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$[Cu_2(ox)(dien)_2](NO_3)_3$, a precursor for preparation of CuO nanoparticles: Synthesis, structural, Hirshfeld surface analyses, and physico-chemical investigations

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ABSTRACT

$[Cu_2(ox)(dien)_2](NO_3)_3$ with (ox=oxalate, dien=diethylenetriamine) has been synthesized and characterized by single-crystal X-ray diffraction as well as FTIR and UV–Vis spectroscopic techniques. The complex crystallizes in the monoclinic space group (P2/c) with the following cell parameters (Å, °): a = 23.7888(10), b = 6.7055(3), c = 12.7842(6) and β = 95.534(2). The 3D network consists of (C_2O_4) groups bridging binuclear Cu(II) cations, in which the copper atoms are in a distorted square-pyramidal coordination environment. Experimental and computed FT-IR results confirmed the presence of characteristic bands of diethylenetriamine tridentate, nitrate and oxalate bidentate groups. UV–Vis spectrum of the complex was recorded and the characteristic transitions were determined. TG-DSC measurements revealed thermal stability of the studied complex until 473 K. Calcination of the complex under air led to the production of CuO nanoparticles. Moreover, the morphology and the size of the complex and its CuO nanoparticles were monitored by scanning electron microscopy (SEM). Magnetization and a.c. susceptibility were measured and discussed. The complex molecular structure was optimized and the simulated geometric parameters compared with the crystal structure values. *Hirshfeld* surface and topological analyses were performed to describe the intermolecular interactions and to simplify the 3D networks of $[Cu_2(ox)(dien)_2](NO_3)_3$. Moreover, its antioxidant activity was assessed using DPPH, ferric reducing power tests and phosphomolybdenum assay.

1. Introduction

The chemistry of oxalate-containing compounds has shown a large interest and has become an active area of research in the past decades, thanks to its relevant coordination properties and its ability to bind many metal ions in bis-chelating bridging mode. The oxalate anion $(C_2O_4)^{2-}$ has a wide application area such as catalysis [1,2] and biological chemistry [3]. In this last field, the oxalate ion $(C_2O_4)^{2-}$ has been investigated to analyze its capability to mediate the exchange coupling between first-row transition metal ions separated by more than 5 Å in both homo- [4–7] and heteropolynuclear [8–10] compounds. A great number of oxalato-bridged binuclear complexes have been characterized so far [11–16]. Such as oxalato-bridged homometallic copper (II) compounds [17–36], where (C_2O_4) is binding the two metallic centers in bis-bidentate bridging mode [17,19-34]. Analysis of the factors that influence the extent of the coupling through oxalate allowed to tune the value of the singlet-triplet energy gap (J) in oxalato-bridged binuclear copper (II) in which the separation distance between the metal ions is in the range 5–6 Å, as reported in the literature [23,31-33] ($|J|$= 12–76 cm^{-1}). However, large J values ($|J|$ = 274–424 cm^{-1}) were reported in the following papers

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