New Method for Extracting and Purifying Dihydromyricetin from Ampelopsis grossedentata

Hongchao Hu, Fan Luo, Mingjie Wang, Zhihuan Fu, and Xugang Shu*

ABSTRACT: Dihydromyricetin (DMY) is a kind of flavone. It has a variety of physiological effects, and its content in Ampelopsis grossedentata is as high as 35%. There are two shortcomings in the traditional batch extraction process commonly used in a laboratory: long extraction time and low extraction rate. In this study, a new chelating extraction method was proposed, that is, ZnCl₂ was introduced into the extraction and purification process to chelate with DMY, and the yield and purity were taken as evaluation indices for a comparative study with the traditional batch extraction method. In addition, ¹H NMR, single-crystal X-ray diffraction, IR, and UV were used to analyze the product structure; thermogravimetry and differential thermal analysis was utilized to examine the thermal stability of DMY. The results were shown as follows. Compared with the batch extraction method, the chelation extraction method could effectively avoid the oxidation of DMY by air during the extraction and purification process, and the yield of the DMY also increased. Furthermore, this method was time-saving. Through investigating the extraction process and characterizing the structure and thermal stability of DMY, the chelation extraction method could be considered to provide a reference for commercial applications of DMY.

1. INTRODUCTION

Many natural active compounds have the pharmacological effects of chemical compounds such as antibiotics and hormones, in addition they are green and nontoxic, with low residue, and without drug resistance. Therefore, they can play an increasingly important role in the area of medicine, food, animal husbandry, and so on. Ampelopsis grossedentata has been used as a herbal medicine for thousands of years in China. It was regarded to have the effects of “regulating Zhong, replenishing Qi and circulating blood and Qi” in the “Compendium of Material Medica.” The active component in A. grossedentata is dihydromyricetin (DMY), which is as high as 35% in young stalks and leaves. DMY has remarkable characteristics of antioxidation, anticancer, anti-inflammation, and enhanced immunity. It can improve the metabolism of glucose and lipids and play an anti-inflammatory role in nonalcoholic fatty liver disease. Qiu et al. revealed that DMY can induce feedback regulation in the keap-1/Nrf2 pathway, weaken liver enzyme release, reduce the malondialdehyde level in the liver, and decrease triglyceride deposition caused by chronic alcohol exposure. Jia et al. deemed that DMY can effectively inhibit the production of a variety of proinflammatory cytokines (IL-1β, TNF-α, and IL-17) and avoid the activation of a TRAF3-p38 signal in macrophages (bone marrow-derived macrophage) so as to greatly relieve the injury caused by acute pancreatitis. The latest clinical report showed that DMY could be used for the treatment of type II diabetes mellitus (T2DM). Liu et al. found that DMY could inhibit the phosphorylation of a peroxisome proliferator-activated receptor γ (PPARγ) through the MEK/ERK mechanism and improve the glucose uptaking and adiponectin secretion in adipocytes.

As DMY is rich in A. grossedentata and has many beneficial effects on organisms, its efficient extraction has a vital significance. DMY is easily soluble in ethanol and other organic solvents but slightly soluble in cold water. Thus, it is usually extracted by Soxhlet extraction and purified by recrystallization. Liao et al. used the difference of solubility of DMY in hot water and cold water to recrystallize DMY eight times and obtained DMY with a purity of 98%. Li et al. adopted microwave-assisted multistage countercurrent extraction technology, which could break plant cells to dissolve DMY, DMY was extracted with water, and a satisfactory result was achieved. Some researchers purified DMY by setting a three-column countercurrent chromatograph using a solvent system, which was composed of n-hexane–ethyl acetate–methanol. The purity of DMY via chromatography was very high, but the efficiency was very low, and the consumption for extracting solvents was large. However, the purity of DMY via batch extraction was low and the purification time was very...
long (usually about 2−3 days or longer). In view of this, it was very crucial to develop a new and effective technology for separating and purifying DMY in order to facilitate the study of the structure and properties of DMY, which would be useful for its application into relevant areas.

DMY is a kind of polyhydroxy compound. It has a high degree of hyperonization, and it is easy to coordinate with metal ions. For example, Zn\(^{2+}\) can form a Zn−O coordination bond with no. 3 −OH on the DMY C ring, which can inhibit the free radical delocalization on the benzene ring and reduce the oxidation resistance of DMY. In this study, we proposed a new extraction and purification method, as shown in Figure 1. In the extraction process, DMY−Zn was obtained by chelating with Zn\(^{2+}\), and then Zn\(^{2+}\) was replaced by EDTA-2Na to get DMY. Taking the yield and purity of DMY as the indices, the experimental results of the chelating extraction method were compared with those of the batch extraction method in terms of extraction time, extraction pH, and extraction temperature. The results are shown in Figure 3. The chelation extraction method can significantly improve the yield of DMY in contrast with the batch extraction method. Because of the complicated operation of the batch extraction method, which needed eight times of recrystallization, the weight of DMY suffered heavy losses. In addition, similar to a majority of flavonoids, DMY was easily oxidized to quinones by air. Thus, the protection of DMY was very important during the extracting and purifying process. DMY and zinc ions could form stable complexes to protect the oxidation of DMY in the process of chelation extraction and purification. Therefore, the production of quinone impurities was reduced.

The effect of extraction time on the purity and yield of DMY is shown in Figure 3a. The yield of DMY can be increased by prolonging the extraction time. When the extraction time was 2.5 h, the yield of DMY was 11.4%, which was the highest value. On the other hand, the extraction time also affected the purity of DMY. When the extraction time was more than 2 h, the purity of DMY decreased. The reason was that the prolonged extraction time led to the increase of impurities generated by the oxidation reaction of DMY with air, which resulted in the decrease of the purity of DMY. Taking the purity and yield of DMY into consideration, when the extraction time was 2 h, the chelating extraction method of DMY extraction worked best.

The effect of extracting pH on the purity and yield of DMY is demonstrated in Figure 3b. The yield of DMY increased with the decrease of pH value. The yield of DMY was 7.2% when pH was 5.5 and 11.3% when pH was 2. The higher pH value led to the hydrolyzation of Zn\(^{2+}\), so the chelation of Zn\(^{2+}\) and DMY was inhibited. Furthermore, as a polyhydroxy compound, DMY was unstable under alkaline conditions, and it was easy to oxidize with air to form quinones. Therefore, the increase of the pH value also decreased the purity of DMY.

**2. RESULTS AND DISCUSSION**

2.1. Evaluation of Extraction and Purification Processes. 2.1.1. HPLC Analysis. The HPLC chromatograms of the DMY standard sample and the DMY sample are shown in Figure 2. The retention time of the DMY standard sample is 6.913 min after mobile phase separation, which was similar to the retention time of DMY reported by Zhao et al. Using a diode array detector, the peak areas of the five DMY standard solutions were 941.2, 1864.4, 2769.6, 3625.2, and 4558.1. The standard curve equation of concentration versus peak area of DMY was \( A = 59.488C + 49.32 (R^2 = 0.9999) \). The retention time of A. grossedentata extracted by using the chelating extraction method is 6.879 min, which was consistent with the retention time of the standard sample. The purity of DMY was 94.3%, which was calculated from the peak area.

Figure 1. Diagram of the chelation extraction process of DMY.

Figure 2. HPLC chromatogram. (a) DMY standard. (b) DMY sample.
The effect of extraction temperature on the purity and yield of DMY is illustrated in Figure 3c. With the increase of temperature, the yield of DMY increased gradually, and the increase of yield for chelate extraction was better than that for batch extraction. When the extraction temperature was 90 °C, the yield of DMY extracted by chelating extraction was the highest, which was 11.3%. Wang et al. conducted TGA on DMY and revealed that DMY began to decompose in an environment higher than 100 °C, so 90 °C was the better extraction temperature.

Compared with the traditional batch extraction method, the chelate extraction method was more advantageous to extract DMY from *A. grossedentata*. When the extraction time was 2 h, pH = 2, and temperature was 90 °C, the average yield and purity of 10 experimental groups of DMY were 12.2 and 94.3%, respectively.

2.2. Mechanism of Extraction and Purification.

2.2.1. UV−Vis Analysis. The UV−vis spectra of DMY and DMY−Zn are shown in Figure 4. DMY had two absorption peaks at 207 and 289 nm, and they were the hallmarks of flavonoids and flavonols (red line), and the result of energy-level transitions of DMY on a benzoyl system (blue area) and a cinnamoyl system (green area), respectively. However, DMY−Zn had two absorption peaks, which appeared at 210−220 and 320−330 nm. The O atom of 3-OH in DMY formed a coordination compound with Zn²⁺. Therefore, the density of an electron cloud on DMY decreased, the electron delocalization increased, and the π → π* transition energy on the aromatic ring decreased, so the absorption spectrum moved to a long wave.

2.2.2. Fourier-Transform Infrared Analysis. Figure 5 shows the infrared spectra of DMY−Zn and DMY. Table 1 illustrates the main infrared absorption frequency of DMY−Zn and DMY. First, it can be noted from Table 1 that the characteristic absorption peaks of DMY obtained by the chelating extraction method coincided with the molecular structure of DMY.

It can be observed from Figure 5a that the strong absorption peak at 3288 cm⁻¹ was assigned to the hydroxyl stretching vibration peak on the DMY benzene ring and the peaks at 1641 and 1339 cm⁻¹ were attributed to the bending vibration of C=O and C−OH on the DMY benzene ring, respectively. While Figure 5b indicates that at the range of 3211−3407 cm⁻¹, there was also a wider hydroxyl vibration peak, which corresponded to the coordination bond formed by an O atom on 3-OH and 5-oxo on the DMY B ring with Zn²⁺; thus, the carbon oxygen double bond became weak. The bonding electron density of C=O decreased when Zn²⁺ was combined with carbonyl oxygen and hydroxyl oxygen. Therefore, the bending vibration peaks of C=O and C−OH...
of DMY·Zn appeared at 1667 and 1360 cm\(^{-1}\), respectively. In addition, compared with DMY, there was a new absorption peak of DMY·Zn at 646 cm\(^{-1}\), which corresponded to the absorption peak of Zn–O.\(^{29}\)

2.2.3. XRD Analysis. The powder XRD patterns of DMY·Zn and DMY are demonstrated in Figure 6. It can be observed from the diffraction pattern that obvious changes had been made in the chemical composition and crystal structure of DMY, which were obtained from the reaction of DMY·Zn with EDTA-2Na. It can be noted from jade 6.5 analysis that many peaks appeared in the range of 5–80°, which meant that DMY·Zn was a crystal molecule.\(^{30}\) Moreover, through comparing the standard card, the diffraction pattern (marked as DMY·Zn in Figure 6) could not be regarded as that of ZnSO\(_4\)·7H\(_2\)O because 2\(\theta\) of Zn(OH)\(_2\) and ZnO in ZnSO\(_4\)·7H\(_2\)O were not correspondent in the pattern. Combined with the analysis of UV–vis and Fourier-transform infrared (FTIR), it can also be proven that the yellow brown precipitate during the extraction process was not produced by the hydrolysis or decomposition of ZnSO\(_4\)·7H\(_2\)O, but the product of chelation of DMY in A. grossedentata with Zn\(^{2+}\). Compared with DMY·Zn, there were many new intensity peaks in the powder diffraction pattern of the product, which was obtained by EDTA-2Na uncoupling (i.e., DMY). This was consistent with the XRD pattern of DMY reported by Liu et al.,\(^{11}\) which meant that DMY was uncoupled from the DMY·Zn after the reaction of DMY·Zn with EDTA-2Na. Furthermore, obvious crystal peaks appeared in the diffraction pattern of DMY, which suggested that DMY itself had high crystallinity.

2.2.4. SEM–EDS Analysis. The DMY·Zn precipitated during the extraction process was a yellowish brown powder (see Figure 7a). With the dissociation of Zn\(^{2+}\) from DMY·Zn and the purification of DMY, DMY took the shape of a continuous silk and turned into yellow white (see Figure 7d). Scanning electron microscopy (SEM) was used to observe the micromorphology and structural characteristics of DMY·Zn and DMY. It was found that DMY·Zn was composed of numerous fine particles, which were closely clustered and arranged, and in the form of irregular three-dimensional clusters (see Figure 7b). While DMY was a regular prismatic crystal (see Figure 7e) with a smooth surface and a uniform size, which was related to the high crystallinity of DMY, and it was suggested that the purity of DMY obtained by using the chelate extraction method was higher. The energy-dispersive X-ray spectroscopy (EDS) analysis of DMY·Zn and DMY

| compounds | \(\nu(\text{O–H})\) cm\(^{-1}\) | \(\nu(\text{C=O})\) cm\(^{-1}\) | \(\nu(\text{C=\(\equiv\)C})\) cm\(^{-1}\) | \(\nu(\text{C–OH})\) cm\(^{-1}\) | \(\nu(\text{C–O–C})\) cm\(^{-1}\) | \(\nu(\text{Zn–O})\) cm\(^{-1}\) |
|-----------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| DMY·Zn    | 3211–3407       | 1667            | 1473            | 1360            | 1171            | 646             |
| DMY      | 3288            | 1641            | 1472            | 1339            | 1171            | 1171            |

Table 1. Main Infrared Absorption Frequency of DMY and DMY·Zn

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Figure 6. Powder XRD patterns of the DMY–Zn complex and DMY.

Figure 7. Morphology and microstructure of DMY–Zn and DMY; (a) photo image of DMY–Zn; (d) photo image of DMY; (b) SEM images of DMY–Zn; (e) SEM images of DMY; (c) EDS spectra and element composition of DMY–Zn; (f) EDS spectra and element composition of DMY.
showed that the main elements of DMY–Zn were C, O, and Zn, and the content of Zn was 20.69% (see Figure 7c), while DMY only contained C and O, and the contents of C and O were 54.21 and 45.79% (see Figure 7e), respectively. In addition, no Zn in the energy spectrum of DMY was detected, which indicated that EDTA-2Na had a stronger replacement ability to combine with all Zn\textsuperscript{2+} in DMY.

2.3. Structural Analysis of DMY. 2.3.1. Single-Crystal X-ray Data Collection and Structure Determination. The X-ray crystallography data analysis of DMY is shown in Table 2.

Table 2. Crystallographic Data and Structure Refinement of DMY Dihydrate

| parameter               | values                                    |
|-------------------------|-------------------------------------------|
| empirical formula       | C\textsubscript{30}H\textsubscript{32}O\textsubscript{20} |
| formula weight          | 712.55                                    |
| crystal system          | monoclinic                                |
| space group             | \(P2_1/\text{c}\)                           |
| \(a\) (Å)               | 15.3733(10)                               |
| \(b\) (Å)               | 7.8961(10)                                |
| \(c\) (Å)               | 24.1511(2)                                |
| \(\beta\) (deg)         | 92.6270(10)                               |
| \(V\) (Å\textsuperscript{3}) | 2928.60(5)                               |
| \(Z\)                   | 4                                         |
| \(\mu\) (mm\textsuperscript{-1}) | 1.202                                    |
| \(D_{\text{calc.}}\) (g/cm\textsuperscript{3}) | 1.616                                    |
| \(F(000)\)              | 1488.0                                    |
| θ (deg)                 | 5.757–155.652                             |
| independent reflections | 6048                                      |
| good Darwin factor      | 0.975                                     |
| final R indices         | 0.0331/0.0883                             |
| R indices (all data)    | 0.0368/0.0907                             |
| largest difference peak | 0.26, −0.27                              |

DMY dihydrate was a monoclinic crystal, which could be categorized as a spatial \(P2_1/\text{c}\) structure. The density of the crystal was 1.616 g/cm\textsuperscript{3}, while \(a = 15.3733\) (10), \(b = 7.8961\) (10), \(c = 24.1511\) (2). The structure of DMY was obtained directly by the shell XT program, and then the \(F^2\) is refined by a full-matrix least-square method.\textsuperscript{31} The single-crystal diffraction data were shown in 190629w (upload to the Cambridge crystalgraphic data center).

The molecular structure of DMY dihydrate is shown in Figure 8. Each crystal cell contained two DMY dihydrate molecules and four crystal water. The molecules were connected by hydrogen bonds. The pyran rings on the inner molecules of DMY dihydrate all existed in the form of plane angle conformation. The bond length of C (11)–C (12) and C (35)–C (36) was 1.5235 and 1.5355 Å, respectively, which was much longer than that of other C–C or C–O bonds in the ring. This special phenomenon was caused by the high degree of hyperdelocalization of DMY itself. The formation of crystal water made the atoms arrange more closely through strong π–π stacking interaction, so that the overall molecular structure had a higher relative stability.\textsuperscript{32,33} Theoretically speaking, DMY had two kinds of chiral centers; there were four potential counterparts. It can be noted from the bond angles of C (10)–C (9)–C (11) (119.69\textsuperscript{3}), O (18)–C (11)–C (9) (108.22\textsuperscript{3}), and O (13)–C (12)–C (11) (108.55\textsuperscript{3}) that the benzoyl system (blue area) and the cinnamoyl system (green area) were not at the same plane, which suggested that DMY was (2S,3S) enantiomers, and it was a kind of tetrahydrate of the racemic D\textsubscript{r} compound.

2.3.2. \(^1\)H NMR Analysis. The hydrogen spectrum of DMY is shown in Figure 9. \(^1\)H NMR (400 MHz, DMSO-\(d_6\)): \(\delta\) 11.90 (s, 1H), 10.82 (s, 1H), 8.91 (s, 2H), 8.22 (s, 1H), 6.39 (s, 2H), 5.90 (d, \(J = 2.1\) Hz, 1H), 5.86 (d, \(J = 2.1\) Hz, 1H), 5.76 (d, \(J = 6.2\) Hz, 1H), 4.90 (d, \(J = 10.9\) Hz, 1H), 4.41 (dd, \(J = 10.9, 6.2\) Hz, 1H).

2.3.3. Thermogravimetry and Differential Thermal Analyses. The thermal stability of DMY dihydrate in the range of 40–900 °C is shown in Figure 10. There were four stages of weight loss as the temperature increased. When the temperature was raised from 40 to 137 °C, the weight loss rate of DMY dihydrate was 11.34%, which was close to the theoretical moisture content of 10.01%. It can be attributed to the loss of free water on the crystal surface and crystal water in the moisture content of 10.01%. It can be attributed to the loss of free water on the crystal surface and crystal water in the

Figure 8. Molecular structure of DMY dihydrate(C\textsubscript{15}H\textsubscript{12}O\textsubscript{8}·2H\textsubscript{2}O).

Figure 9. \(^1\)H NMR analysis of DMY dihydrate.
The thermal degradation of DMY occurred in the temperature from 300 to 400 °C, and the degradation mechanism of dehydration is shown in Figure 11. The theoretical loss was 23.13%, which was close to the actual loss. The weight loss at this stage was the fracture of the C−C chemical bond between the pyran ring and residual benzene ring, which was also the unstable chemical bond in DMY dihydrate.\textsuperscript{35} 2-Hydroxypropanal was the thermal decomposition product, its boiling point was 121.9 °C, and could be evaporated rapidly at 300−400 °C.

3. CONCLUSIONS

The content of DMY in \textit{A. grossedentata} was very rich. The study of new extraction technologies can provide an important reference for commercial application of DMY. The new extraction method (chelating extraction) and the traditional extraction method (batch extraction) of DMY were compared. The yield and purity of DMY were investigated in terms of three factors, that is, extraction time, extraction pH value, and extraction temperature. The results showed that the yield and purity of DMY extracted from \textit{A. grossedentata} by the chelating extraction method were higher, and the oxidation of DMY could be effectively avoided during the extraction and purification process. Compared with the batch extraction method, the chelating extraction method had obvious advantages. When the extraction time was 2 h, pH = 2, and temperature was 90 °C, the average yield and purity of 10 experimental groups of DMY were 12.2 and 94.3%, respectively.

The mechanism of extraction and purification was examined through the analysis of UV−vis, FTIR, and XRD. The results illustrated that in the presence of zinc ions, DMY and Zn\textsuperscript{2+} coordinated to form a DMY−Zn chelate and precipitated. EDTA-2Na was added and competed with DMY to form chelation, and Zn\textsuperscript{2+} on DMY−Zn was completely removed; then, the original conjugation system of DMY was restored. SEM−EDS analyses also showed that the morphology of DMY was very different before and after decoupling with EDTA-2Na, and the appearance of DMY after decoupling was a white silky material.

The crystal analysis of the purified DMY by a X-ray single-crystal diffractometer revealed that the molecular formula of DMY was C\textsubscript{15}H\textsubscript{12}O\textsubscript{8}·2H\textsubscript{2}O, which was a kind of tetrahydrate of the racemic di compound. TGA illustrated that the DMY dihydroxylate can exist stably below 265.93 °C.

4. EXPERIMENTAL SECTION

4.1. Materials. \textit{A. grossedentata} was produced in Wuzhou, Guangxi Zhuang Autonomous Region, China. The DMY standard sample was purchased from Bailingwei Technology Co. Ltd. (China). ZnSO\textsubscript{4}·7H\textsubscript{2}O, HCl, and NaOH used in this study were of analytical grade and obtained from Guangzhou Chemical Reagent Co. Ltd. (China). EDTA-2Na was also of analytical grade and bought from Tianjin Damao Chemical Reagent Co. Ltd. (China). The raw materials and reagents mentioned here were used as-received.

4.2. Extraction and Purification of DMY. The batch extraction method was used based on the method reported by Liao et al.\textsuperscript{15} The dried leaves of \textit{A. grossedentata} were crushed, and tea powder of a size of more than 20 mesh was retained. The \textit{A. grossedentata} powder weighing about 20 g was put into a 500 mL round-bottom flask, and 400 mL of deionized (DI) water (the solid−liquid ratio was 1:20, weight ratio) was poured into the flask; the mixture was heated at 100 °C for 1 h and then filtered while it was hot; and the filtrate was discarded and then recrystallized eight times with DI water.

The chelation extraction method of \textit{A. grossedentata} was as follows. Twenty grams of dried \textit{A. grossedentata} powder, 400 mL of DI water (the solid−liquid ratio was 1:20, weight ratio), and 5 g of ZnSO\textsubscript{4}·7H\textsubscript{2}O powder were put into a 500 mL round-bottom flask and fully stirred; the mixture was adjusted to pH = 2 using 2 mol/L HCl solution and extracted in water at 90 °C for 2 h. The mixture was filtered while it was hot, and the filtrate was retained; the mixture was adjusted to pH = 5 using 1 mol/L NaOH solution, stirred for 5 min, and filtered, and the filter residue was retained. Then, the collected components were washed with DI water three times and
placed in a 500 mL beaker. Then, 300 mL of 0.1 mol/l EDTA-2Na solution was added and stirred for 5 min at 90 °C and filtered while hot, the filtrate was collected, and the DMY crude product was obtained after cooling.

The crude DMY was dissolved in 100 °C water, 5 g of ZnSO₄·7H₂O powder was added, and then the mixture was adjusted to pH = 5 using 2 mol/L HCl solution. Some yellowish-brown precipitation, that is, DMY–Zn, was obtained. After hot filtration and full washing, the filter residues were decoupled using EDTA-2Na (repeated purification 2–3 times), and a white needle-like DMY crystal was obtained.

DMY of 0.5 g was weighed and dissolved in 50 mL of boiling water, then the mouth of the beaker was covered with filter paper and allowed to stand at room temperature for 1 week, and a white needle-like DMY single crystal was obtained.

The ratio of solid to liquid of A. grossedentata powder and DI water was kept as 1:20, and the ratio of A. grossedentata powder to ZnSO₄·7H₂O was fixed as 1:4. The yield and purity of DMY were taken as the evaluation indices; extraction time, extraction pH, and extraction temperature were taken as the factors, and the single-factor experiments were conducted in order to study the influence on the two processes, that is, intermittent extraction and chelating extraction. The levels of each factor are shown in Table 3, and each sample experiment was repeated three times in parallel.

### Table 3. Single-Factor Experimental Design

| experimental factor | level | extraction time (h) | extraction pH | extraction temperature (°C) |
|---------------------|-------|---------------------|---------------|-----------------------------|
|                     | 1     | 0.5                 | 2             | 25                          |
|                     | 2     | 1.0                 | 3             | 50                          |
|                     | 3     | 1.5                 | 4             | 70                          |
|                     | 4     | 2.0                 | 5             | 80                          |
|                     | 5     | 2.5                 | 5.5           | 90                          |

4.3. Measurements. The DMY standard sample and DMY sample were analyzed by Agilent 1260 HPLC. The mobile phase was prepared with 0.1% phosphoric acid and methanol (volume ratio 65:35) as the solvent. The flow rate was 1.0 mL/min, the sample volume was 10 μL, the column temperature was 40 °C, and the detection wavelength was 290 nm.

The UV absorption spectrum in the range of 200–420 nm was determined using a Shimadzu UV-2600 with a SHISEIDO C18 MG UV detector. The preparation of the samples was as follows. The yellow brown precipitate (i.e., DMY–Zn) obtained from the precipitation process was dried to a constant weight in vacuum at 50 °C. Then, 50 mg of DMY and DMY–Zn samples was dissolved in 1 mL of dimethyl sulfoxide (DMSO) solution and then diluted 100 times with DI water.

The chemical structure of DMY was obtained using a PerkinElmer Frontier FT-IR instrument in the wavenumber range of 450–4000 cm⁻¹ with transmission mode, and the sample was prepared by grinding the dry specimens of DMY containing potassium bromide (KBr), and then it was pressed to form disks.

Scanning electron microscopy (Hitachi S-4800) was used to study the micromorphology and main elements of DMY and DMY–Zn samples. Thermal stability of the DMY crystal was investigated by using Mettler-Toledo TGA2 in a flowing nitrogen atmosphere between 40 and 900 °C at a heating rate of 10 °C/min. DMY–Zn and DMY were detected by XRD (X’Pert PRO MPD) at 5–80° (2θ). The structure of the DMY crystal was determined by a single-crystal X-ray diffractometer (Bruker SMART 1000CCD) and ¹H NMR using D-substituted DMSO as the solvent.

**ASSOCIATED CONTENT**

Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acsomega.0c01222.

Crystallographic data of DMY dihydrate (CIF)

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Notes

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