percentage

3.4 UV-Visible Spectroscopy for NiONPs:

The UV-visible spectra of nickel nanoparticles have been measured as a function of wavelength using a UV-visible spectrophotometer (SHIMADZU, Japan) with a range of (200-800) nm. UV-visible spectroscopy was applied for characterization. The UV spectrum reached its maximum at a wavelength of 366 nm, as displayed in Figure 8. This refers to the stable NiONPs structure that absorbs light within the range of (300- 400) nm.[25]

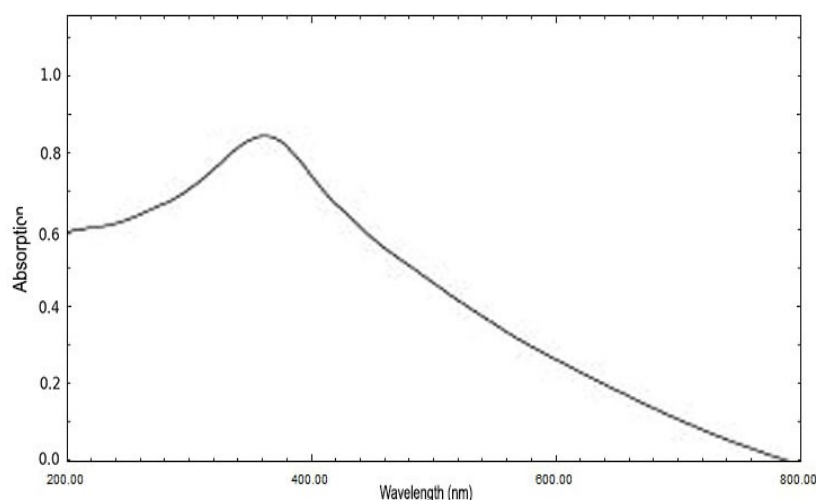


Figure 8. UV-Visible spectroscopy for NiO nanoparticles

4. RESULT AND DISCUSSION:

To study the quality of the produced electrode, the detection limits (LOD), and its comparison with previous works, as well as determine strength, durability, and electrode conditions, and review the characteristics and properties of this nano-sensor. The tests were applied by changing the conditions and concentrations of the solutions post-manufacture to determine electrode response, linear concentration range, slope value, correlation coefficient, lifespan, detection limit, accuracy, compatibility, selectivity, temperature, and pH effect. And its application at work.

4.1 Effect of Temperature:

The first evaluation of this electrode aimed to determine the ideal temperature to work within this range. The effect of temperature changes was measured within the (10 – 70) °C range. The ideal working range for this electrode has been found at (15-40) °C. Low temperatures (below 15 °C) have been observed to affect the recorded voltage measurement. The high temperatures of the solution also influence the electrode paste; the voltage reading gradually drops with a temperature increase above 40 °C. This reduces the electrode's sensitivity in detecting ions. Figure 9

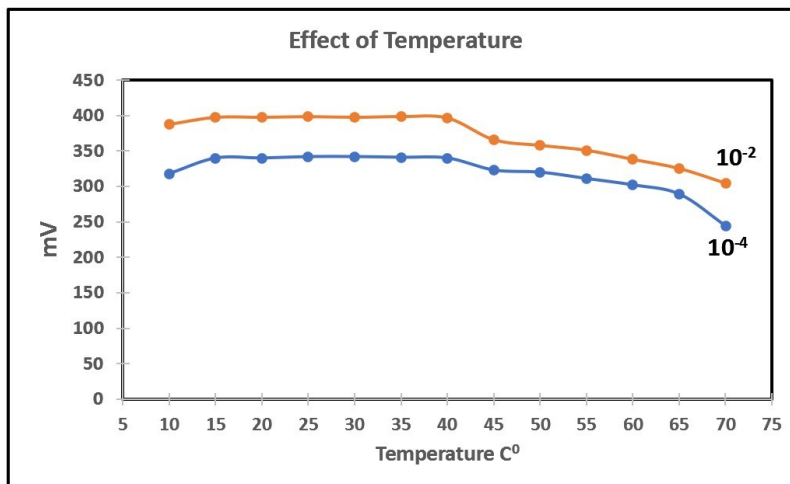


Figure 9. Effect of Temperature on Modified Electrode

4.2 Effect of pH:

After temperature measurement, it was necessary to measure the performance of the electrode when the pH changed. The best pH range where the electrode operated unaffected or changed responding was (5-8). Because the voltage measured for concentrations 10^{-2} and 10^{-4} molar was almost stable and akin to previously measured readings, we changed pH using hydrochloric acid and sodium hydroxide solutions. We found that the acidic medium had less reading voltage than the clearly calculated reading. An excess pH of 8 leads to lower values and voltage instability due to forming a turbid or cloudy solution of nickel hydroxide. Figure 10

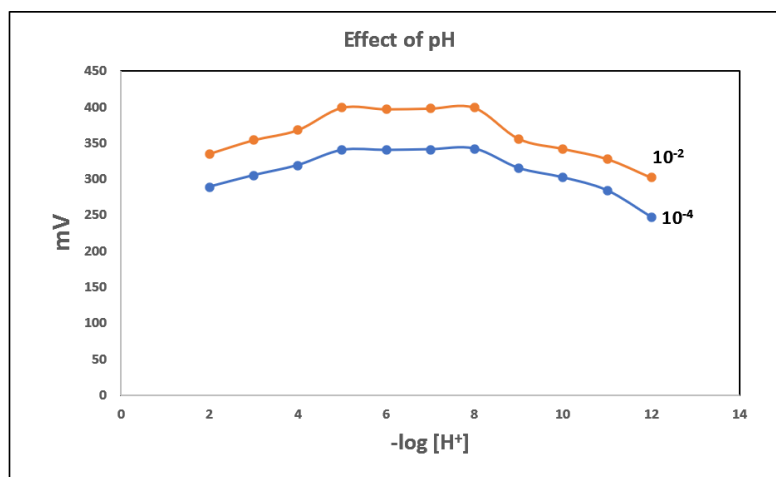


Figure 10. Effect of pH on Modified Electrode

4.3 Response Time

To measure the response time and speed of the manufactured electrode, it was immersed with the reference electrode (calomel) in a group of standard pre-prepared solutions of nickel nitrate with concentrations that gradually decreased from 10^{-1} to 10^{-9} molar. The time taken by the electrode to stabilize the voltage reading was measured for each concentration. The electrode showed a marked variation in time of (2-27) seconds. The readings indicated that the response time was inversely commensurate with the concentration used. The lower the concentration, the longer the electrode stabilizes the response. That is, it needs longer to reach equilibrium. Figure 11

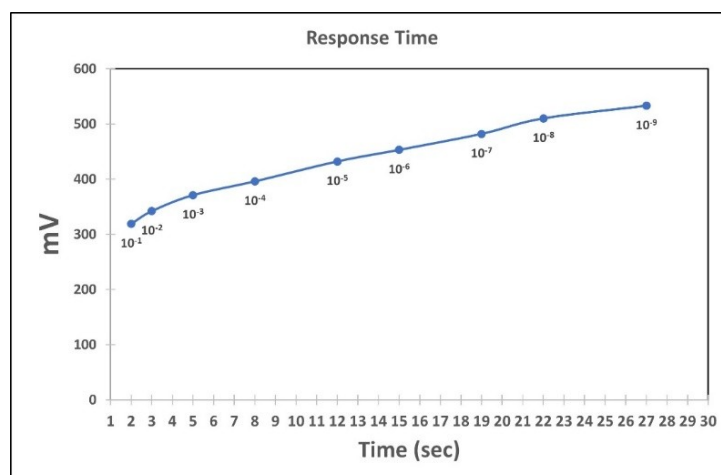


Figure 11. Response Time for Modified Electrode

4.4 The lifespan of the Electrode

The electrode's lifespan was found by measuring the potential difference over 130 days using a standard $\text{Ni}(\text{OH})_2$ solution at a concentration of 1×10^{-2} molar. The results showed that the measured voltage remained stable and consistent. Thus, the electrode's lifespan was approximated to be 130 days. It was observed that the recorded voltage began to decrease after this period, which may have been due to damage to the electrode paste resulting from contamination of the paste or oxidation of the paste components. Figure 12

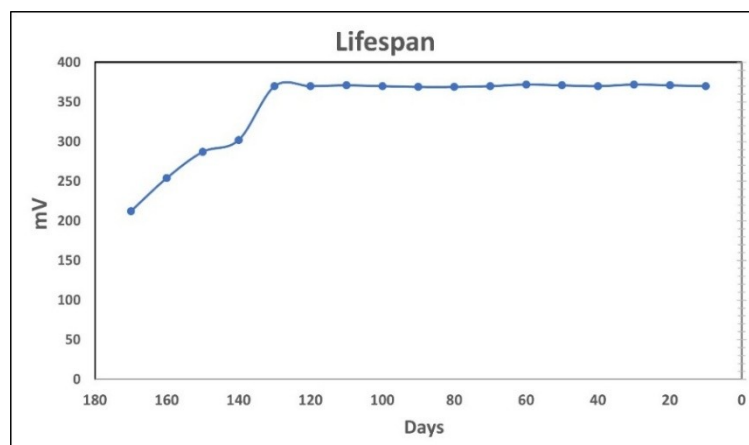


Figure 12. The lifespan of the Electrode

Table 3. The ideal conditions for NiONPs electrode

Parameter	modified carbon paste electrode
pH Effect	5-8
Temperature (C°)	15-40
Response Time (sec)	2-27
Lifespan (day)	130

4.5 Calibration Curve

After determining the ideal electrode conditions at the best temperature of 25 °C and pH 7. The electrode is immersed beside the reference electrode (calomel) in nickel nitrate solution in a 50-milliliter beaker of different concentrations of solutions, which gradually decreases. The voltage for each concentration was recorded and measured several times to draw a calibration curve chart using the straight-line equation, Figure 13, where this curve has demonstrated that the linear range of response is between 10^{-1} and 10^{-9} molar. The correlation coefficient was 0.9987, and the slope value was 28.5 mV/decade, approximating matched the slope value 29 mV/decade as shown in [26].

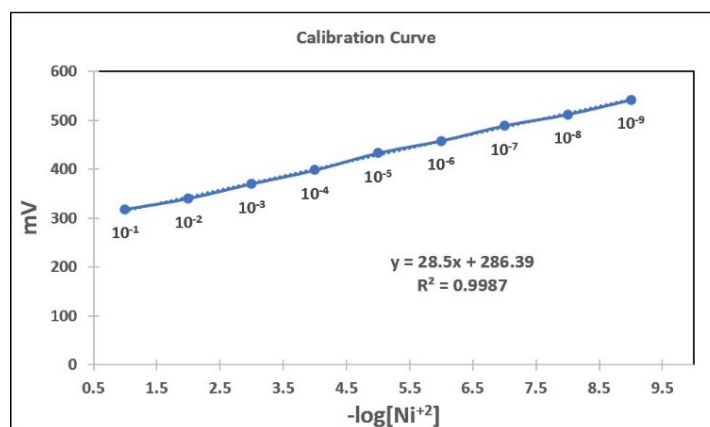


Figure 13. Calibration Curve for Modified Electrode

Table 4. The potentiometric properties for modified carbon paste electrode

Properties	modified carbon paste electrode values
Linear range	$10^{-1} - 10^{-9}$ molar
Slope	28.5 mv/decade
Intercept	286.39 mv
Limit of detection	3.5×10^{-10} molar
correlation coefficient	0.9987

4.6 Accuracy and Precision:

After confirming the standard and ideal conditions for the modified electrode and drawing the calibration curve. The accuracy and precision of the designed electrode using different concentrations of prepared standard solutions required studying. The test was carried out several times for each concentration. Accuracy refers to the degree of proximity or similarity between the values measured by the electrode and the actual or standard values. The electrode shows an accurate reading or is very close to the real concentration of the target ion. The tests proved that the manufactured electrode successfully estimates nickel ions with a low standard deviation and an excellent recovery ratio, as shown in Table 5.

Table 5. Accuracy and Precision Value

Conc. Taken (mol/L)	Conc. Found (mol/L)	Rec%	RSD%
1×10^{-2}	1.004×10^{-2}	100.4	0.5211
1×10^{-4}	1.008×10^{-4}	100.8	0.8323
1×10^{-6}	0.9987×10^{-6}	99.87	0.6497
1×10^{-8}	0.9963×10^{-8}	99.63	0.7985

4.7 Selectivity:

Interferences, such as ions or other substances that affect the response of sensitive electrodes, may give inaccurate signals or interfere with the desired ion. Testing the electrode response and selectivity of ions other than target ions is necessary. The selectivity of the electrode can be determined by finding the K selectivity determination coefficient using equation (1) below. Various concentrations of ions have been added to the nickel nitrate solution.

$$\log K_{A,B}^{\text{pot}} = \frac{E_2 - E_1}{S} \dots\dots\dots (1)$$

Where: $\log K_{A,B}^{\text{pot}}$ is selectivity coefficient, E_1 , E_2 the potential of ions. S is the slope of the calibration curve.

Table 6. Selectivity coefficient values

Type of interference ion	Interference Ion Concentration	$\log K_{A,B}^{\text{pot}}$ Selectivity Coefficient	$K_{A,B}^{\text{pot}}$
CO_3^{2-}	10^{-2} M	-0.24561	0.56805
CO_3^{2-}	10^{-4} M	-0.35088	0.44578
Cd^{+2}	10^{-2} M	-0.24561	0.56805
Cd^{+2}	10^{-4} M	-0.21053	0.61585
SO_4^{2-}	10^{-2} M	-0.28070	0.52396

SO ₄ ⁻²	10 ⁻⁴ M	-0.52632	0.29764
Zn ⁺²	10 ⁻² M	-0.38596	0.41118
Zn ⁺²	10 ⁻⁴ M	-0.31579	0.48329
Fe ⁺³	10 ⁻² M	-0.42105	0.37927
Fe ⁺³	10 ⁻⁴ M	-0.45614	0.34983

As the previous table shows, the constant-value selectivity coefficient with cadmium solutions has the highest number. This is because the cadmium ion interferes with the nickel electrode.

4.8 Limit of Detection (Lod)

LOD is the minimal concentration of an analyte that can be accurately differentiated from the background noise of a measuring instrument. However, it may not be quantified with precision. It denotes the minimal quantity of a chemical that an analytical method can identify with a defined confidence level[27]. This is usually determined using equation (2) below:

$$\text{LOD} = \frac{3.3 \cdot \sigma}{S} \dots\dots\dots (2)$$

Where: σ (sigma) is standard deviation of the background noise or blank sample response, S is the slope of the calibration curve.

$$\text{LOD} = \frac{3.3 \cdot 71.98}{28.5} \times 10^{-9} \cong 8.335 \times 10^{-9} \text{M}$$

4.9 Comparison of Method used in this Research with other Ways of Manufacturing Modified Carbon Electrodes.

It is necessary to compare the method used in this research with previous methods of preparing modified carbon electrodes by nanoparticles. This method has been compared with previous methods in published research and previous studies, and it has turned out that it is better in some aspects. The most important point is the lifespan of the electrode, which allows it to work longer, reaching 130 days. The ideal conditions for this electrode work, such as temperature, pH, and response time, are also good. The table below compares some of the previous methods:

Table 7: Comparison studies of the ISE used in this study versus electrodes reported in the literature

pH	Electrode lifespan	Response Time	detection limit (ion)	Research
5-8	130 days	2-27 sec	3.5×10^{-10} M of Ni	This work
3-8	60 days	50 sec	5.0×10^{-7} of Ni	[28]
3-7.9	Unconfirmed	6 sec	3.2×10^{-7} M of Ni	[29]
3.6-6.8	56 days	40 sec	2.05×10^{-8} M of Ni	[30]
2.7-7.6	180 days	3-5 sec	7.9×10^{-6} M of Ni	[31]
4-8	60 days	25 sec	8×10^{-6} M of Ni	[32]

4.10 Applications:

To experiment with the manufactured electrode, it was used to estimate nickel ions in various water samples, such as well water and industrial water from oil and gas companies in Kirkuk governorate. The ideal pH and temperature of the electrode were adjusted. The measured voltages were applied to the calibration equation of the nickel sensor electrode. The corresponding results are presented in the table below:

Table 8. Direct Method Result

Sample area	Taken Conc	Found Conc	Rec %	RSD %
Sample 1	1×10^{-3}	0.9989×10^{-3}	99.89	0.818
Sample 2	1×10^{-3}	1.006×10^{-3}	100.6	0.207
Sample 3	1×10^{-3}	1.007×10^{-3}	100.7	0.29
Sample 4	1×10^{-3}	1.005×10^{-3}	100.5	0.36
Sample 5	1×10^{-3}	0.9984×10^{-3}	99.84	0.59

The previous table explains that the results indicated that the method of making the electrode is effective and retrievable. The method is considered successful in detecting and estimating nickel ions in water. It is an innovative and simple method using inexpensive and environmentally harmless materials.

5. CONCLUSION

This study employed a green chemistry approach for the synthesis of nickel oxide NiONPs nanoparticles using "Capparis spinosa" leaf extract as a natural reducing and stabilizing agent. This method offers a safe, eco-friendly, and cost-effective alternative to conventional chemical synthesis techniques. The synthesized NiO nanoparticles were characterized using SEM, which revealed their morphology, and XRD, which confirmed their crystalline structure and size. A chemically modified carbon paste electrode incorporating NiO nanoparticles was fabricated and used for the potentiometric detection of Ni^{2+} ions in aqueous solutions. The electrode demonstrated excellent analytical performance, with a Nernstian slope of 28.5 mV/decade, a wide linearity range from 10^{-1} to 10^{-9} mol/l, a low detection limit (LOD) of 3.5×10^{-10} mol/l, a rapid response time of 2–27 seconds, and a long lifespan of approximately 130 days. Moreover, the sensor showed good stability across varying pH and temperature conditions. The novelty of this work lies in the successful use of Capparis spinosa as a green synthesis agent for NiO nanoparticles and their integration into an electrochemical sensing platform.

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