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Electronic Supplementary Information

A One-Pot Solid-State Synthesis of MgO Nanoparticles and their Catalytic, Biological, and Electrochemical Sensor Activities: Hexaaquamagnesium(II) Bis(6-oxo-1,6-dihydropyridine-3-carboxylate) as a Tool

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Experimental

Materials and methods

Commercially available reagents and metal salts of analytical grade were purchased and used without further purification. The 6-hpca was procured from Sigma-Aldrich. Double-distilled water and distilled alcohol were used as solvents for the experimental and analytical processes. The reported metal complex was prepared by the neutralization of

magnesium nitrate in an aqueous medium with 6-hpca at a 1:1 mole ratio. The elemental analysis of H, C, and N was performed using a PerkinElmer Vario-ELIII elemental analyzer. The Mg content of the complex was determined by complexometric ethylenediaminetetraacetic acid (EDTA) titration, upon the destruction of the Mg complex with HNO_3 acid. The melting point of the complex was determined using a Relitech melting-point apparatus. Ultraviolet–visible (UV–VIS) absorbance studies were performed using a Shimadzu UV-2100 spectrophotometer. Transmission electron microscopy (TEM; JEOL 2100F) was employed to determine the surface morphology of the MgO NPs. FT-IR spectra were recorded within the range of 4000–400 cm^{-1} at room temperature using an FT-IR-8300 Shimadzu spectrometer. The ^{1}H NMR spectrum (400 MHz) of the Mg complex was recorded using a multiprobe NMR spectrometer with dimethyl sulfoxide (DMSO) as the solvent and tetramethylsilane (TMS) as the internal reference. The SC-XRD studies for determining the structure of the complex were conducted using a Bruker Venture Duo Photon-II single-crystal X-ray diffractometer. The refinement and structure solution were analyzed using the Shelxtl-Plus software package. We employed the detailed procedure and conditions of our previous method; this is provided in the supplementary information (SI). Thermogravimetry-differential thermal analysis (TG-DTA) measurements were performed using an SDTQ 600 V8.3 and Stanton 781 thermal analyzer at a heating rate of 20°C min^{-1} in air within a temperature range of 27°C–800°C. The antibacterial culture samples were collected from the Department of Biological Science (Microbiology Laboratory), RS Puram, Coimbatore, India.

Single-crystal X-ray diffraction studies

A crystal (dimensions of 0.19 × 0.14 × 0.10 mm^3) was mounted on a MiTeGen cryoloop in random orientation. The opening examination and collection of data were performed using a Bruker Venture Duo Photon-II single-crystal X-ray diffractometer. The data were recorded using the Incoatec IμS microfocus source (Cu or Mo) along with multilayer mirror optics. Preface unit-cell constants were resolved from a set of 180° fast phi scans (typically, 1-s exposure, 1° scan). The intensity data collections comprised combinations of ϖ and ϕ scan frames with a typical scan width of 0.5° and a counting time of 1–10 s/frame at a crystal-to-detector distance of 3.7 cm. The collected data were integrated using an orientation matrix determined from the narrow frame scans. The Apex II and SAINT softwares were employed for the collection and integration of data. The analysis of the integrated data did not show any decay. The cell constants were determined by the global refinement of reflections harvested from the complete data set. The data collected were corrected for systematic errors using SADABS [1] based on the Laue symmetry using equivalent reflections.

The data of the crystal and its intensity collection parameters are listed in Table 1. The structure and its refinements were conducted using the Shelxtl-Plus software [2]. The structure was solved using direct methods and successfully refined in the triclinic space group, P-1. Full-matrix least-squares refinements were conducted by minimizing $\Sigma w(F_o^2 - F_c^2)^2$. The nonhydrogen atoms were anisotropically refined to convergence. All the hydrogen atoms located were refined with geometrical restraints (DFIX and SADI).

Antibacterial studies

The antibacterial activity of the Mg(II) complex and MgO NPs was studied *in vitro* using the disk diffusion method with Mueller Hinton plates against *Staphylococcus aureus* (Gram-positive) and *Escherichia coli* (Gram-negative) bacteria. The nutrient agar and broth cultures were prepared and incubated at 37°C. The detailed experimental procedure and conditions were provided in the SI. After incubation, at a suitable time, the plates containing nutrient agar were seeded with a suspension of 30 μL of both bacterial pathogens separately. Thereafter, 30 μL of the testing compounds at a concentration of 1000 μg/mL in DMSO were introduced into the entire well, and the plates were incubated at 37°C for 24 h. The clear zones observed around the wells indicated positive results. The diameter of the inhibition zones was determined using the standard scale and values, which are expressed in millimeters (mm). The DMSO concentration in the process did not affect the growth of the pathogens. The experiments were performed in triplicate to evaluate their reproducibility. Hence, the obtained outcomes were reported as MICs. *Kanamycin* was used as a standard positive control for all the pathogenic studies.

References

1. Bruker Analytical X-Ray, Madison, WI, 2010.

2. G.M. Sheldrick,A short history of SHELX*, ActaCryst.* A64(2008)112-122.

Table S1. Bond lengths [Å] and angles [°] for complex **1**.

Bond lengths [Å]		Bond angles [°]			
Mg(1)-O(5)	2.0312(13)	O(5)-Mg(1)-O(5)#1	180	C(3)-C(4)-C(6)	123.4(3)
Mg(1)-O(5)#1	2.0312(13)	O(5)-Mg(1)-O(4)#1	87.90(6)	N(1)-C(5)-C(4)	121.4(3)
Mg(1)-O(4)#1	2.0343(13)	O(5)#1-Mg(1)-O(4)#1	92.10(6)	N(1)-C(5)-H(5)	119.3
Mg(1)-O(4)	2.0343(13)	O(5)-Mg(1)-O(4)	92.10(6)	C(4)-C(5)-H(5)	119.3
Mg(1)-O(6)	2.0869(15)	O(5)#1-Mg(1)-O(4)	87.90(6)	C(5')-N(1')-C(1')	118.7(10)
Mg(1)-O(6)#1	2.0869(15)	O(4)#1-Mg(1)-O(4)	180	C(5')-N(1')-H(1')	120.6
O(4)-H(4A)	0.8506	O(5)-Mg(1)-O(6)	90.82(7)	C(1')-N(1')-H(1')	120.6
O(4)-H(4B)	0.8506	O(5)#1-Mg(1)-O(6)	89.18(7)	O(1')-C(1')-C(2')	118.9(11)
O(5)-H(5A)	0.8507	O(4)#1-Mg(1)-O(6)	91.43(7)	O(1')-C(1')-N(1')	122.8(12)
O(5)-H(5B)	0.8504	O(4)-Mg(1)-O(6)	88.57(7)	C(2')-C(1')-N(1')	118.1(9)
O(6)-H(6A)	0.8508	O(5)-Mg(1)-O(6)#1	89.18(7)	C(3')-C(2')-C(1')	122.4(12)
O(6)-H(6B)	0.8506	O(5)#1-Mg(1)-O(6)#1	90.82(7)	C(3')-C(2')-H(2')	118.8
O(2)-C(6)	1.242(2)	O(4)#1-Mg(1)-O(6)#1	88.57(7)	C(1')-C(2')-H(2')	118.8
O(3)-C(6)	1.257(2)	O(4)-Mg(1)-O(6)#1	91.43(7)	C(2')-C(3')-C(4')	119.3(15)
O(1)-C(1)	1.267(3)	O(6)-Mg(1)-O(6)#1	180	C(2')-C(3')-H(3')	120.4
N(1)-C(5)	1.350(3)	Mg(1)-O(4)-H(4A)	124.7	C(4')-C(3')-H(3')	120.4
N(1)-C(1)	1.381(3)	Mg(1)-O(4)-H(4B)	130.7	C(5')-C(4')-C(3')	120.1(17)
N(1)-H(1)	0.86	H(4A)-O(4)-H(4B)	104.5	C(5')-C(4')-C(6)	123.7(14)
C(1)-C(2)	1.420(4)	Mg(1)-O(5)-H(5A)	126	C(3')-C(4')-C(6)	116.0(14)
C(2)-C(3)	1.358(4)	Mg(1)-O(5)-H(5B)	127.1	C(4')-C(5')-N(1')	121.2(14)

C(2)-H(2)	0.93	H(5A)-O(5)-H(5B)	104.5	C(4')-C(5')-H(5')	119.4
C(3)-C(4)	1.414(4)	Mg(1)-O(6)-H(6A)	126.1	N(1')-C(5')-H(5')	119.4
C(3)-H(3)	0.93	Mg(1)-O(6)-H(6B)	128.4	O(2)-C(6)-O(3)	124.42(17)
C(4)-C(5)	1.365(4)	H(6A)-O(6)-H(6B)	104.5	O(2)-C(6)-C(4)	120.23(19)
C(4)-C(6)	1.490(4)	C(5)-N(1)-C(1)	124.0(2)	O(3)-C(6)-C(4)	115.33(19)
C(5)-H(5)	0.93	C(5)-N(1)-H(1)	118	O(2)-C(6)-C(4')	111.0(7)
O(1')-C(1')	1.281(8)	C(1)-N(1)-H(1)	118	O(3)-C(6)-C(4')	124.6(8)
N(1')-C(5')	1.399(14)	O(1)-C(1)-N(1)	117.8(2)		
N(1')-C(1')	1.402(13)	O(1)-C(1)-C(2)	127.0(3)		
N(1')-H(1')	0.86	N(1)-C(1)-C(2)	115.2(2)		
C(1')-C(2')	1.394(13)	C(3)-C(2)-C(1)	120.8(3)		
C(2')-C(3')	1.336(13)	C(3)-C(2)-H(2)	119.6		
C(2')-H(2')	0.93	C(1)-C(2)-H(2)	119.6		
C(3')-C(4')	1.404(16)	C(2)-C(3)-C(4)	121.9(3)		
C(3')-H(3')	0.93	C(2)-C(3)-H(3)	119.1		
C(4')-C(5')	1.346(17)	C(4)-C(3)-H(3)	119.1		
C(4')-C(6)	1.55(2)	C(5)-C(4)-C(3)	116.7(3)		
C(5')-H(5')	0.93	C(5)-C(4)-C(6)	119.9(3)		

Table S2. Simultaneous TG-DTA data of **1**.

Compound	DTA peak temp (°C)	Thermogravimetry			Nature of decomposition/ Product formation
		Temp range (°C)	Mass loss (%)		
			Found	Calc.	
$[Mg(H_2O)_6](C_6H_4NO_3)_2$	167(+)	20-205	33.00	33.80	Formation of $Mg(H_2O)_6(C_6H_3NO_3)$
	318(+) 440(+) 511(+) 715(+)	205-800	89.86	90.14	Formation of MgO

Table S3. EDX data of the MgO NPs.

Element	**Weight %**	**Atomic %**
O K	37.16	34.02
Mg K	62.84	65.98
Total	100	