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Synthesis, characterisation and antimicrobial studies of nickel (II) mixed ligand complexes of β-diketones with nitrogen-containing ligands

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Abstract

A series of mixed-ligand Nickel (II) complexes of β-diketones (dibenzoylmethane (dbmH), benzoylacetone (bzacH) and acetylacetone (acacH) with 1,10-phenanthroline (phen), 2,2′-bipyridine (bipy) and Tetramethylethylenediamine (Tmen) associated with NO_3^- counter ion has been synthesised and characterised by conductance measurement, magnetic susceptibility measurement, infrared spectroscopy, electronic spectroscopy and antimicrobial studies. The electronic spectra studies are consistent with the adoption of probable octahedral geometry while the infrared spectra showed characteristic NO_3^- bands in all the complexes. Conductance measurement of the mixed ligand complexes indicated that they were all non-electrolytes, with the ligands and anions within the coordination sphere. The complexes showed good activity in most of the organisms but few were resistant in *P. notatum, R Stolonifer and K. pneumonae.*

Keywords: Conductance measurement; spectral measurements; antimicrobial studies.

Introduction

Metal β-diketonates are amongst the most widely studied coordination compounds and their chemistry has been investigated for most of the metals in the periodic table [1]. The coordination tendencies of the ligand are well established and a large number of articles have been published on these [2-11]. Their very high chelating ability is evident from the fact that their complexes of most metals in the periodic table are known [12].

β-diketonates complexes are used as catalysts in polymerization of styrene [13], hydrogenation of benzene [14], epoxidation of allylic alcohols, Michael additions [15] and coupling of phenols [16]. Metal β-diketonates are widely used as catalyst precursors and reagents [17], as NMR “shift reagents”, as catalysts for organic synthesis and precursors of industrial hydroformylation catalysts [18].

Generally, studies on mixed-ligand systems have always attracted the attention of the scientific community due to the extreme stability afforded by the system and also their peculiar spectroscopic, photochemical, and electrochemical features [19, 20]. While some studies have been reported on mixed-ligand copper (II) complexes of symmetrical β-diketones, e.g. acetylacetone (acacH) containing 1,10-phenanthroline (phen), 2,9-dimethyl-phenanthroline (dmph) and 2,2’-bipyridine (bipy) such as $[Cu(acac)(phen)(NO_3)].H_2O$, $[Cu(acac)(dmph)(NO_3)]$[20], and $[Cu(acac)(bipy)(H_2O)]NO_3.H_2O$ [21] respectively, as well as unsymmetrical β-diketones, e.g. benzoylacetone [19], the nickel (II) mixed ligand complexes of similar composition have not attracted such attention. We hereby report the synthesis of nickel (II) mixed ligand complexes of acetylacetone, benzoylacetone and dibenzoylmethane with nitrogen-containing ligands with the aim of characterising the complexes by metal analysis, conductance measurement, electronic spectra, infrared spectra and antimicrobial studies.

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Materials and methods

Reagents and solvents

Reagents used include nickel (II) nitrate hexahydrate, 1,10-phenanthroline, 2,2′-bipyridine, dibenzoylmethane, benzoylacetone, acetylacetone, tetramethylethylenediamine, methanol, chloroform and ethanol. They are of analytical grade obtained from Aldrich Chemicals and British Drug House (BHD) Chemicals Limited and were utilised without further purification.

Synthesis of $[Ni(acac)(bipy)(NO_3)(H_2O)]$

Nickel nitrate hexahydrate (0.8713 g; 2.99 mmol) dissolved in 2 mL water was added directly to a mixture of 2,2′-bipyridine (0.4680 g; 2.99 mmol) and acetylacetone (0.32 mL; 2.99 mmol) in 3 mL ethanol. Sodium carbonate (0.3175 g, 2.99 mmol) dissolved in 1 mL water was added to the mixture and stirred for one hour and the precipitates formed were filtered and washed with water and ethanol and dried in a vacuum desiccator over silica [22]. A similar method was adopted for the preparation of other nickel (II) mixed ligand complexes containing dibenzoylmethane, benzoylacetone and acetylacetone with 1,10-phenanthroline, 2,2′-bipyridine and Tetramethylethylenediamine [22].

Biological studies

The antimicrobial screening of the synthesised complexes were investigated using Gram-positive bacteria, *Staphylococcus aureus* and *Bacillus subtilis,* Gram-negative bacteria, *Pseudomonas aeruginosa, Salmonella enterica, Escherichia coli* and *Klebsiella pneumoniae* and four fungi (*Candida albicans*, *Aspergillus niger, Penicillium notatum, Rhizopus stolonifer*). Each of the six compounds was used at a concentration of 100mg/ml, prepared by dissolving 0.5 g in 5 ml of either sterile distilled water or methanol. Plate cultures were prepared either by seeding (bacteria and yeasts) or spread-plate (mould) using 0.1 ml of 10^{-2} dilution from 18 to 24 hour-old broth culture of each bacterium or 24 to 72 hour-old broth culture of each fungus, in Nutrient agar (bacteria) or Sabouraud dextrose agar (fungi) [11].

Physical measurements

The percentage of metals in the mixed-ligand complexes were determined titrimetrically with EDTA. The molar conductivities of the soluble compounds in nitromethane at room temperature were determined using digital conductivity meter (Labtech). The infrared spectra of the complexes, as pressed KBr disc, were recorded on the Buck 500 Scientific model infrared spectrophotometer in the region 4000-400 cm^{-1}. The electronic reflectance spectra of the complexes in Nujol mulls were determined by a Genesys 10S v1.200 2L7H311008 UV-Spectrophotometer, Figure 1.

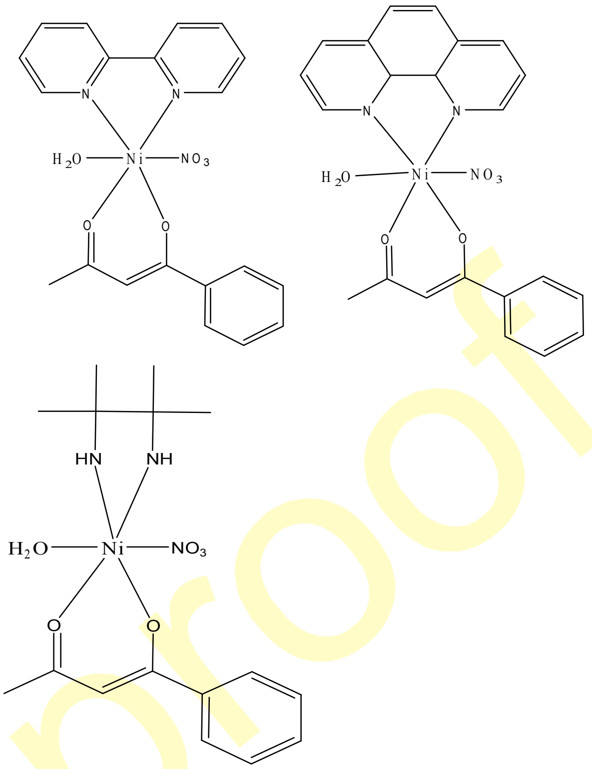

Figure 1. Electronic and infrared-spectral data and magnetic measurement.

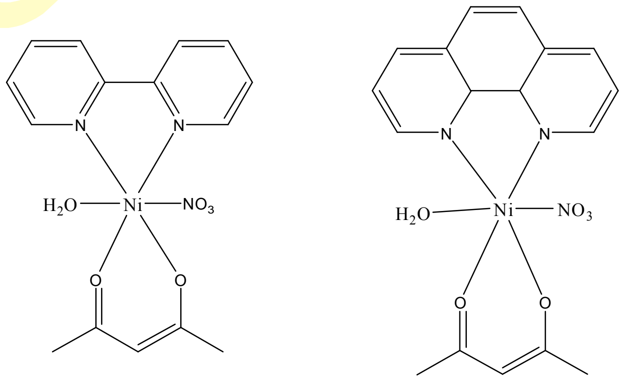

Figure 2. Proposed structure of complexes.

Results and discussion

The analytical data, colours, percentage yields and molar conductance measurements $(\wedge_m)$ of the complexes are given in Table 1. The complexes display various shades of green; they displayed a wide variation in their solubilities in methanol, chloroform, dimethylformamide, dimethylsulphoxide, acetone, petroleum ether, nitromethane and n-hexane, ranging from readily soluble to insoluble.

The mixed ligand complexes were obtained by reaction of appropriate amounts of nickel (II) nitrate with a mixture of β-diketone and the respective diimine ligand; 1,10-phenanthroline, 2,2′-bipyridine and tetramethylethylenediamine in low yields (10.79-49.78%) except [Ni(bzac)(tmen)(NO_3)(H_2O)] and [Ni(dbm)(phen)(NO_3)(H_2O)] with 56.56% and 70% respectively. The formation of the complexes is given by the following equations:

$$Ni(NO_3)_2.6H_2O + \beta\text{-diketone} + N\text{-}N \rightarrow [Ni(\beta\text{-diketone})(N\text{-}N)(NO_3)(H_2O)]$$

where β-diketone = (dbmH, bzacH and acacH) and N-N = 1,10-phenanthroline, 2,2′-bipyridine and tetramethylethylenediamine.

All complexes melted at temperature range of 160-267°C except [Ni(acac)(bipy)(NO_3)(H_2O)] and [Ni(acac)(phen)(NO_3)(H_2O)] which decomposed at 263°C and 264°C respectively. Results are shown in Table 1. The molar conductivities of the complexes were in the range 25-40 ohm^{-1} cm^2 mol^{-1}, showing that they are non-electrolytes in nitromethane [23].

Infrared spectra

Relevant infrared-spectral data of the complexes are presented in Table 2. Assignments of the infrared bands were made by comparing the spectra of the complexes with those of similar compounds in the literature [10, 11, 24]. The broad band observed around 3.500-3.400 cm^{-1} has been assigned to O-H band of water in the complexes. ν(C=O)+ν(C=C) bathochromic shifts of varying magnitudes were observed in the spectra of the complexes compared to the ligands except [Ni(dbm)(phen)(NO_3)(H_2O)] which had hypsochromic shift. The ν(C=O)+ν(C=C) absorption was observed in the range 1.692-1.516 cm^{-1}in [Ni(acac)(phen)(NO_3)(H_2O] while [Ni(acac)(bipy)(NO_3)(H_2O)] had very sharp ν(C=O)+ν(C=C) absorption bands in the 1599-1516 cm^{-1}. The ν(C=O)+ν(C=C) absorptions were observed in the range 1.594-1.516 cm^{-1} for [Ni(bzac)(phen)(NO_3)(H_2O)], [Ni(bzac)(phen)(NO_3)(H_2O)] and [Ni(bzac)(tmen)(NO_3) (H_2O)]. [Ni(dbm)(bipy)(NO_3)(H_2O)] had three bands in the region 1.692-1.595 cm^{-1} which have been assigned to ν(C=O)+ν(C=C) while [Ni(dbm)(phen)(NO_3)(H_2O)] had two very strong bands in the region 1.552-1.519 cm^{-1} which have been assigned to ν(C=O)+ν(C=C) vibration. Metal (II) complexes with phenanthroline were reported to have two strong bands at approximately 725 and 825 cm^{-1}, which were assigned to out of plane motion of the hydrogen atoms on the heterocyclic rings and the middle ring respectively. These two strong bands occurred in the range 711-728 cm^{-1} and 846-852 cm^{-1} in the prepared phenanthroline complexes. In the prepared bipyridine complexes, strong bands in the range 754-766 cm^{-1} have been assigned to out of plane bending of ring hydrogen. The bands in the range 1.384-1.385 cm^{-1} confirmed the presence of NO_3^- in all the complexes. This has been assigned as symmetric stretching of the NO_2 group [10, 25]. Stretching of the π bonds of the N-O linkage produced absorption at 802-867 cm^{-1} while NO_2 bend vibration appeared at 641-758 cm^{-1} [10, 25]. The absorptions below 700 cm^{-1} have been assigned as ν(Ni-O+Ni-N)[26].

Electronic spectra

The solid-state electronic reflectance spectra of the nickel (II) complexes were carried out in nujol and their tentative assignments are presented in Tables 3. π_3-π^*_4 of the acacH, bzacH and dbmH occurred at 30.00, 31.00 and 33,210 cm^{-1} respectively.Upon chelation, π_3-π^*_4 hypsochromic shifts were observed in [Ni(acac)(phen)(NO_3)(H_2O] (31.250 cm^{-1} to 32.150 cm^{-1}), [Ni(acac)(bipy)(NO_3)(H_2O)](31.250 cm^{-1} to 31.330 cm^{-1}), [Ni(bzac)(phen)(NO_3)(H_2O)] (31.060 cm^{-1} to 31,250 cm^{-1}), [Ni(bzac)(bipy)(NO_3)(H_2O)] (31.060 cm^{-1} to 34.990 cm^{-1}) and [Ni(bzac)(tmen)(NO_3)(H_2O)] (31.060 cm^{-1} to 31.250 cm^{-1}). Bathochromic shifts of the π_3-π^*_4 transition were observed in [Ni(dbm)(phen)(NO_3)(H_2O)](35.210 cm^{-1} to 32.880 cm^{-1} and Ni(dbm)(bipy)(NO_3)(H_2O)] (35.210 cm^{-1} to 32.180 cm^{-1}).

Nickel (II) species have a large number of stereochemical forms in which the ion occurs, hence equilibrium between these forms are usually set up which are generally temperature, solvent and sometimes concentration dependent [27]. In solution, Ni (II) β-diketonates sometimes exhibit a monomer ↔ trimer, square planar ↔ octahedral equilibrium [9, 28]. Coordination number five is relatively rare for nickel (II), which more often forms either octahedral high-spin or square-planar low-spin complexes. Three transitions are expected for an octahedral nickel (II) ion in the region 7,000-13,000 cm^{-1}, 11,000-20,000 cm^{-1}, 19,000-27,000 cm^{-1} which are assigned to the $^3A_{2g}(F) \rightarrow {}^3T_{2g}(F)$, $^3A_{2g}(F) \rightarrow {}^3T_{1g}(F)$, $^3A_{2g}(F) \rightarrow {}^3T_{1g}(P)$ respectively [29]. In the reflectance spectrum of the synthesized nickel (II) mixed ligand complexes, bands in the 25,560 cm^{-1}-37,970 cm^{-1} have been assigned to charge transfer transitions and intraligand π-π* transitions. The d-d transitions observed in the range 11,760 cm^{-1}-13,160 cm^{-1} and 16,100 cm^{-1}-18,260 cm^{-1} are assigned to $^3A_{2g} \rightarrow {}^3T_{2g}(F)$ and $^3A_{2g} \rightarrow {}^3T_{1g}(F)$ respectively [9].

Magnetic moment

The spin only ($\mu_{s.o}$) value of 2.83 B.M is expected for nickel (II) complexes with 2 unpaired electrons. Experimental moments of 2.9-3.3 B.M. are normally observed for octahedral nickel (II) complexes due to spin-orbit coupling of the $^3A_{2g}$ and $^3T_{2g}(F)$ terms while moments of 3.2-4.1 B.M. are observed for tetrahedral nickel due to orbital contributions [9], the larger the distortion from a regular tetrahedron, the lower the magnetic moment. A room temperature magnetic moments in the range 2.80-3.51 B.M was observed for thecomplexes, which are for high spin octahedral complexes [30-32].

Antimicrobial activities

All the nickel complexes showed moderate activity in tested fungi except Ni(acac)(phen)(NO_3)(H_2O)] and [Ni(dbm)(phen)(NO_3)(H_2O)] in P. *notatum* and *R.stolonifer* [Ni(acac)(phen)(NO_3)(H_2O)] in *P. notatum* and [Ni(bzac)(bipy)(NO_3)(H_2O)] in *R.stolonifer* that lacked antifungi activiity. All the complexes were active in *Staphylococcus aureus* except [Ni(bzac)(phen)(NO_3)(H_2O)] and [Ni(bzac)(bipy)(NO_3)(H_2O)] which showed moderate activity. [Ni(bzac)(phen)(NO_3)(H_2O)] and [Ni(dbm)(bipy)(NO_3)(H_2O)] were active in *Escherichia coli* while

Table 1. Analytical data of synthesized complexes.

Complexes	Formula weight	Colour	% Ni Observed	% Ni Calculated	% Yield	Melting point (^{0}C)	Conductance Ω^{-1} cm^2mol^{-1}	μ_{eff} (B.M)
[Ni(acac)(phen)(NO_3)(H_2O)]	437.06	Light green	13.31	13.42	49.28	263	38	3.24
[Ni(acac)(bipy)(NO_3)(H_2O)]	431.05	Light green	13.48	13.64	28.65	264	40	3.20
[Ni(bzac)(phen)(NO_3)(H_2O)]	498.13	Green	12.66	11.76	10.79	239-241	37	3.28
[Ni(bzac)(bipy)(NO_3)(H_2O)]	457.09	Green	12.05	12.35	33.49	174-176	25	3.29
[Ni(bzac)(tmen)(NO_3)(H_2O)]	399.01	Green	14.91	14.71	56.56	160-162	17.30	3.11
[Ni(dbm)(phen)(NO_3)(H_2O)]	523.00	Green	11.13	11.22	70.00	240-242	7.37	2.78
[Ni(dbm)(bipy)(NO_3)(H_2O)]	500.01	Light green	11.79	11.73	31.32	265-267	29.10	1.24

Table 2. Relevant infrared spectra bands (cm^{-1}) of the compounds.

Complexes	ν(OH)	ν_{as}(C=O+C=C)	$\nu_{sym}NO_2$	γ(C-H) Phen/Bipy/Tmen	ν(Ni-N/Ni-O/Ni-N+Ni-O)
acacH	3413b	1713s, 1614s			
bzacH	3422b	1599m, 1540b			
dbmH	3447b	1598m, 1540b			
[Ni(acac)(phen)(NO_3)(H_2O]	3428 b	1592s, 1516s,	1384vs	852vs, 728vs	508w, 483w, 416, 357
[Ni(acac)(bipy)(NO_3)(H_2O)]	3436 b	1599vs, 1516vs	1384	766vs	632, 565, 419, 374
[Ni(bzac)(phen)(NO_3)(H_2O)]	3429 b	1594vs, 1562vs, 1516s	1384vs	851vs, 711vs	448, 427, 372
[Ni(bzac)(bipy)(NO_3) H_2O)]	3435 b	1594vs, 1561vs, 1516vs	1384vs	764m	527, 451, 427, 374
[Ni(bzac)(tmen)(NO_3) (H_2O)]	3401 b	1597vs, 1512vs, 1571vs	1385vs	717vs	448, 435, 469, 366
[Ni(dbm)(bipy)(NO_3)(H_2O)]	3437 b	1692vs, 1664vs, 1595m	1384vs	755s	507, 487, 427, 357
[Ni(dbm)(phen)(NO_3) (H_2O)]	3420 b	1552vs, 1519vs	1384vs	847vs, 725vs	522, 497, 452. 380

Table 3. Solid reflectance spectra of the complexes in kilokaser; 1kk = 1000cm^{-1}.

Complexes	C.T/π- π* transitions (kk)	d-d transitions/assignment (kk)
acacH	31.25, 45.46	–
bzacH	31.06, 37.45, 44.64	–
dbmH	35.21,42.92	–
[Ni(acac)(phen)$(NO_3)(H_2O)$]	25.82, 29.15, 32.13, 37.97	12.86; $3A_{2g} \rightarrow 3T_{2g}$ (F) ν_1 17.39; $3A_{2g} \rightarrow 3T_{1g}$ (F) ν_2
[Ni(acac)(bipy)$(NO_3)(H_2O)$]	26.16, 29.79, 31.33, 36.90	12.86; $3A_{2g} \rightarrow 3T_{2g}$ (F) ν_1 17.36; $3A_{2g} \rightarrow 3T_{1g}$ (F) ν_2
[Ni(bzac)(phen)$(NO_3)(H_2O)$]	26.15, 30.31, 31.25, 37.83	12.88; $3A_{2g} \rightarrow 3T_{2g}$ (F) ν_1 16.67; $3A_{2g} \rightarrow 3T_{1g}$ (F) ν_2
[Ni(bzac)(bipy)$(NO_3)(H_2O)$]	26.35, 30.35, 34.99, 37.83	11.76; $3A_{2g} \rightarrow 3T_{2g}$ (F) ν_1 16.00; $3A_{2g} \rightarrow 3T_{1g}$ (F) ν_2
[Ni(bzac)(tmen)$(NO_3)(H_2O)$]	26.18, 30.35, 31.25, 36.50	11.76; $3A_{2g} \rightarrow 3T_{2g}$ (F) ν_1 16.43; $3A_{2g} \rightarrow 3T_{1g}$ (F) ν_2
[Ni(dbm)(phen)$(NO_3)(H_2O)$]	25.56, 30.39, 32.88, 37.82	12.99; $3A_{2g} \rightarrow 3T_{2g}$ (F) ν_1 16.10; $3A_{2g} \rightarrow 3T_{1g}$ (F) ν_2
[Ni(dbm)(bipy)$(NO_3)(H_2O)$]	26.17, 30.40, 32.18, 36.78	12.99; $3A_{2g} \rightarrow 3T_{2g}$ (F) ν_1 17.85; $3A_{2g} \rightarrow 3T_{1g}$ (F) ν_2

Table 4. Antimicrobial activity of the synthesized complexes.

Complexes	*S. aureus*	*E. coli*	*B. subtilis*	*P. aeruginosa*	*S. typhi*	*K. pneumonae*	*C. albican*	*A. niger*	*P. notatum*	*R. stolonifer*
[Ni(acac)(phen) $(NO_3)(H_2O]$	24	18	20	18	16	20	16	16	R	R
[Ni(acac)(bipy) $(NO_3)(H_2O)$]	20	18	22	16	14	14	14	14	R	14
[Ni(bzac)(phen) $(NO_3)(H_2O)$]	14	24	14	20	20	18	16	16	18	16
[Ni(bzac)(bipy) (NO_3) $H_2O)$]	18	16	18	16	R	R	14	16	14	-
[Ni(bzac)(tmen) (NO_3) (H_2O)]	24	22	18	20	20	18	18	16	18	18
[Ni(dbm)(bipy) $(NO_3)(H_2O)$]	20	18	20	20	22	18	18	16	R	R
Gentamicin	16	18	20	16	18	20	ND	ND	ND	ND

Notes:

Growth inhibition zone in millimeters (mm)

10-19-moderate, 20-29-active, 20-29-active, 30 and above-v.active.

R = organism resistant to the extract.

ND = not determined.

S. aur = Staphylococcus aureus.

B. sub = Bacillius subtilis.

K. pne = Klebsiella pneumonia.

E. coli = Escherichia coli.

S. ent = Salmonella enterica.

P. aer = Pseudomonas aeruginosa.

Ca = Candida albicans.

An = Aspergillus niger.

Pen = Penicillium notatum.

Rhiz =Rhizopus. Stolonifer.

others were moderately active. Ni(acac)(phen)$(NO_3)(H_2O)$], [Ni(acac)(bipy)$(NO_3)(H_2O)$] and [Ni(dbm)(phen)$(NO_3)(H_2O)$] were also active in *Bacillus subtilis* while [Ni(bzac)(phen)$(NO_3)(H_2O)$], Ni(bzac)(bipy)$(NO_3)(H_2O)$] and [Ni(dbm)(bipy)$(NO_3)(H_2O)$] showed moderate activity. Ni(bzac)(bipy)$(NO_3)(H_2O)$] was resistant in *S. typhi* and *K. pneumonae* while [Ni(bzac)(phen)$(NO_3)(H_2O)$], [Ni(dbm)((phen)$NO_3)(H_2O)$] and [Ni(dbm)(bipy)$(NO_3)(H_2O)$] were active in *S. typhi* but moderately active in K. *pneumonae*. The complexes were all active *in P. aeruginosa* except Ni(acac)(phen)$(NO_3)(H_2O)$] [Ni(acac)(bipy)$(NO_3)(H_2O)$] and [Ni(bzac)(bipy)$(NO_3)(H_2O)$] which showed moderate activity. The activities of the complexes were comparable to the standard drug, Gentamicin used at 8 and 4 mg/mL but at 2 mg/mL, gentamicin had very weak activity.

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