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Research Article

Effect of Lanthanum Doping on the Microstructure, Thermal Stability, and CO_2 Adsorption Property of ZIF-8

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The reaction materials $La(NO_3)_3{\cdot}6H_2O$, $Zn(NO_3)_2{\cdot}6H_2O$, and 2-methylimidazole were mixed in a certain proportion. A research process innovatively adopts the parallel flow-drop solvothermal method and a lanthanum-doping method to achieve the synthesis of metal organic frameworks. In this study, we successfully introduce lanthanum into the framework of ZIF-8 to stabilize the spatial structure and improve its performance. The structure and properties of La-ZIF-8 were characterized by the X-ray diffraction (XRD), scanning electron microscope (SEM), thermogravimetry analysis (TGA), and fourier transform infrared spectroscopy (FTIR). The relationship between microstructure stability and macroscopical properties is illustrated. The results show that the doping of lanthanum is beneficial for improving the thermal stability and CO_2 adsorption property of ZIF-8 because of the improvement in the microstructure. The introduction of lanthanum to the ZIF-8 is also beneficial for forming porous frameworks and raising the thermal stability and CO_2 adsorption properties. The crystallinity, structure, morphology, and thermal stability of La-ZIF-8 are optimal at the La content of 2 atom.%.

1. Introduction

Metal-organic frameworks (MOFs) are the new regulable and modifiable porous materials combined by the coordination chemistry and the materials science [1–3]. The diversity of metal elements, organic ligands, and coordination modes leads to the various types and functions of MOFs. Zeolite imidazolate frameworks (ZIFs), a new subclass of MOFs, have attracted significant attention as they combine the advantages from both zeolites and conventional MOFs. ZIFs are the nanoporous materials with the topological structure of zeolite, and ZIFs have many advantages including high specific surface area, diverse structure, regulable pore size, and modifiable framework [4, 5]. ZIFs have been widely applied in various fields including gas storage and separation, optical materials, magnetic materials, catalytic materials, and biology [6, 7]. ZIFs have been a research focus of interdisciplinary areas involving energy, materials, and life science. During the past decade, research works have focused on synthesizing new ZIFs for their applications in gas capture and storage.

One of the most widely researched ZIF materials is ZIF-8, which has high CO_2 selectivity and capacity [8, 9]. However, some ZIF-8 materials also have low thermal stability during the synthesis and drying process because the bond energies of coordination bonds formed between the metal and organic ligands are less than those of covalent bonds and metal bonds. In the removal of a large number of free solvent molecules in the framework, these frameworks with an open structure undergo obvious torsion and deformation, losing part of their crystallinity. Some structures even collapse, destroying the crystal structure of the ZIF-8 material and causing loss of the topological structure forming an amorphous powder [10]. Most of the flue gas in the metallurgical industry and thermal power industry is high temperature, which is an acid-mixed gas containing water, sulfide, nitrides, fluoride, and chloride. This makes the ZIF-8 material unable to achieve its ideal CO_2 adsorption effect. The thermal stability, CO_2 adsorption properties, and excellent selectivity are also important performance parameters for the practical application of ZIF-8 materials. The thermal stability of the reported MOF materials is mostly around 400°C and less than 500°C [11–14].

Therefore, the thermal stability and CO_2 selectivity and adsorption properties of ZIF-8 materials should be further improved, expanding new application fields to meet the needs of the metallurgical industry and thermal power industry. It is therefore highly desirable to develop novel adsorption materials with high uptake capacity for CO_2 and thermal stability of ZIF-8. However, systematic information regarding the thermal stability of ZIF-8 under these conditions remains to be reported. ZIF-8 can be used to adsorb and separate CO_2 from smelting flue gas to improve thermal stability [15].

ZIF-8 has a more flexible framework, which is helpful in the field of surface modification. Recent studies have shown that metal cations doped can significantly enhance gas uptake and thermal stability. For example, increasing the gas-storage capacities of $CuSb_2S_4$ by postsynthetic modification with LiCl [16] shows that the CO_2 uptake of $CuSb_2S_4$ is about 0.63 mmol/g at 273 K, while $LiCl@CuSb_2S_4$ shows a maximum CO_2 uptake of 2.31 mmol/g at 273 K, which is remarkably increased by ~360%, compared with the unmodified chalcogel. The introduction of alkali metal and heavy metal into the ZIFs framework are now well documented [17–20]. Therefore, substitutional introduction of lanthanide cations in the crystalline lattice has far less been studied. A major strategy is to dope ZIF-8 with lanthanide because of its low cohesive energies, which can avoid the clustering problem.

In this paper, the lanthanum doping and parallel flow-drop solvothermal method were used to solve the problem of low thermal stability and CO_2 adsorption property of ZIF-8. We also compared the thermal stability and microstructure of the undoped and lanthanum-doped ZIF-8 under the different conditions to determine the optimum doped content on ZIF-8. To the best of our knowledge, this work is the first report to apply ZIF-8 to adsorb and separate CO_2 materials which expands the range of applications for ZIF-8. In particular, in the treatment of CO_2 adsorption and separation in smelting flue gas, the optimum conditions included an acidic mixture with high temperature and containing water, sulfide, nitride, fluoride, and chloride.

2. Experimental

2.1. Reagents and Instruments. Zinc nitrate, lanthanum nitrate, 2-methylimidazole, *N*,*N*′-dimethylformamide (DMF), and so on were chemically pure. The instruments utilized for the analysis and characterization included a JSM-6700F-type SEM, an IFS-type 66v/S infrared spectrometer, a laser particle analyzer, an X'Pert PRO-type X-ray power diffractometer, and a DSC200F3-type differential scanning calorimeter (DSC).

2.2. Synthesis Method of La-ZIF-8. ZIF-8 was synthesized using the parallel flow-drop solvothermal method by taking zinc nitrate, 2-methylimidazole, and DMF as the zinc source, organic ligand, and organic solvent, respectively. Firstly, defined amounts of $Zn(NO_3)_2{\cdot}6H_2O$ and $La(NO_3)_3{\cdot}6H_2O$ were weighted and dissolved in distilled water, and a certain amount of 2-methylimidazole was weighted and dissolved in DMF solution. A precise concentration of DMF solution was placed in a stainless-steel reactor lined with polytetrafluoroethylene (PTFE) and the mixed solution as the bottom liquid. The solution of zinc nitrate, lanthanum nitrate and 2-methylimidazole was dripped into the bottom liquid, heated to a certain temperature, and stirred vigorously. The drop acceleration of zinc nitrate and 2-methylimidazole was adjusted in this process. The mixture was separated to the liquid and La-ZIF-8 by filtration after the reaction finished. The filter cake was aged for 4 hours using 20 mL of acetone, before being refined and washed by DMF for 3 times and dried at 100°C for 12 hours. Finally, white La-ZIF-8 powder was obtained. The advantage of this method was that the reactant had a high degree of supersaturation by controlling the drop acceleration. In addition, the reaction temperature, time, and pH were easy to control. This new method can provide a higher degree of supersaturation of the solution, the nucleation rate was faster than the growth rate, and the obtained ZIF-8 crystal had a small grain size and high surface area.

3. Results and Discussion

3.1. Synthesis of La-ZIF-8. Solution A consisted of $Zn(NO_3)_2{\cdot}6H_2O$ (5.95 g, 0.02 mol) and $La(NO_3)_3{\cdot}6H_2O$ (0.09 g, 0.2 mmol; 0.17 g, 0.4 mmol; 0.26 g, 0.6 mmol) dissolved in 20 mL distilled water. Solution B was 2-methylimidazole (4.93 g, 0.06 mol) dissolved in DMF. 20 mL DMF solution was placed in a reactor as the bottom liquid. Then, a mixed solution of A and B was dripped into the bottom liquid, heated to a certain temperature, and stirred vigorously. The drop acceleration of solution A and solution B was adjusted in this process. The synthesis conditions involved 80°C with stirring for 1 hour. Then, the La-ZIF-5 samples were reacted at different temperatures (100, 120, 140, 160, and 180°C) in a programmable oven for 14 hours, before being cooled to room temperature naturally. The material was dried at 100°C for 12 hours prior to analysis.

3.2. TGA of La-ZIF-8. Using a molar ratio of the metal ions to the ligands of 1 : 3 at 160°C, with a lanthanum content of 2 atom.%, thermal stability of ZIF-8 was studied by TGA. From the TGA curve of La-ZIF-8 presented in Figure 1, it can be seen that weight loss before 100.32°C was caused by the decomposition of crystalline water in ZIF-8. The corresponding endothermic enthalpy was 0.4916 mW/mg. The weight loss at 286.65°C and 331.30°C was caused by the decomposition and desorption of *N*,*N*′-dimethylformamide. The corresponding endothermic enthalpies were 0.2747 mW/mg and 0.5211 mW/mg. At 508.94°C, ZIF-8 began to collapse and showed an obvious endothermic peak with a corresponding enthalpy of adsorption of 1.06 mW/mg. After decomposition, the residual amount was 36.57% (the theoretical quantity is 35.45%), which was close to the theoretical decomposition residue. This showed that the initial decomposition temperature for La-ZIF-8 remained stable at 528.36°C, and the purity was high.

3.3. XRD Analysis of La-ZIF-8. Figure 2 shows the XRD patterns of undoped and lanthanum-doped ZIF-8. The XRD

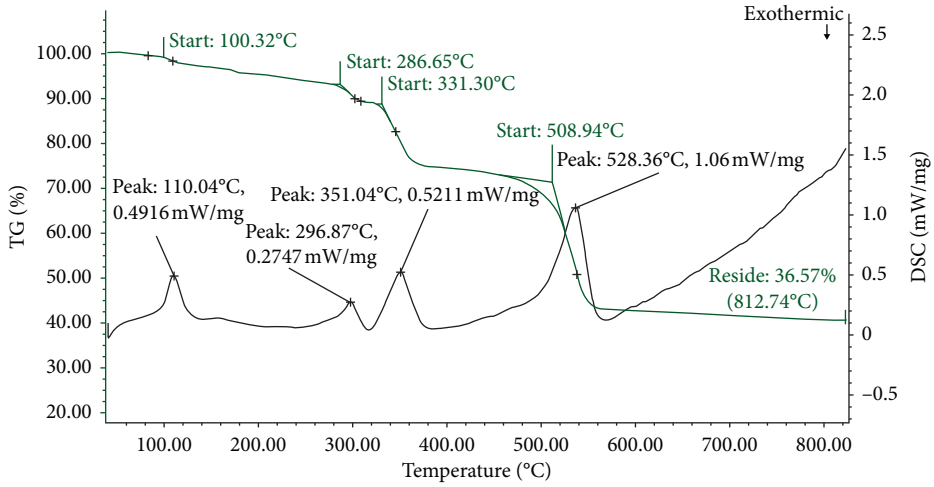

Figure 1: TGA curve of La-ZIF-8.

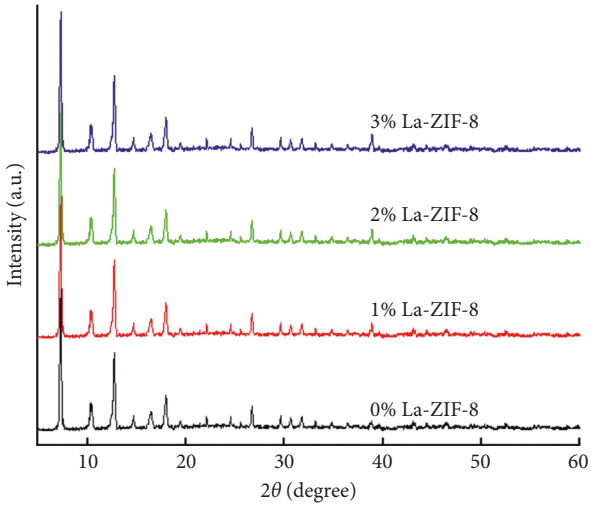

Figure 2: XRD patterns of ZIF-8 and La-ZIF-8.

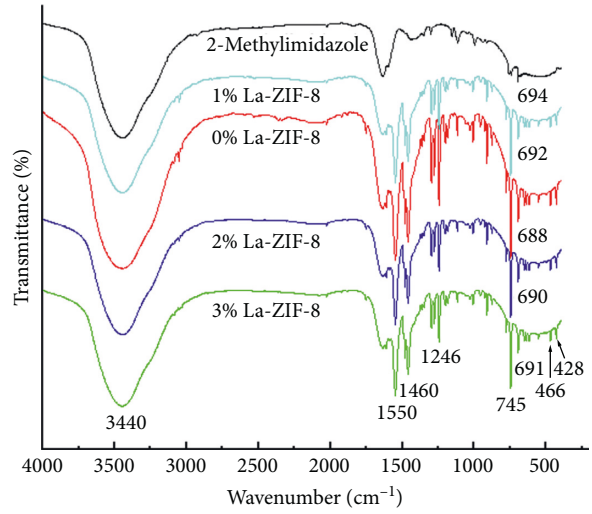

Figure 3: FTIR spectra of ZIF-8 and La-ZIF-8.

pattern of La-ZIF-8 matched that reported in previous literatures [21]. The main diffraction peaks $2\theta = 7.3^{\circ}$, 10.3°, and 12.7° indicated the La-ZIF-8 cubic lattice. The characteristic peaks of ZIF-8 synthesized under different lanthanum-doped contents were small and had typical characteristic peaks of ZIF-8. In addition, all peaks of lanthanum-doped ZIF-8 showed an obvious change in the baseline at $2\theta = 38.9^{\circ}$, corresponding to the most important diffraction signal in the patterns. This indicated that doping with lanthanum ions introduced to lattice expansion in the doped ZIF-8. The doped lanthanum ions should be well incorporated into the framework and substitute partial Zn(II) ions in the $C_8H_{10}N_4Zn$, as observed in the lanthanum-doped ZIF-8. In addition, there was no significant loss of crystallinity in X-ray diffraction patterns, and no supplementary Bragg peaks appear. The introduction of lanthanum into the ZIF-8 causes no other extra peaks associated with a secondary phase such as La_2O_3 and lanthanum nitrate [22]. In contrast, the intensities of the diffraction peak at the doped content of 1 atom.% and 3 atom.% were weakened, probably due to a small number of bond breakages during drying or synthesis of La-ZIF-8, which indicated that the optimum doped content was 2 atom.%. The results strongly implied that doped lanthanum ions into the framework could enhance the structural stability of ZIF-8.

3.4. FTIR Analysis of La-ZIF-8. As demonstrated in the infrared spectra of 2-methylimidazole, undoped and lanthanum-doped ZIF-8 in Figure 3. For example, the adsorption peak at 3440 cm^{-1} was attributed to the aromatic and aliphatic C-H stretch of the imidazole in the ZIF-8 structure. The adsorption peak at 1550 cm^{-1} was caused by the C-N stretch mode. The adsorption peak in the spectral region of 600–1500 cm^{-1} is associated with the entire ring stretching or bending, while the band at 428 cm^{-1} and 466 cm^{-1} is ascribed to the Zn-N stretch; this stretching vibration peak is confirmed by red shift. It is showed that the acid is completely protonated, and the Zn^{2+} and the 2-methylimidazole formed ZIF-8 [23]. Meanwhile, compared with the infrared spectra of 2-methylimidazole, undoped

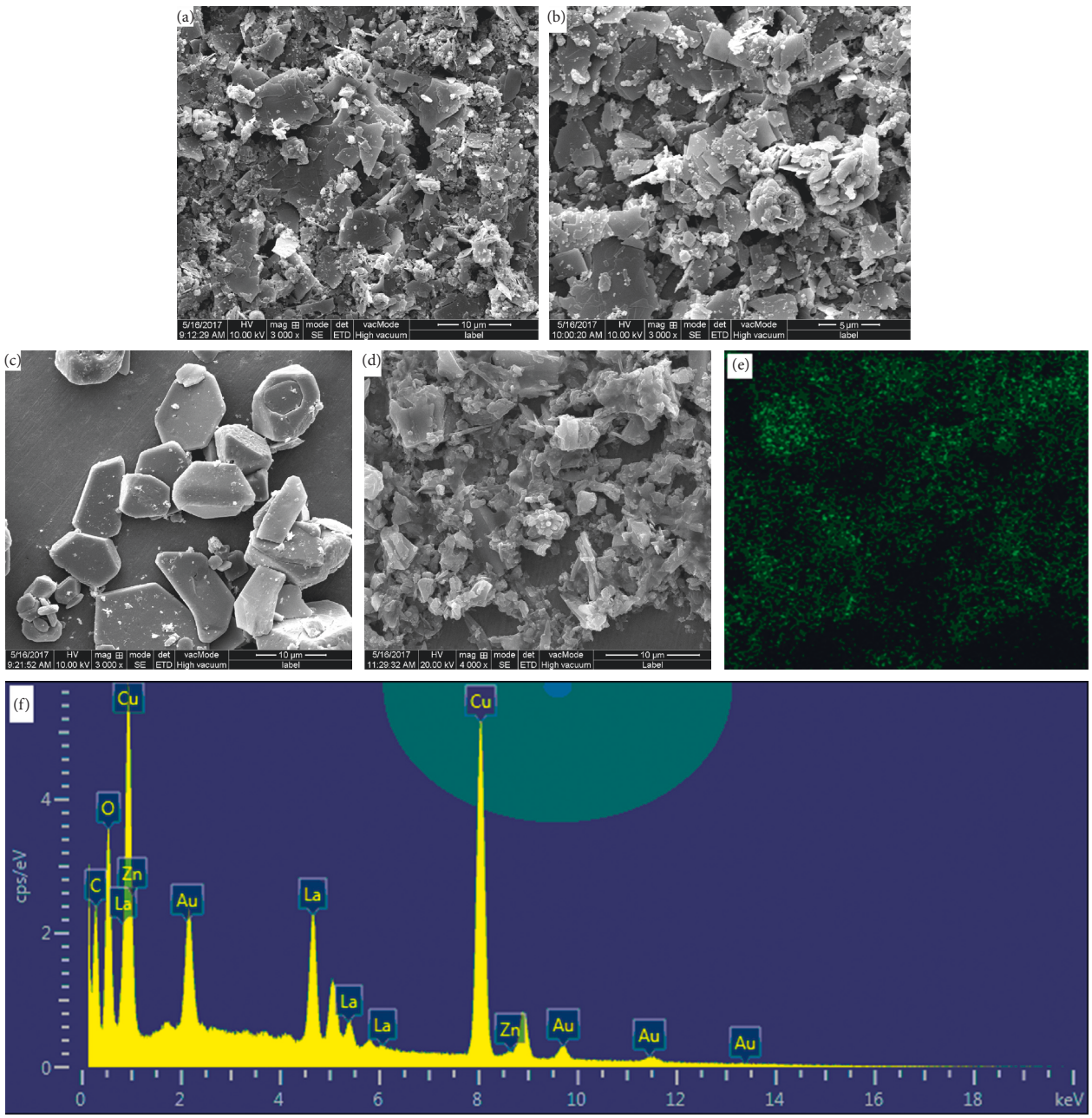

Figure 4: SEM of La-ZIF-8.

and lanthanum-doped ZIF-8 at 694–688 cm^{-1}, this stretching vibration peak is confirmed by red shift. It is shown that the 2-methylimidazole is completely deprotonated, and the Zn_{2+} and the 2-methylimidazole formed ZIF-8. The peaks at 1550–1246 cm^{-1} of 1 atom.% and 3 atom.% were relatively weak, wide, and defective. This indicates that a small content of bond breaks led to a decrease in stability. Therefore, the optimum doped content was 2 atom.%.

3.5. SEM Analysis of La-ZIF-8. Further investigation was conducted on the microstructures of La-ZIF-8 at a molar ratio of the metal ions to the organic ligands of 1 : 3 at 160°C and the lanthanum content of 1, 2, and 3 atom.%. As shown in the SEM of La-ZIF-8 in Figures 4(a) and 4(b), the lanthanum content was 1 and 3 atom.%, and the synthesized materials presented layered growth with an incomplete and broken lamellar structure. The size of the crystal varied from 0.5 to 5 μm. As shown in Figure 4(c), the lanthanum content was 2 atom.%. The synthesized crystal had a complete structure consisting of cubic blocks and hexahedrons with size ranging between 2 and 12 μm. The increase of cubic blocks could increase the thermal stability of the ZIF-8 material frame structure. The results were in match with the cubic lattice of La-ZIF-8 in XRD analysis. To further confirm the chemical compositions of La-ZIF-8, the energy dispersive spectroscopy (EDS) surface scanning analysis was

carried out, as shown in Figure 4(d). The EDS spectrum confirmed the presence of lanthanum in the La-ZIF-8 (Figures 4(e) and 4(f)). Lanthanum elemental mapping shows a homogeneous distribution of lanthanum ions, and the lanthanum content was 1.9 atom.%. This indicated that it was beneficial for synthesizing ZIF-8 when the proportion of lanthanum ions was at the stoichiometry required by the coordination [24, 25].

3.6. The CO_2 Adsorption Property of La-ZIF-8. As shown in Figure 5, all of the samples show a steep initial increase at low pressures and saturation at higher pressures. This was characteristic of the microporous material with high amounts of CO_2 adsorption. The sample prepared with the lower lanthanum content has the smallest Langmuir specific surface areas. The low Langmuir specific surface areas of La-ZIF-8 of this work may be due to the low dimension. The sample prepared with the higher lanthanum content has the largest Langmuir specific surface areas. The large Langmuir specific surface areas of La-ZIF-8 of this work may be due to the multidimension. The results show that the dimensionality of La-ZIF-8 increased when the lanthanum content was raised, which can be explained that the gas adsorption property of La-ZIF-8 microcrystals can vary according to the crystal morphology or exposed surfaces. Figure 5 shows the small SEM image at the lanthanum content of 2 atom.%. Particle morphology, size, and CO_2 adsorption property proved that the optimum doped content of lanthanum had promoted the coordination of the metal ions and organic ligands, and it was beneficial for synthesizing the crystal La-ZIF-8. Therefore, the decrease of crystal size induces the increase in the crystal surface area and leads to the large amount of CO_2 adsorption in the relative high-pressure region, which has important potential application in gas adsorption and storage. The result also suggests that a certain lanthanum content may be beneficial for forming multidimensional frameworks with large voids. The optimum doped content was 2 atom.%.

3.7. Effect of Doped Content on Thermal Stability of La-ZIF-8. The synthetic La-ZIF-8 process condition was the molar ratio of the metal ions to the organic ligands of 1 : 3, and the thermal stability of undoped and lanthanum-doped ZIF-8 studied by TGA is shown in Figure 6. The results show that the thermal stability of undoped and lanthanum-doped ZIF-8 increased at first and then decreased with increasing reaction temperature under different doped contents. The doped content had an effect on the thermal stability of ZIF-8 when the reaction temperature ranged from 100 to 180°C. With increasing reaction temperature, the probability of collision between the reactants increased. A large number of crystal nuclei instantly reacted and generated in this process. At this point, the activation energy of the grain growth was small, and the grains easily grew. The degree of crystallization of ZIF-8 was improved with more complete grain growth and better thermal stability [26–29]. When the reaction temperature is higher than 160°C, the grain growth needs to overcome the energy barrier, requiring a larger

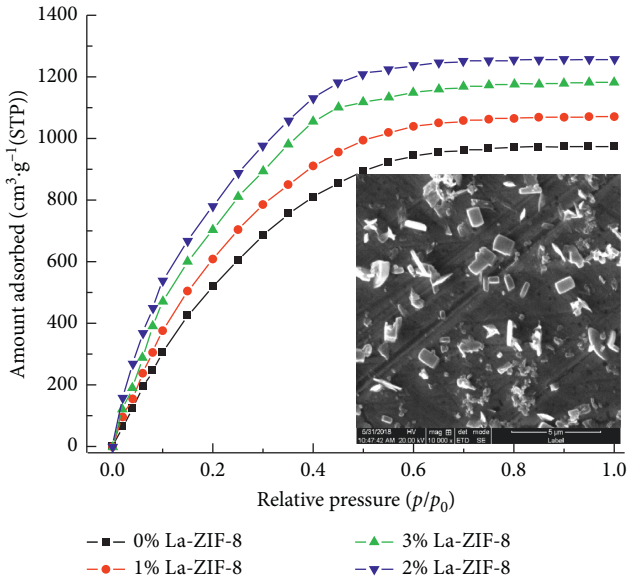

Figure 5: The CO_2 adsorption property of ZIF-8 and La-ZIF-8.

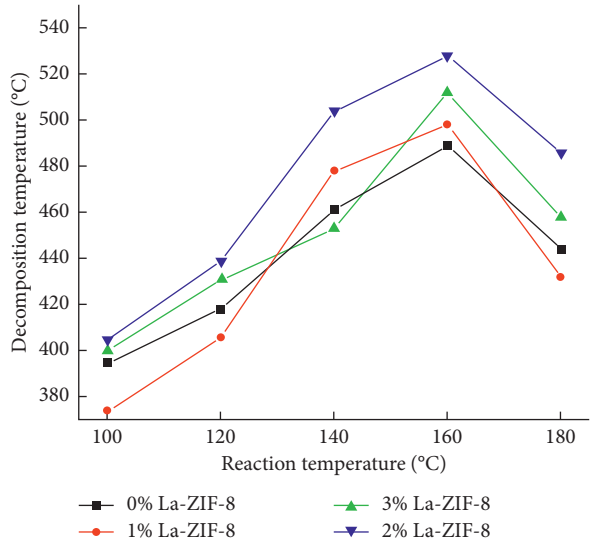

Figure 6: Thermal stability of ZIF-8 and La-ZIF-8.

activation energy. Crystals nucleation and grain growth were relatively difficult, and ZIF-8 growth was not complete with reduced thermal stability [30–32]. These clearly indicate that the thermal stability of ZIF-8 increased gradually with the addition of $La(NO_3)_3{\cdot}6H_2O$. Based on this result, it was confirmed that the ZIF-8 structure was more stable under a lanthanum content of 2 atom.%.

4. Conclusions

The parallel flow-drop solvothermal method was utilized to synthesize the crystal of La-ZIF-8 by taking the molar ratio of the metal ions to the organic ligands of 1 : 3 at 160°C and the lanthanum content of 2 atom.%. SEM showed that the La-ZIF-8 endowed with the cubic blocks and hexahedrons structure. XRD showed that the doped lanthanum ions were

incorporated into the framework and partially substituted Zn(II) ions in $C_8H_{10}N_4Zn$. TGA showed that it was thermally stable up to a temperature of 528.36°C. FTIR showed that the acid was completely protonated. Zinc ions, lanthanum ions, and 2-methylimidazole formed La-ZIF-8. In addition, an O-La adsorption peak also existed in ZIF-8. The thermal stability and CO_2 adsorption of ZIF-8 increased by doped lanthanum ions.

Data Availability

The data used to support the findings of this study are included within the article.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

Acknowledgments

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