



Synthesis, physical properties, and carbon dioxide uptake of new metal-sulfamethoxazole complexes

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ABSTRACT

Considerable research is currently being undertaken to reduce atmospheric CO₂ levels, and a promising approach is capturing and storing the gas using adsorbents. In this regard, the synthesis and investigation of the potential use of new materials as CO₂ storage media has attracted attention from both industry and academia. Metal-organic frameworks have a range of unique chemical and physical properties with many applications. Reported here is the synthesis of three new sulfamethoxazole-metal complexes and their use as models for investigation of the influence of the metal on their ability to absorb CO₂. A new Schiff base was first synthesized, in 89 % yield, from the condensation of sulfamethoxazole and 4-(dimethylamino)benzaldehyde under acidic conditions. The reaction of the Schiff base with metal (nickel, copper, and cobalt) chlorides in ethanol under reflux gave the corresponding sulfamethoxazole-metal complexes in 71–80 % yield. Several experiments were conducted to assess the uptake of CO₂ under different conditions. The complexes have low surface areas (1.36–5.82 m²/g) and average pore volume and diameters of 0.008–0.018 cm³/g and 2.17–4.08 nm, respectively. They showed some ability to adsorb CO₂ (323 K and 40 bars), and the storage capacity was 11.2–26.1 cm³/gm. The cobalt-containing complex had the highest CO₂ storage capability (26.1 cm³/g) due to its relatively high surface area (5.82 m²/g), pore volume (0.018 cm³/g), pore diameter (4.08 nm), and surface roughness (11.6) compared to the nickel and copper complexes.

1. Introduction

The growth in essential industries, such as the chemical and pharmaceutical sectors, adds to the increasingly unsustainable levels of harmful gases emitted into the atmosphere. For example, large quantities of fossil fuels are burned to meet the growing energy demand with a corresponding increase in CO₂ levels [1]. The current CO₂ level in the atmosphere is approximately 50 % higher than that recorded in the 19th century [2]. High levels of CO₂ are unfortunately linked to environmental and associated socioeconomic problems [3]. The ecological impact of large quantities of CO₂ includes raised temperature of the earth's surface with a decline in the level of ice in the north and south poles [4,5], leading to higher sea levels. An additional risk to marine life is increased ocean acidity.

The challenge is the moderation of CO₂ levels in the atmosphere, and a combination of measures can address this. One method is using

alternative energy sources, mainly green and renewable, to replace fossil fuels [6,7]. Indeed, using solar energy, wind power, nuclear power stations, and biomass is an attractive and clean route toward reducing CO₂ emissions [8]. However, these renewable sources cannot meet all the current energy needs and, in addition, are yet to offer an overwhelming financial advantage over fossil fuels [9]. An alternative measure is to use CO₂ storage media to limit release into the atmosphere [10].

The conception and generation of materials that can trap CO₂ is an active area of research [11–13]. Suitable materials should adsorb CO₂ selectively, be cheap to produce, safe for humans and the environment, and be chemically stable and recyclable. These attributes are associated with optimal physical properties, including surface area, pore volume and diameters, and rough surface [14]. Many materials have been explored as CO₂ adsorbents [15–21]. An example is using ammonia and ethanamine to extract CO₂ from natural gas [22]. However, amines are

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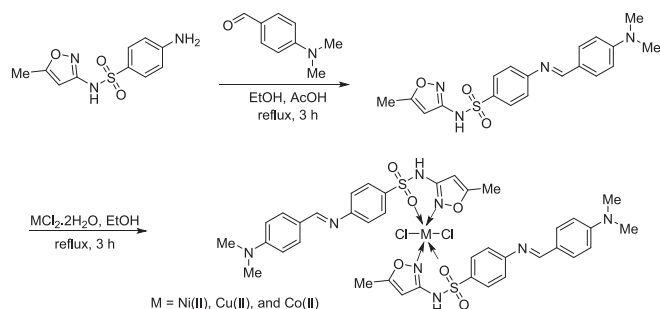
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Scheme 1. Synthesis of sulfamethoxazole-metal complexes.

volatile and toxic and pose a danger to the environment, limiting their applicability.

The capture of CO₂ generally involves separation followed by adsorption at high pressure over porous absorbent materials [23–26]. Porous materials have a variety of qualities that enable the capture of CO₂ [27–30]. The most common CO₂ adsorbents include zeolites, carbon-based materials, cross-linked polymers, and inorganic materials [31–34]. However, these materials can have disadvantages. For example, metal oxides have a limited adsorption capacity [35,36], while activated carbons have poor selectivity and cannot be used for flue gases [37–41]. Organic polymers can have high surface areas [42–44], but the procedures used for their synthesis are not green and generate waste harmful to humans and the environment [45]. In addition, severe environmental problems (e.g., toxicity, recyclability, and reuse) are associated with using many CO₂ adsorbents [46]. Recently, advances have been reported in using metal complexes to capture CO₂, but further studies are still needed to improve the process [47–50].

Sulfamethoxazole is a heterocycle rich in aromaticity and heteroatoms and has valuable applications [51]. For example, it has a broad spectrum of pharmacological activities and acts as a sulfonamide antibiotic [52]. It is solid with a relatively high melting point (169 °C), molecular weight (253.28 g/mol), and stability. Three new metal complexes containing nickel, copper, cobalt, and a sulfamethoxazole moiety are investigated in the current research. The complexes were then used as models to probe the influence of the identity of the metal on CO₂ uptake.

2. Experimental section

2.1. Materials and methods

Sulfamethoxazole (≥98 %), 4-(dimethylamino)benzaldehyde (98 %), hydrated nickel(II) chloride (NiCl₂; 98 %), copper(II) chloride (CuCl₂; 97 %), cobalt(II) chloride (CoCl₂; 97 %), and solvents were supplied by Merck (Gillingham, UK). A Vario EL III elemental analyzer performed an elemental analysis of sulfamethoxazole-metal complexes. Fourier Transform Infrared (FTIR) data were recorded on a Bruker Alpha spectrometer. ¹H and ¹³C NMR spectra (DMSO-d₆, TMS) were recorded using a Bruker DRX300NMR spectrometer. The CO₂ adsorption was determined using an H-sorb 2600 high-pressure volumetric adsorption instrument. The pressure was adjusted to 40 bar and the temperature to 323 K, based on previous related work [47–50]. The surface morphology of sulfamethoxazole-metal complexes was investigated using a Bruker

Table 1
The melting point (MP), yield, and elemental content of sulfamethoxazole-metal complexes.

Complex	MP (°C)	Yield (%)	Calculated (Found; %)				
			C	H	N	S	M
Ni	213–215	78	50.80 (51.01)	4.49 (4.55)	12.47 (12.32)	7.14 (7.06)	6.53 (6.45)
Cu	183–185	80	50.52 (50.65)	4.46 (4.56)	12.40 (12.22)	7.10 (7.01)	7.05 (6.96)
Co	196–198	71	50.78 (50.92)	4.49 (4.55)	12.47 (12.29)	7.13 (7.05)	6.56 (6.49)

XFlash® 6|10 detector for the energy dispersive x-ray (EDX) measurements, a ZEISS Sigma VP microscope for the field emission scanning electron microscopy (FESEM), and a Veeco instrument for the atomic force microscopy (AFM).

2.2. Synthesis of Schiff base

A mixture of 4-(dimethylamino)benzaldehyde (0.75 g, 5 mmol) and sulfamethoxazole (1.27 g, 5 mmol) in dry ethanol (EtOH, 10 mL) containing glacial acetic acid (AcOH, 0.1 mL) was heated under reflux for 3 h. The solid formed was separated by filtration, rinsed with EtOH, and dried to yield the corresponding Schiff base (Scheme 1) as an off-white powder in 89 % yield, MP: 176–178 °C. FTIR (KBr): 1155 (S = O_{sym}), 1363 (S = O_{asym}), 1596 (C = N), 1660 (C = N), 3282 (NH) cm⁻¹. ¹H NMR (500 MHz): 2.31 (s, 3H, Me), 3.04 (s, 6H, 2 OMe), 6.17 (s, 1H, Ar), 6.80 (d, *J* = 8.5 Hz, 2H, Ar), 7.68 (d, *J* = 8.5 Hz, 2H, Ar), 7.76 (d, *J* = 8.5 Hz, 2H, Ar), 7.84 (d, *J* = 8.5 Hz, 2H, Ar), 11.40 (s, exch., 1H, NH). ¹³C NMR (125 MHz): 12.5, 40.1, 95.9, 111.9, 122.0, 123.6, 125.0, 128.6, 131.3, 153.3, 154.7, 158.1, 162.8, 170.8. Anal. Calcd. For C₁₉H₂₀N₄O₃S (384.45): C, 59.36; H, 5.24; N, 14.57; S, 8.34. Found: C, 59.41; H, 5.28; N, 14.59; S, 8.35 %.

2.3. Synthesis of sulfamethoxazole-metal complexes

A mixture of Schiff base (1.92 g, 5 mmol) and metal chlorides (M(II) Cl₂; 10 mmol) was stirred in boiling EtOH (20 mL) for 3 h. The solids formed were separated by filtration, washed (boiling EtOH), and dried to yield the complexes (Scheme 1) as orange powders in high yields (Table 1). The melting points (°C), yields (%), and elemental content (%) of the synthesized metal complexes are shown in Table 1.

2.4. N₂ adsorption measurements

The N₂ adsorption isotherms of sulfamethoxazole-metal complexes were performed using a MicroActive TriStar II Plus Version 2.03 analyzer (Shanghai, China). The complexes were dried for 6 h at 50 °C before the measurements. N₂ adsorption isotherms (77 K) were used to determine the surface areas of materials using the Brunauer-Emmett-Teller (BET) method. The size and volume of pores were measured using the Barrett-Joyner-Halenda (BJH) method.

2.5. CO₂ uptake measurements

A sample of appropriate sulfamethoxazole-metal complex (1 g) was first degassed for 6 h at 50 °C under vacuum to remove trapped traces of either water or solvent within the pores of materials. A portion of the metal complex and a quantity of CO₂ were injected into the measuring tube till equilibrium was achieved. The system for the adsorption

Table 2
Selected FTIR absorption bands (ν, cm⁻¹) of sulfamethoxazole-metal complexes.

Complex	NH	C = N	C = N	S = O _{asym}	S = O _{sym}	M–N	M–O
Ni	3285	1650	1583	1367	1156	559	424
Cu	3322	1640	1582	1366	1156	563	425
Co	3368	1655	1590	1369	1159	565	467

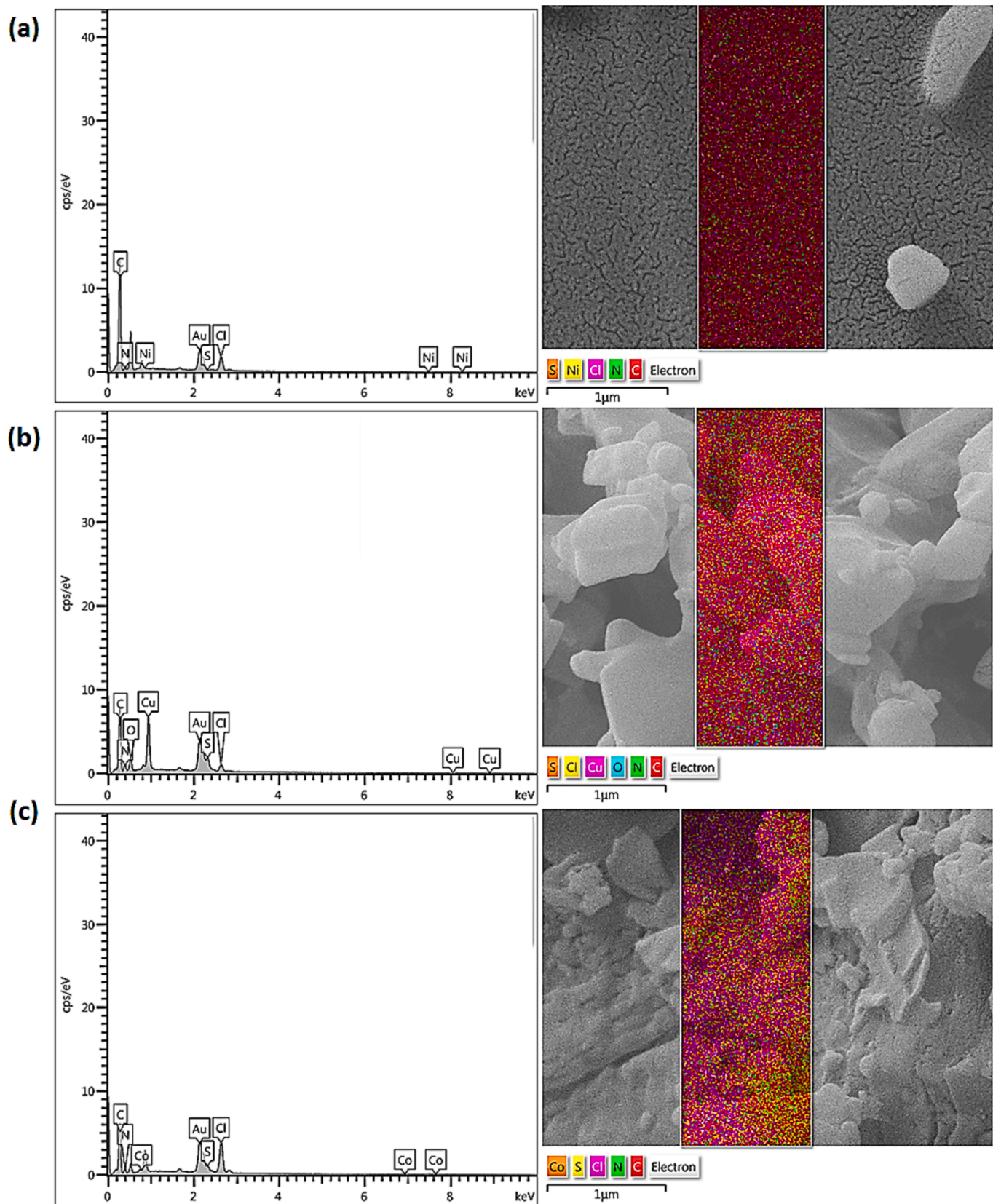


Fig. 1. EDX mapping of sulfamethoxazole-metal complexes containing (a): Ni, (b): Cu, and (c): Co.

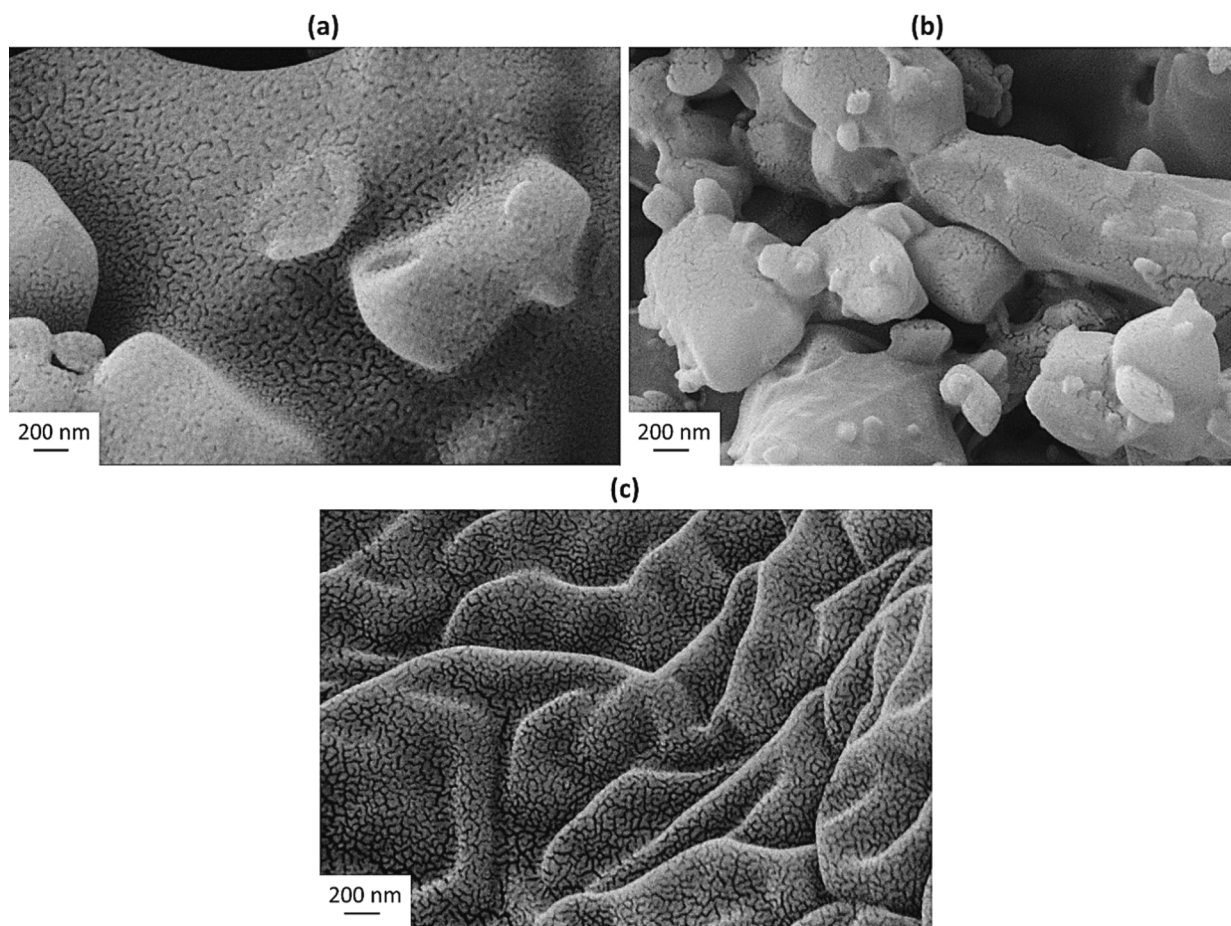


Fig. 2. FESEM images of sulfamethoxazole-metal complexes containing (a): Ni, (b): Cu, and (c): Co.

measurements includes a gas cylinder and a gas compressor connected through a pipe. The method and apparatus design details for the CO₂ uptake experiments were previously reported [38,53].

3. Results and discussion

3.1. Synthesis of sulfamethoxazole-metal complexes

The reaction of Schiff base (two-mole equivalents) and metal chlorides (One mole equivalent) in boiling EtOH for 3 h (Scheme 1) gave the corresponding metal complexes as orangish powders in high yields. Table 2 shows selected FTIR absorption bands for the synthesized sulfamethoxazole-metal complexes. The FTIR spectra showed absorption in the 3285–3368 cm⁻¹ region assigned to the N–H group. The imine group's C–H stretching vibration appeared at ca. 2800 cm⁻¹. The HC = N group of the Schiff base absorption band appeared at the 1640–1655 cm⁻¹ region. At the same time, the absorption band of the C = N bond of the oxazole ring appeared in the 1582–1590 cm⁻¹ region. The asymmetrical and symmetrical vibrations of the S = O bonds appeared in the 1367–1369 and 1156–1159 cm⁻¹ regions, respectively. In addition, appearing in the 559–565 cm⁻¹ and 424–467 cm⁻¹ regions were absorption bands corresponding to M–N and M–O, respectively.

3.2. EDX spectra of sulfamethoxazole-metal complexes

The EDX analysis provides a rough map of the elemental distribution of the sulfamethoxazole-metal complexes. The EDX mapping (Fig. 1) revealed the existence of all the elements expected within the metal complex structures. Clearly, the images showed bands corresponding to the metals (nickel, copper, and cobalt) in addition to heteroatoms

(nitrogen, oxygen, and sulfur) within the organic moiety [54,55].

3.3. FESEM of sulfamethoxazole-metal complexes

The surface morphology of the sulfamethoxazole-metal complex particles was investigated using FESEM, which provides undistorted images for the surface of materials [56]. FESEM images indicate the uniformity, amorphous nature, and roughness of surfaces. The FESEM images (Fig. 2) show a heterogeneous structure of the surface of the complexes. In addition, the surface revealed the presence of pores that have different shapes and diameters. The particle size was 20.1–473.7 nm, 44.7–253.1 nm, and 22.3–60.3 nm for Ni, Cu, and Co complexes, respectively. It should be noted that the complex containing cobalt has a wrinkled surface that could be ideal for gas uptake.

3.4. AFM of sulfamethoxazole-metal complexes

The surfaces of the sulfamethoxazole-metal complexes were examined further using the AFM. The AFM two- and three-dimensional images prove materials' homogeneity, roughness, and porosity [57]. In addition, the AFM reveals the geometric characteristics of particles and any structural deformation. It can be used advantageously without evacuation (i.e., in a vacuum environment). Fig. 3 shows that the sulfamethoxazole-metal complexes are porous and rough. Materials with a rough surface are likely to be more efficient for the adsorption of gases with increased potential to act as storage media. The high surface roughness indicates a high roughness factor (Rq) and a mesoporous structure. The cobalt complex has the highest Rq and porosity. The Rq for the Ni, Cu, and Co complexes was 4.1, 6.8, and 11.6, respectively.

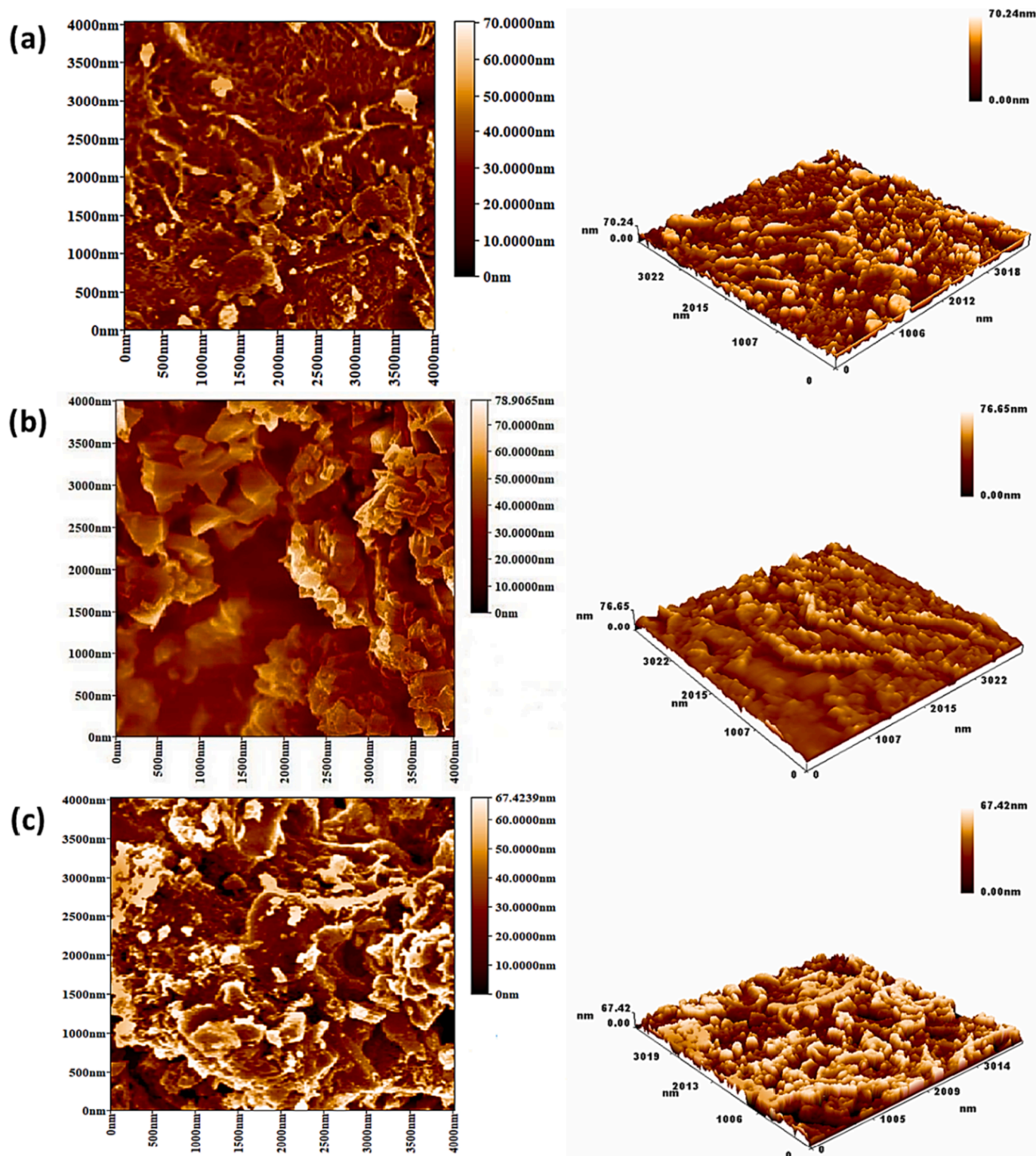


Fig. 3. AFM images of sulfamethoxazole-metal complexes containing (a): Ni, (b): Cu, and (c): Co.

3.5. N_2 adsorption and pore size of sulfamethoxazole-metal complexes

The N_2 adsorption and desorption of sulfamethoxazole-metal complexes were performed at 77 K. The gas volume-relative pressure isotherms were acquired, and the BET technique was used to determine the surface areas from the N_2 adsorption isotherms [58]. The pore diameters of metal complexes were determined using the N_2 adsorption method. The pore size distributions of the sulfamethoxazole-metal complexes are

shown in Fig. 4.

The surface area depends on the pore size rather than the specific pore volume. Small pore size is associated with a large surface area. The structures of metal complexes and their adsorption capacity are linked to the specific surface area and pore volume [59]. The data related to the surface of the metal complexes are summarized in Table 3. The results are consistent with mesoporous structures for the complexes. The cobalt complex has a higher specific area ($5.82 \text{ m}^2/\text{g}$), larger pores volume

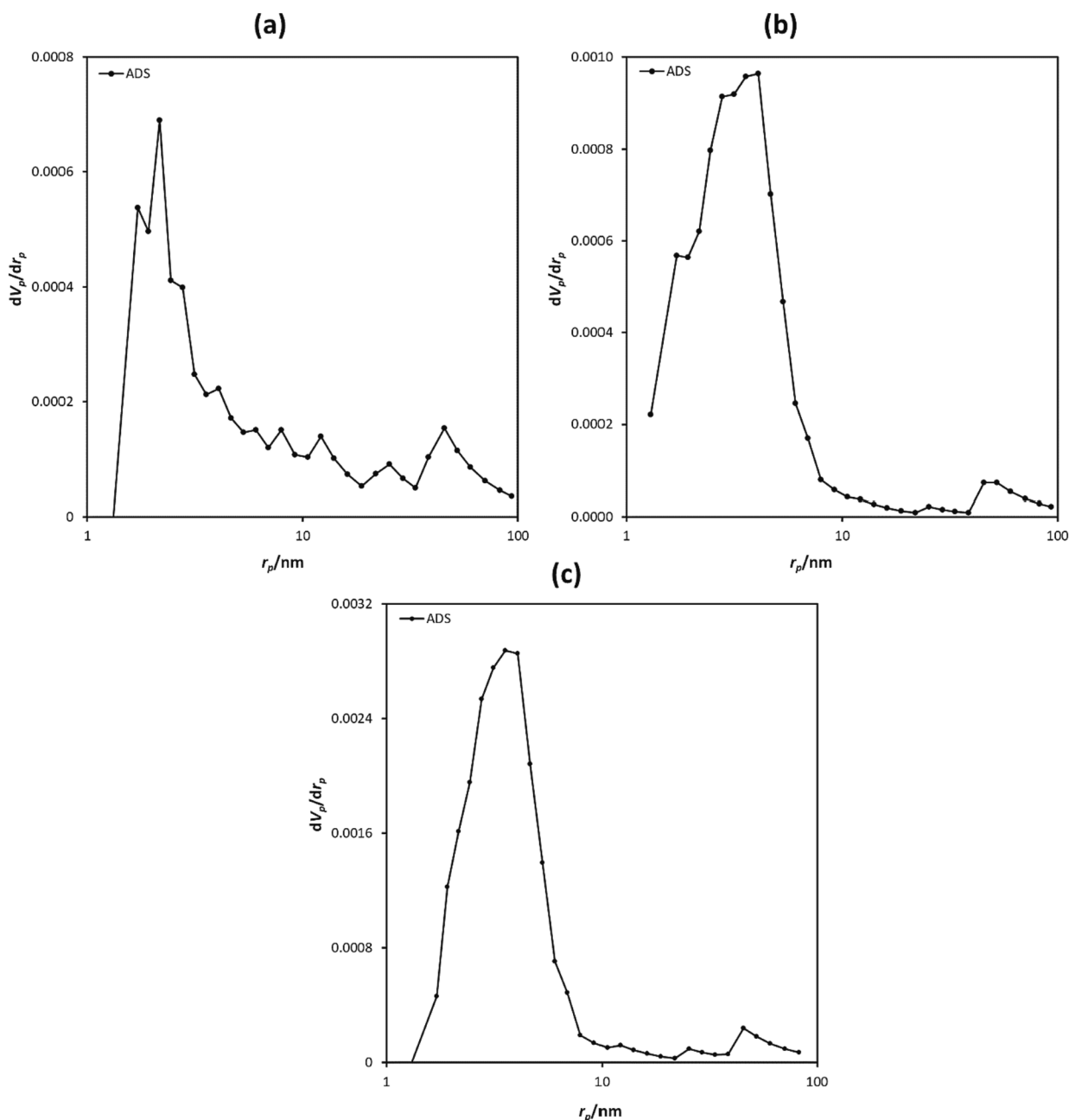


Fig. 4. Pores size distribution of sulfamethoxazole-metal complexes containing (a): Ni, (b): Cu, and (c): Co.

Table 3

Surface area and pore size distribution of sulfamethoxazole-metal complexes.

Complex	S_{BET} (m^2/g)	Pore volume (cm^3/g)	Average pore diameter (nm)
Ni	1.36	0.009	2.17
Cu	2.40	0.008	3.58
Co	5.82	0.018	4.08

(0.018 cm^3/g), and average pores diameter (4.08 nm) compared to the nickel and copper complexes.

CO_2 gas sorption was measured at elevated pressures of up to 40 bar. Fig. 5 shows that the isotherms were of type III, with no monolayers. The isotherms indicated relatively weak interactions between the gases and sulfamethoxazole-metal complex adsorbents [60]. The isotherms were observed to start at the origin, suggesting similar heats of both adsorption and condensation. Gas adsorption on the sulfamethoxazole-metal

complexes' surface was favorable and increased steadily as the pressure increased.

3.6. CO_2 uptake of sulfamethoxazole-metal complexes

Several factors (e.g., pressure, temperature, surface area, and pore volume of the adsorbents) control the CO_2 adsorption process. In addition, strong interaction between polarized bonds in both CO_2 and the adsorbents is vital [61–63]. Several preliminary experiments were conducted in the 1–40 bar pressure range before the highest pressure was adopted for the results. Fig. 6 shows the CO_2 adsorption isotherms of sulfamethoxazole-metal complexes at the optimized conditions (temperature = 323 K and pressure = 40 bar).

The efficiency of adsorbents towards CO_2 depends on the appropriateness of pore properties in shape, diameter, and volume, along with the strength of van der Waals and other interactions between

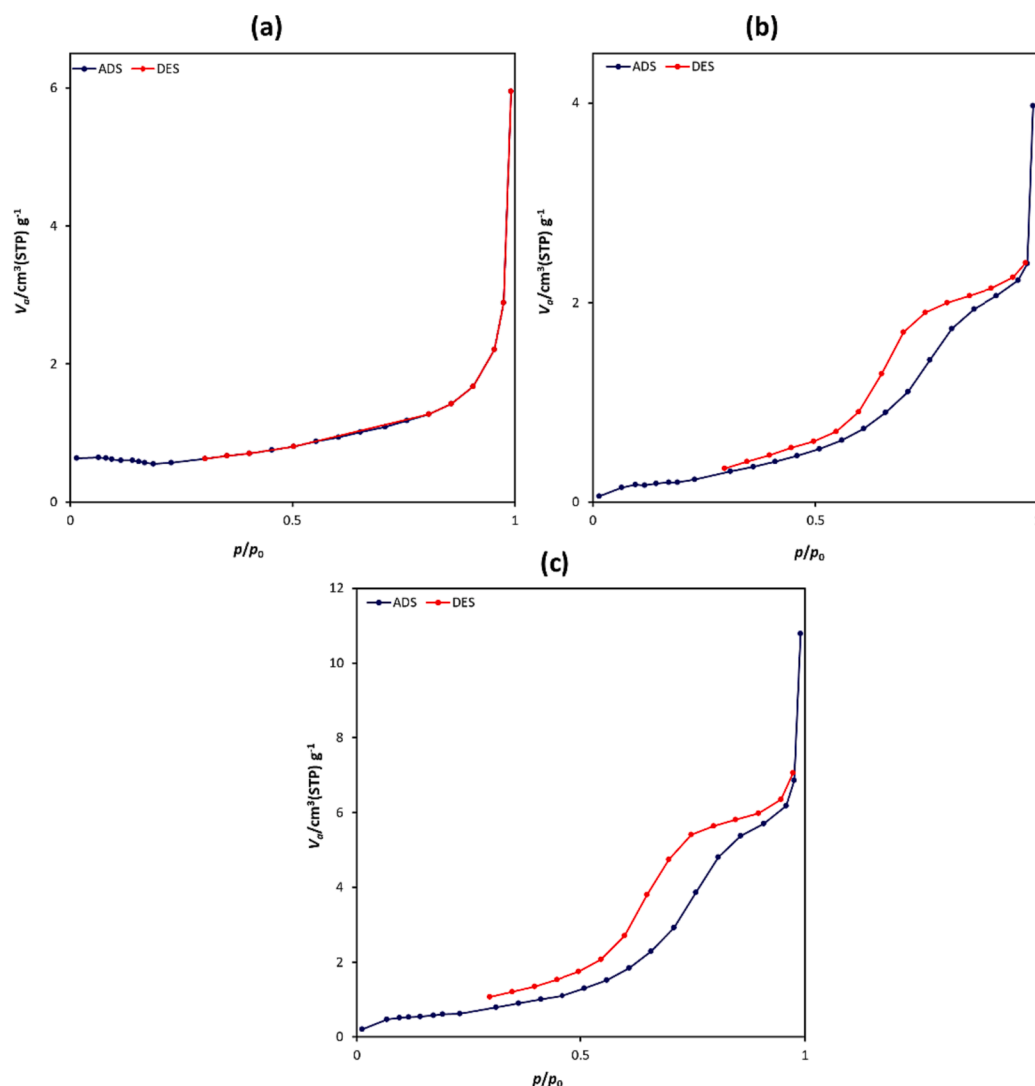


Fig. 5. N_2 adsorption (ADS, black curves) and desorption (DES, red curves) isotherms of sulfamethoxazole-metal complexes containing (a): Ni, (b): Cu, and (c): Co. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

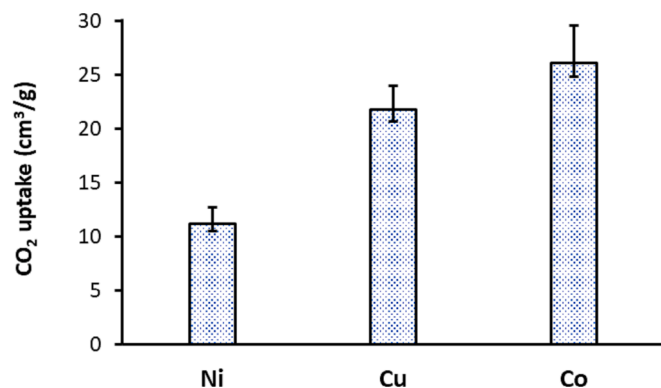


Fig. 6. CO_2 adsorption isotherms of sulfamethoxazole-metal complexes.

sulfamethoxazole-metal complexes and the gas. Hydrogen bonding is possible between the hydrogen atom of the NH group and the oxygen atoms of the CO_2 molecule, which may enhance the adsorption of the gas. In addition, the polar nature of bonds formed by the heteroatoms (N, S, and O) in the organic moiety within the adsorbents can facilitate the interactions with CO_2 . The metals can also act as strong Lewis acids

Table 4

The surface area of several metal complexes and their CO_2 uptake at 323 K.

Adsorbent	S_{BET} (m^2/g)	CO_2 (cm^3/g)	P (bar)	Reference
Sulfamethoxazole-metal complexes	1.4–5.8	11.2–26.1	40	current
Carvedilol-metal complexes	6.1–9.1	10.5–18.2	40	47
Valsartan-metal complexes	16.0–22.8	24.1–34.5	40	48
Metal-fusidate complexes	31.2–46.9	32.2–34.8	50	49
Telmisartan-metal complexes	32.4–130.4	16.5–35.0	50	50

and are therefore capable of coordinating with CO_2 and facilitating its capture. Indeed, porous materials containing metals and heteroatoms have been used to capture CO_2 [47–50]. The CO_2 uptake was 11.2, 21.8, and 26.1 cm^3/g for the Ni, Cu, and Co complexes, respectively. The cobalt-containing complex had the highest CO_2 storage capability, attributed to its higher surface area (5.82 m^2/g), pore volume (0.018 cm^3/g), pores diameter (4.08 nm), surface roughness ($R_q = 11.6$) compared to the nickel and copper complexes. In addition, it contains grooves on the surface, which enhance its capacity to adsorb CO_2 .

The CO_2 uptake (cm^3/g) by different materials containing both metals and organic moieties is shown in Table 4. Despite the low surface

area for the synthesized complexes compared with those reported for other materials [47–50], the capacity for CO₂ uptake by the complexes was similar to those materials. Physisorption likely plays a significant role in the adsorption of CO₂ by the sulfamethoxazole-metal complexes. Physisorption would mainly be through interaction between the heteroatoms of the adsorbents and the oxygen of CO₂ [64].

4. Conclusion

A simple process for synthesizing three sulfamethoxazole-metal complexes containing nickel, copper, and cobalt in high yields was developed. The physical properties and surface morphology of the synthesized complexes were assessed. They have a small surface area (1.36–5.82 m²/g), pores volume (0.008–0.018 cm³/g), and diameters (2.17–4.08 nm). The metals can act as strong Lewis acid centers and promote electrostatic interactions with carbon dioxide. The highest carbon dioxide uptake was seen when the cobalt complex was used. The cobalt-containing complex has the most prominent pores' volume and diameter and the surface area and roughness compared to the other complexes. The results achieved can help in the future design of an efficient carbon dioxide storage media. A prospective study is still needed to assess the possibility of regeneration and reuse of metal complexes.

CRediT authorship contribution statement

Noor Emad: Investigation, Writing – original draft, Writing – review & editing. **Gamal A. El-Hiti:** Conceptualization, Methodology, Software, Validation, Formal analysis, Resources, Data curation, Funding acquisition, Writing – original draft, Writing – review & editing. **Emad Youisif:** Conceptualization, Methodology, Software, Validation, Formal analysis, Resources, Data curation, Supervision, Project administration, Writing – original draft, Writing – review & editing. **Dina S. Ahmed:** Methodology, Software, Validation, Formal analysis, Data curation, Writing – original draft, Writing – review & editing. **Benson M. Kariuki:** Software, Validation, Formal analysis, Writing – original draft, Writing – review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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