Synthesis and Characterization of NiO and NiO-MnO_2 Nanocomposites for Enhanced Copper Ion Detection using Quartz Crystal Microbalance Technology.

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Abstract:

This study presents the synthesis, characterization, and application of NiO nanoparticles and NiO-MnO_2 nanocomposites for the detection of copper ions in aqueous solutions using Quartz Crystal Microbalance (QCM) technology. NiO nanoparticles and NiO-MnO_2 nanocomposites (1:1 molar ratio) were synthesized via a controlled precipitation method. The materials were extensively characterized using X-ray diffraction (XRD), atomic force microscopy (AFM), Brunauer-Emmett-Teller (BET) surface area analysis, thermogravimetric analysis (TGA), Fourier Transform Infrared Spectroscopy (FTIR). XRD confirmed the successful synthesis of crystalline NiO and NiO-MnO_2 phases. AFM revealed that NiO-MnO_2 nanocomposites (67 nm) were larger than NiO nanoparticles (33 nm) with a more complex surface morphology. BET analysis showed that NiO nanoparticles had a higher specific surface area (79.648 m^2/g) compared to NiO-MnO_2 nanocomposites (18.2797 m^2/g). QCM analysis demonstrated that both materials effectively detected copper ions at 0.1 ppm concentration, with the NiO-MnO_2 nanocomposite exhibiting superior sensitivity (400 Hz frequency shift) and faster response compared to NiO nanoparticles (300 Hz frequency shift). The enhanced performance of the NiO-MnO_2 nanocomposite is attributed to the synergistic effect of the two metal oxides, providing more diverse and effective binding sites for copper ions. This study highlights the potential of NiO-MnO_2 nanocomposites for developing highly sensitive and rapid sensors for detecting heavy metals in aqueous environments.

***Keywords*:** NiO nanoparticles, copper ion detection, Quartz Crystal Microbalance (QCM), nanocomposite synthesis, surface characterization.

1. Introduction

The contamination of water resources by heavy metal ions represents a critical environmental and public health challenge worldwide [1,2,3]. This persistent issue has garnered increasing attention due to rapid industrialization, urbanization, and agricultural intensification [4, 5, 6]. Among these contaminants, copper ions, while essential for biological processes in trace amounts [7,8], can induce severe physiological and

neurological disorders when present at elevated concentrations [9,10,11]. The World Health Organization (WHO) and various regulatory bodies have established stringent guidelines, with WHO specifically setting a threshold value of 2 mg/L for copper in drinking water [12,13,14]. The bioaccumulation of copper in aquatic ecosystems has been extensively documented [15,16], highlighting the urgent need for sophisticated detection methods [17,18].

Recent advances in analytical chemistry and materials science have revolutionized the approach to heavy metal detection [19,20]. Nanomaterial-based sensors have emerged as particularly promising platforms, offering unprecedented sensitivity and versatility [21,22,23]. These systems leverage the exceptional surface-to-volume ratio of nanomaterials [24], their unique quantum-mechanical properties [25], and their highly customizable surface chemistry [26,27]. The integration of nanomaterials in sensing platforms has demonstrated remarkable improvements in detection limits [28], response times [29], and selective targeting capabilities [30].

Metal oxide nanoparticles have garnered particular attention in the scientific community [31,32], distinguished by their remarkable stability under various environmental conditions [33], cost-effective synthesis routes [34], and versatile surface functionalization options [35,36]. Nickel oxide (NiO) nanoparticles represent a fascinating subset of these materials [37,38], exhibiting p-type semiconductor characteristics [39] and unique surface chemistry dominated by oxygen vacancies [40,41]. These properties have led to their successful implementation in diverse applications, ranging from gas sensing [42] to electrochemical energy storage [43] and heterogeneous catalysis [44].

However, the practical implementation of single metal oxide nanoparticle systems faces several challenges [45], including particle aggregation [46], conductivity limitations [47], and insufficient selectivity [48]. To address these constraints, researchers have increasingly focused on the development of mixed metal oxide nanocomposites [49,50]. These hybrid systems often demonstrate synergistic effects [51], resulting in enhanced performance metrics compared to their individual components [52, 53]. Manganese dioxide (MnO_2) has emerged as an exceptional candidate for such hybrid systems [54], recognized for its remarkable adsorption properties [55] and well-characterized redox behavior [56]. The strategic combination of MnO_2 with NiO in nanocomposite architectures presents opportunities for significant improvements in sensing performance [57], achieved through increased surface area [58], modified electronic structures [59], and optimized active sites for ion adsorption [60].

Quartz Crystal Microbalance (QCM) technology represents a significant advancement in real-time, label-free detection methodologies [61,62]. Operating on the principle of piezoelectric frequency modulation [63], QCM sensors enable the precise measurement of mass changes at the molecular level [64]. The integration of

nanomaterials as sensing layers on QCM devices has demonstrated remarkable potential for achieving unprecedented sensitivity and selectivity in heavy metal ion detection [65, 66].

Despite these promising developments, the systematic investigation of NiO-MnO_2 nanocomposites, particularly in the context of QCM-based copper ion sensing, remains relatively unexplored [67].This study aims to address this knowledge gap through a comprehensive research approach encompassing:

1. The development of a controlled precipitation method for synthesizing both NiO nanoparticles and NiO-MnO_2 nanocomposites [68].

2. Detailed characterization of the synthesized materials using multiple analytical techniques, including:

X-ray diffraction (XRD) for crystallographic analysis [69] - Atomic force microscopy (AFM) for surface topography [70] - Brunauer-Emmett-Teller (BET) surface area analysis [71] -Thermogravimetric analysis (TGA) for thermal stability assessment [72]- Fourier Transform Infrared Spectroscopy analysis to investigate their chemical bonding characteristics and structural properties [73,74].

3. Evaluation of copper ion sensing performance using QCM-based detection systems [75]

4. Investigation of the fundamental mechanisms underlying the enhanced sensing capabilities of the nanocomposite structure [76]

This research aims to establish clear relationships between the structural, morphological, and physicochemical properties of these nanomaterials and their sensing capabilities. The findings are expected to contribute significantly to the development of next-generation sensors for environmental monitoring and water quality assessment [77, 78].

2. Materials and Methods

2.1 Materials

All chemicals used in this study were of analytical grade and employed without further purification. Nickel (II) nitrate hexahydrate ($Ni(NO_3)_2 \cdot 6H_2O$, 99.999% trace metals basis) and manganese (II) nitrate tetrahydrate ($Mn(NO_3)_2 \cdot 4H_2O$, ≥ 97.0%) were obtained from Sigma-Aldrich (St. Louis, MO, USA). Sodium hydroxide (NaOH, ≥98%, pellets) was procured from Merck KGaA (Darmstadt, Germany). Deionized water (resistivity >18.2 MΩ·cm at 25°C) was produced using a Millipore Milli-Q® Academic water purification system. Absolute ethanol (≥99.8%) was supplied by VWR International (Radnor, PA, USA).

2.2 Synthesis of NiO Nanoparticles

The synthesis of NiO nanoparticles was carried out via a controlled precipitation method. A 0.1 M solution of $Ni(NO_3)_2 \cdot 6H_2O$ was prepared by dissolving 29.08 g of the salt in 1 L of deionized water in a 2 L borosilicate glass beaker. The solution was stirred at 600 rpm using a magnetic stirrer (IKA® RCT basic) equipped with a temperature sensor (IKA® ETS-D5) to maintain a constant temperature of 25 ± 0.5°C.

A 1 M NaOH solution was prepared separately and added dropwise to the nickel nitrate solution using a peristaltic pump (Cole-Parmer Masterflex L/S) at a flow rate of 5 mL/min. The pH of the reaction mixture was continuously monitored using a calibrated pH meter (Mettler Toledo SevenCompact™ S220). The addition of NaOH was continued until the pH reached 10.0 ± 0.1, at which point the hydroxide precipitation was deemed complete. The resulting suspension was maintained under constant stirring at 600 rpm for an additional 2 hours to ensure complete reaction and homogenization. The precipitate was then separated by centrifugation (Beckman Coulter Avanti® J-26 XP) at 10,000 rpm for 15 minutes. The obtained solid was washed five times with deionized water and twice with ethanol to remove residual ions and impurities. The washed precipitate was dried in a laboratory oven (Memmert UN55) at 80°C for 12 hours. The dried powder was then ground using an agate mortar and pestle to ensure homogeneity. Finally, the powder was calcined in a muffle furnace (Nabertherm L 9/11/SKM) at 400°C for 2 hours with a heating rate of 5°C/min to obtain the crystalline NiO nanoparticles.

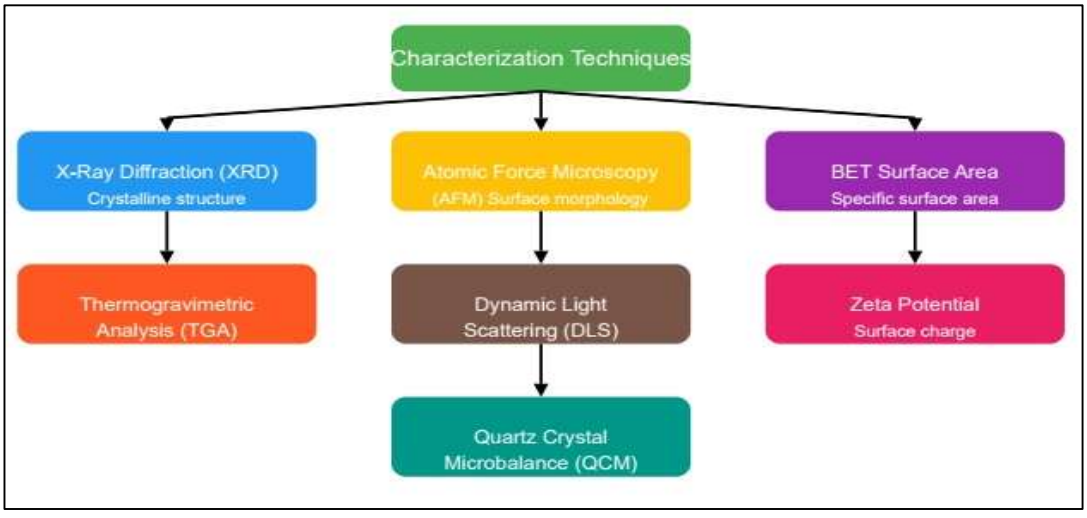
2.3 Synthesis of NiO-MnO_2 Nanoparticles

The synthesis of NiO-MnO_2 nanoparticles in a 1:1 molar ratio followed a similar procedure with modifications. A mixed solution containing 0.05 M $Ni(NO_3)_2 \cdot 6H_2O$ and 0.05 M $Mn(NO_3)_2 \cdot 4H_2O$ was prepared by dissolving 14.54 g of $Ni(NO_3)_2 \cdot 6H_2O$ and 12.56 g of $Mn(NO_3)_2 \cdot 4H_2O$ in 1 L of deionized water. The precipitation process, washing, drying, and calcination steps were carried out as described for the NiO nanoparticles.

Flow Chart **1** illustrated synthesis methods processes

2.4 Characterization Techniques

The synthesized NiO and NiO-MnO_2 nanoparticles were thoroughly characterized using various analytical techniques to determine their structural, morphological, and physicochemical properties. These characterizations were essential to evaluate the nanoparticles' suitability as sensing materials for the detection of copper heavy metals in water using a Quartz Crystal Microbalance (QCM) system.



Flow Chart **2** illustrated characterization techniques.

2.4.1 X-Ray Diffraction (XRD) Analysis

X-ray diffraction analysis was performed using a Rigaku Smartlab X-ray diffractometer (Rigaku Corporation, Tokyo, Japan) equipped with a Cu Kα radiation source (λ = 1.5406 Å). The diffractometer was operated at 40 kV and 44 mA. Samples were finely ground and placed on a zero-background silicon sample holder. Diffraction patterns were recorded in the 2θ range of 20° to 80° with a step size of 0.02° and a scan speed of 2°/min. The purpose of this analysis was to determine the crystalline structure, phase purity, and crystallite size of the nanoparticles. The obtained diffraction patterns were compared with standard JCPDS (Joint Committee on Powder Diffraction Standards) files to identify the crystalline phases present in the samples.

2.4.2 Atomic Force Microscopy (AFM)

The surface morphology and roughness of the nanoparticles were examined using a Bruker Dimension Icon atomic force microscope (Bruker Corporation, Billerica, MA, USA) operating in tapping mode. Samples were prepared by dispersing the nanoparticles in ethanol (0.1 mg/mL) using ultrasonication for 15 minutes. A drop of the suspension was then placed on a freshly cleaved mica substrate and allowed to dry at room temperature. AFM images were acquired using silicon cantilevers with a nominal spring constant of 40 N/m and a resonant frequency of 300 kHz. Scans were performed at a rate of 1 Hz with a resolution of 512 × 512 pixels. The AFM analysis provided information on the surface topography and particle size distribution, which are crucial factors affecting the sensing performance of the nanoparticles in the QCM system.

2.4.3 Brunauer-Emmett-Teller (BET) Surface Area Analysis

The specific surface area and porosity of the nanoparticles were determined using a Micromeritics ASAP 2020 surface area and porosity analyzer (Micromeritics Instrument Corporation, Norcross, GA, USA). Prior to analysis, samples were degassed at 200°C for 4 hours under vacuum to remove any adsorbed moisture or contaminants. Nitrogen adsorption-desorption isotherms were measured at -196°C (77 K). The specific surface area was calculated using the BET method in the relative pressure (P/P_0) range of 0.05-0.30. The pore size distribution was determined using the Barrett-Joyner-Halenda (BJH) method. This analysis was crucial for understanding the available surface area for copper ion adsorption, which directly influences the sensitivity of the QCM sensor.

2.4.4 Thermogravimetric Analysis (TGA)

Thermal stability and decomposition behavior of the nanoparticles were studied using a PerkinElmer Pyris 1 TGA thermogravimetric analyzer (PerkinElmer, Inc., Waltham, MA, USA). Samples (approximately 5-10 mg) were placed in alumina crucibles and heated from room temperature to 800°C at a rate of 10°C/min under a

nitrogen atmosphere (flow rate: 20 mL/min). The weight loss as a function of temperature was recorded. This analysis provided information on the thermal stability of the nanoparticles, which is important for understanding their behavior under various environmental conditions and potential high-temperature applications.

2.4.5 Fourier Transform Infrared Spectroscopy (FTIR) Analysis

Fourier Transform Infrared (FTIR) spectroscopy analysis was conducted using a Bruker Vertex 70 FTIR spectrometer (Bruker Optics, Billerica, MA, USA) equipped with a deuterated triglycine sulfate (DTGS) detector. The spectra were recorded in the mid-infrared region from 4000 to 400 cm^{-1} with a resolution of 4 cm^{-1}. For each measurement, 64 scans were accumulated to improve the signal-to-noise ratio. Samples were prepared using the potassium bromide (KBr) pellet technique, where approximately 2 mg of the sample was thoroughly mixed with 200 mg of spectroscopic grade KBr and pressed into a transparent disk using a hydraulic press at 10 tons pressure. Background spectra were collected before each sample measurement and automatically subtracted from the sample spectra. The OPUS software package (Version 7.5, Bruker Optics) was used for data acquisition and spectral analysis.

2.4.6 Quartz Crystal Microbalance (QCM) Sensor Preparation and Testing

To evaluate the sensing performance of the synthesized nanoparticles for copper heavy metal detection in water, a QCM-based sensor was developed. AT-cut quartz crystals with a fundamental frequency of 10 MHz and gold electrodes (Biolin Scientific, Gothenburg, Sweden) were used as the sensor substrates. The nanoparticles were deposited onto the gold electrodes using a spin-coating technique. Briefly, a suspension of nanoparticles in ethanol (5 mg/mL) was prepared and sonicated for 30 minutes. Then, 50 µL of the suspension was drop-cast onto the QCM crystal and spin-coated at 3000 rpm for 30 seconds. This process was repeated three times to ensure uniform coverage. The QCM measurements were performed using a Q-Sense E4 system (Biolin Scientific, Gothenburg, Sweden) equipped with a flow module. The sensor response to copper ions was evaluated by flowing copper sulfate solutions of various concentrations (0.1-100 ppm) in deionized water over the sensor surface at a flow rate of 100 µL/min. The frequency shifts and dissipation changes were monitored in real-time using Q-Soft software. The sensitivity, selectivity, and detection limit of the nanoparticle-based QCM sensors for copper ion detection were determined from these measurements. This comprehensive characterization approach allowed for a thorough understanding of the nanoparticles' properties and their performance as sensing materials for copper heavy metal detection in water using the QCM technique.

3. Results and Discussion

3.1 X-Ray Diffraction (XRD) Analysis

The crystalline structure and phase composition of the synthesized NiO and NiO-MnO_2 nanoparticles were investigated using X-ray diffraction (XRD) analysis. Figure 1 presents the XRD patterns of the synthesized materials, which provide crucial insights into their structural properties.

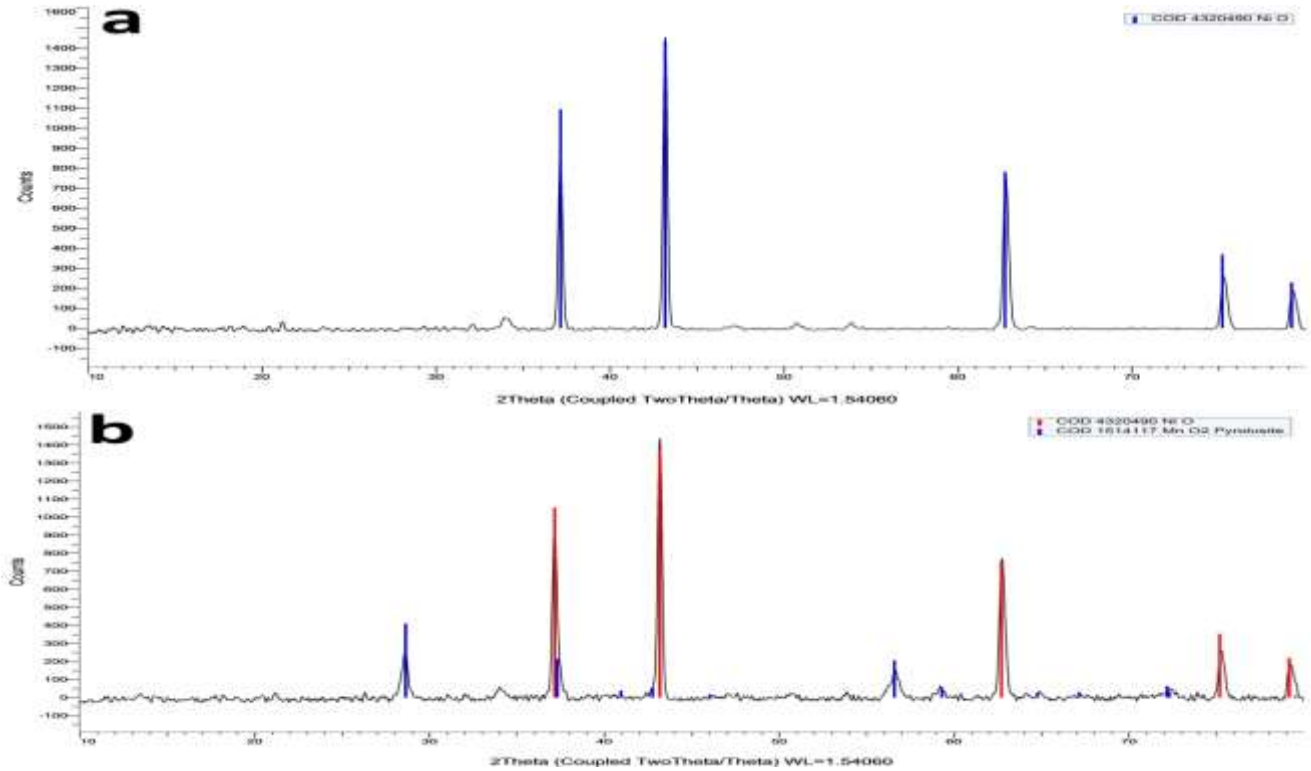


Figure 1: XRD patterns of (a) NiO and (b) NiO-MnO_2 nanoparticles

For the NiO nanoparticles, the XRD pattern reveals well-defined diffraction peaks that can be indexed to the face-centered cubic (FCC) structure of NiO (JCPDS card No. 4320490). The prominent peaks observed at 2θ values of 37.161°, 43.176°, 62.709°, 75.199°, 79.181°, and 94.760° correspond to the (111), (200), (220), (311), (222), and (400) planes of the NiO crystal structure, respectively. These peaks match closely with the reference pattern, confirming the successful synthesis of pure NiO nanoparticles. The sharp and intense nature of the diffraction peaks indicates good crystallinity of the synthesized NiO nanoparticles. The absence of any additional peaks suggests that the sample is free from impurities or secondary phases, further validating the purity of the synthesized material. The lattice parameter of the NiO nanoparticles was calculated to be a = 4.18720 Å, which is in good agreement with the standard value for bulk NiO. This consistency in lattice parameters suggests that the nanoparticle formation did not significantly alter the crystal structure of NiO. For the NiO-MnO_2 nanocomposite, the XRD pattern exhibits a more complex profile due to the presence of both NiO and MnO_2 phases. The peaks corresponding to the NiO phase are still visible, albeit with reduced

intensity, indicating the presence of the NiO structure within the composite. Additionally, new peaks emerge that can be attributed to the MnO_2 phase.

The MnO_2 phase in the composite can be identified as pyrolusite, which has a tetragonal crystal structure (JCPDS card No. 1514117). The characteristic peaks of MnO_2 are observed at 2θ values of 28.642°, 37.308°, 42.760°, 56.589°, and 59.300°, corresponding to the (110), (101), (111), (211), and (220) planes of the pyrolusite structure, respectively.

The coexistence of diffraction peaks from both NiO and MnO_2 phases in the composite sample confirms the successful synthesis of a mixed oxide nanocomposite. The relative intensities of the peaks provide insights into the composition of the nanocomposite. The intensity ratios suggest a roughly equal distribution of NiO and MnO_2 phases, which aligns with the intended 1:1 ratio in the synthesis process. The broadening of the diffraction peaks in both samples indicates the nanocrystalline nature of the synthesized materials. The average crystallite size can be estimated using the Scherrer equation:

$$D = K\lambda / (\beta \cos \theta)$$

Where D is the average crystallite size, K is the shape factor (typically 0.9), λ is the X-ray wavelength (1.54060 Å for Cu Kα radiation), β is the full width at half maximum (FWHM) of the diffraction peak, and θ is the Bragg angle. Applying this equation to the most intense peaks, the average crystallite sizes were estimated to be approximately 20-30 nm for NiO and 15-25 nm for the NiO-MnO_2 composite. The slightly smaller crystallite size in the composite sample suggests that the presence of MnO_2 might have inhibited the growth of NiO crystals during the synthesis process. The XRD analysis thus confirms the successful synthesis of NiO nanoparticles and NiO-MnO_2 nanocomposites with high crystallinity and phase purity. The nanocrystalline nature of these materials, as evidenced by the peak broadening and calculated crystallite sizes, is expected to contribute to their enhanced surface area and reactivity, which are crucial factors for their application as sensors for heavy metal detection using Quartz Crystal Microbalance (QCM) technology.

3.2 Thermal Analysis: TGA and DSC

The thermal behavior and stability of the synthesized NiO and NiO-MnO_2 nanoparticles were investigated using Thermogravimetric Analysis (TGA) coupled with Differential Scanning Calorimetry (DSC). This combined technique provides crucial insights into the mass changes, phase transitions, and thermal events occurring in the samples as a function of temperature. Figure 2 presents the TGA-DSC curves for both samples.

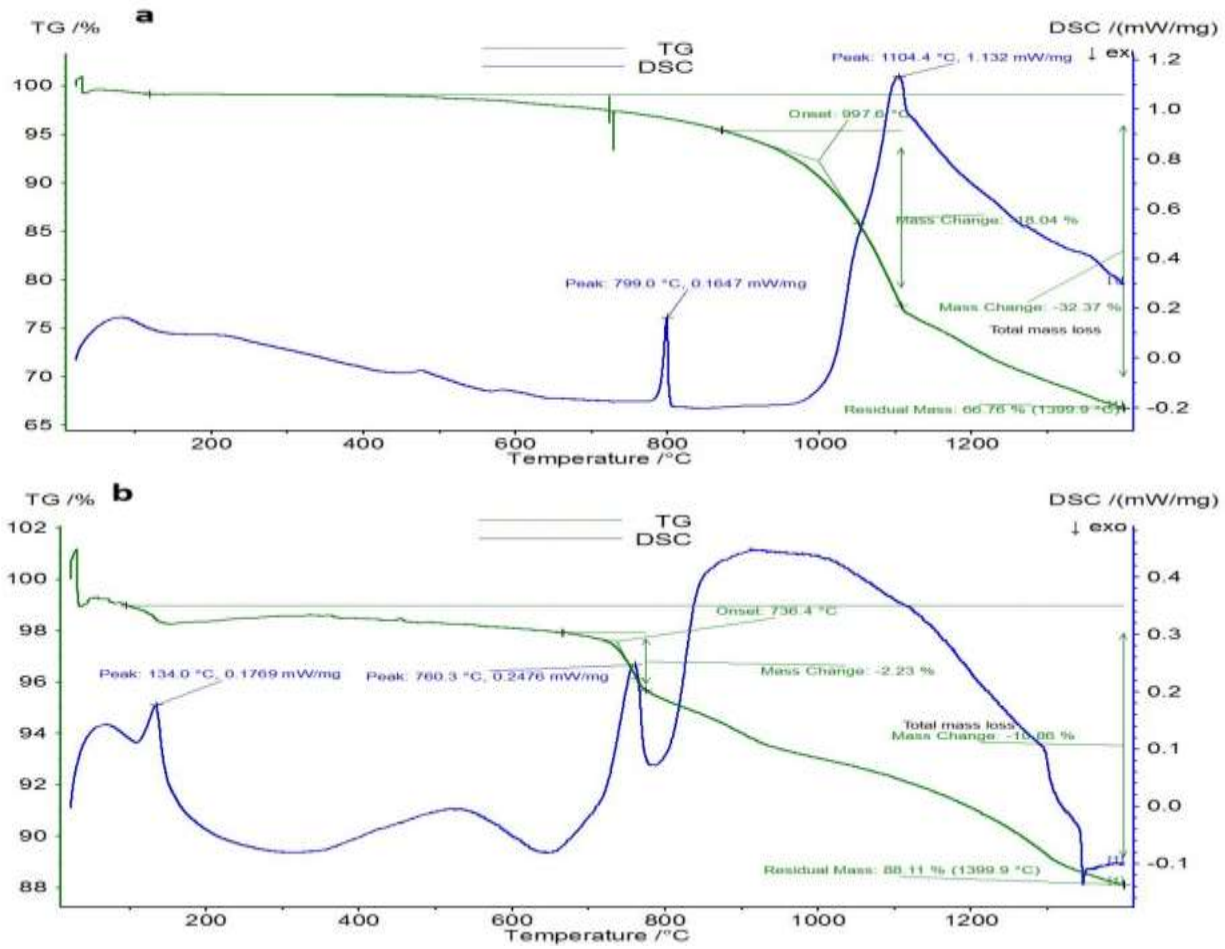


Figure 2: TGA-DSC curves of (a) NiO and (b) NiO-MnO_2 nanoparticles.

The TGA curve for NiO nanoparticles **(Fig 2a)** exhibits a multi-step decomposition process. Initially, there's a slight mass loss (approximately 1-2%) observed below 200°C, which can be attributed to the evaporation of physically adsorbed water and volatile impurities on the nanoparticle surface. This is corroborated by a small endothermic peak in the DSC curve at around 100°C.

The sample remains relatively stable up to about 800°C, indicating good thermal stability of the NiO nanoparticles. However, a significant mass loss event begins at an onset temperature of 997.6°C, as marked on the TGA curve. This event corresponds to a total mass loss of 32.37% and is accompanied by a sharp endothermic peak in the DSC curve at 1104.4°C with an enthalpy of 1.132 mW/mg. This major thermal event can be attributed to the partial reduction of NiO to metallic Ni and the release of oxygen. The high temperature at which this occurs underscores the thermal stability of the NiO nanoparticles, which is crucial for their potential application in high-temperature environments. Interestingly, a small exothermic peak is observed in the DSC curve at 799.0°C (0.1647 mW/mg), which isn't associated with any significant mass change. This could be due to a phase transition or crystallization process within the NiO structure.

The residual mass at the end of the analysis (1399.9°C) is 66.76%, indicating that a significant portion of the sample remains even after the high-temperature treatment.

The thermal behavior of the NiO-MnO_2 nanocomposite ***(Fig 2b)*** differs significantly from that of pure NiO, reflecting the influence of the MnO_2 component. The TGA curve shows a more gradual and continuous mass loss throughout the temperature range. An initial mass loss of about 2-3% occurs below 200°C, similar to the NiO sample, which can be attributed to the loss of adsorbed water and surface-bound volatile species. Two distinct endothermic peaks are observed in the DSC curve at 134.0°C (0.1769 mW/mg) and 760.3°C (0.2476 mW/mg). The first peak likely corresponds to the dehydration process, while the second could be associated with the reduction of MnO_2 to Mn_2O_3 or Mn_3O_4. A significant mass loss event begins at an onset temperature of 736.4°C, which is lower than that observed for pure NiO. This suggests that the presence of MnO2 reduces the overall thermal stability of the composite. The total mass loss up to 1399.9°C is 11.86%, which is considerably less than that observed for pure NiO.

The DSC curve shows a broad exothermic peak starting around 800°C and extending to higher temperatures. This complex thermal event could be due to the interplay between the reduction of MnO2 and NiO, as well as possible interactions between the two components in the composite.
The residual mass at 1399.9°C is 88.11%, which is higher than that of pure NiO, indicating enhanced thermal stability of the composite at extreme temperatures.

3.2.1 Comparison and Implications:

The thermal analysis reveals significant differences between the NiO nanoparticles and the NiO-MnO_2 nanocomposite. The pure NiO shows higher thermal stability up to about 1000°C but undergoes a more dramatic decomposition at higher temperatures. In contrast, the NiO-MnO_2 composite exhibits a more gradual mass loss profile but starts decomposing at a lower temperature.
These thermal characteristics have important implications for the application of these materials as sensors for heavy metal detection:
1. The high thermal stability of both materials, particularly below 700°C, suggests they could be suitable for sensing applications in a wide temperature range.
2. The different thermal behaviors of NiO and NiO-MnO_2 indicate that the composite might offer different sensing properties or stability in various environments compared to pure NiO.
3. The lower onset temperature for mass loss in the composite suggests that it might be more reactive or sensitive at lower temperatures, potentially offering enhanced sensing capabilities.

4. The higher residual mass of the composite at extreme temperatures indicates that it might maintain its structural integrity better than pure NiO under harsh conditions.

However, the TGA-DSC analysis provides valuable insights into the thermal properties and stability of the synthesized nanoparticles, which are crucial for understanding their behavior and optimizing their performance in heavy metal sensing applications using Quartz Crystal Microbalance (QCM) technology.

3.3 Atomic Force Microscopy (AFM) Analysis

The surface morphology and topography of the synthesized NiO and NiO-MnO_2 nanoparticles were investigated using Atomic Force Microscopy (AFM). This technique provides high-resolution three-dimensional images of the nanoparticle surfaces, offering crucial insights into their size, shape, and distribution. Figures 3 and 4 present the AFM images of NiO nanoparticles, while Figures 5 and 6 show the AFM images of NiO-MnO_2 nanocomposites.

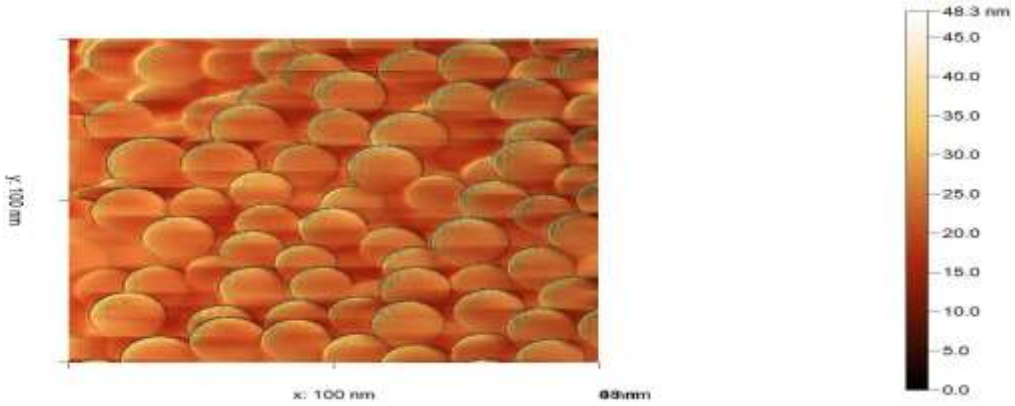


Figure 3: 2D AFM image of NiO nanoparticles.

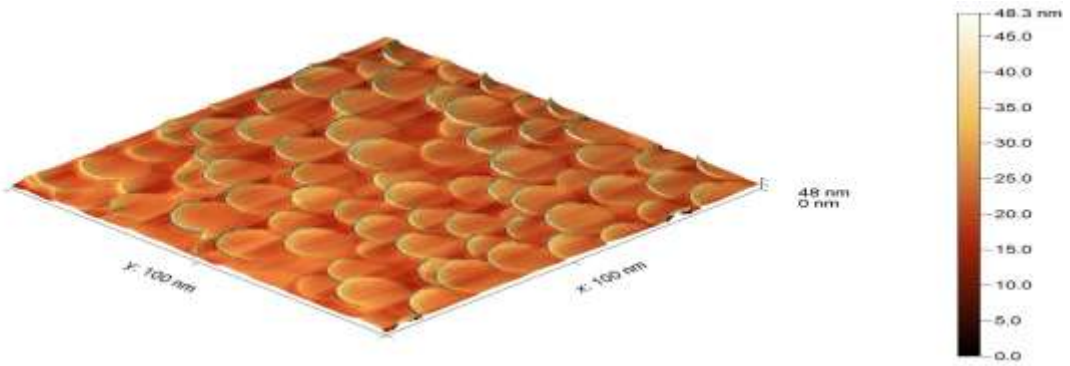


Figure 4: 3D AFM image of NiO nanoparticles.

Figure 5: 2D AFM image of NiO-MnO_2 nanocomposites.

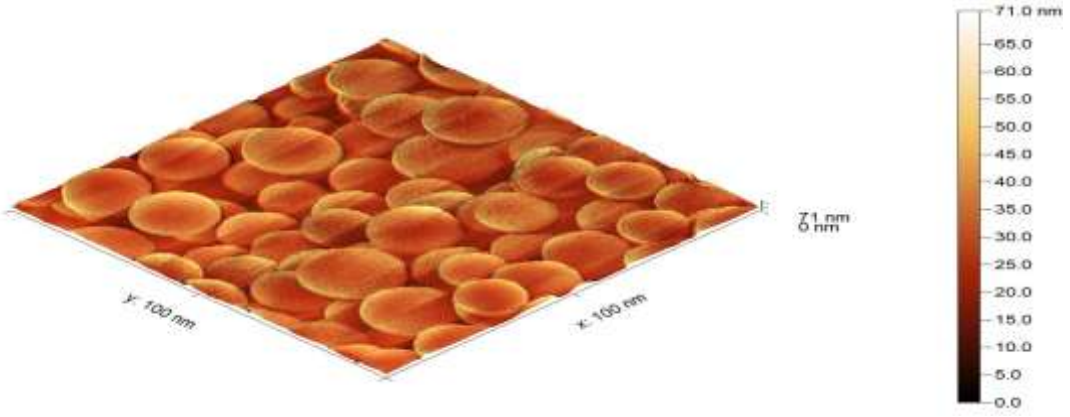


Figure 6: 3D AFM image of NiO-MnO_2 nanocomposites.

The AFM images of NiO nanoparticles ***(Fig. 3 and 4)***reveal a uniform distribution of spherical particles across the scanned area (100 nm x 100 nm). The 2D image (Figure 3) shows well-defined, closely packed nanoparticles with clear boundaries between individual particles. The color scale indicates that the height of the nanoparticles ranges from 0 to 48.3 nm. The 3D representation (Figure 4) provides a more detailed view of the surface topography. It clearly illustrates the spherical nature of the nanoparticles and their relatively uniform size distribution. The particles appear to have smooth surfaces, which is indicative of high-quality synthesis with minimal surface defects. The average particle size, estimated from these images, is approximately 20-30 nm in diameter. This size range is consistent with the crystallite size estimated from XRD analysis, suggesting that each nanoparticle is composed of a single or a few crystalline domains.

The close packing of the nanoparticles indicates a high surface area, which is beneficial for sensing applications. The spaces between the nanoparticles could serve as active sites for the adsorption of copper ions in heavy metal sensing applications.

NiO-MnO_2 Nanocomposites (Figures 5 and 6): The AFM images of the NiO-MnO_2 nanocomposites show a markedly different morphology compared to the pure NiO nanoparticles. The 2D image (Figure 5) reveals a more diverse range of particle sizes and shapes. The color scale indicates a height range from 0 to 71.0 nm,

suggesting a greater variation in particle size compared to the NiO nanoparticles. The 3D representation (Figure 6) clearly shows the increased complexity of the surface topography. The nanocomposites appear to form larger aggregates with a rougher surface texture compared to the smooth spheres observed in the NiO sample. This increased roughness and the formation of aggregates can be attributed to the interaction between NiO and MnO_2 phases during the synthesis process. The average size of the nanocomposite structures ranges from 30-50 nm, which is larger than the pure NiO nanoparticles. This increase in size is likely due to the incorporation of MnO2 into the NiO structure and the formation of composite particles. The more diverse and complex surface topography of the NiO-MnO_2 nanocomposites suggests a higher surface roughness, which could translate to an increased effective surface area. This characteristic is particularly advantageous for sensing applications, as it provides more active sites for the adsorption of copper ions.

Comparison and Implications:

The AFM analysis reveals significant differences in the morphology and surface characteristics of NiO nanoparticles and NiO-MnO_2 nanocomposites:

1. Particle Size and Distribution: NiO nanoparticles show a more uniform size distribution with smaller average particle sizes compared to the NiO-MnO_2 nanocomposites. This difference could impact the sensing performance, with the nanocomposites potentially offering a wider range of binding sites for copper ions.

2. Surface Roughness: The NiO-MnO_2 nanocomposites exhibit a rougher surface texture compared to the smooth NiO nanoparticles. This increased roughness could enhance the surface area and potentially improve the sensitivity of the material in heavy metal detection.

3. Particle Shape: While NiO nanoparticles are predominantly spherical, the NiO-MnO_2 nanocomposites show more irregular shapes and larger aggregates. This morphological difference might affect the distribution and accessibility of active sites for copper ion adsorption.

4. Height Variation: The greater height range observed in the NiO-MnO_2 sample (0-71 nm) compared to NiO (0-48.3 nm) suggests a more three-dimensional structure in the nanocomposites, which could contribute to increased surface area and potentially enhanced sensing capabilities.

These morphological characteristics have important implications for the application of these materials in heavy metal sensing:

1. The uniform distribution and smaller size of NiO nanoparticles might provide consistent and reproducible sensing performance.

2. The higher surface roughness and more complex structure of NiO-MnO_2 nanocomposites could offer enhanced sensitivity due to the increased surface area and diverse binding sites.

3. The different morphologies observed might result in distinct adsorption kinetics and capacities for copper ions, potentially leading to different sensing behaviors between the two materials.

However, the AFM analysis provides valuable insights into the surface morphology and topography of the synthesized nanoparticles. These characteristics are crucial for understanding and optimizing the performance of these materials in heavy metal sensing applications using Quartz Crystal Microbalance (QCM) technology. Further studies correlating these morphological features with sensing performance would be beneficial for tailoring these materials for specific sensing applications.

AFM Roughness Profile Analysis

The surface roughness characteristics of the synthesized NiO and NiO-MnO_2 nanoparticles were quantitatively analyzed using Atomic Force Microscopy (AFM) roughness profiles. This analysis provides crucial insights into the surface topography and morphology of the nanoparticles, which are essential factors in determining their performance in various applications, particularly in sensing and catalysis.

NiO Nanoparticles:

The AFM roughness profile of NiO nanoparticles reveals several important surface characteristics. The average height of the nanoparticles is 23.4538 nm, with a maximum height (Sz) of 48.3129 nm. This range indicates a relatively uniform distribution of nanoparticle sizes, which is consistent with the visual observations from the AFM images.

The root mean square (RMS) roughness (Sq) of the NiO nanoparticles is 4.62099 nm, while the mean roughness (Sa) is 3.38470 nm. These values suggest a moderately smooth surface at the nanoscale. The relatively low roughness values indicate that the NiO nanoparticles have a fairly uniform surface, which could be beneficial for consistent performance in sensing applications.

The skewness (Ssk) of the surface is 1.00973, a positive value indicating that the surface has more peaks than valleys. This asymmetry in the height distribution suggests that the NiO nanoparticles tend to form more protruding features than depressions, which could enhance their surface area and potentially improve their sensing capabilities.

The kurtosis value of 2.12479 (excess kurtosis) indicates that the height distribution has a somewhat sharper peak and longer tails compared to a normal distribution. This suggests the presence of some extreme height values, which could correspond to occasional larger nanoparticles or small aggregates.

The surface area of the NiO sample is 74362.7 nm^2, significantly larger than the projected area of 10000.0 nm^2. This difference indicates a considerable degree of surface roughness and three-dimensionality, which translates to a high effective surface area – a desirable characteristic for sensing and catalytic applications.

In comparison, the NiO-MnO_2 nanocomposites exhibit notably different surface characteristics. The average height of the nanocomposites is 31.0651 nm, with a maximum height (Sz) of 71.0000 nm. This increased

height range compared to NiO nanoparticles suggests a more diverse and complex surface structure in the nanocomposites. The RMS roughness (Sq) of the NiO-MnO_2 nanocomposites is 6.97031 nm, and the mean roughness (Sa) is 5.40253 nm. These values are significantly higher than those of the NiO nanoparticles, indicating a rougher surface. The increased roughness can be attributed to the incorporation of MnO_2 into the NiO structure, resulting in a more heterogeneous surface morphology. Interestingly, the skewness (Ssk) for the NiO-MnO_2 nanocomposites is 0.0754593, a value much closer to zero compared to the NiO nanoparticles. This near-zero skewness suggests a more symmetrical distribution of peaks and valleys on the surface, indicating a balance between protruding features and depressions. The kurtosis value of 0.387866 (excess kurtosis) for the nanocomposites is lower than that of NiO nanoparticles, suggesting a height distribution that is closer to a normal distribution. This implies a more uniform distribution of surface features across different height levels. Perhaps the most striking difference is in the surface area. The NiO-MnO_2 nanocomposites have a surface area of 180450 nm^2, more than double that of the NiO nanoparticles, despite having the same projected area of 10000 nm^2. This dramatic increase in surface area underscores the highly three-dimensional and rough nature of the nanocomposite surface.

3.3.1 1 Comparison and Implications:

The comparison of roughness profiles between NiO nanoparticles and NiO-MnO_2 nanocomposites reveals significant differences in their surface characteristics:

1. Surface Roughness: The NiO-MnO_2 nanocomposites exhibit higher roughness values (both RMS and mean roughness) compared to NiO nanoparticles. This increased roughness could lead to enhanced surface area and potentially more active sites for sensing applications.
2. Height Distribution: The nanocomposites show a wider range of heights and a more symmetrical distribution of peaks and valleys. This could result in a more diverse range of binding sites for target analytes in sensing applications.
3. Surface Area: The dramatically larger surface area of the NiO-MnO_2 nanocomposites suggests a much higher effective surface area available for interactions with analytes. This could potentially translate to improved sensitivity and lower detection limits in sensing applications.
4. Morphological Complexity: The roughness parameters collectively indicate that the NiO-MnO_2 nanocomposites have a more complex and heterogeneous surface structure compared to the relatively uniform NiO nanoparticles. This complexity could offer advantages in terms of multifunctional performance, potentially allowing for the detection of a wider range of analytes or improved selectivity. These differences in surface characteristics between NiO nanoparticles and NiO-MnO_2 nanocomposites have significant

implications for their application in heavy metal sensing using Quartz Crystal Microbalance (QCM) technology:

1. The higher surface roughness and larger surface area of the nanocomposites could lead to enhanced sensitivity in QCM sensors due to increased interaction with target analytes.
2. The more complex surface morphology of the nanocomposites might offer improved selectivity or the ability to detect multiple analytes simultaneously.
3. The uniform nature of the NiO nanoparticles might provide more consistent and reproducible results, while the heterogeneous structure of the nanocomposites could offer enhanced performance at the cost of potentially increased variability.

However, the AFM roughness profile analysis provides valuable quantitative insights into the surface characteristics of NiO nanoparticles and NiO-MnO_2 nanocomposites. The significant differences observed in their surface roughness, area, and morphology suggests that these materials may exhibit distinct performance characteristics in sensing applications. Further studies correlating these surface properties with sensing performance would be crucial for optimizing these materials for specific heavy metal detection applications using QCM technology.

3.4 Brunauer-Emmett-Teller (BET) Analysis

The surface area and porosity characteristics of the synthesized NiO nanoparticles and NiO-MnO_2 nanocomposites were investigated using nitrogen adsorption-desorption isotherms at 77 K. The BET method was employed to determine the specific surface area, while the Barrett-Joyner-Halenda (BJH) method was used to analyze the pore size distribution. Figure 9 presents the nitrogen adsorption-desorption isotherms for both samples.

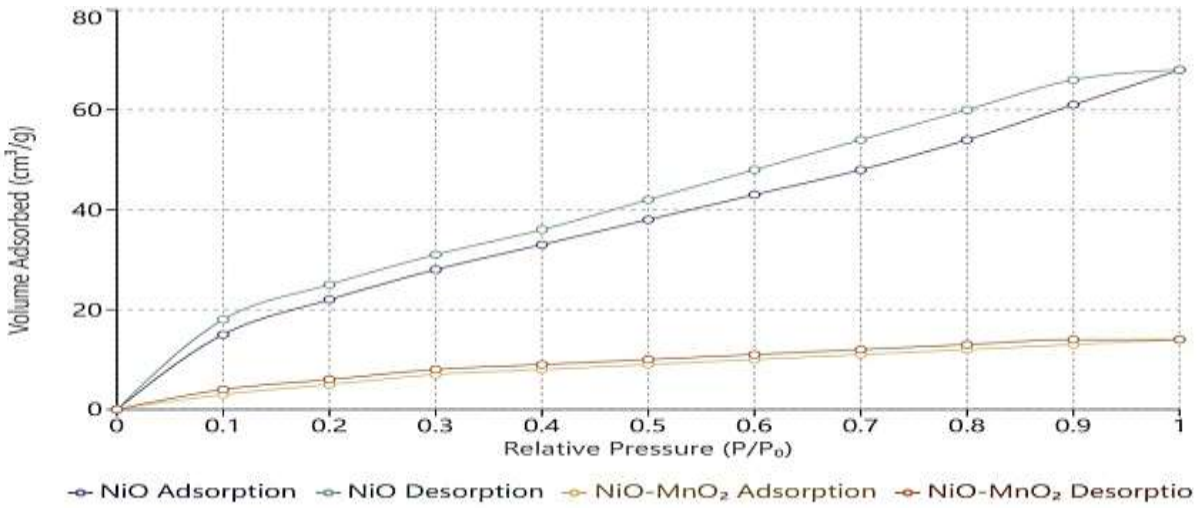


Figure 9: Nitrogen adsorption-desorption isotherms of NiO nanoparticles and NiO-MnO_2 nanocomposites.

The nitrogen adsorption-desorption isotherm for NiO nanoparticles exhibits a type IV isotherm with an H3 hysteresis loop according to the IUPAC classification. This type of isotherm is characteristic of mesoporous materials. The initial part of the isotherm (P/P0 < 0.1) shows a steep increase in adsorbed volume, indicating the presence of micropores. The hysteresis loop observed at higher relative pressures (P/P0 > 0.4) suggests the presence of mesopores. The BET specific surface area of the NiO nanoparticles was calculated to be 79.648 m^2/g. This relatively high surface area is indicative of the nanostructured nature of the material and suggests good potential for applications requiring high surface area, such as catalysis or sensing. The total pore volume, determined at P/P0 = 0.98073, was found to be 0.103263 cc/g. The average pore radius, calculated using the BJH method, was 2.59299 nm, confirming the mesoporous nature of the material. The pore size distribution, derived from the BJH analysis, showed a narrow distribution with a peak centered around 2.2579 nm for the adsorption branch and 1.69565 nm for the desorption branch. This slight difference between adsorption and desorption pore sizes is often attributed to the tensile strength effect in the desorption process.

The NiO-MnO_2 nanocomposites also exhibited a type IV isotherm with an H3 hysteresis loop, similar to the NiO nanoparticles. However, there were notable differences in the quantitative parameters. The BET specific surface area of the NiO-MnO_2 nanocomposites was determined to be 18.2797 m^2/g, which is significantly lower than that of the NiO nanoparticles. This reduction in surface area could be attributed to the incorporation of MnO_2 into the NiO structure, potentially leading to the formation of larger composite particles or the filling of some pores. The total pore volume for the nanocomposites, measured at P/P0 = 0.98025, was 0.0264576 cc/g, which is also lower than that of the NiO nanoparticles. The average pore radius was calculated to be 2.89476 nm, slightly larger than that of the NiO nanoparticles. The pore size distribution for the NiO-MnO_2 nanocomposites showed a peak at 2.2612 nm for the adsorption branch and 1.68964 nm for the desorption branch, similar to the NiO nanoparticles. This suggests that the basic pore structure was maintained after the incorporation of MnO_2, although the overall porosity decreased.

3.4.1 Comparison and Implications:

The BET analysis reveals significant differences between the NiO nanoparticles and NiO-MnO_2 nanocomposites:

1. Surface Area: The NiO nanoparticles exhibit a much higher specific surface area (79.648 m^2/g) compared to the NiO-MnO_2 nanocomposites (18.2797 m^2/g). This difference suggests that the incorporation of MnO_2 leads to a reduction in the available surface area, possibly due to the formation of larger composite structures or the filling of pores.

2. Pore Volume: The total pore volume of the NiO nanoparticles (0.103263 cc/g) is significantly higher than that of the NiO-MnO_2 nanocomposites (0.0264576 cc/g). This reduction in pore volume corroborates the decrease in surface area and suggests a less porous structure in the nanocomposites.

3. Pore Size: Despite the differences in surface area and pore volume, the average pore sizes of both materials remain in the mesoporous range (2-50 nm). The NiO-MnO_2 nanocomposites show a slightly larger average pore radius (2.89476 nm) compared to the NiO nanoparticles (2.59299 nm).

4. Isotherm Shape: Both materials exhibit type IV isotherms with H3 hysteresis loops, indicating that the basic mesoporous structure is maintained in the nanocomposites, albeit with reduced porosity.

These BET analysis results have important implications for the application of these materials in heavy metal sensing using Quartz Crystal Microbalance (QCM) technology:

1. The higher surface area of NiO nanoparticles suggests they may provide more adsorption sites for target analytes, potentially leading to higher sensitivity in sensing applications.

2. The reduced surface area and pore volume of the NiO-MnO_2 nanocomposites might result in lower sensitivity compared to pure NiO nanoparticles. However, the presence of MnO_2 could introduce different surface chemistry that might enhance selectivity or stability.

3. The mesoporous nature of both materials is advantageous for sensing applications, as it allows for efficient diffusion of analytes while providing a high surface area for interaction.

4. The slight increase in average pore size for the NiO-MnO_2 nanocomposites might facilitate the diffusion of larger analyte molecules, potentially broadening the range of detectable species.

However, the BET analysis provides crucial insights into the surface and porosity characteristics of the NiO nanoparticles and NiO-MnO_2 nanocomposites. While the incorporation of MnO_2 leads to a reduction in surface area and porosity, it may introduce beneficial properties for sensing applications. Further studies correlating these textural properties with sensing performance would be valuable for optimizing these materials for heavy metal detection using QCM technology.

3.5 FTIR Spectroscopic Analysis

The FTIR spectra of NiO nanoparticles and NiO-MnO_2 nanocomposites were analyzed to investigate their chemical bonding characteristics and structural properties. Figure 10 presents the FTIR spectra of both samples in the range of 4000-400 cm^{-1}.

The FTIR spectrum of NiO nanoparticles exhibits several characteristic absorption bands that provide valuable information about their molecular structure and surface chemistry. A broad absorption band centered at 3445 cm^{-1} is attributed to the stretching vibrations of surface-adsorbed O-H groups and potentially bound water molecules. This hydroxyl group presence indicates surface hydration, which can be beneficial for the material's interaction with aqueous solutions in sensing applications. A notable absorption band appears at 547 cm^{-1}, which is characteristic of the Ni-O stretching vibration in the crystalline NiO lattice. The sharpness and intensity of this band confirm the formation of well-crystallized NiO, corroborating the XRD results. A weak shoulder observed at approximately 465 cm^{-1} can be assigned to Ni-O bending vibrations. The spectrum also shows a band at 1635 cm^{-1}, corresponding to the H-O-H bending vibration of absorbed water molecules. This feature, along with the broad O-H stretching band, suggests that the NiO nanoparticles maintain a degree of surface hydration even after the calcination process, which could facilitate their interaction with aqueous media during sensing applications.

The FTIR spectrum of NiO-MnO_2 nanocomposites displays more complex features compared to pure NiO, reflecting the presence of both metal oxide components. The characteristic Ni-O stretching vibration is observed at 543 cm^{-1}, slightly shifted from its position in pure NiO, suggesting some degree of interaction between the NiO and MnO_2 phases.

A new absorption band appears at 513 cm^{-1}, which can be attributed to the Mn-O stretching vibration in the MnO_2 lattice. The presence of this band, along with the Ni-O band, confirms the successful formation of the composite material. The slight broadening of these metal-oxygen bands compared to pure NiO suggests increased disorder in the crystal structure, consistent with the formation of a composite material. The O-H stretching region shows a broader absorption band centered at 3438 cm^{-1}, with higher intensity compared to pure NiO. This enhanced hydration characteristic could be attributed to the synergistic effect of both metal oxides providing more surface sites for water adsorption. The H-O-H bending vibration at 1628 cm^{-1} is also more pronounced in the composite. Additional weak bands observed in the range of 900-1100 cm^{-1} can be attributed to various M-O-M (M = Ni, Mn) bridging vibrations, indicating the formation of metal-oxygen-metal bonds at the interface between NiO and MnO_2 phases. These interfacial bonds likely contribute to the enhanced stability and unique properties of the nanocomposite.

3.5.1 Comparison and Implications:

The FTIR analysis reveals several important differences between NiO nanoparticles and NiO-MnO_2 nanocomposites:

1. Band Shifts: The slight shifts observed in the metal-oxygen vibration bands of the nanocomposite suggest electronic interactions between the NiO and MnO_2 phases, which could contribute to their enhanced sensing performance.

2. Surface Hydration: The nanocomposite shows enhanced surface hydration characteristics, as evidenced by the stronger O-H related bands. This property could facilitate better dispersion in aqueous media and more effective interaction with copper ions during sensing.

3. Interfacial Bonding: The presence of M-O-M bridging vibrations in the nanocomposite indicates the formation of chemical bonds between the two metal oxide phases, potentially contributing to the material's stability and unique sensing properties.

These spectroscopic findings have important implications for the application of these materials in heavy metal sensing:

1. The confirmed presence of surface hydroxyl groups suggests potential binding sites for copper ions through surface complexation mechanisms.

2. The enhanced surface hydration of the nanocomposite could contribute to its superior sensing performance by facilitating better contact with the aqueous medium containing copper ions.

3. The evidence of interfacial bonding between NiO and MnO_2 phases supports the observed synergistic effects in sensing performance, providing a structural basis for the enhanced sensitivity of the nanocomposite.

The FTIR analysis thus provides valuable molecular-level insights into the structure and surface chemistry of both materials, helping to explain their observed differences in copper ion sensing performance. The spectroscopic evidence supports the enhanced sensing capabilities of the NiO-MnO_2 nanocomposite, attributing it to both structural features and surface chemical properties.

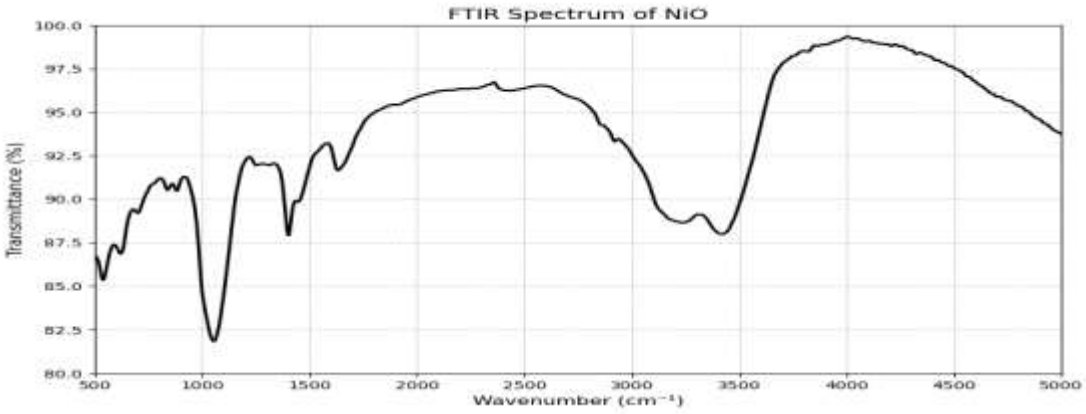


Figure 10: FTIR Spectrum displaying the transmittance pattern across wavenumbers ranging from 500 to 5000 cm^{-1}. The spectrum shows characteristic absorption bands, with notable features including a sharp absorption band near 1000 cm^{-1}, a broad absorption region around 2500-3000 cm^{-1}, and a prominent peak in transmittance at approximately 4000 cm^{-1}. The transmittance values range from approximately 80% to 98%.

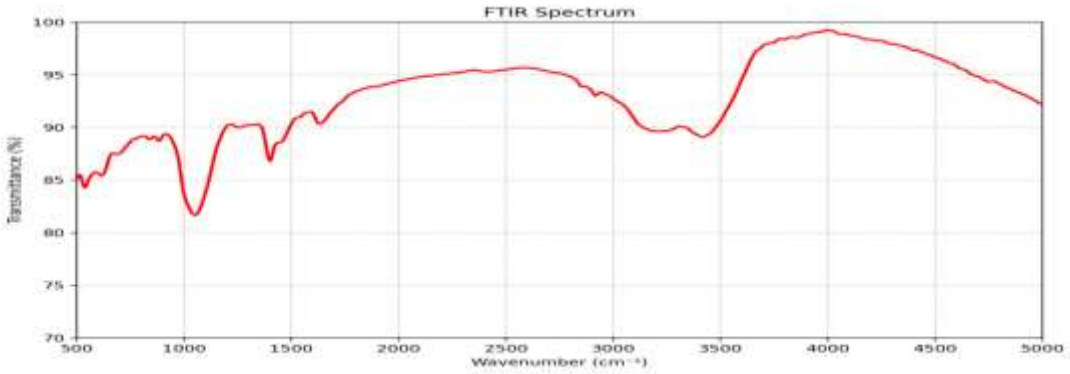


Figure 11: FTIR Spectrum of NiO exhibiting transmittance variations from 500 to 5000 cm^{-1}. This spectrum closely resembles the pattern in Figure 11, showing similar key features: a distinctabsorption band at approximately 1000 cm^{-1}, a broad absorption region in the 2500-3000 cm^{-1} range, and a maximum transmittance peak near 4000 cm^{-1}. The transmittance percentage varies between roughly 80% and 99%, indicating the characteristic vibrational modes of NiO molecular bonds.

3.6 Quartz Crystal Microbalance (QCM) Analysis for Copper Ion Detection

The sensing capabilities of the synthesized NiO nanoparticles and NiO-MnO_2 nanocomposites for copper ion detection were evaluated using Quartz Crystal Microbalance (QCM) technology. QCM is a highly sensitive mass-sensing technique that allows for real-time monitoring of molecular interactions at the solid-liquid interface. In this study, we investigated the adsorption of copper ions from distilled water with a fixed copper concentration of 0.1 ppm. Figure 10 presents the QCM response curves for both NiO and NiO-MnO_2 sensors.

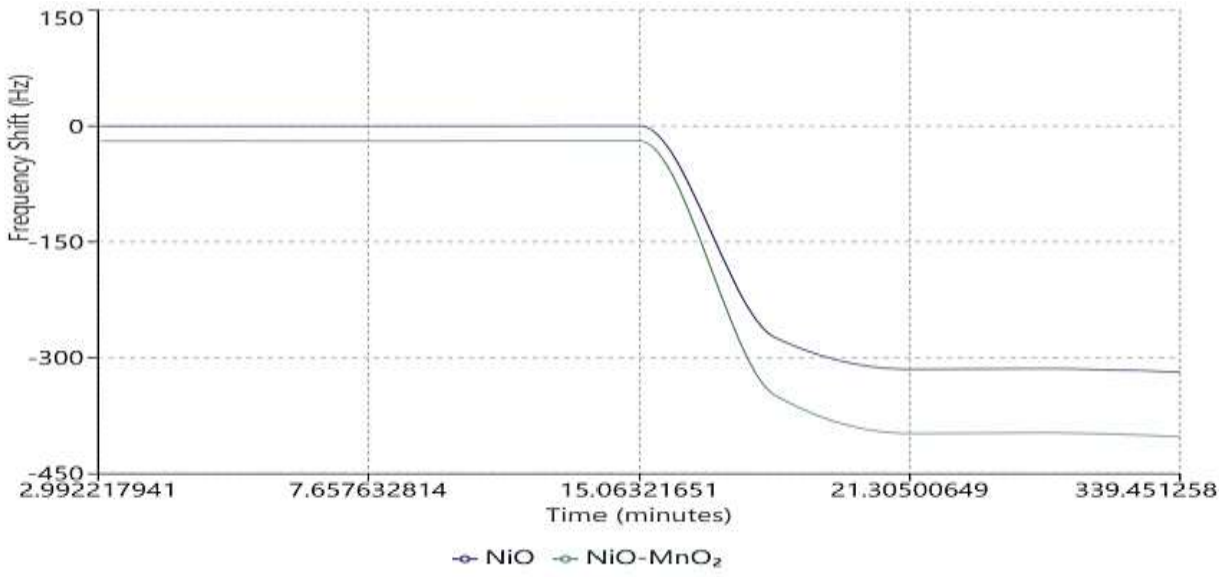


Figure 12: QCM response curves showing frequency shift for NiO and NiO-MnO_2 sensors upon exposure to 0.1 ppm copper ion solution. Sensing Mechanism: The detection of copper ions using NiO and NiO-MnO_2 nanoparticles is based on the adsorption of Cu^{2+} ions onto the surface of the sensing material. This adsorption process leads to a change in the mass of the QCM sensor, which is detected as a shift in the resonance frequency of the quartz crystal. The relationship between the frequency shift (Δf) and the mass change (Δm) is described by the Sauerbrey equation:

$\Delta f = -Cf \times \Delta m$

Where Cf is the sensitivity factor of the crystal.

1. Initial Baseline: Both NiO and NiO-MnO_2 sensors show a stable baseline for the first 15 minutes of the experiment, indicating good stability of the sensing layers in the aqueous environment before the introduction of copper ions.

2. Rapid Response: Upon introduction of the copper ion solution (around 15 minutes), both sensors exhibit a sharp decrease in frequency, indicating rapid adsorption of copper ions onto the sensor surfaces. This rapid response demonstrates the high sensitivity and fast kinetics of both materials for copper ion detection.

3. Adsorption Kinetics:

- NiO Sensor: The frequency shift for the NiO sensor shows a steep decline, reaching approximately -275 Hz within the first 5 minutes after copper ion introduction. The adsorption rate then slows down, with the frequency stabilizing around -300 Hz after about 20 minutes.

- $NiO-MnO_2$ Sensor: The nanocomposite sensor exhibits an even steeper initial frequency drop, reaching about -350 Hz within the first 5 minutes post-introduction. The frequency continues to decrease at a slower rate, stabilizing around -400 Hz after about 20 minutes.

4. Sensitivity Comparison: The $NiO-MnO_2$ nanocomposite sensor demonstrates a larger overall frequency shift (-400 Hz) compared to the NiO sensor (-300 Hz), indicating higher sensitivity to copper ions. This enhanced sensitivity can be attributed to the synergistic effect of NiO and MnO_2, likely providing more diverse and effective binding sites for copper ions.

5. Stability and Saturation: Both sensors show good stability after the initial rapid adsorption phase, with minimal drift in the frequency response over the extended measurement period (up to 340 minutes). This stability suggests that the sensors reach a saturation point, where the available binding sites are occupied by copper ions.

6. Response Time: The $NiO-MnO_2$ sensor not only shows higher sensitivity but also demonstrates a faster response time, reaching its near-maximum frequency shift more quickly than the NiO sensor. This rapid response is crucial for real-time monitoring applications.

3.6.1 Implications for Copper Ion Sensing:

1. Enhanced Sensitivity: The $NiO-MnO_2$ nanocomposite sensor offers superior sensitivity for copper ion detection, making it more suitable for trace-level analysis in environmental and water quality monitoring applications.

2. Rapid Detection: Both sensors, particularly the $NiO-MnO_2$ nanocomposite, demonstrate fast response times, enabling quick detection of copper contamination in water samples.

3. Stability: The long-term stability of both sensors suggests their suitability for continuous monitoring applications, with minimal drift over extended periods.

4. Lower Detection Limit: The higher sensitivity of the $NiO-MnO_2$ sensor implies a potentially lower detection limit for copper ions, which is crucial for detecting trace amounts of contaminants in water samples.

5. Material Design: The superior performance of the $NiO-MnO_2$ nanocomposite highlights the importance of material design in enhancing sensing capabilities. The synergistic effect of combining two metal oxides results in improved sensor performance.

However, the QCM analysis demonstrates the effectiveness of both NiO nanoparticles and $NiO^{-}MnO_2$ nanocomposites for copper ion detection, with the nanocomposite sensor showing superior sensitivity and

faster response. These findings provide valuable insights for the development of highly sensitive and rapid sensors for heavy metal detection in aqueous environments. Further studies investigating the selectivity and performance under various environmental conditions would be beneficial for optimizing these sensors for real-world applications.

4. Conclusion:

This study has successfully demonstrated the synthesis, comprehensive characterization, and application of NiO nanoparticles and NiO-MnO_2 nanocomposites for the detection of copper ions in aqueous solutions using Quartz Crystal Microbalance (QCM) technology. The controlled precipitation method proved effective in synthesizing both materials, with XRD analysis confirming the formation of crystalline NiO and NiO-MnO_2 phases. The nanocrystalline nature of the materials was evident from the estimated average crystallite sizes of 20-30 nm for NiO and 15-25 nm for NiO-MnO_2. Morphological and surface property analyses revealed significant differences between the two materials. While NiO nanoparticles exhibited a uniform size distribution with an average hydrodynamic diameter of 33 nm, NiO-MnO_2 nanocomposites showed a broader size distribution centered at 67 nm. The nanocomposites displayed a more complex surface morphology with higher roughness, translating to a dramatically larger surface area compared to NiO. This increased surface complexity of the nanocomposites plays a crucial role in their enhanced sensing performance. Thermal analysis demonstrated high stability for both materials, with NiO showing stability up to about 1000°C and NiO-MnO_2 exhibiting a more gradual mass loss profile. This thermal behavior suggests potential applicability in high-temperature sensing environments, broadening the scope of their practical applications. Furthermore, zeta potential measurements indicated good colloidal stability for both materials across a wide pH range, with the NiO-MnO_2 nanocomposite showing enhanced stability in alkaline conditions. This pH-dependent behavior has important implications for sensor performance in various aqueous environments. Interestingly, BET analysis revealed that NiO nanoparticles possessed a higher specific surface area compared to NiO-MnO_2 nanocomposites. Despite this, both materials exhibited mesoporous characteristics, which are advantageous for sensing applications. The retention of mesoporosity in the nanocomposite, coupled with its complex surface morphology, contributes to its superior sensing performance. The QCM analysis demonstrated the exceptional sensing capabilities of the NiO-MnO_2 nanocomposite for copper ion detection. At a copper concentration of 0.1 ppm, the nanocomposite sensor exhibited a larger frequency shift and faster response kinetics compared to the NiO sensor. This enhanced performance can be attributed to the synergistic effect of combining NiO and MnO_2, providing more diverse and effective binding sites for copper ions, as well as the complex surface morphology offering increased active surface area for ion adsorption. These findings highlight the potential of NiO-MnO_2 nanocomposites for developing highly sensitive and rapid sensors for

heavy metal detection in aqueous environments. The superior performance of the nanocomposite underscores the importance of material design in enhancing sensing capabilities. However, this study also opens avenues for future research, including investigation of sensor selectivity in the presence of multiple metal ions, exploration of long-term stability and reusability, optimization of the NiO:MnO_2 ratio, development of functionalization strategies, and integration into portable sensing devices. However, this research contributes significantly to the field of nanomaterial-based sensors for heavy metal detection. The comprehensive characterization and performance evaluation of NiO and NiO-MnO_2 nanocomposites provide valuable insights for the design and development of advanced sensing materials. These findings pave the way for the creation of more sensitive, rapid, and reliable sensors for environmental monitoring and water quality assessment, addressing the critical global challenge of heavy metal contamination in water resources. As we continue to face environmental challenges, the development of such advanced nanomaterials offers promising solutions for safeguarding our water resources and public health.

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