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Research Article

Preparation of Schiff Base Derived from 3,4-Dimethoxybenzaldehyde and *p*-Aminobenzoic Acid by Supersonic Speed Gas Impacting Method

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A Schiff base derived from 3,4-dimethoxybenzaldehyde and *p*-aminobenzoic acid (SBDA) was synthesized by novel supersonic speed gas impacting method. The morphology and geometric structure of the synthesized Schiff base SBDA were investigated, the results showed that the particles of SBDA were not completely regular and the HOMO and LUMO of SBDA optimized geometry structure using the semiempirical method PM3 were −9.115 eV and −1.191 eV, respectively, and some bond length of synthesized SBDA became short and bone angle became large compared to the reactant. The thermogravimetric analysis results indicated that SBDA began to decompose above 280°C; the decomposition temperature and thermogravimetric rate could vary with the different heating rate.

1. Introduction

Schiff bases, as the most widely used organic compounds, have been widely used in synthesis of intermediates [1, 2], biological actions [3, 4], polymers [5, 6], and so forth and obtained a lot of progress. Schiff bases have been shown to exhibit a broad range of biological activities. Chen et al. [7] reported that a series of Ru(III) tetrahydro-Schiff base complexes were encapsulated in the supercages of zeolite Y by flexible ligand method. Upon encapsulation in zeolite Y, Ru(III) tetrahydro-Schiff base complexes exhibited higher activity for the hydrogenation of benzene than the corresponding Ru(III)-Schiff base complexes. This indicates that hydrogenation of the C=N bond of Schiff base ligands led to a modification of the coordination environment of the central Ru(III) cations. Nair et al. [8] reported that Co(II), Ni(II), Cu(II), and Zn(II) complexes of the Schiff base derived from indole-3-carboxaldehyde and *m*-aminobenzoic acid were synthesized. The electronic spectral and magnetic moment results indicated that Co(II) and Ni(II) complexes had tetrahedral geometry, while Cu(II) complex was square planar. The results of antimicrobial activity of the synthesized ligand and its complexes show that the metal complexes were found to be more active than the ligand.

Up to now, many methods for the synthesis of Schiff bases had been described; the most widely used method involves the condensation of a carbonyl compound with an amine by liquid reaction, and other innovations and new techniques including grinding, high energy ball mill, and microwave irradiation have been reported with development of science technology in recent years. In our previous work, we had reported a novel solvent-free synthesis method using supersonic speed gas impacting method, and some compounds including Schiff bases were synthesized by this method [9–12]. To synthesize more compounds and confirm the advantage of this technology, in this paper, we synthesized

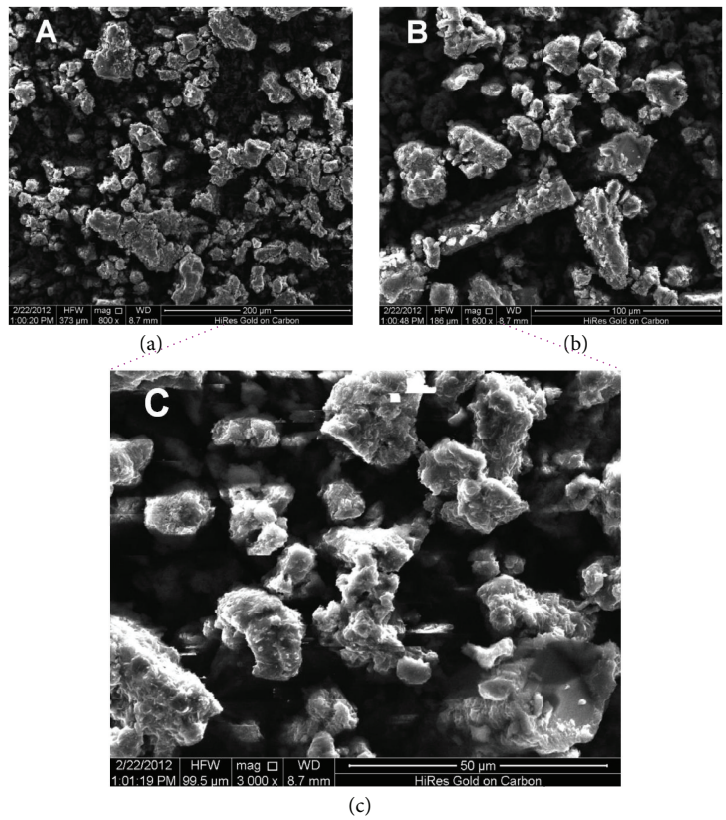

Figure 1: SEM of SBDA.

Schiff base derived from 3,4-dimethoxybenzaldehyde and *p*-aminobenzoic acid (SBDA) by supersonic speed gas impacting method, and the morphology, geometric structure, and thermal stability of synthesized SBDA were investigated.

2. Experimental Section

2.1. Materials. 3,4-Dimethoxybenzaldehyde and *p*-aminobenzoic acid in this study were of analytical grade (AR).

2.2. Synthesis of SBDA. The type synthesis process of Schiff base has been described in our previous paper [9–12], and the SBDA was synthesized as shown in Scheme 1. 3,4-Dimethoxybenzaldehyde and *p*-aminobenzoic acid, in a 1 : 1 molar ratio, were mixed; then the mixture was accelerated to supersonic and reacted at a fixed target. The product was collected after quantitative reaction and dried in a vacuum at 60°C. IR (KBr, V_{max}, cm^{-1}): 3455.1 (O–H), 2836.2 (C–H), 1674.4 (C=O), 1625.9 (N=C), 1577.3, 1510.1, 1452.7 (Ar), 1168.4 (C–O), 809.5, 771.9 (Ar–H); ^{1}H NMR (d6-DMSO, 500 MHz, ppm) δ: 12.06 (s, 1H, Ar–COOH), 8.54 (s, 1H, CH=N), 6.58~7.63 (m, 7H, Ar), 3.86 (s, 6H, Ar–OCH_3).

2.3. Characterization. Fourier transform infrared spectra (FT-IR) were recorded on a Bio-Rad FTS135 spectrophotometer from 4000 to 400 cm^{-1}. The sample of SBDA was mixed with KBr powders and pressed into a disk suitable for IR measurement.

The ^{1}H nuclear magnetic resonance (^{1}H NMR) of SBDA was recorded on Brucker AVANCE 300 spectrometers. The solvent was dimethyl sulphoxide (DMSO).

The morphologies of SBDA were examined by XL-30 ESEM FEG, Philips, in 15–20 kV accelerating voltage (tungsten filament).

Thermogravimetric analysis (TGA) was performed using a simultaneous thermal analysis Q500 (TA instrument USA) with a heating ramp of 5, 10, and 20°C/min under nitrogen flow (50 mL/min) from room temperature to 600°C.

3. Results and Discussion

3.1. Morphology and Geometric Structure of SBDA. Morphology of synthesized SBDA was observed by SEM (B and C are the partly amplificatory images of A, resp.). As seen in Figure 1, the particles of SBDA are not completely regular. However, most of the particles are column or lump, and the thickness of lump particles is about 10 μm. It is a fact that there exist large and small particles, resulting from uneven mixing during reaction and purification or van der Waals and Coulombic forces between the particles. The results of SEM also indicate that it is difficult to control the morphology of synthesized compound by supersonic speed gas impacting method.

The optimized geometric structure of SBDA was carried out by the theoretical calculation, and the calculations were performed with the program VAMP using

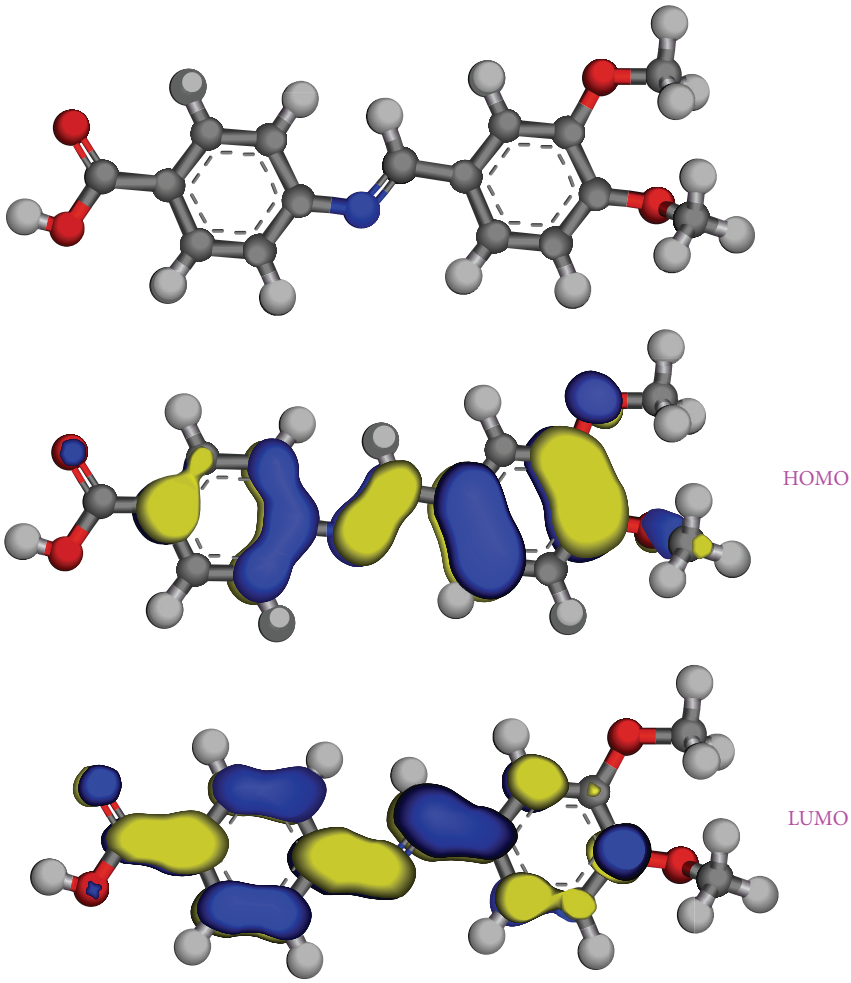

Figure 2: Optimized geometric structure, HOMO, and LUMO of SBDA.

Scheme 1: Synthesis of SBDA.

the semiempirical method PM3 as our previous work [13]. The optimized geometry structure of SBDA is shown in Figure 2, and the HOMO and LUMO of SBDA are −9.115 eV and −1.191 eV, respectively. As shown in Figure 2, the active electronics of HOMO focus on side of C=N, especially the benzene of 3,4-dimethoxybenzaldehyde. However, the electronics receptor of LUMO disperses in the whole compound compared to the active electronics of HOMO.

Some bond lengths and bond angles of optimized SBDA were listed in Table 1. Compared to the bond length of C3–C5 of 3,4-dimethoxybenzaldehyde, the bond length of C3–C5 of the synthesized SBDA decreases from 1.485 nm to 1.466 nm, and the bond angle of ∠H4C3C5 of the synthesized SBDA is larger than that of 3,4-dimethoxybenzaldehyde. These results indicate that *p*-aminobenzoic acid makes C3–C5 of 3,4-dimethoxybenzaldehyde extruded and the geometry structure becomes wide. Meantime, the bond length of C1–N2 of the synthesized Schiff base decreases from 1.423 nm to 1.432 nm compared to that of *p*-aminobenzoic acid.

Table 1: Some bond lengths and bond angles of SBDA.

Bond length/Å		Bond angle/°	
C1–N2	1.432	∠C1N2C3	121.844
N2–C3	1.297	∠N2C3H4	121.441
C3–H4	1.109	∠H4C3C5	117.352
C3–C5	1.466		

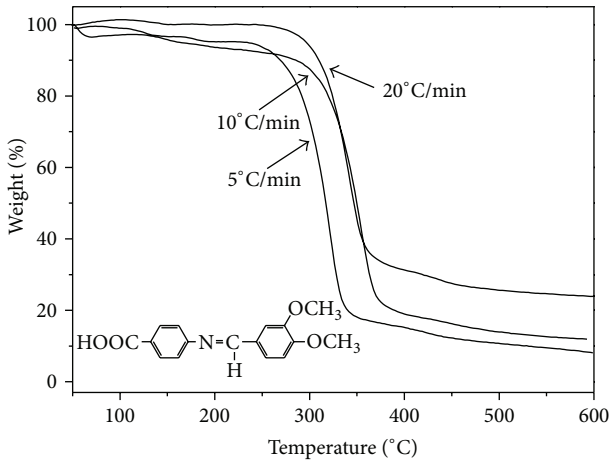

Figure 3: TGA of SBDA with different heating rate.

3.2. Thermal Properties of SBDA. Usually, thermal properties of materials can obviously affect the application; thus, it is necessary to investigate the thermal properties of compound. Figure 3 shows the thermal stability of SBDA with a heating ramp of 5, 10, and 20°C/min under nitrogen flow from room temperature to 600°C. As seen in Figure 3, SBDA begins to decompose above 280°C, which suggests that SBDA has good thermal stability under mild conditions, and it is clear that the decomposition temperature increases with the increasing of heating rate; similar results can be found in decomposition process of other compounds [9, 10, 14, 15]. The reason of this phenomenon is that decomposition of SBDA is not achieved at set temperature because of rapid heating rate. Meanwhile, the program temperature has got into the following set temperature, resulting in decomposition achieving at higher temperature. The results of decomposition process significantly indicated that the decomposition temperature and thermogravimetric rate of SBDA are affected by heating rate.

4. Conclusion

In this paper, a Schiff base derived from 3,4-dimethoxybenzaldehyde and *p*-aminobenzoic acid was successfully synthesized by novel supersonic speed gas impacting method. We investigated the morphology, geometric structure, and thermal stability of synthesized Schiff base, SEM showed the particles of SBDA were not completely regular, and the HOMO and LUMO of SBDA were −9.115 eV and −1.191 eV, respectively. TGA result indicated good thermal stability of SBDA under mild conditions.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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