ORIGINAL PAPER

Two-Dimensional Copper Coordination Polymer Assembled with Fumarate and 5,5'-Dimethyl-2,2'-bipyridine: Synthesis, Crystal Structure and Magnetic Properties

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Abstract

$[[Cu(fum)(dmb)] \cdot H_2O]_n$ (**1**) (fum = fumarate; dmb = 5,5'-dimethyl-2,2'-bipyridine) was obtained by a self-assembly solution reaction, at ambient conditions, and characterized by elemental analysis, IR spectroscopy and X-ray single crystal diffraction. Crystallographic studies show that **1** crystallizes in a triclinic system with a *P-1* space group, with $a = 8.2308(2)$ Å, $b = 9.7563(2)$ Å, $c = 10.3990(2)$ Å; $\alpha = 80.3444(4)°$, $\beta = 77.9517(4)°$, $\gamma = 82.0440(5)°$; $V = 800.45(3)$ Å^3. The Cu(II) centers are five-coordinated with a distorted square pyramidal configuration. The formation of a two-dimensional (2D) array in **1** can be explained by the presence of two different coordination modes in the fumarate ligand: $\mu\text{-}\eta^1\text{:}\eta^0$ and $\mu_2\text{-}\eta^2\text{:}\eta^0$, both in a bridging monodentate manner, the latter generating distinctive rhombic-dinuclear units. The thermal stability of **1** has also been analyzed. Magnetic measurements revealed that this polymer exhibits weak antiferromagnetic ordering.

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Graphical Abstract

[[Cu(fum)(dmb)]·H2O]n, exhibiting weak antiferromagnetic interactions, displays a two-dimensional array comprised of rhombic dinuclear units, where the carboxylate moieties of fumarate bridging ligand displays monodentate and oxo-bridging coordination modes connecting two Cu centers.
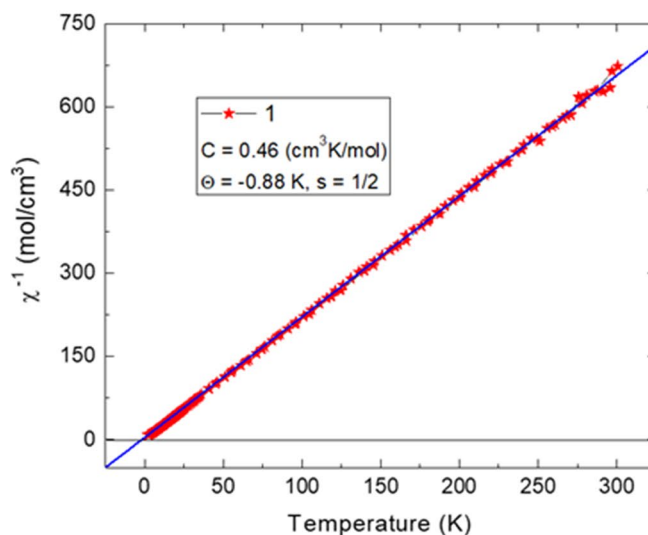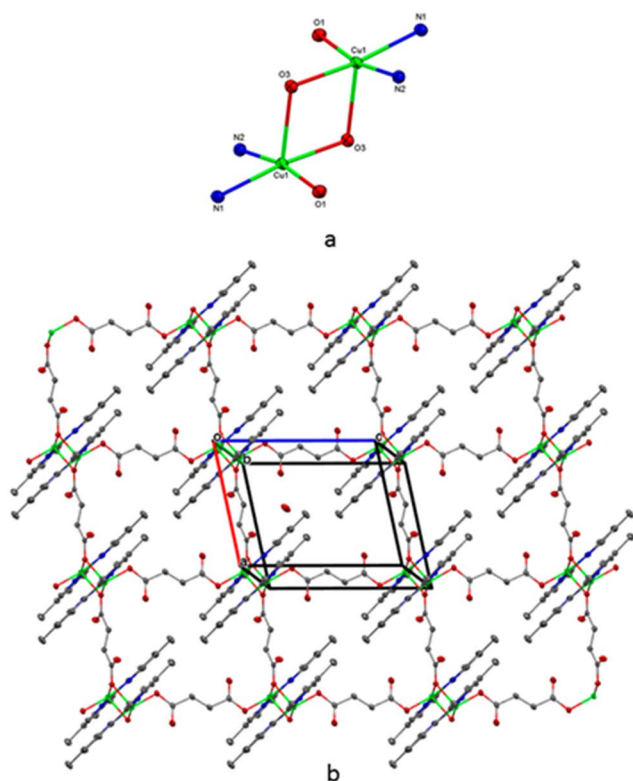
Introduction

For several decades, coordination polymers have been developed with a twofold purpose, to gain basic structural knowledge about these hybrid materials, and to study their properties, such as catalytic, magnetic, and luminescent, trying to make them useful in scientific and technological applications [1–3]. Many researchers in the field, have used the hydrothermal and solvothermal methods as the primary ones to obtain a great number of crystalline coordination polymers [4]; nonetheless, we believe that the self-assembly synthetic strategy at ambient conditions yields, also, interesting crystalline structures, which usually display supramolecular arrays that are involved in the intrinsic properties of these coordination compounds. Dicarboxylate ligands continue being one of the most employed bridging ligands to achieve coordination networks [5]. Moreover, fumarate ligand has been widely used for the formation of complexes [6] and coordination polymers [7]. As it is well known, carboxylate ligands can bind to metal ions in different modes:

monodentate, chelate, monoatomic bridges, bridging bidentate (μ-η^1:η^1), bridging tridentate (μ_3-η^1:η^2), and the less common multiple bridging mode [8]; these divergent coordination modes, make these ligands very versatile regarding the structural dimensionality that can be obtained in the final coordination compound. Thus, previous works have used the combination of carboxylate bridging ligands and 2,2'-bipyridine [9–11] or di-alkyl-2,2'-bipyridines as ancillary ligands to generate 1- and 2-D coordination polymers mainly. Even though, ancillary ligands such as the di-alkyl-2,2'-bipyridines can promote the crystalline stability of the polymers, via supramolecular interactions, and the study of the steric hindrance influence in the dimensionality of the final compound is of significance, few studies have been published using them [12, 13].

Herein, we describe the synthesis, crystalline molecular structure, thermal analysis and magnetic properties of the novel 2D Cu(II) coordination polymer (**1**) bearing fumarate (fum) as bridging ligand and 5,5'-dimethyl-2,2'-bipyridine (dmb) as ancillary ligand.

Experimental

Materials and Measurements

All chemicals were of analytical grade, purchased commercially (Aldrich) and were used without further purification. Synthesis was carried out in aerobic and ambient conditions. Elemental analyses for C, H, N were carried out by standard methods using a Vario Micro-Cube analyzer. IR spectra of the complexes were determined as KBr disks in an Avatar 360 FT-IR Nicolet spectrophotometer from 4000 to 400 cm^{-1}. Thermogravimetric analyses were performed in a TA Instruments equipment, under N_2 atmosphere, at a heating rate of 10 °C min^{-1}, from 20 to 800 °C. Magnetic characteristics of **1** were determined in a MPMS Quantum Design magnetometer with measurements performed at zero field cooling (ZFC) and field cooling (FC) from 2 to 300 K and decreasing. The applied magnetic field was 1000 Oe, and the total diamagnetic corrections were estimated using Pascal´s constants as -245×10^{-6} $cm^3 mol^{-1}$.

Synthesis of 1

A methanol solution (10 ml) of 5,5'-dimethyl-2,2'-bipyridine (0.0184 g; 0.1 mmol) was added to an aqueous solution (5 ml) of sodium fumarate (0.0160 g; 0.1 mmol), under stirring. To this solution, $Cu(NO_3)_2 \cdot 2.5H_2O$ (0.0211 g; 0.1 mmol) in 5 ml of de-ionized water was added. A blue solution was obtained. After three days, blue crystals were obtained as needles, then filtered and washed with a 50:50 deionized water–methanol solution and air-dried. Yield: 72% based on metal precursor. Anal. calc. for $C_{16}H_{16}CuN_2O_5$ (FW = 379.85): C, 50.65; H, 4.22; N, 7.38%. Found: C, 50.25; H, 4.04; N, 7.30%. IR (cm^{-1}): 3530 (vs, sh), 3480 (s, sh), 3050 (s, sh), 1960 (w), 1910 (w), 1860 (w), 1650 (m, sh), 1590 (vs), 1480 (m), 1370 (m), 1250 (m), 1200 (m), 1050 (m, sh), 985 (m), 930 (w), 841 (m, sh), 790 (m), 690 (s, sh), 582 (m), 536 (m), 482 (m), 420 (m).

Crystal Structure Determination

Crystallographic data for **1** were collected on a Bruker SMART APEX DUO three-circle diffractometer equipped with an Apex II CCD detector using MoKα (λ = 0.71073 Å, Incoatec IμS microsource) at 100 K [14]. The crystal was coated with hydrocarbon oil, picked up with a nylon loop, and mounted in the cold nitrogen stream (100 K) of the diffractometer. The structure was solved by direct methods (SHELXS-97) and refined by full-matrix least-squares on F^2 [15] using the shelXle GUI [16]. The hydrogen atoms of the C–H bonds were placed in idealized positions whereas the hydrogen atoms from H_2O moieties were localized from the difference electron density map, and their position was refined with U_{iso} tied to the parent atom with distance restraints at standard distances (0.84 Å for O–H bond) using distance restraints (DFIX). The crystallographic data and refinement details for polymer **1** are summarized in Table 1. Selected bond distances and bond angles are listed in Table 2.

Results and Discussion

Synthesis

Using a very simple methodology of self-assembling solution reaction, equivalent amounts of sodium fumarate (fum), $Cu(NO_3)_2 \cdot 2.5H_2O$ and 5,5'-dimethyl-2,2'-bipyridine (5dmb), were mixed in water–methanol solution, under ambient conditions. Slow evaporation of solvents yielded

Table 1 Crystal data and structure refinement parameters for **1**

Empirical formula	$C_{16}H_{16}CuN_2O_5$
Formula weight	379.85
Temperature (K)	100(2)
Wavelength (Å)	0.71073
Crystal system	Triclinic
Space group	P-1
a (Å)	8.2308(2)
b (Å)	9.7563(2)
c (Å)	10.3990(2)
α (°)	80.3444(4)
β (°)	77.9517(4)
γ (°)	82.0440(5)
Volume ($Å^3$)	800.45(3)
Z	2
D_{calc} (Mg/m^3)	1.576
Absorption coefficient (mm^{-1})	1.393
F(000)	390
Crystal size (mm^3)	$0.399 \times 0.204 \times 0.056$
Theta range for data collection (°)	2.023 to 25.349
Index ranges	$-9 \leq h \leq 9$, $-11 < = k \leq 11$, $-12 \leq l \leq 12$
Reflections collected	13,144
Independent reflections	2931 [R(int) = 0.0195]
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	2931/3/225
Goodness-of-fit on F^2	1.076
Final R indices [I > 2sigma(I)]	R1 = 0.0210, wR2 = 0.0551
R indices (all data)	R1 = 0.0217, wR2 = 0.0556
Largest diff. peak and hole (e.$Å^{-3}$)	0.340 and -0.245

Table 2 Selected bond distances (Å) and angles (°) for **1**

	Bond lengths (Å)		
Cu(1)-O(1)	1.9498(11)	Cu(1)-N(2)	2.0111(13)
Cu(1)-N(1)	1.9983(14)	Cu(1)-O(3)#1	2.2294(11)
Cu(1)-O(3)	1.9994(11)		
	Angles (°)		
O(1)-Cu(1)-N(1)	92.48(5)	O(3)-Cu(1)-N(2)	95.99(5)
O(1)-Cu(1)-O(3)	89.02(5)	O(1)-Cu(1)-O(3)#1	90.33(4)
N(1)-Cu(1)-O(3)	171.01(5)	N(1)-Cu(1)-O(3)#1	112.75(5)
O(1)-Cu(1)-N(2)	168.72(5)	O(3)-Cu(1)-O(3)#1	76.10(5)
N(1)-Cu(1)-N(2)	81.03(5)	N(2)-Cu(1)-O(3)#1	100.66(5)
	D-H…A	d(H…A)	d(D…A)	<(DHA
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O(5)-H(5B)…O(4)#3	0.851(16)	1.998(16)	2.8379(18)	169(2)
O(5)-H(5A)…O(2)	0.836(15)	2.024(16)	2.8572(18)	175(2)

Symmetry transformations used to generate equivalent atoms:

[#]1 $-x$, $-y + 1$, $-z$ [#]2 $-x$, $-y + 1$, $-z + 1$ [#]3 $-x + 1$, $-y + 1$, $-z$

blue crystals of **1**. These crystals are insoluble in common solvents and, air and moisture stable.

Crystal Structure of 1

[[Cu(fum)(dmb)]·$H_2O]_n$ (**1**) crystallizes in a triclinic system with a *P-1* space group and forms an infinite two-dimensional (2-D) coordination polymer. The asymmetric unit consists of one Cu^{2+} ion, two halves of fumarate ligand, one bipy ligands and one guest H_2O molecule (Fig. 1a). The Cu center is five-coordinated and surrounded by three oxygen atoms from the fum ligands and two nitrogen atoms from one dmb ligand. The metal center displays a slightly distorted square pyramidal configuration (Figs. 1b and 2a). In this coordination geometry, the basal plane is defined by O1, O3, N1 and N2, from the fum and dmb ligands, respectively. The apical position is occupied by O3, from one fum carboxylate. Towards the apical ligating atom, the metal ion is deviated from the corresponding basal plane by 0.170 Å. The basal plane is found to be tetrahedrally distorted with a τ value of 0.208 [τ = (171.01–168.72)/60 = 0.038] [16] (Fig. 2a). In this polymer, all fum ligands bind the metal centers in a monodentate bridging fashion; however, a unique Cu(II) dinuclear repeat unit appears when two fumarate ligands bind, with one oxygen atom (O3) each, to two different Cu ions (μ₂-oxo), forming a rhombic cluster made of two Cu ions and two oxygen atoms (Figs. 1b and 2). The angles O3-Cu-03 = 76.10° and Cu1-O3-Cu1 = 103.91° corroborate the rhombic and planar (dihedral angle of 0.0°) geometry of the dimer. The distance between Cu•••Cu ions is 3.33 Å, which is larger than those found for paddle-wheel type compounds (2.58–2.65 Å) [17], although it is closer to those originated in complexes having bridging Cu–O–Cu square

dimeric clusters (3.05–3.12 Å) [18, 19]. Thus, these two Cu(II) centers are triple bridged by two fum anions, one with μ₂-η²:η⁰, and the other with μ-η¹:η⁰ coordination modes, via monoatomic bridging fashion (Fig. 1b). This kind of dimeric cluster is, to some extent, rare, since few coordination complexes or polymers have been reported showing this type of Cu dimeric unit [19–21], and only one of them exhibiting the fum ligand [21]; in that reported 2D polymer, the Cu… Cu distance is 3.45 Å, which is closer to that obtained for **1**. In concordance to the carboxylate-bridges coordination modes, this dimeric unit in **1** possesses a *syn–syn* configuration and an equatorial-equatorial conformation arrangement. In a broader view, another fum ligand binds to these Cu centers in a bridging monodentate manner, as mentioned above, connecting the rhomboid Cu dimers to larger fusion-cycles made of four Cu centers all linked and bridged by four fum ligands. Moreover, these coordination modes of fum ligand give rise to a 2-D array in the coordination polymer **1** (Fig. 2b). The 2D structure of **1** can be classified as uninodal 4-connected layer with the sql (Shubnikov tetragonal plane net) topology and the point symbol of {4⁴.6²} [22]. The crystal packing of **1** is stabilized also by hydrogen bonds. Adjacent complex units are connected by hydrogen bonds between the non-coordinated, or lattice, water molecule and the non-coordinated carboxylate oxygen atoms of fum: O(5)–H(5B)–O(4) and O(5)–H(5A)–O(2) (Table 2). Nevertheless, these supramolecular interactions do not affect the structural dimensionality of **1**. Accordingly to previous results in the synthesis and characterization of coordination polymers bearing dicarboxylate bridging ligands and 2,2'-bipyridine, and its dimethyl derivatives, as ancillary ligands [10], the position of the methyl groups in the bipyridine rings seems to have effect in the dimensionality of
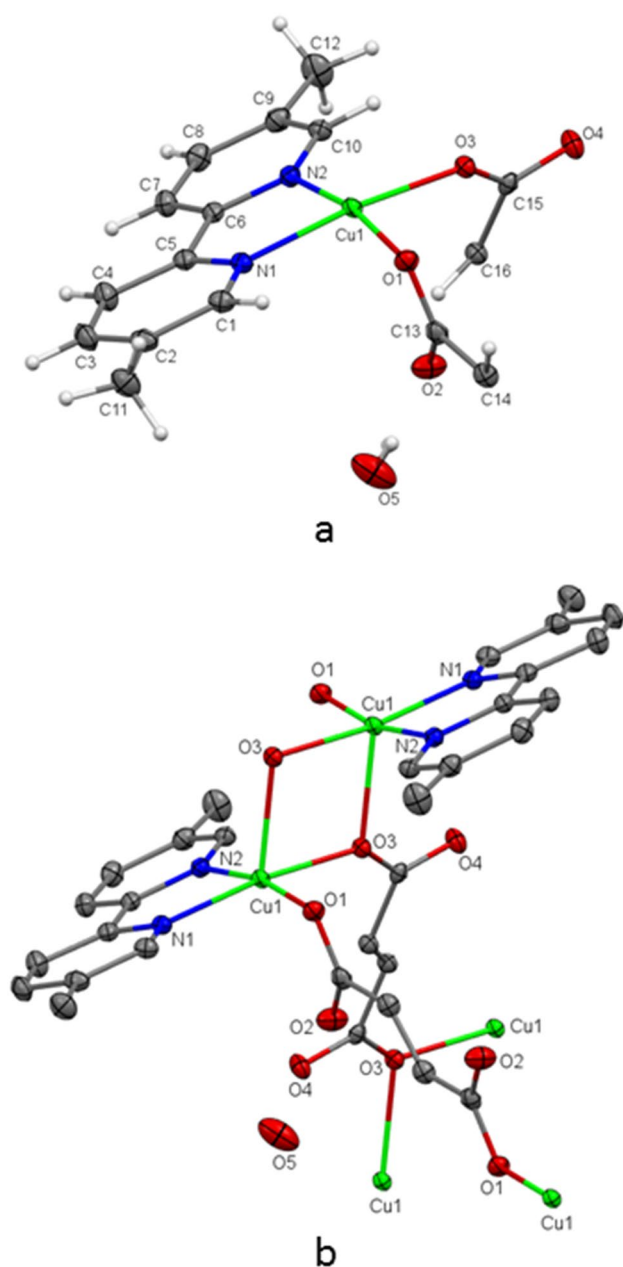
Fig. 1 Asymmetric unit of [[Cu(fum)(dmb)]·H$_2$O]$_n$ (**1**) (**a**). Molecular structure of **1** (**b**). (Ellipsoids shown at 60% probability). Hydrogens are omitted for clarity

the polymer [12, 13]. Commonly, when an ancillary ligand with larger steric hindrance (5,5'-dimethyl-2,2'-bipyiridine; 4,4'-tert-buthyl-2,2'-bipyridine) is utilized, 1D coordination polymers are typically formed and, when 2,2'-bipyridine, and, sometimes, 4,4'-dimethyl-2,2'-bipyridine are employed, 2D coordination arrays can be attained. Consequently, **1** is one of the few examples where, regardless of the steric effect of the methyl moieties in dmb, a 2D network is accomplished. It is important to emphasize that the coordination modes of dmb, above mentioned, also significantly

influences the dimensionality of the resultant coordination polymer **1**.

Thermogravimetric Analysis of 1

To examine the thermal stability of the polymer, thermal analysis was performed for **1** between 20 and 800 °C (Fig. S1, Supplementary Data). Polymer **1** exhibits mainly four decomposition stages. The first weight loss (3.78%) for **1** occurs between 120 and 160 °C, the second one, with a weight loss of 42.75% of the initial weight, takes place approximately between 190 and 242 °C; the third stage corresponds to a weight loss of 31.88% and occurs between 254 and 390 °C. The last weight loss (6.42%) occurs at 395 °C where only ~ 19% of the initial sample weight remains at 800 °C. The first decomposition stage can be endorsed to the loss of non-coordinated water; the rest of the stages can be attributed practically to the combined weight loss of the fum (calcd. 30.03%) and dmb (calcd. 48.50%) ligands. The residual of the initial weight loss, at 800 °C, can be assigned to CuO (calcd. 20.94%).

Magnetic Properties of 1

DC magnetic susceptibility χ(T), in terms of cm^3/mol, was determined and plotted in Fig. 3, at zero field cooling (ZFC) and field cooling (FC) modes, from 2 to 300 K and decreasing, in an applied field of 1000 Oe. χT value at room temperature is 0.44 cm^{3}mol^{-1} K, which is close to the value expected for one magnetically isolated Cu^{2+} ion, Pascal constants were added. Because of the occurrence of dinuclear Cu(II) clusters along the 2D polymer structure in **1**, and the results of its experimental magnetic studies, we thought that this compound would present small antiferromagnetic interactions within the dimeric Cu(II) unit. Thus, the experimental data were fit using Bleaney–Bowers expression for a coupled $S = 1/2$ dimeric unit [23]. It is important to mention that this model has been applied for coordination polymers having similar dinuclear units as in our compound [24]. Nevertheless, the fitting results gave unphysical results: $J/k_B = 102.9$ cm^{-1}, $g = 0.025$, and $\theta = 2$ K, here J is the magnetic spin exchange, k_B is the Boltzmann constant. The Hamiltonian used was H$= -2 J(S_1 S_2)$, for Cu magnetic moments. Figure 3 shows the results of this fitting with Bleaney–Bowers and Curie–Weiss. Green line corresponds to the fitting of the complete Bleaney–Bowers equation [24], including the two terms both, at high and low temperature. As seen, the fitting is not good. The fitting using only the first term of the same equation, still is out of the experimental curve (red line), although is slightly better to the experimental data. This gives more representative physical parameters, $J/k_B = -1.21$ cm^{-1} and $g = 0.5$, this last g value is still unrealistic, and must be close to $g = 2$. Fitting
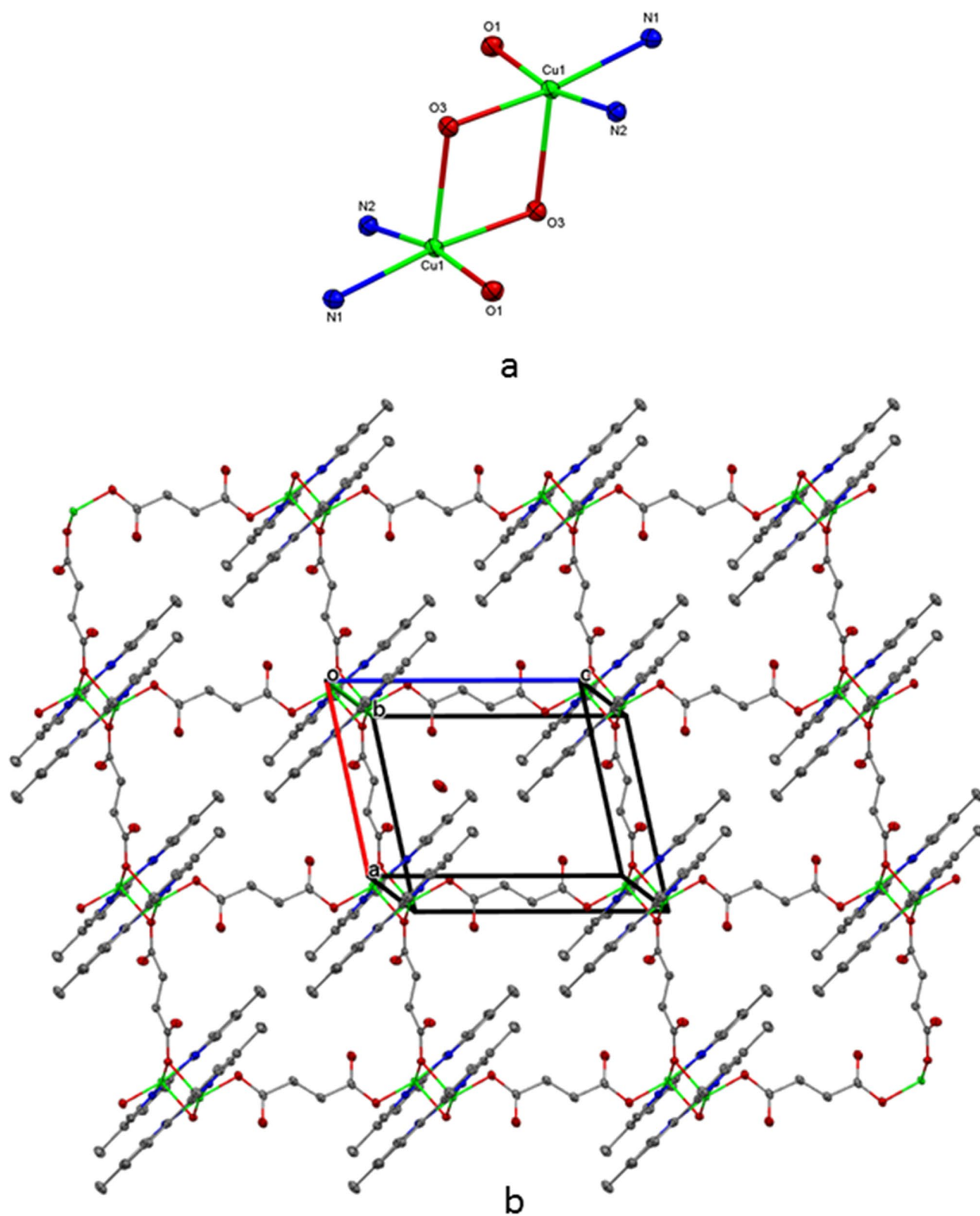
Fig. 2 Detail of the rhombic Cu(II) dinuclear unit with distorted square pyramidal coordination geometry (**a**) and 2D polymer crystalline structure, view looking down almost *b* axis (**b**), of **1**. (Ellipsoids shown at 60% probability). Hydrogens are omitted for clarity
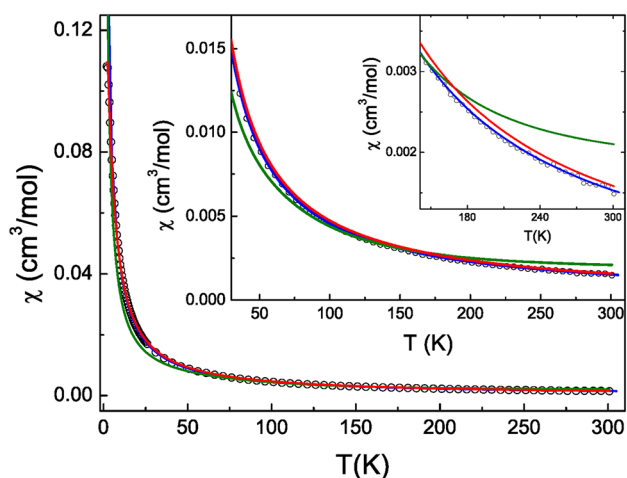
Fig. 3 χ vs. T plots for **1**. Experimental data fitting to Curie–Weiss law (blue line), and two variations of Bleaney–Bowers model (red and green lines). Inset plots show the Bleaney–Bowers model deviations from experimental values at specific magnetic susceptibility and temperature intervals (Color figure online)
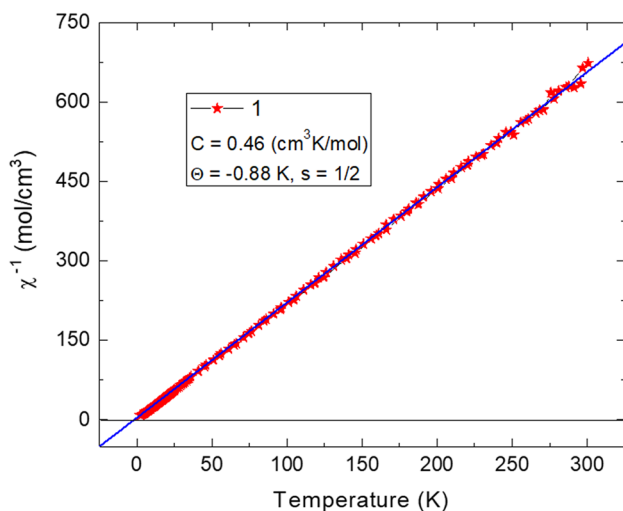

Fig. 4 χ^{-1} vs. T plot for **1**. Blue line corresponds to the fitting of experimental data to Curie–Weiss law (Color figure online)

results reported by authors using different magnetic models for similar 2D Cu(II) systems [20, 21], have not given good results. Thus, the best fitting is obtained with Curie–Weiss law, the blue line in Fig. 3 shows the fit of the experimental data with Curie–Weiss. The obtained parameters with the inverse susceptibility, $\chi^{-1}(T)$, see Fig. 4, shows that $C = 0.46$ cm^{3}Kmol^{-1}, and $\theta_{(C-W)} = -0.88$ K, which indicates weak coupling antiferromagnetic interactions. This obtained Curie–Weiss temperature can be used to estimate the value of the magnetic interaction by using a mean-field expression as: $\theta = zJS(S+1)/3k_B$ [25]; where z is the number of nearest-neighbor ions, J is the exchange integral, S is the spin. The estimated values for $zJ = -2.45$ cm^{-1}. Comparable J values have been obtained for other weak antiferromagnetic systems ($J = -2.9$ and -3.0 cm^{-1}), where metal ions are bridged by fum ligands. It is believed that the long distance between metal centers precludes the intramolecular magnetic coupling between metal ions [26]. So, the magnetic behavior of **1** agrees well with a week antiferromagnetic interaction between Cu(II) ions, which has been usually found for the *syn–syn*, equatorial-equatorial arrangement in carboxylate bridges of metal ions in similar dinuclear clusters [19, 21, 27]. The reported magnetic properties of a 2D polymer with Cu(II) dimer unit including the fum ligand, similar to **1**, were also described as having an antiferromagnetic behavior at low temperature; no magnetic parameters were specified for that polymer [21]. It is also known that the magnitude of coupling constants in oxygen bridged Cu(II) dimers can be correlated to several structural characteristics, such as: O–Cu–O angle, including planarity of the bridging oxygen atom, Cu…Cu distance, tetrahedral distortion of the N2CuO2 plane, and magnitude of the axial interactions [28].

Conclusion

A copper coordination polymer based on fumarate (fum) bridging ligand, and 5,5'-dimethyl-2,2'-bipyridine (dmb) as co-ligand, has been synthesized by self-assembly, solution reaction, under ambient conditions. The crystalline structure of **1** displays a two-dimensional array, composed of rhombic dinuclear units, where the carboxylate moieties of fum ligand exhibits two different coordination modes in a bridging monodentate fashion: monodentate and oxo-bridging connecting two Cu(II) ions. This last coordination motif generates the uncommon dinuclear unit. Furthermore, polymer **1** possesses weak antiferromagnetic properties, as determined by fitting experimental magnetic susceptibility data to Curie–Weiss law, with $\theta_{(C-W)} = -0.88$ K and $zJ = -2.45$ cm^{-1}. Studies of coordination polymers like **1** are underway also using fumarate and larger dicarboxylate bridging ligand, such as muconate, and different metals: Mn, Co and Zn; besides the determination of their magnetic and photophysical properties, respectively, these hybrid materials will be proven as potential photocatalysts in the elimination of organic pollutants in water.

Supplementary Data

CCDC-1013317 contains supplementary crystallographic data for **1**. 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].

Disclosures

Conflict of interest The authors declare that they have no conflict of interest.

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