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Enhancement thermal stability and CO₂ adsorption property of ZIF-8 by pre-modification with polyaniline

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Abstract

In this study, the thermal stability and CO₂ adsorption property of ZIF-8 were effectively improved by using parallel flow-drop solvothermal method. -NH₂ was successfully introduced into the surface of ZIF-8 and their structures by pre-modification with polyaniline. XRD analysis shown that the intensity of ZIF-8 characteristic peak increased with the increase doped content of polyaniline, but the crystal structure of ZIF-8 remained unchanged. FTIR analysis shown that the two new peaks at 1609 and 690 cm⁻¹ can be attributed to the -NH₂ bonding of ZIF-8. TGA analysis shown that the thermal stability of NH₂-ZIF-8 was 632.71 °C. SEM analysis shown that the increase of CO₂ adsorption property was due to the increase of NH₂-ZIF-8 cubic crystal system. It was found that BET surface area was 789.7 m²g⁻¹, micropore volume was 0.55 cm³g⁻¹, pore size was 0.99 nm by taking the molar ratio of metal ions to ligands being 1:2, and the molar ratios of polyaniline to metal ions being 12%.

1. Introduction

The efficient comprehensive utilization of blast-furnace gas is a breakthrough for energy saving and low carbon emission reduction in iron and steel industry. The main reasons for low utilization rate of blast furnace gas are high inert gas content, low calorific value and poor combustion stability. There are flame instability and extinction phenomena in the combustion process [1, 2]. The specific reason is that CO₂ in blast furnace gas component neither participates in combustion nor supports combustion. On the contrary, it absorbs heat and causes the actual temperature to be lower (1300 °C) [3]. If the inert gas CO₂ in blast furnace gas can be separated beforehand, the problem of low utilization rate of blast furnace gas can be solved, and adsorption is the main method to solve this problem. At present, if the porous adsorbents have been used in steel industry, the common problems are that they are sensitive to CO₂ adsorption conditions, low thermal stability, low selectivity and low adsorption capacity. This problem has become a bottleneck in the application of such porous materials. MOFs (Metal-organic frameworks) provide new research ideas for CO₂ adsorption and desorption. MOFs materials have many advantages (large specific surface area, diversity of structure, adjustable pore size, unsaturated metal position and modifiable frameworks) and are used in many fields (gas storage and separation, optical materials, magnetic materials, catalytic materials, sensing material and biological materials) [4–7]. ZIF-8 has attracted extensive research interest because of its simple synthesis and various formulations, easy modification, and its potential applications in catalysis and gas separation [8–10]. Several researchers have reported the reason why ZIF-8 has high CO₂ adsorption performance. For instance, both experimental and simulated adsorption isotherms have generally revealed that the ZIF-8 prefers to adsorb CO₂. Meanwhile, two preferential sites of CO₂ adsorbed in ZIF-8 were found to be on both sides of 6-ring windows and around the aromatic rings of MeIMs, respectively. However, for the ZIFs materials which adsorb and separate CO₂ from flue gas in steel industry, their working environment is mostly high temperature, and they are mixed gases containing many kinds of compounds, which makes the MOFs materials unable to achieve their ideal CO₂ adsorption effect [11–13]. Therefore, the thermal stability and CO₂ adsorption property of ZIF-8 materials need to be further improved in
order to expand new application fields [14, 15]. Selecting special functional groups to synthesize and modify MOFs materials is an effective way to further increase its stability and CO2 adsorption property. Introducing -NH2 in the synthesis process or after the synthesis of MOFs materials can make CO2 molecule attack the free electron pair of nitrogen atom, which will form new -COO and -NH3+ groups. This measure will greatly improve the adsorption performance of CO2. [16, 17]. Merten Kobalz [18] the effect of synthetic materials on the CO2 adsorption of MOFs. It was found that the CO2 adsorption property increased slowly with the increase of nitrogen content of product components, which indicated that the use of some nitrogen containing organic ligands was conducive to the construction of MOFs materials with better CO2 adsorption property and thermal stability.

A new post-synthetic modification (PSM) method for synthesizing CuBtc materials was developed by Nadeen [19]. The economical dopant of molecular glycine with amine and a carboxyl group was found to be able to saturate OMSs in the parent CuBTC MOFs materials. Thus, the hydrothermal stability and CO2:N2 selectivity are improved. It was also found that the adsorption capacity, CO2:N2 selectivity, and hydrothermal stability of Gly-CuBTC MOFs materials depend on the percentage of saturation of OMSs in the parent MOFs materials. In addition, Nousheen Iqbal [20] further studied the effect of nitrogen containing modifiers on the structure of MOFs materials and the change of their CO2 adsorption properties. The results showed that the chemical stability, thermal stability and CO2 adsorption property of MOFs materials can be improved by introducing functional groups with target functions or different polarities into the framework of organic ligands to modify MOFs materials. Herein, we report our systemic work on the preparation of NH2-ZIF-8 by the parallel flow-drop solvothermal method. And the introduction of -NH2 under different conditions is modified to improve the CO2 adsorption performance and thermal stability of ZIF-8. The resulting NH2-ZIF-8 materials are comprehensively characterized by a variety of techniques. The influence of synthetic method, different modifier, doped content and surface chemistry on the CO2 adsorption property and thermal stability of the NH2-ZIF-8 is discussed for the first time. The research results provide new ideas for the synthesis and modification of ZIF-8 matrix composites, and provide theoretical guidance and technical support for the application of ZIF-8 in the treatment of CO2 in flue gas of steel industry and thermal power industry.

2. Experiment

2.1. Reagents and measurements instruments

Zinc nitrate hexahydrate (Zn(NO3)2·6H2O, 99.9%), ethanol (C2H5OH, 99.7%) were obtained from xiya reagent Co., Ltd Ethanolamine (MEA, C2H7NO, 99%), tetraethylenepentamine (TEPA, C8H23N5, 99.5%), polyaniline (PANI, C14H13N4, 99%) were obtained from taizhou yateng chemical materials Co., Ltd N,N-dimethylformamide (DMF, C3H7NO, 99.9%) and 2-methylimidazole (2MI, C3H4N2, 99.5%) were obtained from shanghai shifeng biotechnology Co., Ltd All the chemicals were used as received without any further treatment. Deionized water was obtained from a water purification system (Makepolo, SPT-20) in the laboratory.

X-ray diffraction (XRD, JSM-7600F diffractometer) analysis was performed using a Cu Ka radiation (30 kV - 40 mA) at step time 1 s and step size of 0.1° by scanning 2θ angle between 2° and 40°, to investigate phase composition and crystal structure of materials. The structures and morphologies of materials were examined by scanning electron microscopy (SEM, SU8020), the materials were spattered with gold before imaging and photographing by employing a scanning electron microscope with a potential of 10 kV under magnification ranging from 1000 to 20 000. All materials have been checked by a FTIR spectrometer (Perkin-Elmer 100 FTIR) for intensities of functional groups. The spectrum was scanned from 400 to 4 000 cm−1 with a resolution of 4 cm−1 using an attenuated total reflectance (ATR) technique. Thermogravimetric analysis (TGA, DSC200F3) was used to characterize the thermal stability of materials. TGA records the weight changes of the sample when heated continuously. The materials were heated from 0 °C to 800 °C at a heating rate of 10 °Cmin−1 under a nitrogen atmosphere with a nitrogen. The specific surface area and pore volume of materials were measured using a micromeritics gas adsorption analyzer ASAP2010 instrument equipped with commercial software for calculation and analysis. Specific surface areas were calculated according to the Brunauer–Emmett–Teller ( BET) equation, and pore size distributions were computed using Barrett–Joyner–Halenda (BJH) method assuming the cylinder pore geometry from the desorption data.

2.2. Synthesis of NH2-ZIF-8

The parallel flow-drop solvothermal method and pre-modification method was utilized to synthesize NH2-ZIF-8 by taking the molar ratio of metal ions to ligands being 1:2, and the molar ratios of polyamiline to metal ions being 6%, 12% and 18%. Metal salts, organic ligands and modifiers were dissolved in different solvents. Solution A was the solution of zinc nitrate hexahydrate (5.95 g, 0.02 mol) dissolved in 20 ml distilled
water. Solution B was polyaniline (0.266 g, 1.2 mmol; 0.533 g, 2.4 mmol; 0.799 g, 3.6 mmol) and 2-methylimidazole (3.284 g, 0.04 mol) dissolved in 20 ml DMF. 20 ml DMF solution placed in a stainless-steel reactor lined with polytetrafluoroethylene as the bottom liquid, and the bottom liquid was heated to a certain temperature and stirred violently. Then, solution A and solution B was dripped into the bottom liquid at the same time. The reaction system reacted at different temperatures in a programmable oven for 18 h, and the temperature of the furnace naturally decreased to room temperature after reaction. After filtering and washing, the solids (NH₂-ZIF-8) were dried in an electric vacuum drying oven at 90 °C for 12 h, and collected sample.

3. Results and discussion

3.1. The effect of doped content on the XRD of NH₂-ZIF-8

Figure 1 shown the XRD patterns of the NH₂-ZIF-8 samples by taking the different molar ratio of the metal ions to the organic ligands (1:1, 1:2, 1:3) and temperature (100 °C, 120 °C, 140 °C). Figure 1 shown that the condition is utilized to synthesize the crystal of ZIF-8 by taking the molar ratio of the metal ions to the organic ligands of 1:2 at 120 °C. The characteristic peak strength of the synthesized material is significantly higher than that of the products synthesized under any other experimental conditions. The XRD spectrum is also the most consistent with the XRD pattern of ZIF-8 reported by Mojgan [21]. Meanwhile, the part of the impurity peaks at the diffraction angle of 2θ = 30–40° were eliminated.

Figure 2 shown the XRD of ZIF-8 modified by polyaniline with different doped content. The XRD pattern for ZIF-8 and NH₂-ZIF-8 matches with the XRD pattern of ZIF-8 reported by Mojgan [21]. The NH₂-ZIF-8 have some typical characteristic peaks at 2θ = 7.3°, 10.4°, 12.7°, 16.4° and 18.0°, correspond to planes 110, 200, 211, 310, and 222. This result confirmed the formation of crystalline ZIF-8 phase and shown that the ZIF-8 and NH₂-ZIF-8 have a higher degree of crystallization. The synthesized NH₂-ZIF-8 exhibits (200) preferred growth, which indicated that they have a tendency to form cubic crystals [22]. Cubic crystals usually have larger specific surface area and higher microstructural stability, which helps to improve gas adsorption property. As shown in figure 2, the crystal structure of ZIF-8 is remained after the incorporation of polyaniline. Moreover, it is observed that the intensity of the characteristic peaks of ZIF-8 increased with polyaniline doped content increased, which may be ascribed to a fairly strong binding of polyaniline to ZIF-8. In summary, polyaniline has a good effect on the modification of ZIF-8, and the introduction of -NH₂ has no significant effect on the structure of ZIF-8.

3.2. The effect of doped content on the FTIR of NH₂-ZIF-8

Figure 3 corresponds the FTIR spectra of the NH₂-ZIF-8 compared with polyaniline, 2-methylimidazole and ZIF-8 samples. The bands at 2927 cm⁻¹ indicated the aromatic C-H stretching vibrations of the 2-methylimidazole. The bands at 1432, 1145, 759 cm⁻¹ are associated with the entire ring stretching or bending of 2-methylimidazole stretching. While the band at 1010 cm⁻¹ of the 2-methylimidazole and 995 cm⁻¹ of the ZIF-8 and NH₂-ZIF-8 is ascribed to Zn-N stretch, this stretching vibration peak is confirmed by red shift. It is shown that the 2-methylimidazole is completely protonated, and the Zn²⁺ and the 2-methylimidazole are

Figure 1. XRD of different temperature and molar ratio synthesized NH₂-ZIF-8.
formed ZIF-8 [23]. Meanwhile, the peak at 423 cm$^{-1}$ shown the distinct stretching vibration of Zn-N, these peaks also appear in NH$_2$-ZIF-8.

As compared with the ZIF-8 of spectra, two new peak appearing at 1309 and 690 cm$^{-1}$ could be attributed to -NH$_2$ bonding of ZIF-8 molecules, thus suggesting that -NH$_2$ have been successfully introduced to the ZIF-8. On the other hand, compared with the infrared spectra of different doped content of polyaniline, the peaks of 6% and 18% were relatively weak, wide and defective. Simultaneously, 12% doped content of polyaniline has led to the intensification of the FTIR absorbance peaks coming from 1432, 1145, 759 cm$^{-1}$. The results show that the ratio of polyaniline to metal ion is too low or too high to modify zif-8 effectively. The reason is that the content of polyaniline has a great influence on the deproton of 2-methylimidazole [24]. In summary, the results of 12% doped content shown that the modification of ZIF-8 by polyaniline was successful.

3.3. The effect of doped content on the thermal stability of NH$_2$-ZIF-8

The TGA curve of the ZIF-8 and NH$_2$-ZIF-8 are shown in figure 4. On the whole, in the TGA curve of four samples shown four distinct weightlessness steps. The first step of the weight loss at 5% occurred up to 150 °C and it is attributed to the removal of moisture molecules trapped in the ZIF-8. Compared with ZIF-8 and NH$_2$-ZIF-8, the weight loss of ZIF-8 is much faster than that of NH$_2$-ZIF-8. The second step of the weight loss
about 10% occurred up to 250 °C and it is attributed to the removal of solvents molecules trapped in the ZIF-8. The third step of the weight loss about 20% occurred up to 450 °C and it is attributed to the removal of residual solvents molecules and polyaniline in the ZIF-8. The four step of the weight loss about 30% occurred up to 650 °C and it is attributed to the collapse of the framework with the release of organic materials. The TGA curve of NH2-ZIF-8 obtained by 12% doped content of polyaniline can be seen (the inset in figure 4), the four weightlessness temperatures are 138.94 °C, 224.36 °C, 412.38 °C, 632.71 °C. The four weightlessness rates are 5.04%, 6.94%, 20.94%, 32.13%, and the four DSC are 1.135 W/mg, 1.135 W/mg, 1.135 W/mg, 1.135 W/mg. As the residue from the decomposed ZIF-8 crystal is almost entirely due to the zinc oxide, the average residue of NH2-ZIF-8 after decomposition is 35.93%, and that of ZIF-8 after decomposition is 34.92% (theoretical 35.45%), which is close to theoretical decomposition residue. It can be seen that NH2-ZIF-8 decomposition residue is higher than ZIF-8 decomposition residue, indicating that the introduction of -NH2 improves the binding efficiency of 2-methylimidazole with zinc ions [25]. Compared with the maximum decomposition temperatures of four samples, the thermostability of host framework is increased gradually with the addition of polyaniline. It may be that the introduction of -NH2 improves the thermal stability of ZIF-8 frame. Compared with other studies, the CO2 absorption amount of NH2-ZIF-8 is relatively not very high. This phenomenon is attributed to two reasons [26]. First, this is because the numbers of adsorption sites on the ZIFs increased with the addition of amino functional groups, which also increased the framework mass (1.7%). Second, the initial weight drops at ~150 °C is inapparent compared to ZIF-8, owing to the incorporation of -NH2 that significantly increased the hydrophilicity of NH2-ZIF-8 and thus its ability to adsorb water. Overall consideration, the decomposition temperature of 12% doped content of polyaniline slightly increased to 632.71 °C, suggesting their higher thermal stability.

3.4. SEM analysis of NH2-ZIF-8

Figure 5 presents SEM images of particle shape and microstructure of the ZIF-8 and NH2-ZIF-8. Pure ZIF-8 (figure 5(a)), ZIF-8 modified by ethanolamine (figure 5(b)) and tetraethylenepentamine (figure 5(c)) is cubic or prismatic morphology, with better dispersion and smaller particle size. The samples of ZIF-8 modified by polyaniline is four prism, trapezoid morphology, and the particle size of the sample is almost the same. As show in figures 5(d), (e) and (f), the polyaniline content is 6%, 12% and 18%. It is clearly observable that the average size of NH2-ZIF-8 increases to about 10 μm when polyaniline is introduced, which is the result of (200) crystallographic planes growth. The (200) crystallographic planes growth shown that the synthesized NH2-ZIF-8 crystal is a cubic crystal system. When the content of polyaniline is 6% and 18%, the crystalline morphology of NH2-ZIF-8 is blurred and the content of small particles is increased. The particle size difference is large and the dispersion is poor. When the content of polyaniline is 12%, the particle size of NH2-ZIF-8 changes little, the size of NH2-ZIF-8 tends to be uniform, the structure is clearer and the dispersion is better. It can be seen that polyaniline as a modifier and growth inhibitor plays a role in avoiding timely contact between zinc ions and dimethylimidazole. It controls the nucleation rate of NH2-ZIF-8, inhibits the growth direction of NH2-ZIF-8 on some surfaces, drives its directional growth, and effectively controls the size of NH2-ZIF-8 in the reaction system [27]. Polyaniline is too few to inhibit the growth direction of NH2-ZIF-8 on some surfaces. Too much polyaniline does not drive its directional growth. In addition, x-ray diffraction analysis shown that the
synthesized NH2-ZIF-8 crystal is cubic, and cubic crystals have larger specific surface area. In conclusion, the types of modifiers and appropriate polyaniline content in the reaction system are very important for the synthesis of NH2-ZIF-8 with CO2 adsorption property. Therefore, the optimum doped content is 12%.

3.5. Effects of different modifier on the CO2 adsorption property of NH2-ZIF-8

The CO2 adsorption properties of NH2-ZIF-8 synthesized with tetraethylenepentamine, polyaniline, ethanolamine as modifiers are shown in table 1. As shown in table 1, polyaniline has the best modification effect at the same modifier content. The main reason for this difference is that the BET surface area of polyaniline is the highest (789.7 m2 g⁻¹) and its stability is the highest (632.71 °C) at the same modifier content. Large specific surface area and large pore volume are the reasons for improving CO2 adsorption property. In addition, the drying temperature (90 °C) of NH2-ZIF-8 was close to the volatilization point of polyaniline (82 °C), unstable compounds and polyaniline volatilized during drying, and some metal active sites were obtained, which increased the adsorption capacity of CO2. Compared with the experimental results of pre-modification and post-modification, it is found that the pre-modification data is significantly better than the post-modification data. Therefore, ZIF-8 is modified by pre-modification. The above analysis shown that the modification of ZIF-8 by pre-modification with polyaniline has the best CO2 adsorption property and thermal stability.

In order to further analyze the effect of polyaniline modification on the CO2 adsorption property of the ZIF-8 and NH2-ZIF-8 samples, the results of different contents of polyaniline were shown in figure 6. As shown in figure 6, all samples shown a steep initial increase at lower pressures and a relatively flat increase at higher pressures. The results shown that the CO2 adsorption capacity of ZIF-8 modified with polyaniline was improved by 4%–17% compared with that of unmodified ZIF-8, and the modification effect was obvious. Introducing functional groups into ZIF-8 can effectively change the electrostatic force between ZIF-8 and CO2, thus improving the CO2 adsorption property of ZIF-8 [28]. It can be seen that CO2 adsorption property of ZIF-8

| Material  | Modification method | Modifier | BET surface area/m²g⁻¹ | Micropore volume/cm³g⁻¹ | Pore size/nm |
|-----------|---------------------|----------|-------------------------|--------------------------|-------------|
| ZIF-8     | No                  | No       | 635.2                   | 0.34                     | 1.32        |
| NH2-ZIF-8 | Pre-modification    | TEPA     | 682.3                   | 0.48                     | 1.21        |
|           |                     | MEA      | 710.4                   | 0.52                     | 1.16        |
|           |                     | PANI     | 789.7                   | 0.55                     | 0.99        |
|           | Post-modification   | TEPA     | 654.1                   | 0.43                     | 1.29        |
|           |                     | MEA      | 662.4                   | 0.45                     | 1.19        |
|           |                     | PANI     | 776.9                   | 0.53                     | 0.98        |

Figure 5. SEM of NH2-ZIF-8.
depends on its interaction with CO2. ZIF-8 was modified with -NH2, and some new adsorption sites and new pore structure were formed to enhance the interaction between NH2-ZIF-8 and CO2. This phenomenon is attributed to two reasons. First, the -NH2 have a high affinity toward acidic CO2 mainly through the interaction with the lone electron pair on nitrogen. Second, quadrupole-π electron interaction between CO2 molecule and imidazole ring is strengthened by -NH2 [29]. In conclusion, the introduction of -NH2 functional group is an effective way to improve the CO2 adsorption property of ZIF-8. The CO2 adsorption and desorption curve of NH2-ZIF-8 obtained by 12% doped content of polyaniline can be seen (the inset in figure 6). According to the analysis of figure 6, 12% polyaniline is the best modifier.

4. Conclusion

The effects of pre-modification of polyaniline on the thermal stability and CO2 adsorption property of ZIF-8 were studied. The parallel flow-drop solvothermal method was utilized to synthesize the crystal of NH2-ZIF-8 by taking the molar ratio of the metal ions to the organic ligands of 1:2 for 18 h at 120 °C, 12% doped content of polyaniline. -NH2 was successfully introduced into the surface of ZIF-8 and their structures were confirmed by FTIR, TGA, XRD and BET tests. The polyaniline plays an important role in structure orientation and size control of NH2-ZIF-8 controllable growth. Polyaniline is too few to inhibit the growth direction of NH2-ZIF-8 on some surfaces. Too much polyaniline does not drive its directional growth. Among the NH2-ZIF-8 explored, NH2-ZIF-8 has the best thermal stability and CO2 adsorption property when the doped content is 12%. The improvement of thermal stability and CO2 adsorption property of ZIF-8 have effectively expanded the application field of MOFs materials.

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Conflicts of interest

The authors declare that they have no conflicts of interest.

Author contribution statement

Sheng Wang wrote and substantively revised the main manuscript text. Jiaming Cui completed the experiment and obtained the experimental data. Shengquan Zhang designed the experimental scheme. Xianfei Xie analyzed and explained the data. Wenke Xia analyzed and drafted various charts.
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