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# Synthesis and Properties of New Metal Complexes Containing Heterocyclic Moieties and Investigation of the Role of the Metal in Carbon Dioxide Gas Capture

## ABSTRACT

*The continuous release of carbon dioxide ($CO_2$) into the atmosphere will inevitably lead to greater environmental damage. The capture and storage of $CO_2$ is one strategy to mitigate the harm associated with its high concentrations in the atmosphere. The design and synthesis of new materials to act as storage media for $CO_2$ is currently an important challenge for researchers. In this regard, the investigation into the synthesis of new organometallic materials and their potential as $CO_2$ storage media is reported. Therefore, the current work aimed to produce new materials using a simple procedure and investigate their properties, including factors affecting their $CO_2$ adsorption. Four metal complexes containing heterocyclic units were synthesized using a simple method, and their structures were confirmed using several techniques. The surface morphology of the materials was inspected by microscopy. The metal complexes exhibited tunable particle sizes with diameters that ranged from 16.77 to 97.62 nm and a Brunauer–Emmett–Teller surface area of 1.20–4.01 $m^2$/g. The materials can capture $CO_2$ at 323 K and 40 bars, with the manganese-containing complex showing the highest $CO_2$ storage capacity (13.1 $cm^3$/gm).*

*Keywords: synthesis, metal complexes, carbon dioxide capture, surface area, surface morphology*

## 1. INTRODUCTION

The level of carbon dioxide ($CO_2$) in the atmosphere is on an upward curve correlated with the growing demand for energy and resources (e.g., power stations and the pharmaceutical industry) and the associated high demand for fossil fuels [1]. Currently, the concentration of $CO_2$ in the atmosphere is over 50% higher than the level at the end of the 19th century [2]. The increase in $CO_2$ levels is linked to rises in the surface temperature of the Earth, sea level, and the acidity of ocean water, for example, leading to serious environmental and socioeconomic problems [3–6]. It is challenging to reduce $CO_2$ emissions into the atmosphere rapidly due to the continuous high demand for fossil fuels, and so a multifaceted strategy is needed to control the levels. One approach is to find reliable alternative sources of energy to replace the use of fossil fuels, and, indeed, much attention has been paid to renewable sources such as wind, solar, nuclear, and biomass as a strategy to reduce $CO_2$ emissions into the atmosphere [7, 8]. However, renewable sources currently fall far short of the capacity required to meet the global demand for energy and generally cannot compete with fossil fuels in terms of cost [9].

Another way to address the unsustainable levels of the gas in the atmosphere is $CO_2$ capture. For this to be successful, the design and use of new adsorbent materials to capture $CO_2$ requires exploration [10–13]. Several processes have been used

to capture $CO_2$ from the atmosphere [14–20]. For example, amines (e.g., ethanolamine and ammonia) have been employed to remove $CO_2$ from natural gas [21]. However, there are associated risks with the use of amines since they are volatile and hazardous. The process of $CO_2$ capture often involves the separation of the gas and its adsorption at high pressure [22]. Thus, many new porous materials have been tested as storage media [23, 24]. Porous materials designed for $CO_2$ capture should meet certain requirements; they should be stable, polar, have large enough surface area and pore size, be easy to synthesize, be cheap to produce, have a long shelf life, be recyclable, and not pose a danger to the environment [25–28]. Examples of materials that have been tested as $CO_2$ adsorbents include metal-organic frameworks, carbon-based materials, zeolites, cross-linked polymers, and inorganic materials [29–32]. Metal oxides have been used with metal carbonates as materials in a chemical looping process, but their adsorption capacity is limited [33,34]. Additionally, carbon materials (e.g., activated carbons) are not suitable for flue gases due to their hydrophilic properties and poor selectivity [35–39]. Porous materials, including organic polymers, are used as $CO_2$ adsorbents due to their low heat of adsorption and high surface area [40–42]. However, the synthetic procedures for these materials are not environmentally friendly [43].

Organometallic compounds (e.g., metal–organic frameworks) have been used in many applications, such as storage and separation media, catalysts, and sensors [44]. However, there are problems associated with the use of organometallics that need to be addressed to enable their commercialization. The most common issues are their toxicity, regeneration, and reuse. Some success has been reported with organometallic regeneration and reuse [44]. However, further research is still needed to improve materials efficiency, cost of production, cleanliness, and simplicity of the process. Recently, success has been achieved in capturing $CO_2$ using polymeric materials [45–47], heterocycles [48,49], and organometallics [50–53]. Heterocycles such as thiophenes have useful applications in medicine, optoelectronic conductive devices, catalysis, and the production of dyes, plasticizers, and resins [54,55]. The current work explores the synthesis of new complexes containing different metals and heterocyclic units, such as thiophene. While recognizing that they do not show the levels required for application, an additional aim is to use the compounds as a model to explore how the metal identity influences the $CO_2$ absorption in such materials.

## EXPERIMENTAL

### General

Chemicals, solvents, and reagents (analytical grades) were obtained from Merck (Gillingham, UK) and used as received. Melting points were measured on a Gallenkamp melting point apparatus. The FTIR spectra (KBr disks) were recorded on a Shimadzu IR Prestige-21 single-beam Fourier Transform Infrared spectrophotometer (400–4000 cm⁻¹). A Shimadzu-1800 double-beam UV-Vis spectroscopy was used to record the electronic absorption spectra (200–1100 nm; $10^{-3}$ M in DMF). A Bruker BM6 magnetic monitor was used to measure the magnetic susceptibility of complexes. Molar conductance ($10^{-3}$ M in DMF) was calculated using a WTW ProfiLine Oxi3205 conventional portable meter. The NMR spectra were recorded on Bruker DRX300N-MR spectrometry in DMSO-$d_6$. The electron impact (70 eV) mass spectrum of the ligand was conducted on a Shimadzu GCMS-QP2010-plus mass spectrometer that was equipped with a DB-5MS column (30 m × 0.25 mm I.D. × 0.1 μm). The injector temperature was kept at 260 °C, and the oven temperature started at 50 °C for 2 minutes, increased to 210 ° C (3 °C/min), and then to 270 °C (10 °C/min). The carrier gas used was helium, with a pressure of 69 kPa. The complexes were dried for 4 hours at 60 °C using a vacuum oven to dry samples. The specific surface area of complexes was measured using the Brunauer-Emmett-Teller (BET) method. Pore sizes and volumes were measured via the Barrett–Joyner–Halenda (BJH) method. The $CO_2$ uptake measurements (three times) were performed on an H-sorb 2600 high-pressure volumetric adsorption instrument. The complex (1 g) was first degassed for 1 h at 50 °C in a vacuum oven to remove any traces of either water or solvent that are trapped within the pores of the complexes. Based on our previous work, the pressure and the temperature were set at 40 bar and 323 K, respectively [48–50]. The gas uptake experiment was then replicated three times under identical conditions to determine the optimum pressure. A ZEISS Sigma VP (10 kV) was used to record the field emission scanning electron microscopy (FESEM) images. The energy dispersive x-ray (EDX) and mapping tests for the complexes were performed on Oxford Instruments equipment.

A Veeco instrument was used to record the atomic force microscopy (AFM) images.

Synthesis of 3-hydrazone-1,3-dihydroindole-2-one (2)

A solution of 1$H$-indole-2,3-dione (**1**; 1.00 g, 6.8 mmol) in MeOH (10 mL) was added dropwise to a stirred solution of hydrazine monohydrate (0.34 g,6.8 mmol) in MeOH (10 mL). The mixture was refluxed for 6 hours, and the yellow solid formed was filtered, washed with MeOH, and dried at 50 °C to give pure **2** (Scheme 1) in 80% yield, M.P. 187–188 °C. FTIR (cm$^{-1}$): 3369, 3197, 1689, 1560, 1461.
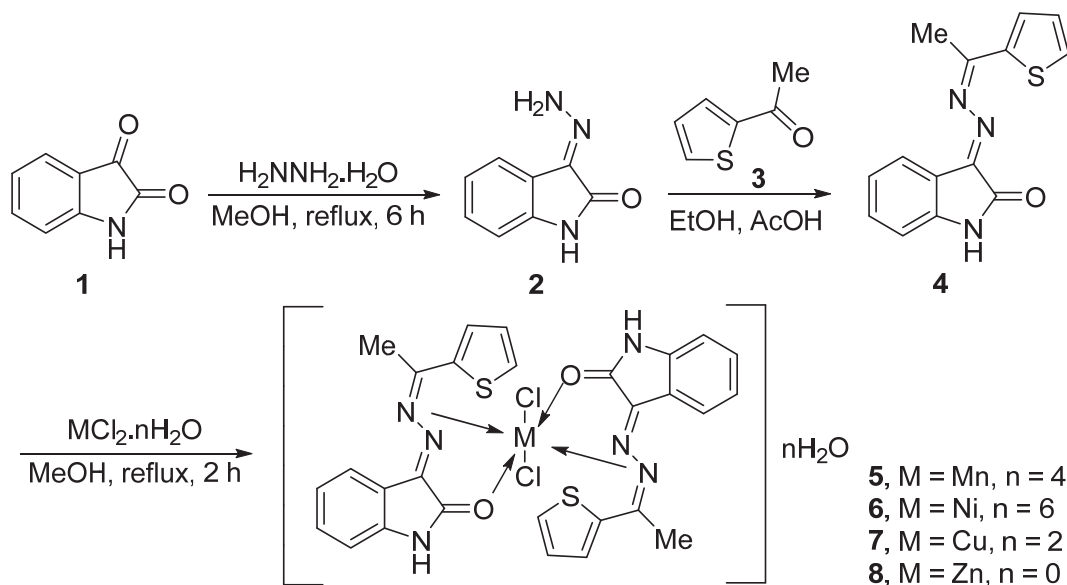


Scheme 1. Synthesis of metal complexes 5–8.

**5**, M = Mn, n = 4
**6**, M = Ni, n = 6
**7**, M = Cu, n = 2
**8**, M = Zn, n = 0

Synthesis of 3-[(1-Thiophen-2-yl-ethylidene) hydrazone]-1,3-dihydroindol-2-one (4)

A solution of **2** (1.00 g, 6.2 mmol) in EtOH (10 mL) was added dropwise to a stirred solution of 1-(thiophen-2-yl)ethan-1-one (**3**; 0.78 g, 6.2 mmol) in EtOH (10 mL) containing glacial AcOH (0.5 mL). The mixture was refluxed for 4 hours, and the orange solid formed was collected by filtration, washed with EtOH, recrystallized using a mixture of MeOH and EtOH (1:1 by volume), and dried at 50 °C to give pure **4** (Scheme 1) in 75% yield, M.P. 234–236 °C. FTIR (cm$^{-1}$): 3372, 3201, 1688, 1612, 1465. UV (nm): 249, 313. $^1$H NMR (400 MHz; DMSO-d$_6$): 2.51 (s, 3H, Me), 6.86 (t, 8 Hz, 1H, Ar), 6.97 (t, 8 Hz, 1H, Ar), 7.16 (d, 8 Hz, 1H, Ar), 7.22 (d, 8 Hz, 1H, Ar), 8.56, (d, 5 Hz, 1H, thiophene), 8.75–8.78 (m, 2H, thiophene), 10.70 (s, 1H, NH). $^{13}$C NMR (100 MHz; DMSO-d$_6$): 39.3, 110.1, 117.9, 121.8, 122.7, 123.2, 126.7, 127.5, 129.0, 129.2, 139.1, 141.0, 163.3, 166.4. EI-MS (m/z, %): 270 (M$^+$, 100), 197 (15), 160 (55).

Synthesis of metal complexes 5–8

A mixture of metal chloride (1.8 mmol) and **4** (0.97 g, 3.6 mmol) in MeOH (10 mL) was refluxed for 2 h. The colored complexes obtained were collected by filtration, washed with MeOH, and dried to give **5–8** (Scheme 1) in good yields (Table 1). The solids obtained are highly insoluble in most solvents, and therefore, no crystallization process was performed.

Table 1. Color, melting point, yield, and elemental analysis of metal complexes 5–8

| Complex | Metal | Color | M.P (°C) | Yield (%) | Analysis (%); Found (Calculated) | | |
|---|---|---|---|---|---|---|---|
| | | | | | C | H | M |
| **5** | Mn | Reddish brown | 213–215 | 66 | 45.56 (45.66) | 4.09 (4.11) | 7.29 (7.46) |
| **6** | Ni | Yellow | 200–203 | 65 | 43.20 (43.32) | 4.37 (4.41) | 7.37 (7.56) |
| **7** | Cu | Dark brown | 177–179 | 60 | 47.31 (47.43) | 3.65 (3.70) | 8.87 (8.96) |
| **8** | Zn | Light yellow | 218–221 | 75 | 49.74 (49.83) | 3.34 (3.29) | 9.52 (9.69) |

*Results and Discussion*

Synthesis of metal complexes

Compound **4** was obtained in 75% yield, as shown in Scheme 1. The mass spectrum of **4** showed a pseudo molecular ion peak (MH$^+$) at m/z = 270. Its $^1$H NMR spectrum showed the presence of the methyl and NH protons as singlet signals at 2.51 and 10.70 ppm, respectively. The $^{13}$C NMR spectrum of **4** showed the carbonyl carbon at 166.4 ppm. The reaction of **4** with metal chlorides leads to the formation of metal complexes **5–8** (Scheme 1) in rea-

sonable yields (Table 1). The structures of **5–8** were confirmed by the FTIR (Table 2), electronic (Table 3), and NMR (Table 4) spectral data.

The coordination took place between the metal and oxygen rather than the sulfur. The lone pairs of electrons of the sulfur atom are involved in thiophene aromaticity and are not available for coordination. In addition, the high melting points of the synthesized complexes indicate their high stability in the air. The complexes decomposed at temperatures higher than 200 °C.

*Table 2. Selected FTIR absorption bands (cm–1) of 5–8*

| Complex | Metal | OH | NH | C=O | C=N | M–N | M–O | M–Cl |
|---|---|---|---|---|---|---|---|---|
| **5** | Mn | 3360 | 3161 | 1762 | 1658 | 524 | 501 | 289 |
| **6** | Ni | 3360 | 3163 | 1730 | 1658 | 524 | 501 | 289 |
| **7** | Cu | 3450 | 3196 | 1732 | 1618 | 480 | 457 | 277 |
| **8** | Zn | — | 3161 | 1691 | 1652 | 524 | 501 | 289 |

FTIR spectroscopy of 5–8.

The FTIR spectra of **5–8** showed the presence of absorption bands corresponding to the C=N bonds in thiophene that appeared in the regions of 1618–1658 cm$^{-1}$ (Table 2). They also showed absorption bands that are corresponding to the carbonyl group at 1691–1730 cm$^{-1}$. There was a shift in the carbonyl group position compared with compound **4,** indicating that coordination has taken place. Indeed, new absorption bands corresponding to the M–N, M–O, and M–Cl bonds appeared in the regions of 480–534, 457–501, and 277–289 cm$^{-1}$, respectively [56, 57].

Molar conductivity, magnetic moment, and UV spectroscopy of 5–8

The electronic spectra (200–1100 nm) of **5–8** were measured in DMF at 20 °C. The UV spectrum of the Mn complex showed bands at 267 and 317 nm corresponding to the π–π* and n–π* transitions, respectively (Table 3). The spectrum does not show d–d transitions except a band at 395 nm, which was attributed to charge transfer (CT) since the multiplicities in the ground and excited states are different. This result is consistent with the magnetic moment

($\mu_{eff}$ = 6.26 BM), indicating an octahedral geometry [58,59].

The UV spectrum of the Ni complex showed bands at 260 and 312 nm that were assigned to the π–π* and n–π* transitions, respectively. In addition, three bands in the visible region at 400, 500, and 810 nm were attributed to the spin-allowed transitions $^3A_2g(F) \rightarrow {}^3T_1g(P)$, $^3A_2g(F) \rightarrow {}^3T_1g(F)$, and $^3A_2g(F) \rightarrow {}^3T_2g(F)$, respectively.

The magnetic moment (2.68 BM) of the Ni complex was in the normal range for octahedral arrangement [58, 59]. For the Cu complex, three absorption bands appearing at 211, 268, and 380 nm were attributed to π–π*, n–π* transitions, and the CT, respectively. The broad band centered at 635 nm was attributed to $^2Eg \rightarrow {}^2T_2g$ transitions. The d–d bands were strongly indicative of the distorted octahedral geometry, which was confirmed by magnetic susceptibility (1.89 BM) [60].

The electronic spectrum of the Zn complex showed a charge transfer band at 407 nm as well as two bands at 275 and 329 nm due to π–π* and n–π* transitions, respectively [58]. The molar conductivity recorded indicates that complexes **5–8** are non-electrolytes.

*Table 3. Molar conductivity, magnetic moment, and electronic spectral data of metal complexes 5–8*

| Complex | Metal | Conductivity (Ohm$^1$ cm$^2$ mol$^{-1}$) | μeff (BM) | Band (nm) | Assignment |
|---|---|---|---|---|---|
| **5** | Mn | 10 | 6.26 | 267 | π–π* |
| | | | | 317 | n–π* |
| | | | | 395 | CT |
| **6** | Ni | 20 | 3.68 | 260 | π–π* |
| | | | | 312 | n–π* |
| | | | | 400 | $^3A_2g(F)\rightarrow{}^3T_1g(P)$ |
| | | | | 500 | $^3A_2g(F)\rightarrow{}^3T_1g(F)$ |
| | | | | 800 | $^3A_2g(F)\rightarrow{}^3T_2g(F)$ |
| 7 | Cu | 32 | 1.89 | 211 | π–π* |
| | | | | 268 | n–π* |
| | | | | 280 | CT |
| | | | | 635 | $^2Eg\rightarrow{}^2T_2g$ |
| **8** | Zn | 3 | Diamagnetic | 275 | π–π* |
| | | | | 329 | n–π* |
| | | | | 407 | CT |

### NMR Spectroscopy of 5–8

The structures of **5–8** were confirmed by the NMR spectral data (Table 4). It was not possible to record the NMR spectrum of the Mn complex using the standard technique at room temperature due to its poor solubility in a variety of deuterated solvents [58,61]. The $^1H$ NMR spectra of the metal complexes showed the methyl protons as singlets in the 2.18–2.38 ppm region. In addition, the NH protons appeared as singlets at the 10.01–10.70 ppm region. The carbonyl carbons appeared downfield (166.2–168.6 ppm) in the $^{13}C$ NMR spectra, while the methyl carbons appeared at high field (27.2–27.8 ppm). It should be noted that the mass spectra of the synthesized complexes **5–8** were very poor and provided no useful information, presumably due to their low volatility.

*Table 4. NMR spectral data of metal complexes 6–8*

| Complex | Metal | $^1H$ NMR | $^{13}C$ NMR |
|---|---|---|---|
| 6 | Ni | 2.38 (s, 3H, Me), 6.79 (t, 8 Hz, 1H, Ar), 6.83 (t, 8 Hz, 1H, Ar), 6.99 (d, 8 Hz, 1H, Ar), 7.19 (d, 8 Hz, 1H, Ar), 8.60, (d, 4 Hz, 1H, thiophene), 8.82–8.87 (m, 2H, thiophene), 10.70 (s, 1H, NH) | 27.3, 110.4, 117.8, 122.7, 126.7, 127.4, 129.2, 134.4. 135.4, 139.0, 144.5, 145.4, 163.2, 168.6 |
| 7 | Cu | 2.38 (s, 3H, Me), 6.72 (t, 8 Hz, 1H, Ar), 6.87 (t, 8 Hz, 1H, Ar), 7.14 (d, 8 Hz, 1H, Ar), 7.31 (d, 8 Hz, 1H, Ar), 8.65, (d, 4 Hz, 1H, thiophene), 8.72–8.77 (m, 2H, thiophene), 10.70 (s, 1H, NH) | 27.8, 110.0, 112.6, 118.2, 121.6, 123.2, 125.1, 129.7, 133.1, 138.8, 144.5, 151.1, 159.8, 166.2 |
| 8 | Zn | 2.18 (s, 3H, Me), 6.82 (t, 8 Hz, 1H, Ar), 6.82 (t, 8 Hz, 1H, Ar), 6.91 (d, 8 Hz, 1H, Ar), 7.13 (d, 8 Hz, 1H, Ar), 7.91, (d, 4 Hz, 1H, thiophene), 8.69–8.79 (m, 2H, thiophene), 10.01 (s, 1H, NH) | 27.2, 110.5, 117.9, 121.5, 122.7, 126.3, 127.5, 129.3, 134.3, 135.3, 139.1, 140.9, 163.3, 166.4 |

### EDX Mapping of 5–8

The elemental contents of **5–8** were confirmed using the energy-dispersive X-ray analysis (EDX). The EDX images provide a rough estimate of metal contents. However, it is not as precise as the results obtained from the elemental analysis. Figure 1 shows the EDX mapping graphs for the synthesized metal complexes, and they reveal all the elements within the structures of metal complexes. Figure 1 showed a lower percentage for Zn compared with those shown for appeared for the other metals. It should be noted that the structures and quantity of water for the metal complexes (Scheme 1) were suggested based on the elemental analysis (Table 1).
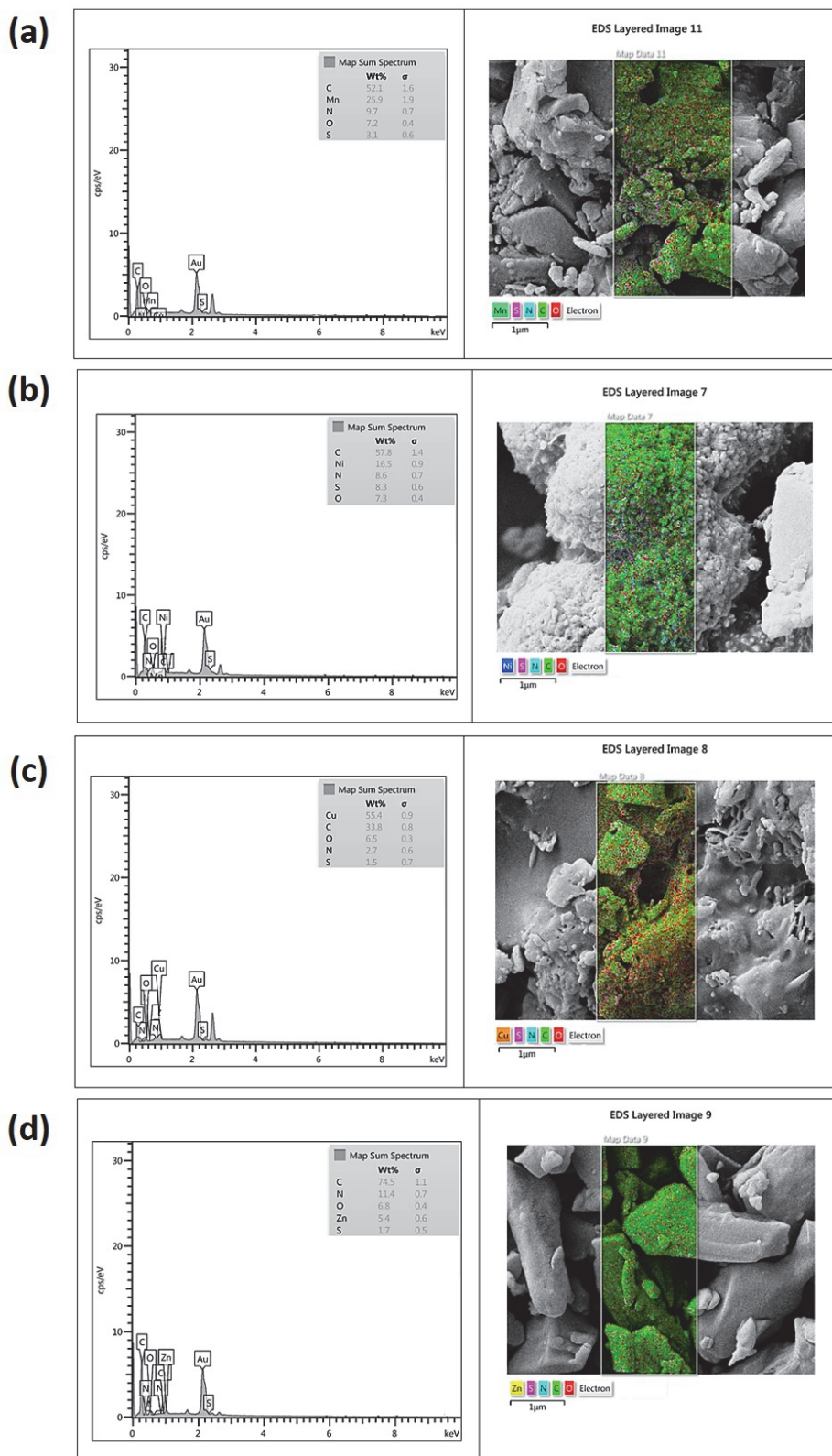
Figure 1. EDX mapping images of complexes containing (a) Mn, (b) Ni, (c) Cu, and (d) Zn.

Surface morphology of 5–8

The morphology, particle sizes, and porosity of metal complexes **5–8** were examined using various microscopy techniques. FESEM provided clear and undistorted images of the surface of complexes **5–8** (Figure 2). FESEM images indicate the uniformity, amorphous nature, and roughness of surfaces. The

roughness of the surface can influence the adsorption of $CO_2$. The increase in the roughness of materials leads to a greater surface area for gas adsorption [62]. In addition, the FESEM images showed the existence of a range of particle sizes. Complexes **5–8** have irregular pore sizes with particle diameters in the range of 37–189, 35–69, 31–263, and 26–241 nm for the complexes containing Mn, Ni, Cu, and Zn, respectively [63].
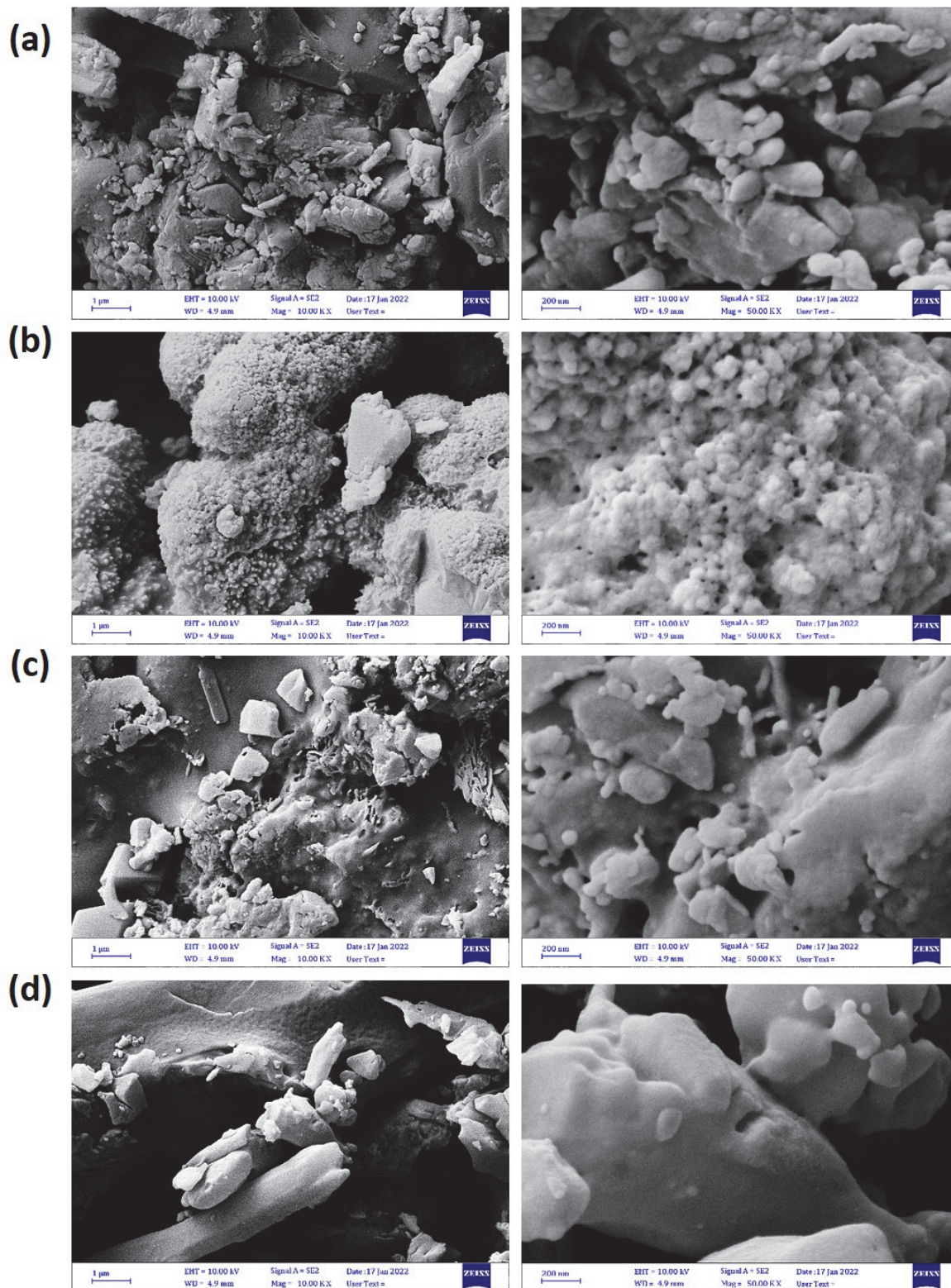


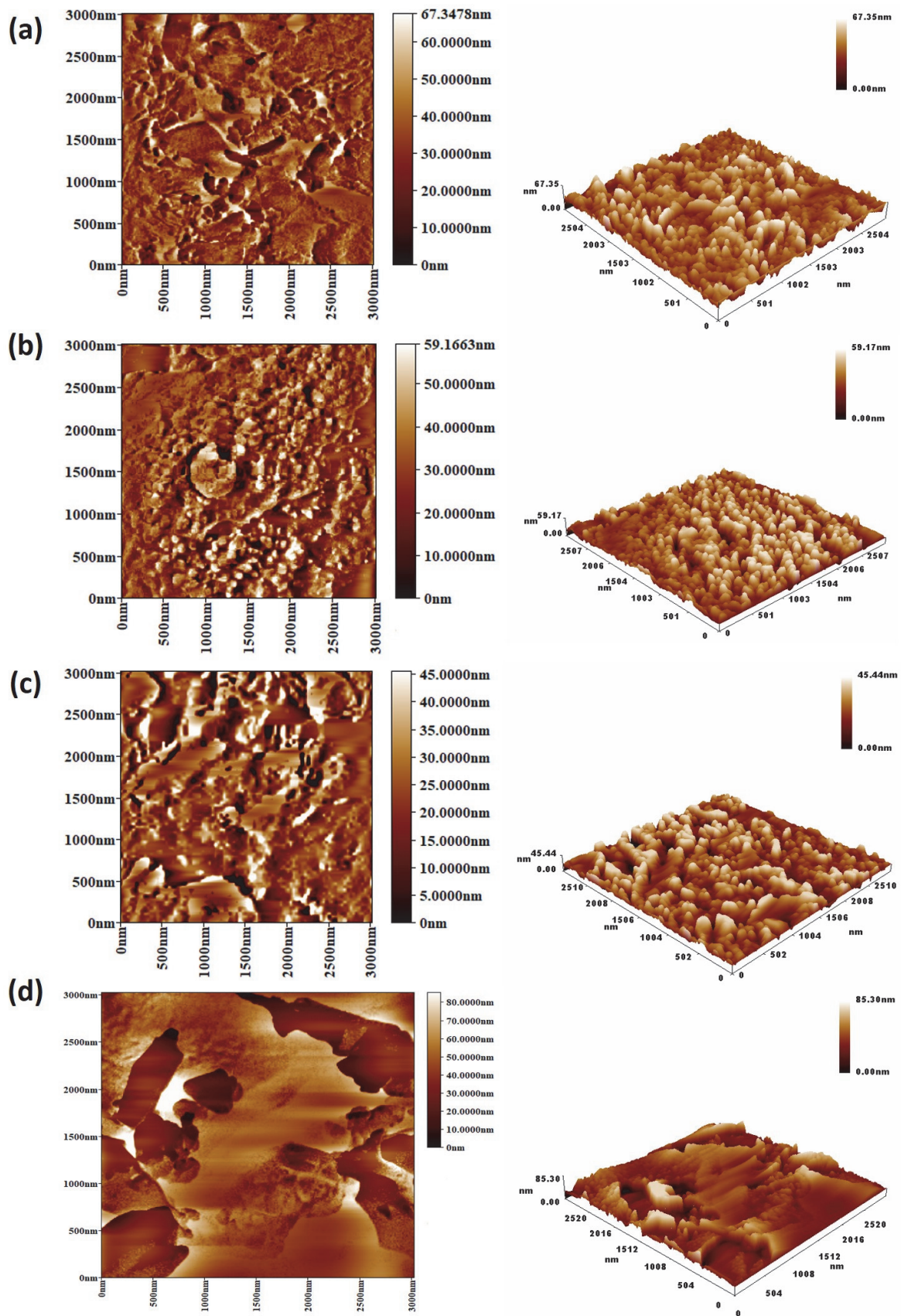*Figure 2. FESEM image of complexes containing (a) Mn, (b) Ni, (c) Cu, and (d) Zn.*

*Figure 3. AFM images of complexes containing (a) Mn, (b) Ni, (c) Cu, and (d) Zn.*

The AFM (two and three-dimensional) images provided information about the surface porosity and roughness (Figure 3). The AFM images indicated that the surface of the metal complexes is rough and highly porous. The roughness factor for the surface of metal complexes containing Mn, Ni, Cu, and Zn was 356, 175, 124, and 238, respectively. The particle sizes were 31.3–207.8, 33.5–117.5, 83.7–386.6, and 67.0–460.9 nm for complexes containing Mn, Ni, Cu, and Zn, respectively. It was clear that the particle sizes measured by the AFM in the Cu and Zn-based samples were more or less correlated with those obtained from the FESEM. However, the AFM provides much larger particle sizes in the Cu and Zn complex-es compared to those provided by the FESEM. It is not clear why this variation took place, but it could be that the particle sizes were just visually estimated from the FESEM.

The BET surface area of 5–8

The BET surface area determines the adsorption capacity of adsorbents, and additionally, the porosity provides information about their chemical and physical interactions with the adsorbed gas. Therefore, the nitrogen isotherms, along with the pore size distribution of complexes containing Mn, Ni, Cu, and Zn, were determined (Figure 4).
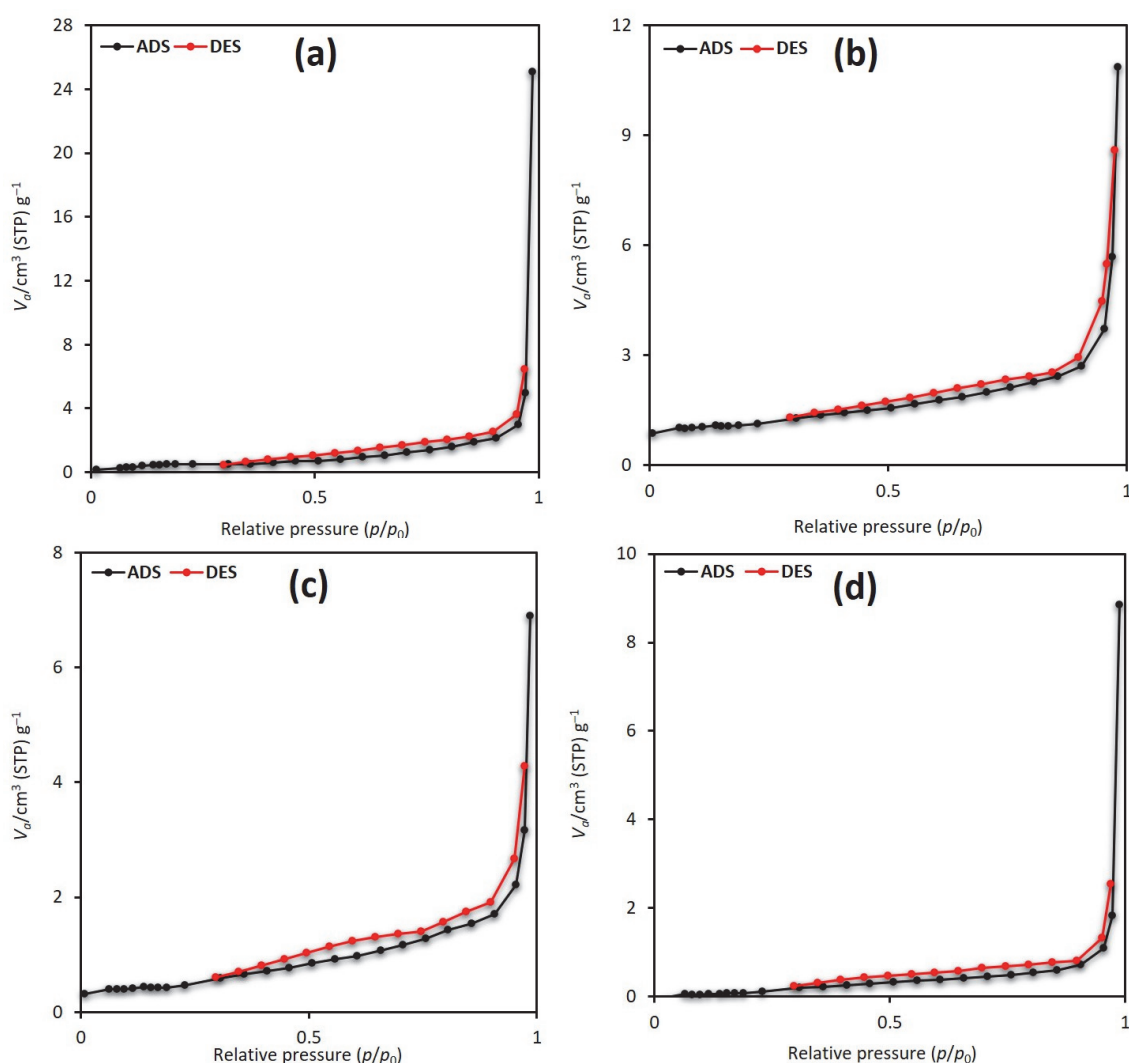


Figure 4. The N2 adsorption isotherms of complexes containing (a) Mn, (b) Ni, (c) Cu, and (d) Zn.

The interactions between the metal complexes and $CO_2$ were relatively weak [64]. The isotherms indicated that the structures of complexes are type IV mesoporous. No identifiable monolayer formation was found. The nitrogen adsorption isotherms ($P/P^o = 0.9$) were used to calculate the BET surface area and pore volume. The desorption data was used to estimate the average pore diameter (Table 5). The BET surface area of metal complexes was low (1.20–4.01 m²/g), and the pore volume and diameter were in the ranges

0.011–0.039 $cm^3$/g and 16.77–97.62 nm, respectively. The Mn complex has the highest BET surface area (4.01 $m^2$/g), pore volume (0.039 $cm^3$/g), and diameter (97.62 nm). It seems likely that interparticle sorption takes place at the high relative pressure used, leading to a steep increase in the uptake graphs.

*Table 5. Surface area and pore size distribution of metal complexes obtained by the N2 adsorption*

| Com-plex | Metal | $S_{BET}$ ($m^2$/g) | Pore volume ($cm^3$/g) | Average pore diameter (nm) |
|---|---|---|---|---|
| 5 | Mn | 4.01 | 0.039 | 97.62 |
| 6 | Ni | 1.20 | 0.011 | 16.77 |
| 7 | Cu | 1.59 | 0.014 | 26.30 |
| 8 | Zn | 1.62 | 0.017 | 45.77 |

The $CO_2$ uptake of 5–8

The pores' size and the type and strength of interactions between adsorbent and $CO_2$ (e.g., Van der Waal forces or hydrogen bonding) control $CO_2$ adsorption. High energy is required to adsorb $CO_2$ if the aperture size is small compared to the molecular size, as repulsive forces could dominate [65]. In addition, the effect of pores' volume of absorbent materials is important for gas uptake capacity [66], with materials with large pores volumes having the space to store more $CO_2$ [67]. The chosen conditions for the $CO_2$ uptake process were based on our previous related work [45–53]. The $CO_2$ uptake recorded at 323 K and 40 bars for the complexes containing Mn, Ni, Cu, and Zn were 13.1, 10.1, 4.3, and 10.8 $cm^3$/g, respectively (Figure 5). In terms of wt %, the $CO_2$ uptake was 2.6, 2.0, 0.9, and 2.1% for the Mn, Ni, Cu, and Zn complexes, respectively. The Mn complex led to the highest $CO_2$ uptake compared to the others. The Mn complex has the highest BET surface area and pore volume and diameter. In addition, the Mn atom has the lowest atomic radii compared with the other metals. Generally, the $CO_2$ uptake by complexes is reasonable since the complexes have a small surface area. This provides a sound starting point for the optimization of the chemical and physical properties to improve the interactions and capacity of the materials. We have not attempted to study the stability of the complexes. However, we believe they are highly stable since physisorption mainly takes place in the adsorption of $CO_2$. Therefore, the complexes should remain intact during and after the adsorption process.
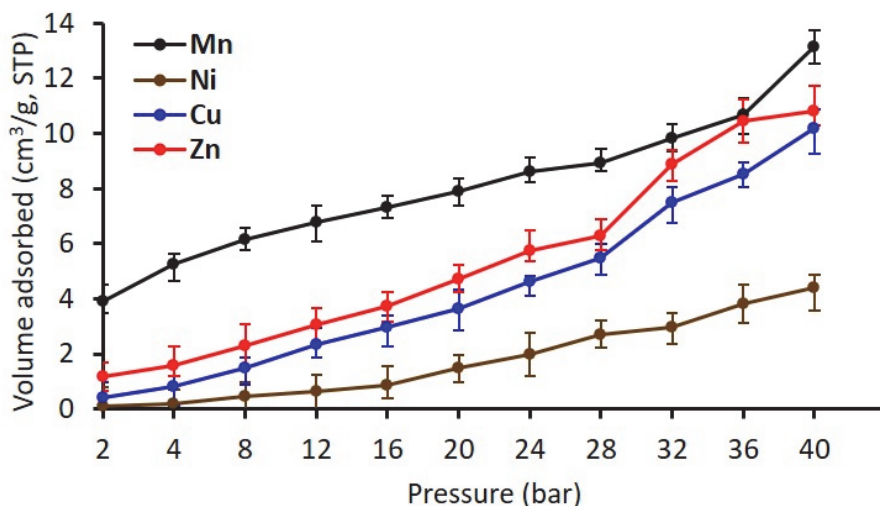


*Figure 5. The CO2 adsorption isotherms for the metal complexes.*

It is believed that the $CO_2$ adsorption mechanism is dependent on both the aromatic moieties, polarity of heteroatoms, and the Lewis acid center in the complexes used. We believe that physisorption is involved in $CO_2$ adsorption by the newly synthesized metal complexes. Physisorption plays an important role in the adsorption of gas due to the interaction between the polarized bonds within both the complexes and $CO_2$ [68]. Table 6 shows the $CO_2$ uptake (wt %) over various storage media that have been reported recently in comparison to those achieved using the currently synthesized metal complexes. Clearly, multiple factors are controlling the adsorbent's efficacy toward $CO_2$ uptake. The most common factors are the surface area, pores volume and diameter, and surface morphology.

*Table 6. Surface area and CO2 uptake of recently synthesized adsorbents*

| Adsorbent | $S_{BET}$ (m²/g) | $CO_2$ (wt) | T (K), P (bar) | Ref |
|---|---|---|---|---|
| Thiophene-metal complexes | 1.2–4.0 | 0.9–2.6 | 323, 40 | Current |
| Polyphosphates–1,4-diaminobenzene | 82.7–213.5 | 2.0–6.0 | 323, 40 | 45 |
| Polysilicates | 8.2–18.0 | 2.2–6.0 | 323, 40 | 46 |
| Polyphosphates–benzidine | 24.8–30.0 | 1.8–14.0 | 323, 50 | 47 |
| Trimethoprim Schiff bases | 4.2–20.3 | 6.0–10.3 | 313, 40 | 48 |
| Melamine Schiff bases | 5.2–11.6 | 6.1–10.0 | 323, 40 | 49 |
| Metal-fusidate complexes | 31.2–46.9 | 6.3–7.2 | 323, 50 | 50 |
| Valsartan-metal complexes | 16.0–22.8 | 4.8–6.8 | 323, 40 | 51 |
| Carvedilol metal complexes | 6.1–9.1 | 2.1–3.5 | 323, 40 | 52 |
| Telmisartan metal complexes | 32.4–130.4 | 3.3–7.1 | 323, 50 | 53 |

## Conclusions

Four complexes containing heterocyclic units and metals were synthesized, and their structures were confirmed spectroscopically. Their physical properties and surface morphology were assessed. The materials have a diameter ranging from 16.77 to 97.62 nm and a specific surface area in the range of 1.20–4.01 m²/g. The highest $CO_2$ storage capacity was achieved with the complex containing manganese, followed by the others in the order of zinc, copper, and nickel. The manganese complex has the largest surface area, pores' volume, and average diameter compared with the others. In addition, the manganese-containing complex has the highest surface roughness factor. The results obtained open gates for designing a better storage media for $CO_2$. However, the toxicity that might be associated with these materials, their regeneration, and reuse need to be assessed.

## Conflicts of Interest

The authors declare no conflict of interest requiring disclosure in this article.

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## IZVOD

## SINTEZA I SVOJSTVA NOVIH METALNIH KOMPLEKSA KOJI SADRŽE HETEROCIKLIČNE GRUPE I ISTRAŽIVANJE ULOGE METALA U HVATANJU GASA UGLJEN-DIOKSIDA

*Kontinuirano oslobađanje ugljen-dioksida ($CO_2$) u atmosferu će neizbežno dovesti do veće štete po životnu sredinu. Sakupljanje i skladištenje $CO_2$ je jedna od strategija za ublažavanje štete povezane sa visokim koncentracijama $CO_2$ u atmosferi. Dizajn i sinteza novih materijala koji će delovati kao medij za skladištenje $CO_2$ je trenutno važan izazov za istraživače. S tim u vezi, objavljeno je istraživanje sinteze novih organometalnih materijala i njihovog potencijala kao medija za skladištenje $CO_2$. Stoga je trenutni rad imao za cilj da proizvede nove materijale koristeći jednostavnu proceduru i istraži njihova svojstva, uključujući faktore koji utiču na njihovu adsorpciju $CO_2$. Četiri metalna kompleksa koji sadrže heterociklične jedinice sintetizovana su jednostavnom metodom, a njihove strukture su potvrđene korišćenjem nekoliko tehnika. Morfologija površine materijala je pregledana mikroskopski. Metalni kompleksi su pokazali podesive veličine čestica sa prečnicima koji su se kretali od 16,77 do 97,62 nm i Brunauer-Emmett-Teller-ovom površinom od 1,20-4,01 $m^2$/g. Materijali mogu uhvatiti $CO_2$ na 323 K i 40 bara, pri čemu kompleks koji sadrži mangan pokazuje najveći kapacitet skladištenja $CO_2$ (13,1 $cm^3$/gm).*

***Ključne reči:*** *sinteza, metalni kompleksi, hvatanje ugljen-dioksida, površina, morfologija površine*

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