

Research Article

Synthesis of Stable Tetraethylenepentamine-Functionalized Mesocellular Silica Foams for CO₂ Adsorption

Shou-Heng Liu,¹ Wei-Che Hsiao,¹ and Chia-Chin Chiang²

¹ Department of Chemical and Materials Engineering, National Kaohsiung University of Applied Sciences, Kaohsiung 80778, Taiwan

² Department of Mechanical Engineering, National Kaohsiung University of Applied Sciences, Kaohsiung 80778, Taiwan

Correspondence should be addressed to Shou-Heng Liu; shliu@kuas.edu.tw

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Various amounts of tetraethylenepentamine (TEPA) loading on mesocellular silica foams (TEPA-MSF-*x*) were fabricated via a chemical grafting method in this study. The performance of dry 15% CO₂ adsorption on the TEPA-MSF-*x* was tested by a microbalance at 348 K under ambient pressure. Experimentally, CO₂ adsorption capacities of the TEPA-MSF-*x* sorbents were found to be 28.5–66.7 mg CO₂/g-sorbent. In addition, TEPA-MSF-*x* sorbents exhibited enhanced durability during repeated adsorption-desorption cycles compared to the conventional sorbents prepared by a physical impregnation method. This significant enhancement in the stability of CO₂ adsorption-desorption process was most likely due to the decreased leaching of TEPA which is covalently tethered to the surface of MSF.

1. Introduction

It is well known that global warming of the earth is caused by the progressive increase of CO₂ concentration in the atmosphere due to the extensive utilization of fossil fuels [1–3]. As a result, development of an efficient and economic method to capture CO₂ from large stationary sources, such as coal-fired power plants and cement and steel factories, is urgent. At present, absorption processes using amine-containing solvents have been generally utilized for postcombustion CO₂ capture. Nonetheless, this technology is restricted by some drawbacks including high capital and operation costs and high-energy consumption during regeneration of the sorbents.

For the past few years, extensive studies have been reported on technologies concerning physisorption by using porous solid sorbents for sequestration of CO₂, such as microporous zeolites [4, 5], nanoporous carbons [6–8], nanoporous coordination polymers [9], and organic nanostructures [10, 11]. However, these sorbents have low CO₂ adsorption capacities which are generally smaller than the

criteria value of ca. 88 mg g⁻¹ sorbent for commercialized applications. Moreover, these materials also encounter difficulties such as poor selectivity, poor tolerance to water, and high-temperature regeneration/activation.

In the recent years, ordered mesoporous silicas (OMSs) are commonly used as gas sorbents because they possess unique properties including tunable pore size (2–50 nm), narrow pore size distribution, high surface area, large pore volume, and good thermal stability. For the purpose of CO₂ capture, surface modification of OMSs with amine functional groups was further performed. At least two methods were reported to prepare amine-functionalized sorbents, including (i) liquid amines physically impregnated onto the OMSs and (ii) amines chemically bonded to the OMSs. In terms of the former method, different amino compounds and polyamines supported on various OMSs, such as MCM-48, KIT-6, MSF, and SBA-15, have been utilized as sorbents for CO₂ capture [12–15]. For instance, ca. 50 wt% polyethylenimine (PEI-) incorporated MCM-41 with a high CO₂ adsorption capacity of 246 mg g⁻¹-PEI is 30 times higher than that of MCM-41 and also ca. 2.3 times that of the pure PEI

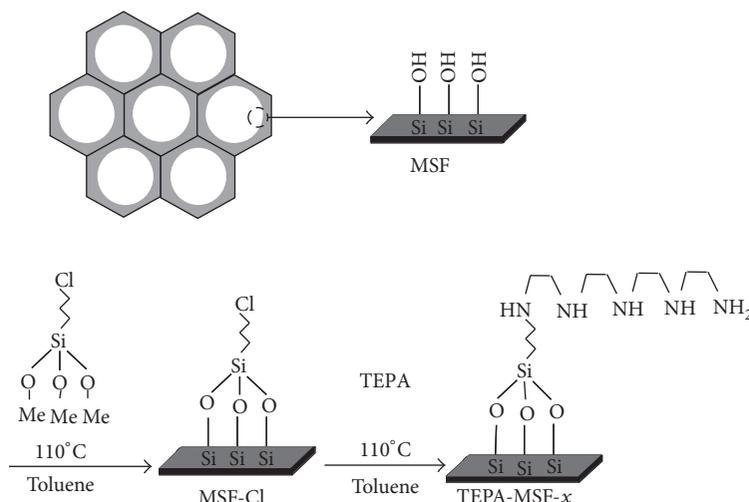


FIGURE 1: Scheme of preparation of TEPA-modified MSF by a chemical grafting method.

[16, 17]. In terms of the latter method, the most common aminosilanes [18–28] used in functionalization of OMSs are including mono(3-aminopropyltrimethoxysilane), di-(3-(2-aminoethyl)aminopropyltrimethoxysilane), and tri-aminosilanes(3-[2-(2-aminoethyl)aminoethyl]aminopropyltrimethoxysilane). Furthermore, grafting amines into the pore-expanded mesoporous silicas was capable of capturing CO_2 by greater amount of amine and also more resistant to moisture while compared to other supports such as activated carbon, silica gel, and pure silica [29–31]. Cyclic adsorption-desorption process also indicated that the above adsorbents have good durability. Hicks et al. [32] prepared a covalently tethered hyperbranched aminosilica (HAS) sorbent which can perform CO_2 adsorption-desorption reversibly with a high capacity of 136.4 mg g^{-1} sorbents at 298 K and cyclic stability. A stepwise method was reported by Bhagiyalakshmi and coworkers [33] to synthesize amino dendrimers grafted SBA-15 sorbents which are efficient for CO_2 capture with thermal durability after seven cycles of adsorption-desorption process.

By far, a variety of amines loaded on various OMSs as sorbents for CO_2 adsorption were widely investigated. Among all sorbents, tetraethylenepentamine (TEPA) incorporated mesoporous silicas [34–37] demonstrate promising applications because of their high CO_2 adsorption capacities and facile preparation route. However, the TEPA-modified sorbents have the shortage regarding the instability of cyclic adsorption-desorption process [38–40]. In this study, a chemical grafting route has been developed to synthesize TEPA-functionalized mesocellular silica foams (MSFs) sorbents as indicated in Figure 1. The resulting TEPA-incorporated MSFs were characterized by a series of different analytical and spectroscopic techniques, including N_2 adsorption/desorption, small angle X-ray scattering (SAXS), and Fourier-transformed infrared (FTIR) spectroscopies. The synthesized sorbents show an extremely enhanced durability after repeated adsorption-desorption cycles, revealing some opportunities for practical and cost-effective applications in industry.

2. Experimental Section

2.1. Materials Preparation. The pure MSF samples were synthesized based on the method reported earlier [41]. In a typical run, 2 g of neutral triblock copolymer surfactant (Pluronic 123; $\text{EO}_{20}\text{PO}_{70}\text{EO}_{20}$, MW = 5800, Aldrich) was dissolved in 75 mL of aqueous 1.6 N HCl at room temperature, followed by adding 12 mg of NH_4F (ACROS) and 1.5 g of 1,2,4-trimethylbenzene (TMB; 98%, ACROS) into the mixture. After stirring for 1 h at 313 K, 4.3 g of TEOS was added to the above mixture. The resultant reaction mixture was stirred at 313 K for 20 h followed by aging at 373 K for 24 h. The solid products of as-synthesized MSF were recovered by filtration and dried at room temperature overnight followed by removal of organic template by calcination at 823 K. TEPA-functionalized MSF adsorbents were prepared by a chemical grafting method. 0.5 g of calcined MSF was dispersed in 25 mL of dry toluene with 50 mM (3-chloropropyl)trimethoxysilane (CPTMS; 98%, ACROS). The reaction mixture was refluxed at 383 K for 24 h, and the final product was filtered, washed with toluene and then alcohol, and dried under a vacuum at 343 K for 8 h. This resultant sample is denoted MSF-Cl. Subsequently, various amounts of TEPA were added to 0.5 g of MSF-Cl to perform surface functionalization via the above-mentioned procedure developed for CPTMS grafting. The obtained samples are denoted as TEPA-MSF- x (x is weight ratios of TEPA/MSF, $x = 0.5\text{--}4.0$).

2.2. Characterization Methods. Small angle X-ray scattering (SAXS) was carried out on a Nanostar U system (Bruker, AXS GmbH). Nitrogen adsorption isotherms were obtained at 77 K on a Micromeritics ASAP 2020 analyzer. Pore size distribution curves were evaluated by the BJH method from the adsorption branch. The Brunauer-Emmett-Teller (BET) specific surface area was calculated from nitrogen adsorption data in the relative pressure (P/P_0) range from 0.05 to 0.2. The total pore volume was estimated from the amount adsorbed at the P/P_0 of 0.99. Fourier transform infrared (FTIR) spectra

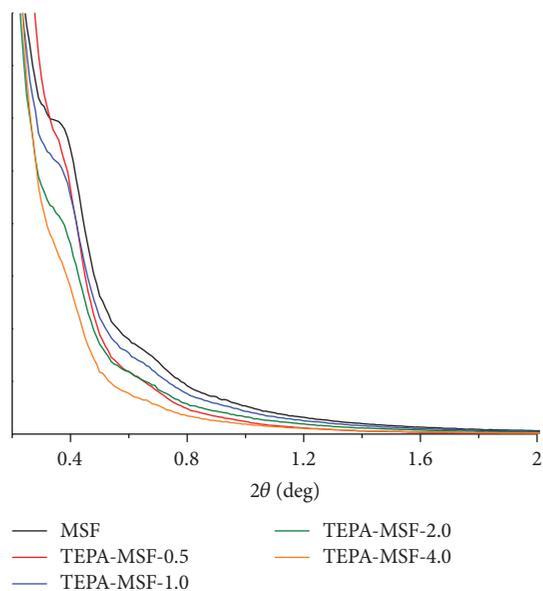


FIGURE 2: Small-angle XRD patterns of pure MSF and various TEPA-MSF-*x* samples.

were obtained on a Bio-rad 165 spectrometer with 4 cm^{-1} resolution using KBr pellets at room temperature.

2.3. CO_2 Adsorption. A modified thermogravimetric analyzer (TGA, PerkinElmer Pyris 6) with a H_2O saturator [42] was applied to evaluate the adsorption and desorption performance of various adsorbents. Typically, about 10 mg of sorbent placed in a sample cell was heated to 373 K under N_2 flow (50 mL min^{-1}), followed by maintaining at that temperature for at least 30 min until no weight loss was observed. Afterward, the sample was cooled down to 348 K, then by introducing 15% dry CO_2 into the TGA cell at a flow rate of 50 mL min^{-1} . After 42 min of adsorption process, the gas was switched to pure N_2 flow (50 mL min^{-1}) to achieve desorption procedure at the same temperature for 78 min. The sensitivity and accuracy of TGA microbalance are $10 \mu\text{g}$ and 0.1%, respectively. The cyclic adsorption/desorption measurements were also studied to evaluate the stability of the sorbents.

3. Results and Discussion

As shown in Figure 2, the small-angle XRD pattern of the pure MSF samples indicates one main intensive (100) feature at 2θ of ca. 0.35° , suggesting the existence of well-ordered hexagonal arrays and two-dimensional (2D) channel structure. However, upon grafting TEPA onto the MSF surface (TEPA-MSF-*x*), substantial decreases in diffraction peak intensities were observed, particularly for the TEPA-MSF-4.0 at which the (100) reflection was almost vanished. The features at low 2θ angles of TEPA-MSF-*x* were found to be decreased, which could be ascribed to the successful incorporation of TEPA compounds, leading to less order of hexagonal structure and/or partial collapse of the wall of mesostructured

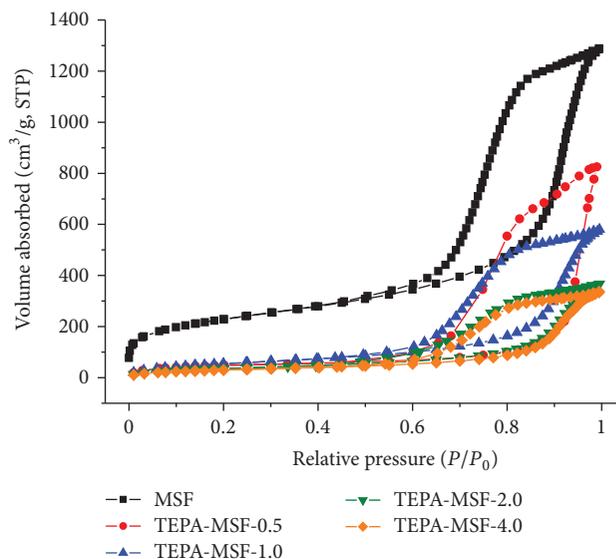


FIGURE 3: N_2 adsorption-desorption isotherms of pure MSF and various TEPA-MSF-*x* samples.

silica [43–45]. As can be seen in Figure 3, N_2 adsorption/desorption curve of the pure MSF samples showed the typical signatures for mesoporosity, namely, type-IV isotherms with well-defined hysteresis loops, consistent with XRD results. However, the decrease of hysteresis loops may be caused by the blockage of mesopore channels upon incorporating a considerable amount of TEPA functional compounds onto the TEPA-MSF-*x* samples. As indicated in Table 1, notable decreases of total pore volume (*V*), BET surface area (*S*), and BJH pore size (*D*) were observed for TEPA-modified samples compared to the pure MSF samples, again suggesting the successive functionalization of TEPA and/or CPTMS onto MSF. To further confirm the presence of TEPA functional groups in the surface-modified MSF samples, the FTIR spectroscopy was measured. As can be seen in Figure 4, the FTIR spectrum of the parent MSF showing characteristic peaks at 1079 and 968 cm^{-1} as assigned as Si-O-Si and Si-OH stretching, respectively. Upon incorporation of TEPA, the peak at 968 cm^{-1} vanished, and three weak peaks appear at 1667 cm^{-1} ($\delta_{\text{N-H}}$), 2931 cm^{-1} ($-\text{CH}_2\nu_{\text{as}}$), and 2818 cm^{-1} ($-\text{CH}_2\nu_{\text{s}}$) which were attributed to the NH_2 scissoring vibration and CH stretching vibration in the amine functional groups, respectively. These results again confirm the successful impregnation of TEPA onto MSF.

CO_2 adsorption/desorption curves of pure MSF and TEPA-MSF-*x* are shown in Figure 5. The measurements were carried out at 348 K by using TGA under atmospheric pressure. The CO_2 adsorption capacities of various TEPA-MSF-*x* are also summarized in Table 1. Unlike the pure MSF samples, which show almost none CO_2 uptake, TEPA-MSF-*x* samples exhibit the adsorption capacities of ca. 28.5 – $66.7 \text{ mg CO}_2/\text{g-sorbent}$, respectively. As proposed previously [46], the mechanism of chemical adsorption pathway between amine active sites and CO_2 in anhydrous conditions is

TABLE 1: Physicochemical properties and CO₂ adsorption performance of parent MSF and various TEPA-MSF-*x* samples.

Sample	V (cm ³ g ⁻¹) ^a	S (m ² g ⁻¹) ^b	D (nm) ^c	CO ₂ uptake (mg g ⁻¹)
MSF	2.34	634	29	—
TEPA-MSF-0.5	0.89	209	18	37.5
TEPA-MSF-1.0	0.56	134	17	53.2
TEPA-MSF-2.0	0.53	113	17	66.7
TEPA-MSF-4.0	0.53	115	17	28.5

^aTotal pore volumes calculated as the amount of N₂ adsorbed at $P/P_0 = 0.99$.

^bBrunauer-Emmet-Teller (BET) surface areas.

^cPore diameters calculated by the Barrett-Joyner-Halenda (BJH) method using the adsorption branches.

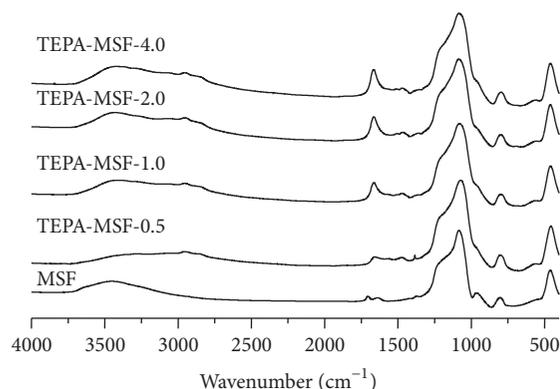


FIGURE 4: FTIR spectra of pure MSF and various TEPA-MSF-*x* samples.

the formation of ammonium carbamate, thus the isolated amine groups are ineffective in CO₂ adsorption due to the stoichiometric CO₂/N ratio of 0.5 as indicated below:



Among all TEPA-MSF-*x* samples, TEPA-MSF-2.0 sorbents have the maximum of CO₂ adsorption capacity. In terms of amine efficiency (CO₂/N), all TEPA-MSF-*x* sorbents were below 0.5 mmol mmol⁻¹, which may be due to the fact that partial primary amino groups of TEPA were reacted with Si-Cl in the MSF-Cl samples during grafting process, leading to the lower surface density of primary amines in the TEPA-MSF-T-*x* sorbents. In the earlier report [47], experimental results indicated that primary amino sorbents had the maximum CO₂ adsorption capacities among primary, secondary and tertiary amines.

In order to be realized for practical applications in industry, CO₂ capture sorbents with a cyclic adsorption-desorption durability under long-term operation is desirable. As shown in Figure 6, it is worthy to note that CO₂ adsorption capacity of our prepared TEPA-MSF-1.0 sorbents exhibits a stable and reversible performance during five repeated runs of adsorption-desorption cycles (i.e., total operation period of ca. 12.5 h) at 348 K under dry 15% CO₂ concentration. In contrast, the TEPA-MSF-1.0P sorbents, which were prepared

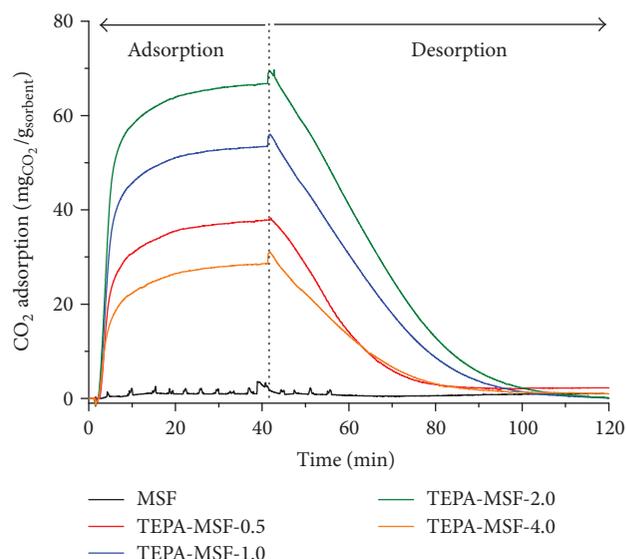


FIGURE 5: CO₂ adsorption on pure MSF and various TEPA-MSF-*x* samples.

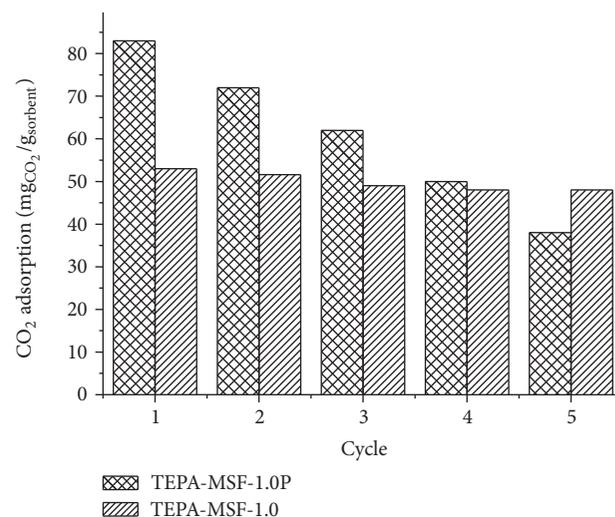


FIGURE 6: Cyclic runs of CO₂ adsorption tests on TEPA-MSF-1.0 and TEPA-MSF-1.0P sorbents at 348 K.

by a conventional physically impregnation method, show a steady decrease with a capacity loss of approximately 54% after the fifth adsorption-desorption cycle. This is probably owing to the extensive leaching of TEPA which is physisorbed on the surface of MSF.

4. Conclusion

In summary, CO₂ adsorption on TEPA-MSF-*x* sorbents prepared by a chemical grafting route shows their CO₂ uptake capacities of ca. 28.5–66.7 mg CO₂/g-sorbent at 348 K under ambient pressure using dry 15% CO₂. Among all TEPA-MSF-*x* samples, TEPA-MSF-2.0 sorbents have the maximum of

CO₂ adsorption capacity. Moreover, the TEPA-MSF-1.0 sorbents possess notably enhanced durabilities during repeated adsorption-desorption cycles compared to TEPA-MSF-1.0P sorbents. This enhancement of durability in CO₂ uptake is probably due to the decreased leaching of TEPA which is chemically modified to the surface of MSF. These TEPA-MSF-*x* sorbents demonstrate reversible and stable properties, revealing a capable CO₂ sorbent for the practical application during cyclic adsorption-desorption processes.

Acknowledgment

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