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Synthesis, Morphology and Thermal Decomposition of Schiff Base Derived from *m*-Hydroxybenzaldehyde and *p*-Aminobenzoic Acid

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Schiff base derived from *m*-hydroxybenzaldehyde and *p*-aminobenzoic acid was synthesized using improved jet milling: rapid efficient reaction without environment pollution and wastes. The compound had been characterized by FT-IR, ^{1}H NMR techniques. SEM showed the Schiff base was rodlike and the average length of the rodlike particles was 1.5 μm, the average width was 250 nm. Accordingly, the aspect ratio of the particles was 6. The thermal stability of Schiff base derived from *m*-hydroxybenzaldehyde and *p*-aminobenzoic acid was investigated by TGA thermal analyzer. The result showed that the Schiff base appeared the maximum decomposition rate from 225-300 ºC and the decomposition reached about 40 %.

Key Words: *m*-Hydroxybenzaldehyde, *p*-Aminobenzoic acid, Thermal decomposition, Jet milling.

INTRODUCTION

With rapid development of science and technology, more and more solvent-free reaction methods were developed[1-6]. The solvent-free reaction has particular advantages *i.e.*, no environment pollution, simple manipulation, high selectivity and productivity, low cost, *etc*.[7]. In the past, the jet milling was just used to produce powder-materials with new and interesting properties. In previous work[5,6], we had reported that the jet milling was improved to applied in chemistry reaction and the solvent-free reaction using improved jet milling not only involves mild conditions, a simple operation and short reaction time, but also gives quantitative yield without waste producing work-up procedures.

Schiff base is very important organic compound due to their facile synthesis and wide applications[8-10]. The previous main synthesis of Schiff base was achieved by liquid reaction[11]. In recent year, grinding[12] and high energy ball mill[13] also were used to synthesize Schiff base. In this paper, we successfully synthesized Schiff base derived from *m*-hydroxybenzaldehyde and *p*-aminobenzoic acid using improved jet milling and the morphology and thermal decomposition of the Schiff base were investigated.

EXPERIMENTAL

All materials used in this study were of analytical grade (AR). The *p*-aminobenzoic acid was purchased from Beijing Chemical Reagents Company (Beijing, China) and the *m*-hydroxybenzaldehyde was purchased from Mianyan Rongshen Chemical Reagents Company (Sichuan Province, China). The reaction equipment is designed and shown in Fig. 1.

Synthesis of Schiff base: Schiff base derived from *m*-hydroxybenzaldehyde and *p*-aminobenzoic acid was prepared as follows: *p*-aminobenzoic acid and *m*-hydroxybenzaldehyde, in a 1:1 molar ratio, were mixed and the specific reaction process have described in previous paper[6]. The product was collected and dried in a vacuum at 90 ºC (Fig. 2).

Schiff base tests (attribute): The infrared spectra were measured on Nicolet 380 FT-IR spectrometer, in the range 4000-400 cm^{-1}. The ^{1}H NMR was recorded on Brucker AVANCE 300 spectrometers. A S4800 scanning electron microscope at 15 kV voltage (Hitachi, Ltd. Japan) was used to determine the size and morphology of the particles. TGA thermal analyzer was performed using a simultaneous thermal analysis Q500 (TA instrument USA) with a heating ramp of 2.5, 5, 10, 15, 20, 30 ºC/min under nitrogen flow (50 mL/min) from room temperature to 500 ºC.

RESULTS AND DISCUSSION

Structure of Schiff base: The FTIR spectra of Schiff base derived from *m*-hydroxybenzaldehyde and *p*-aminobenzoic acid is shown in Fig. 3. In the spectra, the peaks at 3392.9 and 1285.7 cm^{-1} contribute to the absorption of O-H stretching vibration and the plane bending vibration; the absorption peak at 1683 cm^{-1} contribute to C=O stretching vibration of COOH;

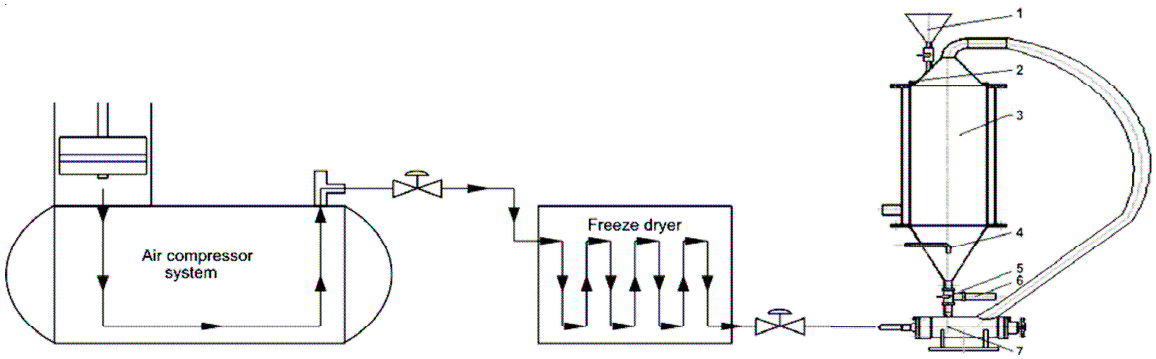

Fig. 1. Schematic drawing of the reaction. 1-Loading hopper; 2-delivery valve; 3-circulation collecting system; 4-adjustable jet pipeline; 5-tee globe valve; 6-outlet port; 7-reaction zone

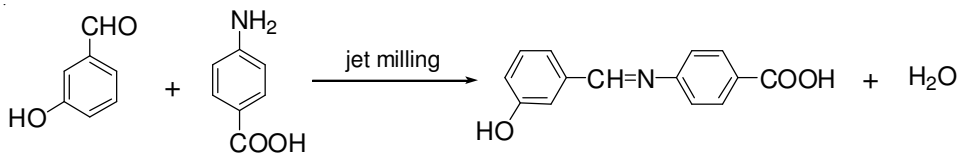

Fig. 2. Synthesis of Schiff base

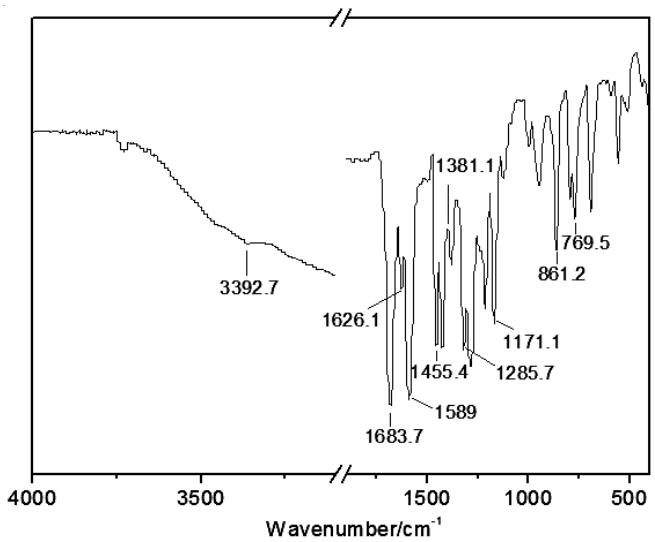

Fig. 3. IR spectra of Schiff base derived from *m*-hydroxybenzaldehyde and *p*-aminobenzoic acid

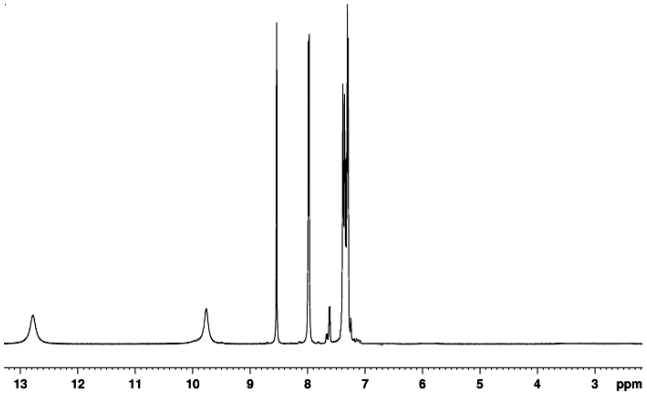

Fig. 4. ^{1}H NMR spectra of Schiff base derived from *m*-hydroxybenzaldehyde and *p*-aminobenzoic acid

owing to conjugated effect, C=N move to lower frequency. Thus, the absorption peak at 1626.1 cm^{-1} contribute to C=N stretching vibration. Then the absorption peaks at 1589 cm^{-1}, 1455 cm^{-1} prove the existence of benzene. The absorption peak at 1171.1 cm^{-1} contribute to C-O stretching vibration of phenol and at 1381.3 cm^{-1} contribute to C-O stretching vibration of COOH, the absorption peak at 861 and 769 cm^{-1} belong to C-H deformation vibration of benzene.

The ^{1}H NMR patterns of Schiff base derived from *m*-hydroxybenzaldehyde and *p*-aminobenzoic acid is depicted in Fig. 4. The single peak at δ_H = 8.53 is CH=N proton resonance peak; the peak at δ_H =12.78 contribute to COOH proton resonance; the single peak at δ_H = 9.76 was Ar-OH proton resonance peak and the multiple peaks at δ_H = 7.3-7.99 are proton resonance peaks of benzene. NMR analysis further confirm that Schiff base derived from *m*-hydroxybenzaldehyde and *p*-aminobenzoic acid was prepared.

Morphology of Schiff base: SEM images (Fig. 5) of the Schiff base particles (B is the partly amplificatory images of A, respectively). The SEM image shows that the particles of the Schiff base are regular. From Fig. 5B, the morphology of most Schiff base is rodlike and the average length of the rodlike particles is 1.5 μm, the average wide is 250 nm. Accordingly, the aspect ratio of the particles is 6, which indicates that crystal growth habit is a linear direction. At the same time, it is clear that there are little larger particles occurred as agglomerates of smaller ones resulting from van der Waals and coulombic forces between the particles. This is due to improved jet milling's crushing effect during reaction. The Schiff base derived from *m*-hydroxybenzaldehyde and *p*-aminobenzoic acid, which was prepared by solvent-free reaction using jet milling, had not only regular morphology, but also excellent distribution.

Thermal decomposition of Schiff base: Fig. 6 shows the TGA curves of Schiff base with a heating ramp of 2.5, 5, 10, 15, 20, 30 ºC/min under nitrogen flow from room temperature to 500 ºC. As seen in Fig. 6, Schiff base derived from *m*-hydroxybenzaldehyde and *p*-aminobenzoic acid appears the

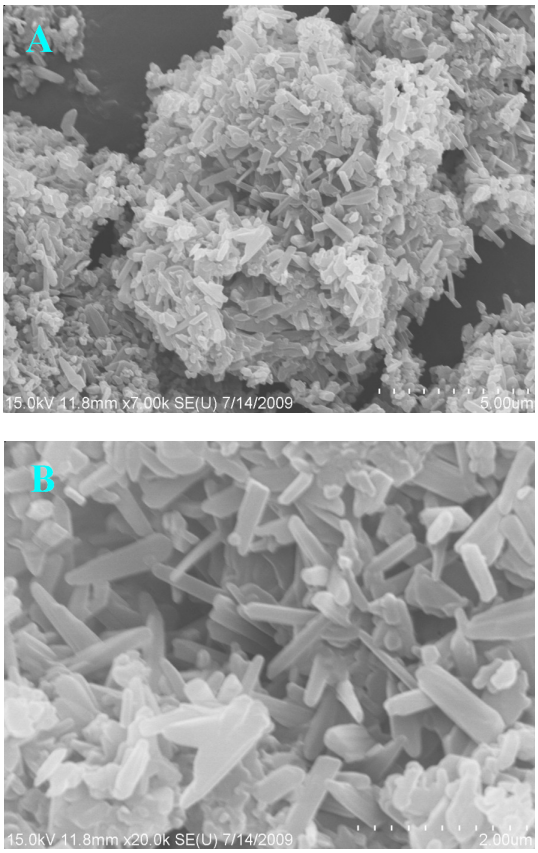

Fig. 5. SEM of Schiff base derived from *m*-hydroxybenzaldehyde and *p*-aminobenzoic acid

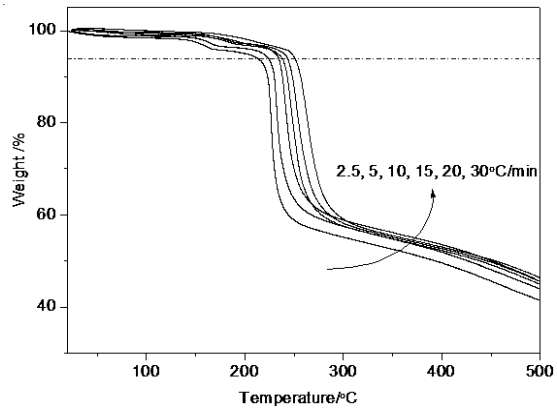

Fig. 6. TGA curves of Schiff base derived from *m*-hydroxybenzaldehyde and *p*-aminobenzoic acid

maximum decomposition rate from 225-300 ºC at different heating rate and degree of decomposition reaches *ca.* 40 %. Then, the decomposition rate becomes slow. What is more, with the increasing of heating rate, the decomposition temperature increases and heating decomposition degree decreases, the reason is that rapid heating rate makes decomposition of Schiff base not achieve at set temperature, at the same time, the temperature has get into the following set temperature, resulting in decomposition achieving at higher temperature.

Conclusion

Schiff base derived from *m*-hydroxybenzaldehyde and *p*-aminobenzoic acid was synthesized by rapid efficient solvent-free reaction. SEM showed the Schiff base was rodlike and the average length of the rodlike particles was 1.5 μm, the average size was 250 nm. Accordingly, the aspect ratio of the particles was 6. The research on thermal stability of the Schiff base showed that the Schiff base appeared the maximum decomposition rate from 225-300 ºC and the decomposition reached about 40 %.

REFERENCES

1. F. Constabel and K.E. Geckeler, *Tetrahedron Lett*., **45**, 2071 (2004).
2. V. Polshettiwar and R.S. Varma, *Tetrahedron Lett*., **49**, 2661 (2008).
3. D. Michaud, J. Hamelin, F. Texier-Boullet and L. Toupet, *Tetrahedron*, **58**, 5865 (2002).
4. E. Colacino, P. Nun, F.M. Colacino, J. Martinez and F. Lamaty, *Tetrahedron*, **64**, 5569 (2008).
5. Y.-H. Cai, R.-F. Peng, S.-J. Chu and J.-B. Yin, *Asian J. Chem*., **22**, 5835 (2010).
6. Y.-H. Cai, D.-M. Ma, R.-F. Peng and S.-J. Chu, *S. Afr. J. Chem*., **61**, 12 (2008).
7. C.F. Jin, S. Jing and X.Q. Xin, *Chin. J. Inorg. Chem*., **18**, 859 (2002).
8. M.H. Klingele and S. Brooker, *Coord. Chem. Rev*., **241**, 119 (2003).
9. M.S. Costa, N. Boechat, E.A. Rangel, F.C. da Silva, A.M.T. de Souza, C.R. Rodrigues, H.C. Castro, I.N. Junior, M.C.S. Lourenco, S.M.S.V. Wardell and V.F. Ferreira, *Bioorg. Med. Chem*., **14**, 8644 (2006).
10. M.S. Karthikeyan, D.J. Prasad, B. Poojary, K.S. Bhat, B.S. Holla and N.S. Kumari, *Bioorg. Med. Chem*., **14**, 7482 (2006).
11. G. Cevasco and S. Thea, *J. Org. Chem*., **64**, 5422 (1999).
12. C. Wang, Y.Q. Zhang, J.C. Li, G.S. Li, X.L. Li, Z. Wang and Y.M. Li, *Chin. J. Org. Chem*., **25**, 1135 (2005).
13. G. Kaupp, J. Schmeyers, M.R. Naimi-Jamal, H. Zoz and H. Ren, *Chem. Eng. Sci*., **57**, 763 (2002).