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Influence of the ancillary ligand on the crystalline structures and magnetic properties of mesaconato-bridged Mn coordination polymers

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{[Mn(mes)(dmb)(H_2O)]·H_2O}$_n$ (**1**) and [Mn(mes)(bpy)]$_n$ (**2**); (mes = mesaconato; dmb = 5,5′-dimethyl-2,2′-bipyridine; bpy = 2,2′-bipyridine), were obtained by self-assembly solution reaction at ambient conditions, and structurally characterized by single crystal X-ray diffraction. **1** crystallizes in an orthorhombic system with a *Pbca* space group and forms a one-dimensional (1D) coordination polymer; the six-coordinated Mn(II) centers display a distorted-trigonal prismatic configuration. **2** crystallizes in an orthorhombic system with a $P2_12_12$ space group yielding a two-dimensional (2D) coordination array, their metal centers having a distorted octahedral geometry. Magnetic susceptibility measurements reveal that **1** is paramagnetic, while **2** shows weak antiferromagnetic coupling.

Introduction

The potential applications of coordination polymers, and metal-organic frameworks (MOFs), in fields such as catalysis, adsorption, luminescence, magnetism, among others, continues encouraging the growing interest in this type of inorganic-organic materials.[1] From the diverse methodologies explored and reported to synthesize these hybrid materials, the self-assembly approach seems to be the easiest and most productive of all, even tough, this method carries uncertainty about the prediction of the chemical structure and its dimensionality. Among the different organic ligands to be used in the generation of coordination polymers, the di- or multi-carboxylate ligands are the predominant ones; this can be understood due to the availability of these bridging ligands and, also, owed to the countless assortment of possible structures resulting from the various coordination modes exhibited by the carboxylate ligands.[2] The mesaconic acid is a dicarboxylic acid which has scarcely been used as bridging ligand to form coordination polymers. To our knowledge, there are few relevant reports of coordination polymers with mesaconato ions in their structure. The reaction between [Zn(tu)$_4$]Cl$_2$ and sodium mesaconato has been shown to yield a 1D zig-zag chain of {[Zn(tu)$_2$(μ-mesaconato)]·2H_2O}$_n$ (tu = thiourea).[3] The 2D [Cu$_3$(μ3-OH)(μ-pz)$_3$(MeFum)(Hpz)] and the 1D [Cu(MeFum)(Hpz)$_2$(H_2O)]·H_2O (Hpz = pyrazole; pz = pyrazolate; MeFum = 2-methylfumarate or mesaconato dianion) coordination polymers were obtained from the solvothermal and ambient reactions, respectively, of copper 2-methylfumarate with Hpz in water.[4] A 1D Cd(II) coordination polymer, [Cd$_2$(mes)$_2$(4-phpy)$_4$] (4-php = 4-phenylpyridine) was synthesized at ambient conditions using the layering methodology; the photoluminescence properties of the polymer were also described.[5] Another paper reported, for the first time, the isomerization of itaconic acid to produce mesaconato as bridging ligand, a 2D Zn(II) coordination polymer, [Zn(mes)(dpa)]$_n$ (dpa = 4,4′-dipyridylamine), was obtained under solvothermal conditions; and the luminescent properties of this polymer were stated.[6] There is also a paper about a 1D Pb(II) coordination polymer bearing the mesaconato ligand, [Pb$_2$(phen)$_2$(cit)(mes)]·2H_2O (phen = o-phenantroline; cit = citraconato), again, the mesaconato ligand was the product of the isomerization of itaconic acid as the synthesis was carried out under solvothermal conditions, the photoluminescent properties of the complex were also reported.[7] Moreover, a zirconium-mesaconato MOF, with an hexanuclear building unit and a fcu topology, was obtained using a microwave oven by reacting mesaconic acid and $ZrOCl_2$·8H_2O in a mixture of water and acetic acid at 95 °C for 5 h under stirring.[8] The 2D polymer {[Zn(mes)(4-nvp)$_2$]·H_2O}$_n$ (4-nvp = 4-(1-naphthylvinyl)pyridine) was synthesized and their crystallography study revealed water dimers inside the rectangular grids of the polymer structure; this polymer showed interesting temperature dependent

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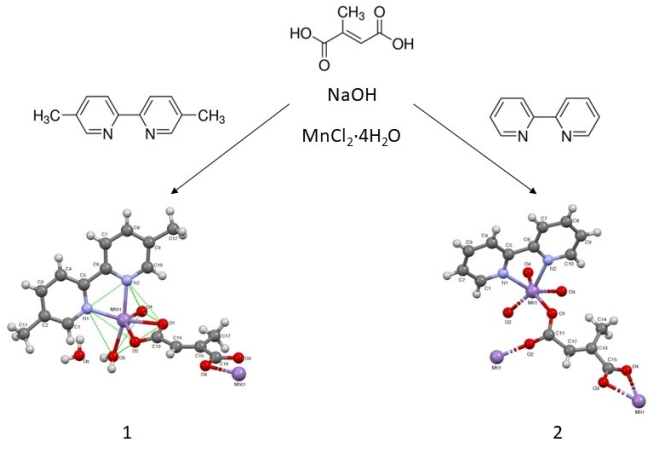
dielectric properties.[9] In a more recent work, the 2D polymer $\{[Zn(mes)(4\text{-}nvp)_2]\cdot H_2O\}_n$ (mes = mesaconato, 4-nvp = 4-(1-naphthylvinyl)pyridine) was structurally described and their properties as luminescent sensor of Pd(II) in water were also detailed.[10] Thus, we could not find a paper reporting a Mn(II)-mesaconato coordination polymer; in addition, there are no investigations reporting magnetic properties of coordination polymers having the mes ligand. Alternatively, the use of ancillary ligands, such as the 2,2′-bipyridine and its derivatives, in coordination polymers have been employed previously as an easy way to modify structural characteristics and, therefore, the intrinsic properties of these materials.[11] We have reported earlier the modification of the structure and the distinct magnetic properties found in a 1D and a 2D Mn(II)-fumarato polymers assembled with different dimethyl-2,2′-bipyridine auxiliary ligands.[12] Research on molecule-based magnetic materials has been relevant in the development of novel materials with unique, and sometimes unexpected, magnetic properties and, furthermore, in the innovation of materials having combined physical properties. These types of magnetic materials could have applications in several technological fields due to properties such as magnetic ordering with critical temperatures above ambient temperature, single molecule quantum effects, magnetic state switching through photo, thermal, or pressure stimulus, among others.[13] Herein, we describe the synthesis, structural characterization, and magnetic properties of polymers **1** and **2**, which exhibit 1D and 2D coordination arrays, respectively.

Results and Discussion

Syntheses of **1** and **2** were carried out at ambient conditions by mixing $MnCl_2\cdot 4H_2O$ with the mes ligand, and dmb and bpy co-ligands, respectively, in a water-methanol solution; polymers structures were determined by elemental analysis and single crystal X-ray diffraction analysis (see Scheme 1).



Scheme 1. Self-assembly solution synthesis of polymers $\{[Mn(mes)(dmb)(H_2O)]\cdot H_2O\}_n$ (**1**) and $[Mn(mes)(bpy)]_n$ (**2**). Molecular crystalline structures are shown.

Polymer **1** crystallizes in an orthorhombic system with *Pbca* space group and forms a one-dimensional (1D) zig-zag chain (Figure 1). The metal center in **1** is hexa-coordinated with a N2O4 coordination sphere arising from two mes ligands, one dmb ligand and one coordinated water molecule, having a distorted trigonal-prismatic geometry (Figure 1). In a perfect trigonal-prismatic geometry, the two triangular faces of the prism should be parallel; however, in **1** the two triangular faces: N1-O2-O5 and N2-O1-O4 are not parallel, and the planes defined by those faces make an angle of 27.4°. It has been reported that if, in certain complexes, the Bailar rotation is less than 30° around a hexa-coordinated sphere then its geometry can be described as distorted trigonal prism; above those degrees, the geometry should be considered as distorted-octahedral or octahedral, as correspond.[14] As a reminder, the Bailar twist is a mechanism suggested for the racemization of octahedral complexes containing three bidentate chelate rings. These complexes typically adopt an octahedral geometry; however, they can also racemize by the formation of a trigonal prismatic intermediate. Furthermore, mes bridging-coordination modes in **1** combine monodentate and bidentate chelate at the carboxylate ends (Figure 1), yielding thus the 1D chain. This kind of coordination is still considered unusual in dicarboxylato-bridging di- or multi-nuclear complexes, and polymers, and it is the first time this coordination mode appears in hybrid polymers assembled with the mes ligand. Besides, there are just one example reported about metaprism complexes with innocent ligands, including an aqua ligand, in their structures,[15] and another showing a Mn(II) coordination polymer having trigonal prismatic geometry.[16] Metaprism is the name given to

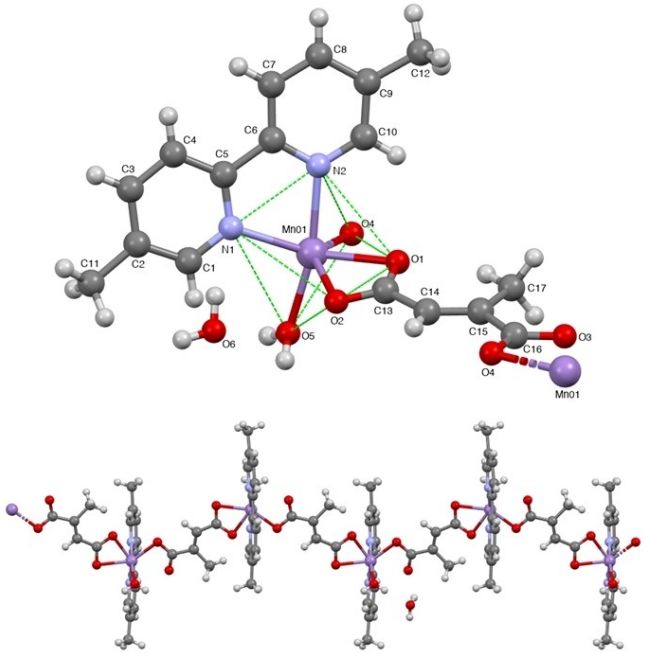


Figure 1. Molecular structure (top) and 1D polymeric structure, view looking down the *c* axis (bottom) of $\{[Mn(mes)(dmb)(H_2O)]\cdot H_2O\}_n$ (**1**).

crystal structures of complexes that lie in between two known coordination geometries, i.e. amid octahedral and trigonal prismatic in hexa-coordinated complexes. The presence of an aqua ligand and a water molecule of crystallization in **1** (Figure 1) promotes hydrogen bonding, which yields a 2D supramolecular structure. This supramolecular array is, mainly, generated by the intermolecular interactions O(5)-H(5B)···O(2), O(6)-H(6 A)···O(3) and O(6)-H(6B)···O(4) (Figure S1). Hence, polymer **1** is another example where the preference of distorted trigonal-prismatic over the characteristic octahedral, or distorted octahedral, coordination geometry might be influenced by supramolecular interactions, as we detailed in a paper published previously.[17]

Polymer **2** crystallizes in an orthorhombic system with a $P2_12_12$ space group generating a 2D coordination array (Figure 2). The metal centers in **2** have a distorted octahedral geometry; the N2O4 hexa-coordinated sphere is composed of three mes ligands and one bpy ligand (Figure 2). The mes ligand coordination modes in **2** occur as an alternation of bidentate chelate and bridging bidentate, this latter coordination approach gives rise to the 2D array (Figure 2). We have found previously the same combination of coordination modes in the 2D polymer $\{[Mn_2(fum)_2(4dmb)_2]\cdot H_2O\}_n$ (fum = fumarate; 4dmb = 4,4′-dimethyl-2,2′-bipyridine).[12] Moreover, polymer **2**

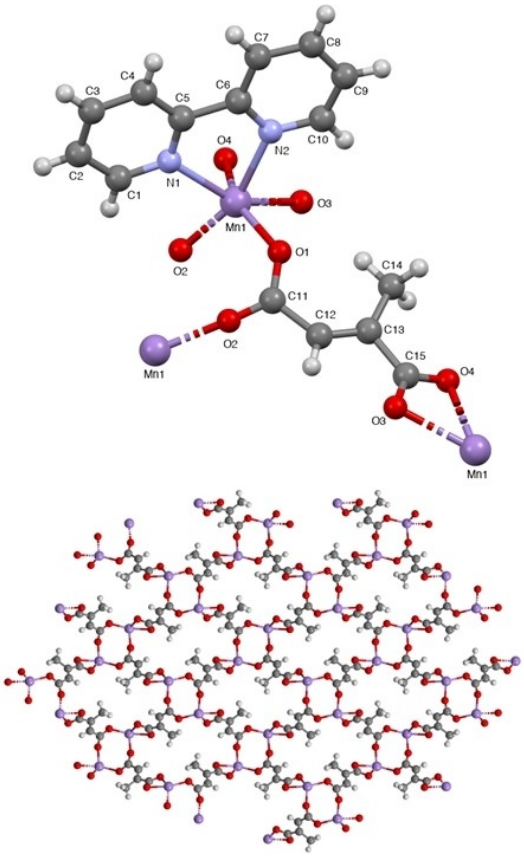


Figure 2. Molecular structure (top) and 2D polymeric structure, view looking down the *c* axis, bpy ligands removed for clarity (bottom) of $[Mn(mes)(bpy)]_n$ (**2**).

possess a ***fes*** topology, accordingly to the TOPOS PRO software analysis performed.[18] Topos PRO imports the data from the cif file of **2**. To restore the connectivity involved in a coordination array this software uses special algorithms, which are based on the Voronoi partition of the crystal space. Then, it is required to simplify the structure; in this route, the structural groups are found, the net is simplified by squeezing these groups to their centers of mass, and finally the topology of the structure is identified and classified it, and usually topological analogues of the analyzed structure are found.[18]

Hence, the different structural dimensionality obtained in **1** and **2** could be ascribed to the dissimilar steric hinderance of the dmb and bpy ligands, being larger in the former ligand. It seems that less steric hinderance in the bpy ligand of **2** allows one carboxylate end of mes ligand to coordinate in a bridging bidentate fashion to different metal centers ($\eta^1{:}\eta^1$), yielding thus a 2D structure. As above mentioned, we have described similar structural effects of the ancillary ligands in crystalline Mn(II)-fumarato coordination polymers.[12]

The magnetic susceptibility, χ, for **1** and **2** was determined by measurements performed at zero field cooling (ZFC) and field cooling (FC) from 2–300 K and decreasing. The applied magnetic field was 1000 Oe. Plots of χ and χ^{-1} versus temperature for **1** and **2** are shown in Figures 3 and 4, respectively.

The magnetic susceptibility data of **1** could be fitted to the Curie-Weiss law, $\chi = C/(T-\theta)$, with $C = 4.3\ cm^3\,K\,mol^{-1}$ and a Curie-Weiss temperature $\theta_{(C\text{-}W)} = 0.3$ K (Figure 3). The values of these magnetic parameters indicate that **1** behaves essentially as a paramagnetic system.

Conversely, observing the comportment of the χ *vs.* T plot for **2** (Figure 4), the magnetic susceptibility starts to increase steadily as temperature decreases, then, it has a sudden increase, until reaching a maximum at around 8 K (T_N), and then it drops again below that temperature. That maximum can be attributed to the Néel temperature (T_N), which usually appears in antiferromagnetic molecular systems.[19] Therefore, to characterize polymer **2** as a low-temperature molecular antiferromagnet, its magnetic susceptibility data were fitted to the Curie-Weiss law and to the Bleaney-Bowers model.[20] The Curie-Weiss plot for **2** gave the following constants: $C = 0.435\ cm^3\,K\,mol^{-1}$ and $\Theta = -26.96$ K, evidencing the weak antiferromagnetic exchange occurring between Mn(II) metal centers in **2**. Applying the Bleaney-Bowers approach to the experimental data obtained for **2**, the best fit for a coupled $S = 5/2$ dimeric unit was achieved with $J/k_B = -3.7$ K ($-2.6\ cm^{-1}$), $g = 0.51$, $\Theta = -27.9$ K and $\rho = 4.1\,\%$ (Figure 4). Although, g value is low, the Bleaney-Bowers model describes adequately the experimental results, corroborating the antiferromagnetic interaction between Mn(II) centers in **2**. Previous works applying this model to coordination polymers,[21] have reported J values determined in antiferromagnetic molecular structures[22] comparable to the one we obtained. The Θ negative values attained for both models are close, proving thus the existence of the low-temperature weak antiferromagnetic coupling in polymer **2**. As a comparison, a Mn(II) 1D coordination polymer, $[Mn(adc)(4\text{-phpy})_2(H_2O)_2]_n$, ($H_2adc$ = acetylenedicarboxylic acid and 4-phpy = 4-phenylpyridine) was synthesized and its magnetic

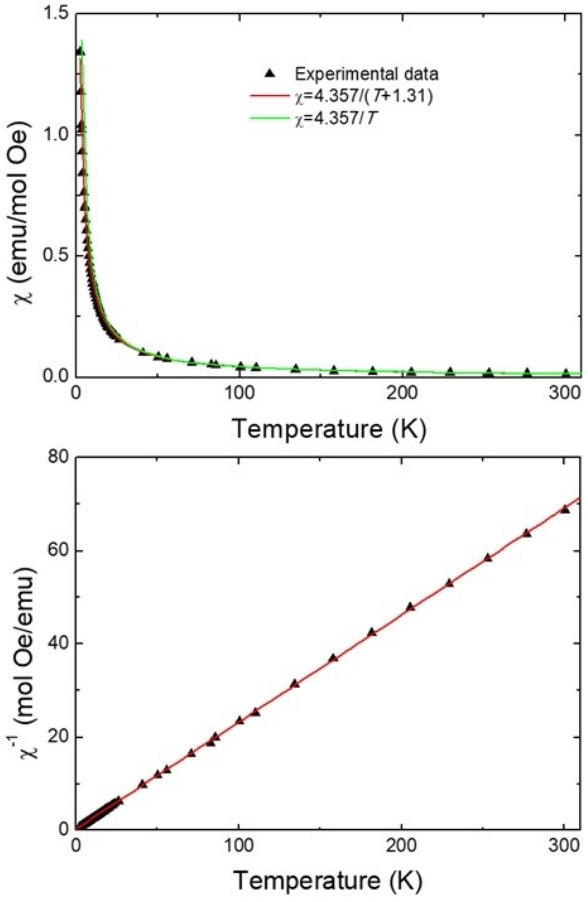


Figure 3. χ vs. T plot (top) and χ-1 vs. T plot (bottom) for {[Mn(mes)(dmb)(H_2O)]·H_2O}$_n$ (**1**). In both plots, lines correspond to Curie-Weiss law fittings.

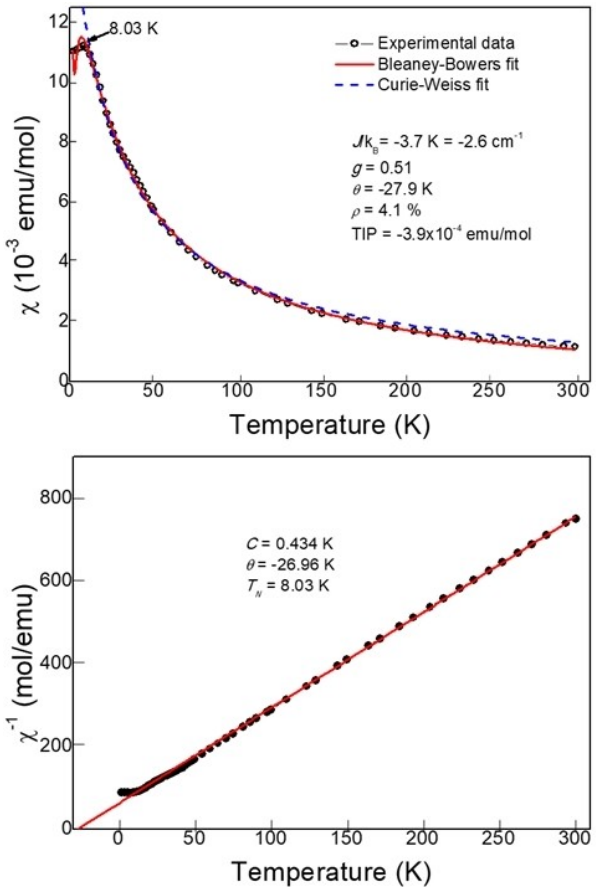


Figure 4. χ vs. T plot (top) and χ-1 vs. T plot (bottom) for [Mn(mes)(bpy)]$_n$ (**2**). In the χ vs. T plot, continue line corresponds to Bleany-Bowers model fitting and discontinue line corresponds to Curie-Weiss law fitting. In the χ-1 vs. T plot (bottom), continue line corresponds to Curie-Weiss law fitting.

studies revealed a very weak antiferromagnetic exchange *via* the Mn-Mn dicarboxylate bridge of the adc ligand.[23] Accordingly, the different magnetic properties determined in polymers **1** and **2** could be attributed to their structural characteristics. Thus, in the 1D polymer **1** the distance between two Mn metal centres is 9.0 Å; while, in its supramolecular structure the distance shortens to 6.1 Å. On the contrary, in the 2D polymer **2** the shortest distance between two metal centres is 4.5 Å, producing thus a better scenery for the antiferromagnetic exchange exhibited.

Conclusions

Two Mn(II) coordination polymers bearing mesaconato (mes) as bridging ligand, and dimethyl-2,2′-bipyridine (dmb) and 2,2′-bipyridine (bpy) as ancillary ligands, have been synthesized by self-assembly reactions at ambient conditions. Single crystal X-ray diffraction studies showed that in polymer **1** the mes ligand coordinates as η^1:η^0, yielding a 1D polymer, while in **2**, the η^1:η^1 and μ_2-η^1:η^1 coordination modes of mes alternate generating a 2D array. We believe that the different dimensionalities acquired by the polymers are due to the diverse steric hinderance found in the ancillary ligands. Less steric hinderance in bpy co-ligand allows a higher dimensionality in polymer **2**. The structural differences in coordination polymers **1** and **2** are revealed in their magnetic properties; consequently, polymer **1** displays a paramagnetic behavior, whereas polymer **2** possess weak antiferromagnetic interactions corroborated by Curie-Weiss law and Bleaney-Bowers model.

Experimental Section

Materials and instrumentation: All chemicals were of analytical grade, purchased commercially (Aldrich) and were used without further purification. Elemental analyses for C, H, N were carried out for standard methods using a Vario Micro-Cube analyzer. Crystallographic data collection for **1** and **2** were performed at 100 K using MoKα radiation (0.71073 Å) on a Bruker APEX II CCD from an Incoatec ImuS source and Helios optic monochromator.[24] Suitable crystals were coated with hydrocarbon oil, picked up with a nylon loop, and mounted on the cold nitrogen stream of the diffractometer. The structures were solved by direct methods[25] and refined by full-matrix least-

squares on F2 using the shelXle GUI.[26] For **1** and **2**, the hydrogen atoms of the C–H bonds were placed in idealized positions whereas the hydrogen atoms from O–H moieties (water molecules) were localized from the difference electron density map and their position was refined with Uiso = aUeq (where a is 1.5 for –CH3 and 1.2 for O–H moieties and the others). Compound **1** presents a positional disorder in the dicarboxylate ligand that was modeled in two positions in ratio 90/10 of occupancy, while **2** presents a positional disorder in the dicarboxylate ligand that was modeled in two positions in ratio 54/46 of occupancy. The disorder in compound **1** was modeled using SIMU, DELU and SAME instructions, and for **2** using SIMU, RIGU and SAME implemented in shelXle GUI, and their occupancy was calculated using free variable. The compound **2** crystallized in chiral space group $P2_12_12$ with absolute structure parameter of 0.031(8). Magnetic susceptibility measurements of **1** and **2** were performed in a MPMS Quantum Design magnetometer with measurements made at zero field cooling (ZFC) and field cooling (FC) from 2–300 K and decreasing. The applied magnetic field was 1000 Oe, and the total diamagnetic corrections were estimated using Pascal's constants as -255×10^{-6} cm^3 mol^{-1}.

CCDC deposition numbers 2301207 and 2301208 contain supplementary crystallographic data for 1 and 2, respectively. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving/html, or from Cambridge Crystallographic Data Center (CCDC), 12 Union Road, Cambridge CB2 1EZ, UK [Fax: (+44) 1223–336-033; Email: deposit@cdc.cam.ac.uk].

Synthesis of 1: A 10 ml solution of sodium hydroxide (1 mmol) was added to an ethanol solution (10 ml) of mesaconic acid (0.5 mmol) while stirring. Then, a deionized water solution (10 ml) containing 5,5′-dimethyl-2,2′-bipyridine (0.5 mmol) and $MnCl_2\cdot4H_2O$ (0.5 mmol) was poured to the initial solution while stirring. A light-yellow translucent solution was obtained. After five days, yellow crystals were obtained then filtered, washed with a 50:50 deionized water-methanol solution and air-dried. Yield: 56% based on metal precursor. Anal. calcd. for $C_{17}H_{20}MnN_2O_6$: C, 50.63; H, 4.53; N, 6.94%; found: C, 50.27; H, 4.91; N, 6.81%.

Synthesis of 2: A 10 ml solution of sodium hydroxide (1 mmol) was added to an ethanol solution (10 ml) of mesaconic acid (0.5 mmol) while stirring. Then, a deionized water solution (10 ml) containing 5,5′-dimethyl-2,2′-bipyridine (0.5 mmol) and $MnCl_2\cdot4H_2O$ (0.5 mmol) was poured to the initial solution while stirring. A light-yellow translucent solution was obtained. After five days, yellow crystals were obtained then filtered, washed with a 50:50 deionized water-methanol solution and air-dried. Yield: 62% based on metal precursor. Anal. calcd. for $C_{15}H_{12}MnN_2O_4$: C, 53.11; H, 3.56; N, 8.26%; found: C, 53.37; H, 3.60; N, 7.98%.

Supporting information (see footnote on the first page of this article): X-ray crystallography data.

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Conflict of Interest

The authors declare no conflict of interest.

Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

Keywords: Mn coordination polymer · mesaconato · 2,2′-bipyridine · antiferromagnetism

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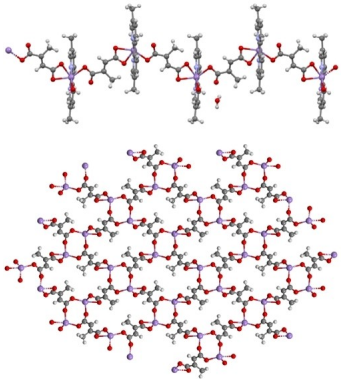

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