



Synthesis and Characterization of Amine-modified Mesoporous Santa Barbara Amorphous-15 for Carbon Dioxide Sequestration at High Pressure and Room Temperature

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Amine-modified mesoporous Santa Barbara Amorphous (SBA)-15 adsorbent has been prepared by impregnation method using tetraethylenepentamine. The samples of this modified SBA-15 have been characterized by small angle X-ray scattering (SAXS), Scanning electron microscopy (SEM), Nitrogen adsorption-desorption isotherm and FT-IR. The adsorption capacity of CO₂ on unmodified and modified samples were measured at high pressure (up to 20 bar) and room temperature and were 2.69, 4.02 and 3.62 mmol CO₂/g for SBA-15, TEPA (55%)-SBA-15 and TEPA (65%)-SBA-15, respectively. The increment in the CO₂ adsorption capacity of amine modified mesoporous material (TEPA (55%)-SBA-15) is attributed to the increased amine mass interaction with CO₂. Further increase of amine content clogs the pores of the sorbent which results in slight reduction of CO₂ adsorption in TEPA (65%)-SBA-15.

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1. INTRODUCTION

The emission of greenhouse gases is serious environmental problem of present era which contributes to the global warming and air pollution [1-4]. Among the greenhouse gases, Carbon dioxide is significantly increased in the recent decades. Since the time of the industrial revolution, global increment of the atmospheric concentration of CO₂ has been about 100 ppm (36%) [5]. Human activities such as burning of fuels at homes and industrial units have increased CO₂ emission to atmosphere [6-9]. The control of CO₂ emission has become an important research topic which is of interest to many researchers [10].

The emission of CO₂ may be reduced through several ways [11-15]. The best way is the replacement of fossil fuels with environmental friendly biofuels and improvement in energy efficiency with capturing CO₂ from fuel sources [9, 16]. In the last few decades,

several methods have been proposed for CO₂ removal [1, 2, 16, 17]. Absorption with chemical reaction using aqueous solutions of alkanolamines is one of the methods for carbon dioxide removal. But this method has disadvantages such as high energy consumption for solvent regeneration, equipment corrosion and amine's flow problems. To overcome these problems [18, 19], functionalization of amine groups with grafting or impregnation methods onto porous supports have been used [20-23].

There are many porous supports such as active carbons, zeolites, silica mesoporous and metal-organic framework which are used for carbon dioxide removal. A recent review by D'Alessandro et al. [2] describes these porous materials used for CO₂ adsorption, discussing important aspects such as their advantage and disadvantage, in addition to adsorption capacity.

During the past few years, silica mesoporous materials such as MCM-41 [5, 10], MCM-48 [14, 21], SBA-15 [24, 25], SBA-12 [18] and KIT-6 [26] were used as porous materials to remove carbon dioxide.

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These materials have several remarkable features such as high surface area, ordered pore size and easily functionalized surface [24, 27], but the CO₂ adsorption capacity of the unmodified mesoporous support is low because there are no induced strong reactions between pure silica surface's residual hydroxyl groups and CO₂ molecules. Therefore, the increment of CO₂ adsorption capacity by grafting or impregnation of amine groups such as mono, di- and tri-amines has been studied by different research groups [16, 24]. However, the available data shows that CO₂ adsorption capacity values vary from 0.5 to 3.43 mmol CO₂ per g adsorbent at low pressure [16], while industrial situations involve with process at high pressure and room temperature.

To investigate the removal of CO₂ in industrial process, we have functionalized the 2-D SBA-15 type silica mesoporous, using different amount of tetraethylenepentamine (TEPA) by impregnation treatment. We have characterized the resulting materials by various techniques and have studied their CO₂ adsorption behavior by volumetric measurements at high pressure and room temperature.

2. MATERIALS AND METHODS

2. 1. Reagents and Materials

All materials such as poly (ethylene oxide)-poly (propylene oxide)-poly (ethylene oxide) block copolymer non-ionic surfactant EO₂₀PO₇₀EO₂₀ (Pluronic P123, Merck), Tetraethyl orthosilicate (TEOS 98%, Merck), tetraethylenepentamine (TEPA 99%, Merck), hydrochloric acid (HCl 37%, Merck), ethanol (EtOH, Merck) used in the synthesis SBA-15 as structure-directing agent, precursor of Si, amine source, catalyst and solvent respectively, were of analytical grade.

2. 2. Synthesis of SBA-15

SBA-15 was prepared by hydrothermal synthesis using dilute solutions of the nonionic block copolymer surfactant Pluronic P123 following the literature [28]. Briefly, P123 (4.0 g) was dissolved in 2 M HCl (150 mL) at room temperature. TEOS (9.6 mL) was added to that solution slowly and stirred for 24 hours at 40°C. The mixture was aged at 100°C for 24 h under static conditions. The solid product was filtered, washed with deionized water and dried for 24 h at 100°C. The white solid product was calcined at 550°C for 8 h with a heating rate of 1°C min⁻¹ to remove the surfactant used in the synthesis.

2. 3. Synthesis of TEPA-SBA-15 Composite

For the functionalization with tetraethylenepentamine (TEPA), different amount of TEPA was dissolved in 15 mL ethanol and stirred for 15 minutes. Then SBA-15 powder (0.5 g) was added to this solution. After that, the mixture is refluxed for 4 hours at ambient temperature. The liquid phase was removed by evaporation at 80°C

and dried for 2 h at 100°C. The prepared samples were termed: TEPA(x)-SBA-15 (x was 55 wt% and 65 wt %).

2. 4. Characterization

Two adsorbents, TEPA (55%)-SBA-15 and SBA-15 support were examined by Small Angle X-ray Scattering using Swaxs Hecus (model S3-MICRO) equipment. The diffraction data were recorded in the 2θ range of 0.6–10° with a scanning rate of 5s/step (step 2θ =0.02°). Nitrogen adsorption-desorption isotherms of the samples were measured at 77K using Micromeritics model ASAP 2010 analyzer. The specific surface area was obtained employing BET method and the pore size distribution was evaluated by Barrett-Joyner-Halenda (BJH) method. Transmission infrared (IR) spectra were obtained by DIGILAB FTS 7000 spectrometer, as KBr discs. Particle morphology and compositional analysis of these adsorbents were carried out using scanning electron microscopy (Philips model XL-30 microscop). The samples were coated with gold in order to increase their conductivity before scanning.

2. 5. CO₂ Adsorption Measurements

The CO₂ adsorption capacities of TEPA (x)-SBA-15 samples were evaluated employing volumetric method and the set-up used is shown in Figure 1. At first, 0.5-1 g of a sample was loaded in the adsorption reactor (HP vessel) and attached to the system, then to degas the system, temperature was increased to 433 K and the valves 4, 3 (see Figure 1) were opened. After that, the vacuum pump was turned on and the system was vacuumed for 1.5 h (without any loss of crystallinity) under the given thermal conditions. After de-gassing, the system was cooled to room temperature. The CO₂ was adsorbed by opening the valves 2 & 3. The pressure of HP vessel decreased due to some dead volume in reactor (including hollow space and the connected tubes) and some adsorption. The dead volumes were calculated via helium test and subtracted from total pressure change. By measuring the dead volumes, one could calculate the exact pressure decrease due to the carbon dioxide adsorption.

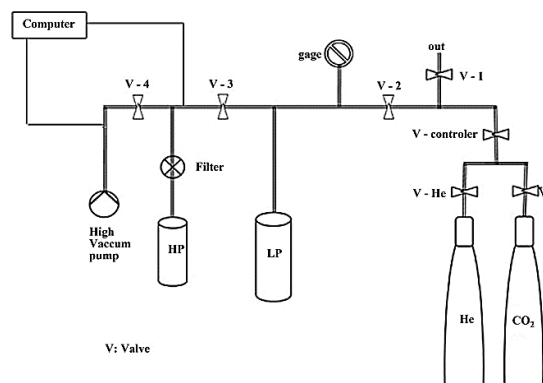


Figure 1. Setup for gas adsorption capacity measurement

CO₂ adsorption amount (mol) was calculated by using real gas equation, $PV=ZnRT$. Where n, P, V are amount (mol), pressure and volume of CO₂ gas adsorbed in HP reactor respectively. T was 298 K, the gas temperature and R denotes the universal gas constant 8.314 J mol⁻¹K⁻¹. Z is compressibility factor at related pressure.

3. RESULT AND DISCUSSION

3. 1. XRD Analysis The small angle X-ray scattering pattern of SBA-15 and TEPA (55 %)-SBA-15 are shown in Figure 2. XRD pattern of two synthesized adsorbents exhibits three peaks (1 0 0, 1 1 0, 2 0 0) at $2\theta < 2^\circ$ which can be indexed as 2D hexagonal lattice [29]. Consequently, modification of hexagonal SBA-15 type mesoporous do not seriously damage the overall ordered structure of the mesoporous silica.

The intensities of the XRD peaks for TEPA (55 %)-SBA-15 decreases slightly and the peaks shifted to a little higher 2θ angles. Hence, the observed decrease and shift of XRD peak intensity is probably due to the pore filling effect of channels in mesoporous support and the anchoring ligands on the outer surface of support [29].

3. 2. N₂ Adsorption-desorption Isotherms Analysis Data on surface area and pore diameter were measured by standard N₂ adsorption isotherms. The BET method provides the information about surface area. The BET-based curves of SBA-15 and TEPA (55%)-SBA-15 are shown in Figure 3. The nitrogen adsorption isotherm of the calcined SBA-15 is typical reversible type IV, and adsorption isotherm (S-shape) and H1 Hysteresis the characteristic of a mesoporous molecular sieve. After amine loading, the IV type isotherm and H1 hysteresis are still preserved. The BET surface area of SBA-15 was 937 m².g⁻¹. After amine loading, surface area was reduced from 937 to 247 m².g⁻¹.

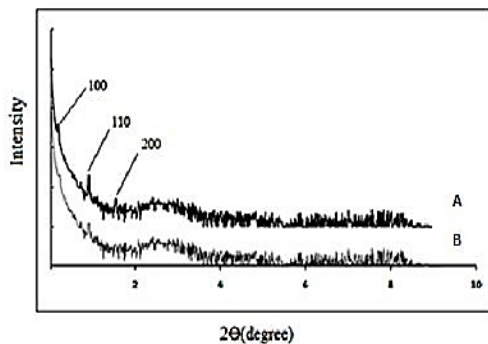


Figure 2. X-Ray diffraction patterns of SBA-15(A) and TEPA (55%)-SBA-15(B)

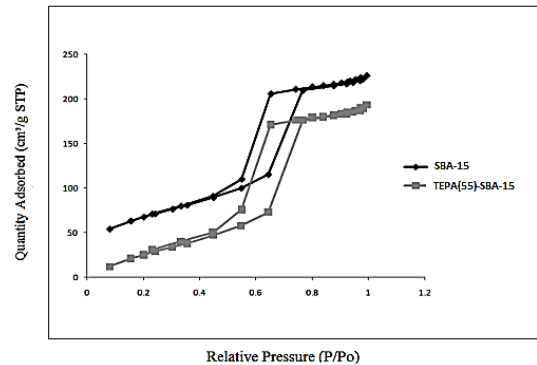


Figure 3. Adsorption isotherms of N₂ on the SBA-15 TEPA (55%)-SBA-15

Pore diameter and pore volume were measured by BJH method. Figure 4 illustrates BJH curve of two synthesized adsorbent and is based on the pore condensation phenomena. This method is applicable only in the mesoporous or microporous region. Pore diameter and pore volume of SBA-15 support was 6.55 nm and 1.17 cm³.g⁻¹, respectively. But, in the TEPA (55%)-SBA-15, these parameters were decreased to 5.65 nm and 0.34 cm³.g⁻¹, respectively. In addition, the pore volume is reduced from 1.17 to 0.34 cm³.g⁻¹ indicating that most likely the pores are blocked by the TEPA groups. Table 1 shows textural properties of SBA-15 support and TEPA (55%)-SBA-15.

TABLE 1. Structural properties of SBA-15 and TEPA (55%)-SBA-15

| Samples | S _{BET} (m ² .g ⁻¹) | Pore diameter (nm) | Pore volume (cm ³ .g ⁻¹) |
|-------------------|--|-----------------------|--|
| SBA-15 | 937 | 6.55 | 1.17 |
| TEPA (55%)-SBA-15 | 247 | 5.65 | 0.34 |

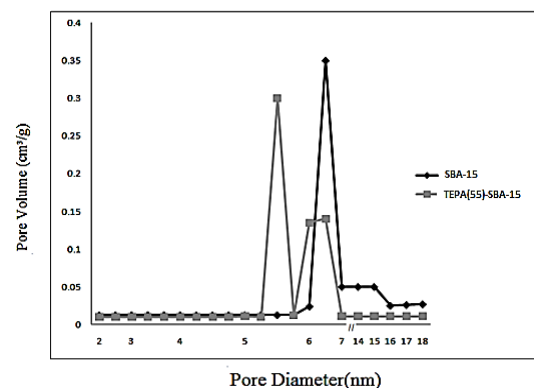


Figure 4. Pore size distribution of SBA-15 and TEPA (55%)-SBA-15

3. 3. FT-IR Analysis Figure 5 shows the FT-IR spectra of calcined SBA-15 and TEPA (55%)-SBA-15 in the scan range from 400 to 4000 cm^{-1} . A broad peak with high intensity around 3450 cm^{-1} on the unmodified SBA-15 indicates the O–H stretching vibrations of silanol groups and adsorbed water molecules. This peak with same intensity was seen on FT-IR spectrum of amine modified SBA-15, which indicates that there is no reaction between TEPA and OH-silanol in SBA-15 support. The band around 960 cm^{-1} can be attributed to Si–O vibration in Si–OH group in siliceous SBA-15. The characteristic band at 1031 cm^{-1} is due to the Si–O in stretching mode in Si–O–Si structure. The IR bands at 3422 and 3277 cm^{-1} are due to asymmetric and symmetric NH_2 stretching. N–H and C–H vibration bands are seen at 1539 and 1463 cm^{-1} respectively. The bands at 2973 and 2854 cm^{-1} are assigned to the CH_2 asymmetric and symmetric stretching modes of the TEPA in amine modified SBA-15 [29]. In addition, two bands at 1180 and 1004 cm^{-1} are related to N–C stretching modes of the TEPA molecules.

3. 4. Scanning Electron Microscopy The morphologies of the prepared unmodified SBA-15 and TEPA (55%)-SBA-15 mesoporous are shown in Figure 6. It can be seen that all the samples are composed of uniform and long fibrous aggregates. No difference in fibrous morphology is seen before and after functionalization of silica mesoporous. This obviously indicates that the shape of particles is not changed and morphology of samples have remained after modification.

3. 5. Isotherms of CO_2 Adsorption Figure 7 shows the CO_2 adsorption capacity of SBA-15, TEPA (55%)-SBA-15 and TEPA (65%)-SBA-15 at 20 bar and room temperature. Unmodified SBA-15 support possesses low CO_2 adsorption capacity about 2.69 mmol/g^{-1} .

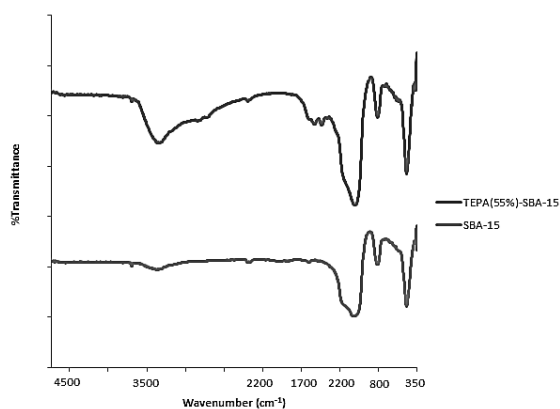


Figure 5. FT-IR spectra of SBA-15 and TEPA (55%)-SBA-15 materials

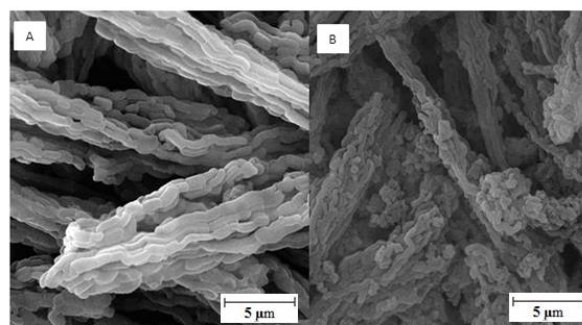


Figure 6. SEM images of SBA-15 (A) and TEPA55%-SBA-15 (B)

By loading amines onto mesoporous SBA-15, adsorption capacity is increased. Increase of carbon dioxide adsorption capacity is due to loading of amine groups. TEPA possess primary amine and secondary amine in their structures. The improvement in CO_2 adsorption over functionalization of SBA-15 with amine groups is because of acid-base interaction between acidic CO_2 basic and amine groups. This is explained by the fact that active alkali $-\text{NH}_2$ group possess lone pair of electrons on N atom which undergoes nucleophilic attack on the C atom of acidic CO_2 , leading to formation of carbamate through carboxyl group. The active H atom in carboxyl group can then form ion pairs through hydrogen bond with the nearby amine group, and hence stabilizes the chemisorption of CO_2 . However, stabilization of carbamate in the course of hydrogen bonding decreases the number of free amine groups. This indicates that, one mole CO_2 stabilization requires two moles of amine groups. TEPA (55%)-SBA-15 and TEPA (65%)-SBA-15 adsorbents show CO_2 adsorption capacity of 4.02 and 3.62 mmol/g^{-1} , respectively. The adsorption capacity of TEPA (65%)-SBA-15 is greater than which measured for SBA-15 support but it is lower than that of TEPA (55%)-SBA-15. This exception is associated with the completely filled pores of this adsorbent and block age of the pores by the additional TEPA groups.

Figure 8 shows CO_2 adsorption isotherms of SBA-15 and all amine modified mesoporous at room temperature and different pressures in the range of 0-20 bar. In the range of 0-8 bar, TEPA (65%)-SBA-15 shows the highest CO_2 adsorption capacity due to amine loading on the outer surface of support and reaction with CO_2 . But in the range of 8-20 bar, free amines on the surface were decreases due to interaction with CO_2 molecules. The CO_2 adsorption isotherm on all synthesized adsorbent is of a typical type IV isotherm according to the IUPAC classification. The adsorption capacity for an isotherm increases with increasing pressure and reaches the saturation state and then remains constant.

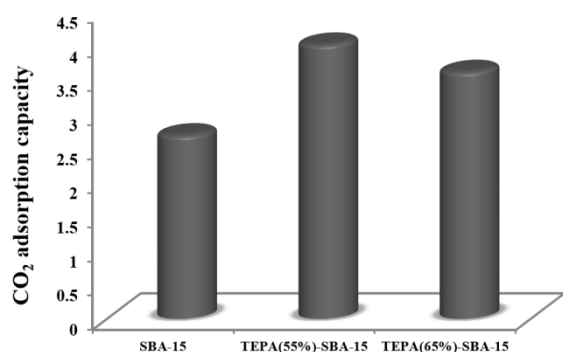


Figure 7. CO₂ Adsorption capacity chart of SBA-15 and TEPA-SBA-15 materials at 20 bar

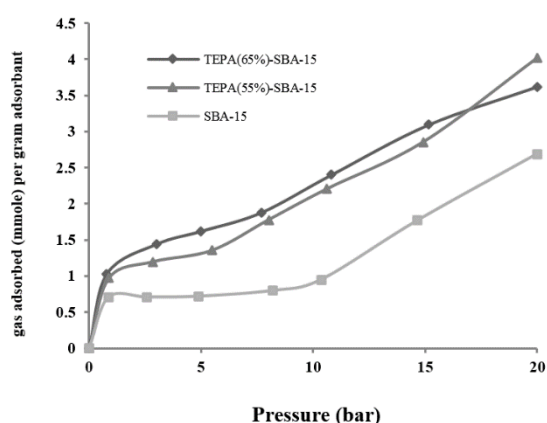


Figure 8. CO₂ adsorption curves on SBA-15 and TEPA(x)-SBA-15 materials at 298 K up to 20 bar

4. CONCLUSION

In this work, amine modified SBA-15 is prepared using tetraethylenepentamine by impregnation method. The resulting sample was characterized by FT-IR, XRD, SEM and BET analysis. CO₂ adsorption capacity of prepared sample is measured by volumetric method. The order of CO₂ adsorption capacities for the prepared samples were SBA-15 < TEPA (65%)-SBA-15 < TEPA (55%)-SBA-15. Increasing the amine mass on SBA-15 increases CO₂ adsorption capacity of amine modified mesoporous material. However, with further increase of amine groups result in slight lowering of CO₂ adsorption capacity of (TEPA (65%)-SBA-15) this is due to the clogging of the pores by amine group in this sample.

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Synthesis and Characterization of Amine-modified Mesoporous Santa Barbara Amorphous-15 for Carbon Dioxide Sequestration at High Pressure and Room Temperature RESEARCH NOTE

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جاذب مزوپوروس SBA-15 اصلاح شده با آمین از طریق روش اشباع سازی با تترا اتیلن پنتا آمین تهیه شد. نمونه‌های SBA-15 اصلاح شده با روش‌های شناسایی مانند پراش اشعه ایکس، میکروسکوپ الکترونی روبشی، تکنیک جذب-وا جذب نیتروژن و اسپکتروسکوپی زیر قرمز تبدیل فوریه مورد بررسی قرار گرفتند. ظرفیت جذب CO₂ ترکیبات اصلاح نشده و اصلاح شده در فشار بالا و دمای اتاق ۲۰۲، ۴ و ۲/۶۲ mmol CO₂/g به ترتیب برای SBA-15، SBA-15 (55%) - TEPA و SBA-15 (65%) - TEPA اندازه گیری شد. افزایش ظرفیت جذب CO₂ در ترکیبات اصلاح شده آمینی (TEPA (SBA-15) (55%) به دلیل برهم‌کنش آمین با CO₂ می‌باشد. افزایش بیشتر آمین باعث انسداد حفرات جاذب، و در نتیجه کاهش جزئی در جذب CO₂ در SBA-15 (65%) - TEPA می‌شود.

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