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# Porous Aromatic Melamine Schiff Bases as Highly Efficient Media for Carbon Dioxide Storage

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Received: 28 November 2019; Accepted: 19 December 2019; Published: 20 December 2019



**Abstract:** High energy demand has led to excessive fuel consumption and high-concentration  $CO_2$  production.  $CO_2$  release causes serious environmental problems such as the rise in the Earth's temperature, leading to global warming. Thus, chemical industries are under severe pressure to provide a solution to the problems associated with fuel consumption and to reduce  $CO_2$  emission at the source. To this effect, herein, four highly porous aromatic Schiff bases derived from melamine were investigated as potential media for  $CO_2$  capture. Since these Schiff bases are highly aromatic, porous, and have a high content of heteroatoms (nitrogen and oxygen), they can serve as  $CO_2$  storage media. The surface morphology of the Schiff bases was investigated through field emission scanning electron microscopy, and their physical properties were determined by gas adsorption experiments. The Schiff bases had a pore volume of  $0.005-0.036 \text{ cm}^3/\text{g}$ , an average pore diameter of 1.69-3.363 nm, and a small Brunauer–Emmett–Teller surface area ( $5.2-11.6 \text{ m}^2/\text{g}$ ). The Schiff bases showed remarkable  $CO_2$  uptake (up to 2.33 mmol/g; 10.0 wt%) at 323 K and 40 bars. The Schiff base containing the 4-nitrophenyl substituent was the most efficient medium for  $CO_2$  adsorption and, therefore, can be used as a gas sorbent.

**Keywords:** porosity properties; adsorption capacity; carbon dioxide storage; melamine Schiff bases; surface area; energy

# 1. Introduction

Fuel consumption has been increasing over the years to meet the high demand for energy required for various human activities. Therefore, CO<sub>2</sub> concentration has increased drastically to unprecedented levels in the atmosphere [1]. Fossil fuel combustion is the main contributor (60%) to the increased CO<sub>2</sub> concentration level in the environment [2]. Chemical, agro, power, and pharmaceutical industries contribute to approximately 70% greenhouse gas emission, which primarily causes climate changes and global warming [3,4]. The rise of sea and ocean levels, increased acidity of water, and drastic global weather changes are the main environmental problems associated with the increased CO<sub>2</sub> emission, which will consequently lead to economic collapse [5,6]. Additionally, the CO<sub>2</sub> levels in the

environment cannot be lowered rapidly due to the large-scale and high consumption of fuels, which are difficult to be reduced. Novel strategies must be developed to not only resolve the environmental problems arising from global warming but also reduce carbon emission at the source. In addition, it is important to devise new technologies and design novel materials that can be used as a media to capture CO2 effectively [7–10].

Various technologies have been developed to capture and store  $CO_2$  that can efficiently reduce its atmospheric level [11–16]. Recently, researchers from both academia and industry directed their attention toward the capture and storage of  $CO_2$  [17–19]. Various chemical absorbents have been used as media for  $CO_2$  capture, in which amines (e.g., ethanolamine) are the most common ones [20]. The use of amines involves a simple process; however, it is limited because of high operational cost, energy requirement, and the use of very volatile chemicals [21]. Therefore, other techniques that involve the use of adsorbents were developed. Such materials reportedly exhibit adsorption capacity of >4.4% by weight, long life duration, recyclability, and reusability [22–24].

Chemical adsorption of CO<sub>2</sub> is a simple as well as cost and energy effective process. Metal-based adsorbents such as metal oxides are known as common capture media for CO<sub>2</sub> because of their basic and ionic nature [25]. For example, calcium and magnesium oxides can adsorb CO<sub>2</sub> stoichiometrically to produce the corresponding metal carbonate through an exothermic reaction [26]. However, the adsorption capacity of materials varies on the basis of kinetic factors [25]. The adsorption capacity of calcium oxide is limited but sufficiently high to facilitate its use as an effective medium for CO<sub>2</sub> capture. Several other materials such as ionic liquids in a solid matrix [27], zeolites [28], silica [29], and those containing activated carbons [30–32] have been evaluated as CO<sub>2</sub> sorbents. Some of these materials possess unique thermal properties, high chemical stability, high surface area, tunable chemical structures, recyclability, and reusability. However, zeolites are not suitable for CO<sub>2</sub> capture from flue gases because of their excellent hydrophilic properties [33]. In addition, materials containing activated carbon selectivity [34].

Activated carbon has been prepared from different materials such as polymers, resins, and biomass and can be used as an efficient adsorbent for  $CO_2$  [30]. Various chemical and physical processes have been conducted to activate and modify the surface area and pore volume of such adsorbents to increase their capacity for  $CO_2$  capture. The chemical process of activation requires the use of a base, while the physical one requires an appropriate carbonization gas [35,36]. The adsorption capacity of activated carbon depends on the distribution of the chemical activator within the matrix. Polyacrylonitrile in the presence of a base (e.g., potassium hydroxide; KOH) was used as an effective medium to capture  $CO_2$ and exhibited good  $CO_2$  uptake at 25 °C and under 1 bar [31]. The  $CO_2$  uptake was even higher for the resorcinol–formaldehyde resin at the same temperature and pressure in the presence of potassium carbonate as an activator [37].

Metal–organic frameworks (MOFs), synthesized from different molecular building units, have been investigated as adsorbents for CO<sub>2</sub> because of their extended surface area [38–40]. The interaction between MOFs and CO<sub>2</sub> is strong because it occurs through hydrogen bonding and requires a low heat of adsorption, similar to that observed for zeolites [25]. The CO<sub>2</sub> storage capacity of MOFs can be enhanced through the addition of polar residues within their surfaces [41]. Porous-organic polymers (POPs) are highly stable chemically and thermally as well as have low density, tunable structure with a desirable surface area, and different functional groups; therefore, they act as good adsorbents for CO<sub>2</sub> [33]. The presence of heteroatoms (e.g., nitrogen, oxygen, sulfur, phosphorus) within the skeleton of POPs enhances CO<sub>2</sub> capture capacity [33]. The surface polarity of POPs can be increased by the addition of organic moieties containing polar groups or inorganic ions, which facilitates the strong interaction between CO<sub>2</sub> and adsorbent materials [33]. Various POPs showed good CO<sub>2</sub> capture capacity; however, the use of metals in the synthesis of POPs produces toxic pollutants. More research is still needed to optimize the synthetic procedures for POP production by employing simple and effective processes [42]. Nitrogen-rich heterocycles such as triazines have potential use in supramolecular applications because they interact with many chemicals through  $\pi$ – $\pi$  interactions, hydrogen bond formation, and chelation [43]. Melamine has a high nitrogen content (66% by weight) and has been used in various applications such as the production of raw materials with high nitrogen content, plastic, medicinal products, metal-free catalysts, and CO<sub>2</sub> adsorbents [44–46]. Melamine Schiff bases can be easily synthesized through the reaction of melamine and aromatic carbonyl compounds in the presence of a catalyst. Recently, we have synthesized various Schiff bases and investigated their use as additives to stabilize polymeric films against irradiation [47–53]. Melamine Schiff bases have all the qualities needed for their use as efficient adsorbents for CO<sub>2</sub>. In this study, we report the use of melamine Schiff bases, which are highly aromatic and porous, as an efficient media for the capture of CO<sub>2</sub> at 40 bars and 323 K.

## 2. Materials and Methods

## 2.1. Materials

Chemicals, reagents, and solvents were purchased from Merck (Schnelldorf, Germany) and were used as received.

## 2.2. Physiochemical Measurements

The surface morphology of Schiff bases was observed through field emission scanning electron microscopy (FESEM, TESCAN MIRA3, Kohoutovice, Czech Republic) at an accelerating voltage of 10 kV. The N<sub>2</sub> adsorption–desorption isotherms of the Schiff bases were recorded on a Quantchrome chemisorption analyzer (Quantachrome Instruments, Boynton Beach, FL, USA) at 77 k. The Schiff bases 1–4 were degassed in a vacuum oven for a long period (6 h) at a high temperature (100  $^{\circ}$ C) under a flow of  $N_2$  gas (Cascade TEK, Cornelius, OR, USA) to ensure the removal of any residues or small molecules such as water from the pores of materials. The surface area of the Schiff bases was calculated using the Brunauer–Emmett–Teller (BET) equation at a relative pressure  $(P/P^{\circ})$  of 0.98. The pore size of the Schiff bases was verified by the Barrett–Joyner–Halenda (BJH) method. The CO<sub>2</sub> uptake was measured at 40 bars and 323 K using the H-sorb 2600 high-pressure volumetric adsorption analyzer (Gold APP Instruments Corp., Beijing, China), which has two degassing and analyzing ports that can be simultaneously operated. The experiment of  $CO_2$  storage was repeated for at least 10 times for pressure optimization. A known quantity of gas was injected into a measurement tube that contained the Schiff base sample until an equilibrium between the adsorbed gas and the Schiff base sample was established. The final equilibrium pressure was recorded automatically using a software program and the adsorbed quantity of gas was calculated from the obtained data.

#### 2.3. Synthesis of Schiff Bases 1-4

Schiff bases **1–4** were synthesized using a reported procedure by the condensation of melamine and 3 molar equivalents of aromatic aldehydes; 4-nitrobenzaldehyde, 2-hydroxybenzaldehyde, 3-hydroxybenzaldehyde, and 4-hydroxybenzaldehyde, in boiling dimethylformamide containing acetic acid as a catalyst under reflux for 6 h [47].

# 3. Results

# 3.1. Synthesis of Schiff Bases 1-4

The spectroscopic data from the <sup>1</sup>H-NMR and FT-IR spectra, elemental analysis results, and physical properties (e.g., melting points and colors) of the synthesized Schiff bases **1–4** were identical to those of the previously reported bases [47]. Figure **1** represents the chemical structures of the synthesized Schiff bases **1–4**. Schiff base **1** contained a nitro group, while Schiff bases **2–4** contained a hydroxyl group with different arrangements (*ortho, meta,* and *para*).



Figure 1. Schiff bases 1-4 [47].

## 3.2. FESEM of Schiff Bases 1-4

The morphologies of Schiff bases 1–4 were investigated through FESEM. Figures 2–5 show that Schiff bases 1–4 had a relatively uniform and amorphous surface with micro-size particles. The pore dimensions of the Schiff base samples varied and were found to be 20–392 nm. It was clear that the particle size of Schiff base 1 (Figure 2) was smaller than those of Schiff bases 2–4. Schiff bases 2 and 4 (Figures 3–5) have the largest pore dimensions. Schiff base 1 had a different morphology compared to the other Schiff bases because it contains a nitro group, which causes more noticeable irregularity in particle size and shape and, thus, a highly porous structure. The presence of the functional group that had a high content of nitrogen (nitro group) could improve not only the porosity but also the surface area and efficiency for  $CO_2$  uptake [33].



Figure 2. Field emission scanning electron microscopy (FESEM) image of Schiff base 1.



Figure 3. FESEM image of Schiff base 2.



Figure 4. FESEM image of Schiff base 3.



Figure 5. FESEM image of Schiff base 4.

The pore dimensions of Schiff bases **1–4** were smaller than those reported for some POPs and larger than those for telmisartan tin complexes [54–56]. For example, POPs containing polyphosphates derived from 1,4-diaminobenzene showed irregular and porous structures with pore dimensions of 49–981 nm [54]. In addition, polyphosphates derived from benzidine showed porous structures with pore dimensions of 28–806 nm [55]. In contrast, the pore dimensions of telmisartan tin complexes ranged from 20 to 51 nm.

#### 3.3. N<sub>2</sub> Adsorption–Desorption of Schiff Bases 1–4

The N<sub>2</sub> adsorption–desorption measurements for the Schiff bases **1–4** were conducted at 77 K. The N<sub>2</sub> isotherms and pore sizes and volumes of Schiff bases **1–4** are represented in Figures 6–9. The shape of the N<sub>2</sub> isotherm for **1** was similar to the type IV isotherm. Schiff bases **2–4** showed N<sub>2</sub> sorption isotherms that are almost identical to the type III isotherm, in which monolayer formation was not identified.



Figure 6. N<sub>2</sub> isotherms and pore size and volume for Schiff base 1.



Figure 7. N<sub>2</sub> isotherms and pore size and volume for Schiff base 2.



Figure 8. N<sub>2</sub> isotherms and pore size and volume for Schiff base 3.



Figure 9. N<sub>2</sub> isotherms and pore size and volume for Schiff base 4.

The BET surface area (S<sub>BET</sub>), pore volumes, and average pore diameters of Schiff bases **1–4** were calculated (Table 1). Among the synthesized Schiff bases, **1** (containing a nitro group) exhibited the highest surface area (S<sub>BET</sub> =  $11.6 \text{ m}^2/\text{g}$ ) and total pore volume (0.036 cm<sup>3</sup>/gm), but the lowest pore diameter (1.69 nm). Schiff base **1** had a mesoporous structure, while, **2–4** (containing a hydroxy group at *ortho-, meta-* and *para*-position of the aryl ring) had microporous structures (pore diameter = 2.44–3.63 nm). Some POPs and tin complexes showed porous structures with similar pore diameters. For example, porous polyphosphates derived from either 1,4-diaminobenzene or benzidine exhibited a pore diameter of 1.96–2.43 nm [54] or 2.43–2.86 nm [55], respectively, compared to that of 2.43 nm for telmisartan tin complexes [56].

Table 1. Porosity	<sup>r</sup> properties	of <b>1–4</b>
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Schiff Base	$S_{BET}$ (m <sup>2</sup> /g)	Pore Volume (cm <sup>3</sup> /g)	Average Pore Diameter (nm)
1	11.6	0.036	1.69
2	5.2	0.004	3.63
3	10.2	0.016	2.44
4	8.5	0.005	3.62

A gravimetric technique was used to detect the gas uptake quantity and, therefore, determine the gas adsorption isotherm [57]. In addition, the gas quantity that has been removed from the gas phase was used to estimate the physisorption isotherms of the gas. The desorption or adsorption branch of

the isotherm can be used to calculate the pore size distribution. The  $CO_2$  sorption isotherms for Schiff bases 1–4 are shown in Figures 10–13 and their  $CO_2$  uptake are reported in Table 2.



Figure 10. Adsorption isotherm of CO<sub>2</sub> for Schiff base 1.



Figure 11. Adsorption isotherm of  $CO_2$  for Schiff base 2.



Figure 12. Adsorption isotherm of  $CO_2$  for Schiff base 3.



**Figure 13.** Adsorption isotherm of CO<sub>2</sub> for Schiff base 4.

Table 2. CO<sub>2</sub> adsorption capacity of Schiff bases 1-4 at 323 K and 40 bars.

Schiff Base	CO <sub>2</sub> Uptake (cm <sup>3</sup> /g)	CO <sub>2</sub> Uptake (mmol/g)	CO <sub>2</sub> Uptake (wt%)
1	52.29	2.33	10.0
2	30.64	1.36	6.1
3	45.15	2.01	9.0
4	32.27	1.44	6.4

As seen in Figures 10–13, Schiff bases 1–4 do not have an apparent adsorption–desorption hysteresis, which indicates the reversible adsorption of CO<sub>2</sub> within the Schiff base pores at the temperature and pressure used (323 K and 40 bars). The CO<sub>2</sub> uptake for Schiff bases 1–4 was high (6.1–10.0 wt%), possibly because of the excellent pore diameter and the strong van der Waals interactions and hydrogen bonding between the Schiff bases and CO<sub>2</sub>. In addition, Schiff bases 1–4 contain strong Lewis base sites that aid the capture of CO<sub>2</sub>. Indeed, porous materials containing heteroatoms such as oxygen, nitrogen, and phosphorous can selectively capture CO<sub>2</sub> over methane and nitrogen gases [54–56].

The surface area for the Schiff bases was relatively low (5.2–11.6 m<sup>2</sup>/g); however, they showed remarkable CO<sub>2</sub> uptake (1.36–2.33 mmol/g; 6.1–10.0 wt%). Similar observations have been previously reported at similar temperature and pressure. For example, porous polyphosphates containing benzidine showed low surface area (27.5–30.0 m<sup>2</sup>/g) and high CO<sub>2</sub> uptake (up to 14.0 wt%) [55]. On the other hand, polyphosphates containing 1,4-diaminobenzene exhibited high surface area (82.7–213.5 m<sup>2</sup>/g), but the CO<sub>2</sub> uptake was limited to 0.6 wt% [54]. Telmisartan tin complexes showed surface area of 32.4–130.4 m<sup>2</sup>/g and up to 7.1 wt% CO<sub>2</sub> uptake [56]. Materials with the highest surface area showed the most effective CO<sub>2</sub> uptake. Polyacrylonitrile carbon fibers in the presence of a base provided a CO<sub>2</sub> uptake of 2.74 mmol/g at room temperature and normal pressure [31]. In contrast, porous nanocarbons with a high surface area (439 m<sup>2</sup>/g) provided a low CO<sub>2</sub> uptake (1.94 mmol/g [30]. Ionic liquids in a silica matrix led to materials having a very small surface area (1–9 m<sup>2</sup>/g) and relatively poor sorption capacity towards CO<sub>2</sub> as 0.35 g of CO<sub>2</sub> per g of adsorbent [27].

## 4. Conclusions

Four melamine Schiff bases have been investigated as potential media for CO<sub>2</sub> storage at 323 K and 40 bars. These Schiff bases have a relatively low surface area ( $S_{BET} = 5.2-11.6 \text{ m}^2/\text{g}$ ) and varied porous structures, showing pore volumes of 0.004–0.036 cm<sup>3</sup>/g and diameters of 1.69–2.63 nm. The Schiff bases showed remarkable CO<sub>2</sub> uptake (6.1–10.0 wt%), possibly because of their high aromaticity and

heteroatom contents. The Schiff base containing a nitro group showed the most effective  $CO_2$  uptake (10.0 wt%) owing to the high content of nitrogen (heteroatom) within the porous material. The Schiff bases containing a hydroxy group have a lower surface area and pore volume, but higher pore diameter compared to the one containing a nitro group. Such Schiff base is inexpensive and easily producible in high yield and, therefore, can be used at an industrial scale.

**Author Contributions:** Conceptualization and experimental design: G.A.E.-H., M.F.A., D.S.A., and E.Y.; Experimental work and data analysis: R.M.O. and E.T.B.A.-T.; writing: G.A.E.-H., D.S.A. and E.Y. All authors discussed the results and have approved the final version of the paper. All authors have read and agreed to the published version of the manuscript.

**Funding:** The authors are grateful to the Deanship of Scientific Research, King Saud University for funding through Vice Deanship of Scientific Research Chairs.

Acknowledgments: We thank Al-Nahrain and Al-Mansour Universities for the technical support.

Conflicts of Interest: The authors declare that they have no conflict of interest.

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