Synthesis and use of carvedilol metal complexes as carbon dioxide storage media

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Abstract

The consequences of increased fossil fuel consumption on the environment presents a challenge. Carbon dioxide capture is a useful technique to reduce global warming. Therefore, three carvedilol metal (nickel, cobalt, and copper) complexes were synthesized as potential carbon dioxide storage media. The structural and textural properties of metal carvedilol complexes have been established using various techniques. The metal complexes have mesoporous structures in which pore size was approximately 3 nm. Particle size ranged from 51.0 to 393.9 nm with a relatively small surface area (6.126–9.073 m²/g). The carvedilol metal complexes have either type-III or IV nitrogen adsorption–desorption isotherm. The complexes showed reasonable capacity towards carbon dioxide uptake (up to 18.21 cm³/g) under the optimized condition (40 bar and 323 K).

Graphical Abstract

Keywords Carvedilol metal complexes · Carbon dioxide storage media · Mesoporous materials · Nitrogen adsorption–desorption isotherms · Pore size · Surface area

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Introduction

The reduction of environmental pollution produced due to the excessive use of energy is a challenge that requires attention [1, 2]. Fossil fuels are the most used energy source.

Their combustion contributes (60%) to the increased level of carbon dioxide (CO_2) and in particular from industrial sources [3]. The high atmospheric CO_2 concentration leads to devastating environmental changes, ultimately contributing to climate change [4]. Hydrogen is a clean energy source and its use can overcome some of the disadvantages associated with fossil fuels. However, it cannot fully replace fossil fuels in the near future. The reduction of CO_2 concentration in the environment through its capture is an important strategy [5]. Various inexpensive materials have been designed to act as storage media for CO_2 [6–10]. The use of chemical absorbents showed some success [11]. However, the process involves the use of volatile absorbents, requires high energy, and has a high operating costs [12]. Therefore, different processes for the capture of CO_2 have been developed. These processes should be simple, efficient and require low energy consumption, [13].

Materials with high adsorption capacity such as ionic liquids, zeolites, silica, and activated carbons have been used to capture CO_2 [14–18]. However, limited progress has been achieved since ionic liquids are expensive to recycle and zeolites are highly hydrophilic and not suitable for flue gases [19]. Materials containing carbon such as resins, biomass, and polymers have high stability and surface area, are cheap to produce, and easy to modify, but they have poor selectivity [20]. In addition, the process involves the use of carbonate or hydroxide as a base [21]. Metal–organic frameworks (MOFs) have been used as adsorbents for CO_2 since they have large surface area [22, 23]. A strong interaction exist between CO_2 and MOFs due to hydrogen bonding. Such interaction requires a low adsorption heat [24]. The MOFs storage capacity of CO_2 can be enhanced through by incorporating polar groups within their skeletons [25]. Porous-organic polymers (POPs) have many qualities to act as adsorbents for CO_2 [19]. They have high surface area, stability, adsorption capacity, tunable pores, and their structures can be easily modified. In addition, various functional groups (organic and inorganic) can be incorporated within their skeletons [26]. Moreover, they contain heteroatoms that facilitate the capture of CO_2 [19]. These qualities make them good candidates to be used as media for the storage of CO_2 [27]. However, the POPs synthetic procedures still need optimization and to be simplified [28]. Recently, various porous materials have been designed, synthesized, and used as gas storage media [29–39]. Similarly, covalent

organic frameworks (COFs) are nanoporous materials that have strong covalent bonds and can be used in gas adsorption and separation [40].

The current work report the synthesis, properties, and use of three metal complexes containing carvedilol as CO_2 storage media. Carvedilol is inexpensive, commercially available, non-toxic, has a high molecular weight and it is expected to have some adsorption characteristics.

Materials and methods

General

Chemicals, reagents, and solvents were obtained from Merck. The melting points were recorded on MPD Mitamura Riken Kogyo apparatus. The FTIR spectra (KBr disc) were recorded on 8300 Shimadzu Spectrophotometer. The magnetic susceptibility was measured on a Bruker BM6 instrument. The conductivity was measured in dimethyl sulfoxide (10^{-3} mol/L) at 25 °C using a WTW ProfiLine Oxi 3205 m. The surface morphology was examined on a TESCAN MIRA3 microscope (15 kV). The CO_2 adsorbed over metal complexes was measured on an H-sorb 2600 high-pressure volumetric adsorption analyzer.

Synthesis

A solution of carvedilol (0.81 g, 2.0 mmol) in methanol (MeOH; 20 mL) was slowly added to a stirred solution of metal chloride ($\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$, $\text{CuCl}_2 \cdot 4\text{H}_2\text{O}$, or $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$; 1.0 mmol) in MeOH (10 mL). The mixture was refluxed for 3 h and then cooled to room temperature. The solid was removed by filtration, washed with MeOH (2×10 mL), and dried to give the corresponding metal complex. The yield (%) and melting point (°C) for each complex is reported in Table 1.

Nitrogen gas adsorption

The metal complexes were dried at 60 °C for 8 h prior to the measurements. The specific surface area was measured from the nitrogen adsorption isotherms at 77 K using the Brunauer–Emmett–Teller (BET) method. Pore volume and

Table 1 Melting point, yield, and elemental analysis of carvedilol metal complexes

Complex	Mp. (°C)	Yield (%)	Calculated (Found; %)			
			C	H	N	M
Ni(II)	130–131	81	66.29 (66.33)	5.80 (5.83)	6.44 (6.45)	6.75 (6.79)
Cu(II)	98–99	77	65.93 (65.95)	5.76 (5.79)	6.41 (6.42)	7.27 (7.32)
Co(II)	91–92	72	66.28 (66.30)	5.79 (5.83)	6.44 (6.45)	6.77 (6.80)

pore size were determined using the Barrett–Joyner–Halenda (BJH) method.

Carbon dioxide storage

A sample of metal complex (1.0 g) was degassed in a vacuum oven (2 h; 40 °C) to remove traces of moisture and solvent that might be trapped in the pores. The CO₂ adsorption experiment was performed at different pressures for optimization.

Results and discussion

Synthesis and characterization

Reaction of excess carvedilol (two mole equivalents) and hydrated metal chlorides (nickel, copper, and cobalt; one mole equivalent) in boiling MeOH for 3 h gave the corresponding carvedilol metal complexes (Scheme 1) as green solids in high yields (Table 1).

The FTIR spectra of carvedilol metal complexes showed no absorption band for the OH group. They showed the appearance of bands corresponding to the M–O bonds (455–470 cm^{−1}) [41] and aliphatic (3394–3410 cm^{−1}) and aromatic N–H (3240–3275 cm^{−1}) bonds (Table 2). The high shift in the stretching vibration of aliphatic N–H indicated that coordination with metals has taken place. Based

on molar conductivity (10–12 μS/cm), the complexes are nonelectrolytes in which no ions exist in solution [42]. The Ni(II) complex has a magnetic moment of 3.24 BM, which indicates a high spin octahedral structure [43]. While Cu(II) complex has a magnetic moment of 1.74 BM which is comparable with a slightly pseudo octahedral geometry [44]. For the Co(II) complex, the magnetic moment was 4.32 BM which is consistent for a tetrahedral geometry [42].

Field emission scanning electron microscopy

The field emission scanning electron microscopy (FESEM) was used to inspect the surface of carvedilol metal complexes (Fig. 1). The surfaces of the complexes were amorphous, uniform, contained grooved networks, and irregular blocks, and have a long range of pore dimensions (93.8–274.5 nm) [45]. The grooves were parallel to each other and perpendicular to the outer surface of complexes. These properties could help the particles' porosity to enable gas capture [32]. The particles sizes of Ni(II), Cu(II) and Co(II) complexes were 51.0–116.6, 93.8–274.5 and 67.5–393.9 nm, respectively.

Porosity

The BET method was used to analyze the texture properties of carvedilol metal complexes. The nitrogen (N₂) adsorption–desorption isotherms of carvedilol metal complexes

Scheme 1 Synthesis of carvedilol metal complexes

Table 2 Selected FTIR data, conductivity, and magnetic moment of carvedilol metal complexes

Complex	Wavenumber (cm ^{−1})				Conductivity (μS/cm)	Magnetic moment (BM)
	NH (Ali)	NH (Ar)	M–O	M–N		
Ni(II)	3410	3240	462	424	11	3.24
Cu(II)	3394	3251	470	439	10	1.74
Co(II)	3402	3275	455	435	12	4.32

Fig. 1 FESEM images of carvedilol metal complexes

were studied at 77 K at different pressures (Fig. 2). The Cu(II) complex has a type-III sorption isotherm with no identifiable monolayer formation [46, 47]. On the other hand, the Ni(II) and Co(II) complexes have type-IV isotherms that contained a sharp bend at the beginning of curves due to monolayer formation followed by hysteresis as a result of van der Waals force increase [31].

Fig. 2 N_2 Adsorption-desorption isotherms of carvedilol metal complexes

The BET method was used to calculate the surface area of complexes (6.126–9.073 m^2/g). While, the BJH model was used to determine pore size. The pore size was approximately 3 nm (Table 3) which indicate mesoporous

Table 3 Porosity properties of carvedilol metal complexes

Complexes	S_{BET} (m^2/g)	V_{Total} (cm^3/g)	Pore size (nm)
Ni(II)	9.073	0.021	3.082
Co(II)	7.803	0.020	3.079
Cu(II)	6.126	0.008	3.078

structures [48]. The pore size distribution of carvedilol metal complexes is shown in Fig. 3. The Ni(II) complex has the highest surface area and total pore volume.

Fig. 3 Pore size distribution of carvedilol metal complexes

CO₂ storage capacity

Carvedilol metal complexes showed a reasonable CO₂ uptake (Fig. 4 and Table 4). The CO₂ uptake depends on the strength of interaction between gas and complex, and surface area and pore dimensions of the adsorbent [49]. The order of CO₂ uptake was Ni > Co > Cu, which is consistent with the order of both surface area and total pore volume. The Ni(II) complex has a high charge density compared to the others [50].

Schiff bases containing melamine showed good CO₂ uptake (up to 10 wt%), however, they have small surface area (up to 11.6 m²/g) [30]. Similarly, porous polyphosphates containing benzidine units in their skeleton have small surface area (up to 30 m²/g), but adsorbed CO₂ efficiently (up to 14 wt%) [36]. On the other hand, polyphosphates that have 1,4-diaminobenzene units have relatively large surface area (up to 213.5 m²/g), but their efficiencies towards adsorption of CO₂ was very low (0.6 wt%) [32]. Tin complexes containing telmisartan have surface area up to 130.4 m²/g and CO₂ uptake over 7 wt% [33]. Clearly, surface area plays a role in adsorption process, but other factors such as pore size and diameter are also important. In principle, materials that have high surface area are better CO₂ adsorbents. For example, polyacrylonitrile carbon fibers adsorbed CO₂ efficiently

Fig. 4 CO₂ adsorption isotherms of carvedilol metal complexes

Table 4 CO₂ uptake of carvedilol metal complexes at 323 K and 40 bar

Complex	CO ₂ uptake		
	cm ³ /g	mmol/g	Wt %
Ni(II)	18.21	0.81	3.5
Cu(II)	10.54	0.47	2.1
Co(II)	17.13	0.76	3.3

(2.74 mmol/g) at room temperature and normal pressure, in the presence of a base [18]. At a similar condition, porous nanocarbons that have large surface area (1114 m²/g) leads to a CO₂ uptake of 4.60 mmol/g in the presence of ethylenediamine and potassium oxalate [17]. On the other hand, porous nanocarbons that have small surface area (439 m²/g) leads to a lower CO₂ adsorption (1.94 mmol/g) [17]. Silica matrix contacting ionic liquids have a very small surface area (up to 9 m²/g) and poor CO₂ adsorption capacity (0.35 g of CO₂ per g of adsorbent) [15].

Conclusions

Three carvedilol metal complexes were synthesized in good yields and their structures and properties were established. The synthesised complexes have mesoporous structures and type-III or IV nitrogen adsorption–desorption isotherm. They have a relatively small surface area, and reasonable carbon dioxide adsorption capacity. Nickel and cobalt complexes showed higher carbon dioxide uptake compared to the copper complexes, possibly due to their relatively high surface area and total pore volume compared to the others.

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