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Research Article

Crystal Structure and Catalytic Activity of Poly[bis(3-bromo-2-hydroxybenzaldehyde)-2-aminopyrimidinemagnesium(II)] for Hydrogenation of 1,3-Butadiene

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Abstract

A new six-coordinated Mn(II) coordination polymer, $[Mn(L_1)(L_2)_2]_n$ (L_1 = 2-aminopyrimidine, HL_2 = 3-bromo-2-hydroxybenzaldehyde) was synthesized by 3-bromo-2-hydroxybenzaldehyde, NaOH, 2-aminopyrimidine and manganese(II) acetate dihydrate. The Mn(II) coordination polymer was structural characterized by elemental analysis and single crystal X-ray diffraction. The results show that each Mn(II) ion is six-coordinated with two phenolic hydroxyl O atoms from two 3-bromo-2-hydroxybenzaldehyde ligands (O1 and O4), two formyl group O atoms from two 3-bromo-2-hydroxybenzaldehyde ligands (O2 and O3), and two N atoms from two 2-aminopyrimidine molecules (N1A and N2), and forms a distorted octahedral coordination geometry. The Mn(II) coordination polymer displays a 1D chained structure by the bridge effect of 2-aminopyrimidine N atoms. The catalytic activities of Mn(II) coordination polymer and Pd@Mn(II) coordination polymer for hydrogenation of 1,3-butadiene have been investigated. The Pd@Mn(II) coordination polymer catalyst shows the good catalytic activity and selectivity in the hydrogenation of 1,3-butadiene. The 1,3-butadiene conversion is 61.3% at 70 °C, and the selectivity to total butene is close to 100%.

Keywords: Mn (II) coordination polymer; Synthesis; Structural characterization; Catalytic activity

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1. Introduction

In the petrochemical industry, the thermal cracking of petroleum fractions usually produces the byproduct 1,3-butadiene (1,3-BD), which must be removed below 10 ppm for polymerization processes because they poison the catalysts and degrade the product quality [1]. The hydrogenation of 1,3-butadiene is an effective strategy to remove 1,3-butadiene in the in the petrochemical industry. Pd supported catalyst is mostly used in the selective hydrogenation of 1,3-butadiene due to the excellent catalytic activity, selectivity, and stability [2,3]. Manganese coordination polymers exhibit excellent activity in many fields, such as: catalytic property [4–7], magnetic properties [8–11], solvent adsorption [12], oxidative dehydrogenation [13], antitumor and antibacterial activities [14,15], lumines-

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cence properties [16–19], electrochemical property [20], and nonlinear optical property [21] and so on. So the researches on the manganese coordination polymer have attracted the attention of scientists. The synthesis of coordination polymers is influenced by ligands, metal centers, solvent, pH and reaction temperature [22]. In addition, the mixed ligands has become an impactful factor in the formation of coordination polymers.

However, there are few studies on hydrogenation of 1,3-butadiene catalyzed by manganese coordination polymers. Our research group has studied the catalytic activity of some metal complexes [23–28]. To further explore the catalytic activity of metal complexes, in this paper, a novel Mn(II) coordination polymer has been synthesized by 3-bromo-2-hydroxybenzaldehyde, NaOH, 2-aminopyrimidine and manganese(II) acetate dihydrate. The structure of Mn(II) coordination polymer has been determined by elemental analysis and single crystal X-ray diffraction. The catalytic activities of Mn(II) coordination polymer and Pd@Mn(II) coordination polymer for hydrogenation of 1,3-butadiene have been investigated. The Pd nanoparticles were loaded on the surface of Mn(II) coordination polymer, and the catalytic active center of Pd can be completely exposed to the reactants of 1.0 vol%1,3-butadiene/N_2 and H_2. Furthermore, the catalytic activity is not affected by the size of reactants. Interestingly, the Mn(II) coordination polymer catalyst exhibits very low catalytic activity with 1,3-butadiene conversion less than 1% toward the 1,3-butadiene hydrogenation, however, the Pd@Mn(II) coordination polymer catalyst shows the higher conversion (61.3% at 70 °C) than Mn(II) coordination polymer.

2. Materials and Methods

2.1 Materials and Measurements

3-bromo-2-hydroxybenzaldehyde (A.R.), NaOH (A.R.), 2-aminopyrimidine (A.R.), and manganese(II) acetate dihydrate (A.R.) were purchased from Jilin Chinese Academy of Sciences-Yanshen Technology Co., Ltd.. C, H and N were analyzed using an Elementar Vario III EL elemental analyzer (Hanau, Germany). The crystal data of Mn(II) coordination polymer were collected on a Bruker Smart CCD diffractometer (Bruker, Billerica, MA, USA). The effluent from the reactor was collected and analyzed using an online gas chromatography (GC-6890, Purkinje General Instrument Co., Ltd., China) equipped with a Al_2O_3 capillary column.

2.2 Synthesis of Mn(II) Coordination Polymer

The 3-bromo-2-hydroxybenzaldehyde (0.5 mmol, 0.1005 g), 2-aminopyrimidine (0.5 mmol, 0.4750 g) and NaOH (0.5 mmol, 0.020 g) were dissolved in 15 mL ethanol/H_2O solution (v:v = 2:1) with stirring. After 1 h, 0.0865 g manganese(II) acetate dihydrate (0.5 mmol) solid was added to the above solution. Then the mixture was stirred and kept at *ca.* 75 °C for 6 h. The resultant solution was cooled and filtered, the orange crystals of poly[bis(3-bromo-2-hydroxybenzaldehyde)-2-aminopyrimidinemagnesium(II)] were received from the filtrate in two weeks. Elemental analysis (%) calcd. for $C_{18}H_{13}Br_2MnN_3O_4$: C, 39.27; H, 2.36; N, 7.64. Found (%): C, 39.52; H, 2.19; N, 7.39.

2.3 Synthesis of Pd@Mn (II) Coordination Polymer

The Mn(II) coordination polymer-supported Pd catalysts were synthesized using impregnation method according to the procedures in the literature [29]. First, 0.0074 g of $Pd(CH_3COO)_2$ were melted in 0.5 mL of ethanol. Then, the solution of $Pd(CH_3COO)_2$ was added into Mn(II) coordination polymer (0.070 g). After sonicating for 1 h, the suspension liquid was sustained at room temperature for 12 h. The final homogenous mixture was further dried at 50 °C for 7 h in air dry oven.

2.4 Crystal Structure Determination

A suitable single crystal of poly[bis(3-bromo-2-hydroxybenzaldehyde)-2-amino-Pyrimidinemagnesium(II)] (0.30 mm × 0.20 mm × 0.10 mm) was chosen to collect data on a Bruker Smart APEX CCD diffractometer with graphite-monochromated Mo-*Ka* radiation (λ = 0.71073 Å) at 298(2) K. The structure was solved by direct method using SHELXL program [30] and refined by full-matrix least squares on F^2 by means of the program OLEX2 [31]. The crystallographic data of Mn(II) coordination polymer are summarized in Table 1.

2.5 General Procedure for the Hydrogenation of 1,3-Butadiene

The selective hydrogenation of 1,3-butadiene was performed in a quartz fixed bed at the atmospheric pressure. 0.020 g of the catalyst mixed with 0.480 g quartz sand (40-80 mesh), and then packed into the reactor. Prior to catalysis, the catalyst was treated for at 50 °C for 1 h in a reducing environment with the

H_2 (99.999 %) flow (at 10 mL/min). Then the reactants of 1.0 vol% 1,3-butadiene in 99.0 vol% N_2 (13.0 mL/min) and 99.999 vol% H_2 (6.5 mL/min) flowed through the Pd@Mn (II) coordination polymer catalyst bed. The effluent from the reactor was collected and analyzed using an online gas chromatography (GC-6890, Purkinje General Instrument Co., Ltd., China) equipped with a Al_2O_3 capillary column.

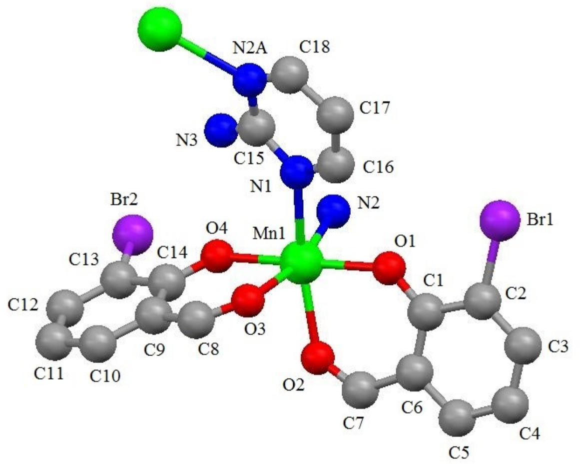

Figure 1. The asymmetric unit of Mn(II) coordination polymer 1.

3. Results and Discussion

3.1 Structural Description of Mn(II) Coordination Polymer

Single-crystal X-ray diffraction shows that the Mn(II) coordination polymer **1** crystallizes in the monoclinic system with the $P2_1$/n space group. The asymmetric unit of Mn(II) coordination polymer **1** is shown in Figure 1. The selected bond lengths (Å) and angles (°) for Mn(II) coordination polymer **1** are given in Table 2. As shown in Figure 1, the asymmetric unit of Mn(II) coordination polymer is made up of one Mn(II) ion, one 2-aminopyrimidine ligand and two 3-bromo-2-hydroxybenzaldehyde ligands. Each Mn(II) ion is six-coordinated with two phenolic hydroxyl O atoms from two 3-bromo-2-hydroxybenzaldehyde ligands (O1 and O4), two formyl group O atoms from two 3-bromo-2-hydroxybenzaldehyde ligands (O2 and O3), and two N atoms from two 2-aminopyrimidine molecules (N1A and N2), and forms a distorted oc-

Table 1. Crystallographic data of Mn(II) coordination polymer 1.

Empirical formula	$C_{18}H_{13}Br_2MnN_3O_4$
Formula weight	550.07
Temperature/K	298(2)
Crystal system	Monoclinic
Space group	$P2_1/n$
a/Å	11.9818(6)
b/Å	7.4995(5)
c/Å	21.5342(9)
α/°	90
β/°	91.650(6)
γ/°	90
Volume/Å^3	1934.21(18)
Z	4
ρ_{calc}, mg/mm^3	1.612
μ/mm^{-1}	4.844
S	1.036
F(000)	1076
Index ranges	$-14 \le h \le 11$, $-8 \le k \le 7$, $-22 \le l \le 25$
Reflections collected	8235
θ/°	1.922–25.011
Independent reflections	3395 [R(int) = 0.0298]
Data/restraints/parameters	3395/0/241
Goodness-of-fit on F^2	1.036
Refinement method	Full-matrix least-squares on F^2
Final R indexes [I>=2σ (I)]	$R_1 = 0.0318$, $wR_2 = 0.0563$
Final R indexes [all data]	$R_1 = 0.0427$, $wR_2 = 0.0599$
Largest diff. peak/hole / e Å^{-3}	0.583 /-0.544

tahedral coordination geometry. The bond angles around Mn(II) in the equatorial plane are O1-Mn1-O3 (103.69(8)°), O3-Mn1-O4 (82.01(8)°), O4-Mn1-N2 (83.99(8)°), O1-Mn1-N2 (90.57(9)°), and the sum of the above bond angles is 360.26°, showing O1, O3, O4, N2 and Mn(II) are coplanar. The Mn-O and Mn-N distances are 2.066(2) Å (Mn1-O1), 2.221(2) Å (Mn1-O2), 2.229(2) Å (Mn1-O3), 2.079(2) Å (Mn1-O4), 2.308(3) Å (Mn1-N1), and 2.310(2) Å (Mn1-N2), which are comparable to other Mn(II) coordination polymers [32,33]. The asymmetric unit of Mn(II) coordination polymer are connected by 2-aminopyrimidine to generate a 1D infinite chain (Figure 2). Finally, the isolated 1D chains assemble an extended 3D supramolecular framework via π-π stacking interactions (Figure 3). It is worth mentioning that 2-aminopyrimidine as a bridge ligand plays an important role in the formation of coordination polymer.

3.2 Synthesis Strategy of Pd@Mn(II) Coordination Polymer Catalyst

Pd@Mn(II) coordination polymer catalysts were synthesized using impregnation method. First, the solution of Pd(CH_3COO)$_2$ was added into Mn(II) coordination polymer. After sonicating for 1 h, the suspension was stored for12 h at room temperature and dried in air dry oven at 50 °C for 7 h. Finally, the catalysts were in-situ reduced at 50 °C for 1 h under H_2 flow at rate of 10 mL/min before catalysis in a quartz fixed bed. Liu *et al.* [29] prepared Pd/MIL-101(Cr) catalysts by impregnation using Pd(CH_3COO)$_2$ as precursor, and followed by H_2 reduction at 50 °C for 2 h. Two obvious

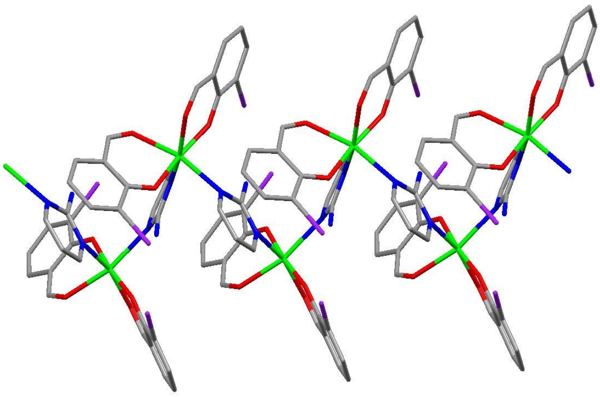

Figure 2. 1D chained structure of Mn(II) coordination polymer 1.

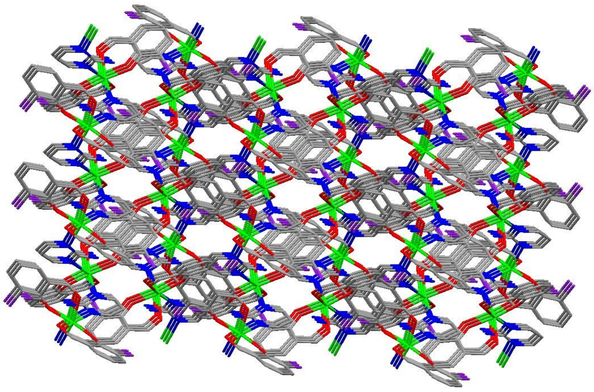

Figure 3. 3D network structure of Mn(II) coordination polymer 1.

Table 2. Selected bond lengths (Å) and bond angles (°) for Mn(II) coordination polymer 1.

Bond	*d*	Angle	(°)
Mn1-O1	2.066(2)	O4-Mn1-O1	173.73(9)
Mn1-O4	2.079(2)	O1-Mn1-O2	81.87(8)
Mn1-O2	2.221(2)	O2-Mn1-O4	96.36(9)
Mn1-O3	2.229(2)	O1-Mn1-O3	103.69(8)
Mn1-N1	2.308(3)	O4-Mn1-O3	82.01(8)
Mn1-N2	2.310(2)	O2-Mn1-O3	83.34(8)
C2-Br1	1.893(3)	O1-Mn1-N1	86.39(9)
C13-Br2	1.906(3)	O4 -Mn1-N1	96.64(9)
C1-O1	1.297(4)	O2-Mn1-N1	162.41(9)
C7-O2	1.242(4)	O3-Mn1-N1	86.78(9)
C8-O3	1.232(4)	O1-Mn1-N2	90.57(9)
C14-O4	1.296(4)	O4-Mn1-N2	83.99(8)
C16-N1	1.340(4)	O2-Mn1-N2	103.82(8)
C15-N1	1.361(4)	O3-Mn1-N2	164.92(9)
C18-N2	1.338(4)	N1-Mn1-N2	89.30(9)

peaks were obtained in the Pd 3d XPS spectra of Pd/MIL-101(Cr) at the binding energy of 341.1 eV and 335.6 eV which were attributed to Pd^0 3d3/2 and Pd^0 3d5/2, respectively [29]. Taking into account that Pd@Mn(II) coordination polymer catalysts were prepared by a similar method to that of Pd/MIL-101(Cr), we may infer that Pd in the Pd@Mn(II) coordination polymer catalysts mainly exist in the metallic palladium (Pd^0).

3.3 Catalytic Studies of 1,3-Butadiene Hydrogenation

The conversions of 1,3-butadiene and product selectivity for the Mn(II) coordination polymer and Pd@Mn(II) coordination polymer catalysts are given in Figure 4 and Figure 5, respectively. The Mn(II) coordination polymer catalyst has very low catalytic activity with 1,3-butadiene conversion less than 1% toward the 1,3-butadiene hydrogenation because of lack of active sites. The Pd@Mn(II) coordination polymer catalyst shows the higher conversion than Mn(II) coordination polymer catalyst. The 1,3-butadene conversions were 44.6%, 50.0%, 61.3%, 63.7%, and 64.1% on Pd@Mn (II) coordination polymer catalyst at 50 °C, 60 °C, 70 °C, 80 °C, ad 90 °C, respectively. The 1,3-butadiene conversion continuously increased with the increasing of reaction temperature from 50 °C to 70 °C. At high temperature range (70-90 °C), there is no much difference between the 1,3-butadiene conversions. The Pd@Mn(II) coordination polymer catalyst shows the higher butene selectivities, the selectivities to total butenes are all close to 100% at 50–90 °C. The selectivity of the Pd@Mn(II) coordination polymer catalyst to different isomers of butene showed the order 1-butene > *trans*-2-butene > *cis*-2-butene. The selectivities to 1-butene, *trans*-2-butene, and *cis*-2-butene are similar at 50–90 °C, indicating that no isomerizations was occurred during the 1,3-butadiene hydrogenation reaction [29,34,35].

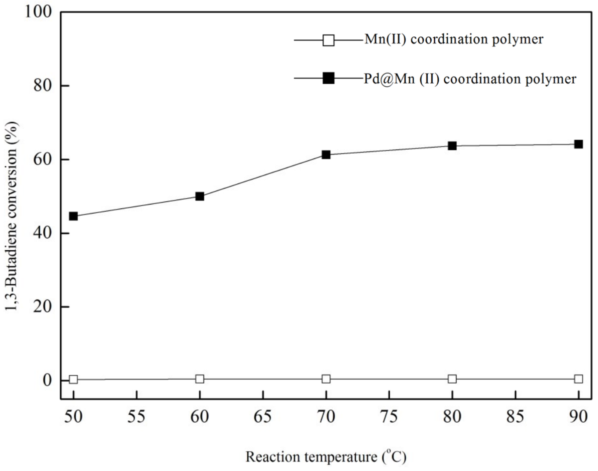

Figure 4. The conversions of 1,3-butadiene for Mn(II) coordination polymer and Pd@Mn(II) coordination polymer catalyst.

4. Conclusions

In summary, we synthesized a new six-coordinated Mn(II) coordination polymer, $[Mn(L_1)(L_2)_2]_n$ by 3-bromo-2-hydroxybenzaldehyde, NaOH, 2-aminopyrimidine and manganese(II) acetate dihydrate. The Mn(II) coordination polymer displays a 1D chained structure by the bridge effect of 2-aminopyrimidine N atoms. The catalytic activities of Mn (II) coordination polymer and Pd@Mn (II) coordination polymer for hydrogenation of 1,3-butadiene have been investigated.

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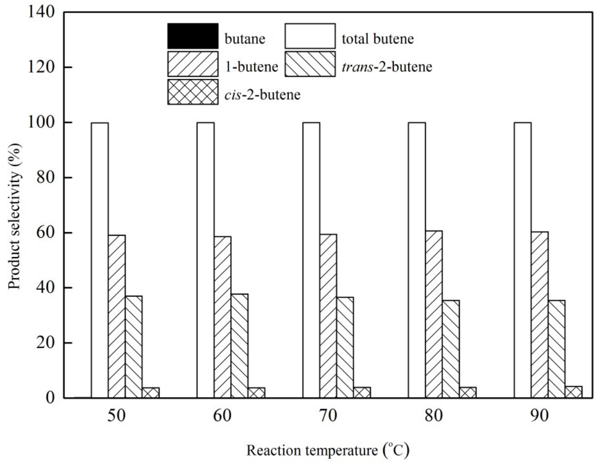

Figure 5. The product selectivity for Pd@Mn(II) coordination polymer catalyst.

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