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ORIENTAL JOURNAL OF CHEMISTRY
An International Open Access, Peer Reviewed Research Journal

www.orientjchem.org

ISSN: 0970-020 X
CODEN: OJCHEG
2021, Vol. 37, No.(2):
Pg. 413-418

Synthesis, Characterization of Novel β-diketone Ni, Fe, Cr, Complexes and Study of Its Antifungal, Antibacterial, Antioxidant and Anti-inflammatory Activities

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http://dx.doi.org/10.13005/ojc/370221

(Received: March 09, 2021; Accepted: April 10, 2021)

ABSTRACT

The β-diketone is synthesized via the named Baker-Venkataraman. In current task, β-diketone ligand mixed with different metal nitrates and form different metal complexes such as Ni(II), Fe(III), Cr(III). Ligand gives tautomerism, this tautomerism process was examined with the help of FTIR. Because of enol assist as ligand in the metal complexes preparation. The prepared metal complexes are described by various techniques like elemental analysis, FTIR, DTA, TGA, magnetic properties and molar conductivities. Different properties of complexes are studied such as antioxidant, antibacterial, anti-inflammatory and antifungal properties.

Keywords: 1,3-β-diketone ligand, β-diketone ligand, Baker-Venkataraman transformation reaction, 1,3-β-diketone metal complex, Antibacterial activity, Antioxidant activity, Antifungal agent, Anti-inflammatory activities.

INTRODUCTION

d-block elements performs crucial part in the development of metal complexes in co-ordination chemistry. Enormous works are exist on the various applications of metal complexes. β-diketone and their metal complexes perform crucial part in the aspect of co-ordination chemistry[1]. Important material in the thin film development which is used in optical device[2], radiopharmaceuticals[3]. β-diketone perform as versatile reactant for different reactions of number of heterocycles[4] pharmacological importance of flavones[5], luminescence assets of pyrazolic[6], isoxazolyl[7], triazolic[8], pyrimidines[9]. β-diketone is utilized as chelating agent[10], toxic metal ions adsorbed on different resin[11], protection from harmful radiation, toxins by cosmetics sunscreen[12], anti-HIV-1 drug design[13]. photoluminescence and photoisomerization property[14]. β-diketone complexes shows Lewis acidity, volatility, idyllic molar enthalpy of formation, idyllic molar heat of sublimation, catalytic property and vapor pressures[15]. β-diketone express medical property like anticancer and antibacterial[16] antimicrobial and antioxidant activity[17], anti-inflammatory activity[18].

β-diketone is useful for the treatment of anti-influenza[19]. β-diketone utilized in organic light emitting diodes (OLED)[20]. β-diketone express enol and keto tautomerization[21] β-diketone shows anti-spreading action such as lubricant oil on the surface of steel[22] β-diketone shows anti-tumor property[23] Since entirely mentioned assets, in current paper, involve β-diketone ligand and their different metal complexes. The prepared metal complexes are tested by different methods such as antioxidant, antifungal and anti-inflammatory and antibacterial action.

EXPERIMENTAL

Merck AR grade chemicals are used in the current research task. Chemicals remain utilized without extra purification. The normal process utilized to purify solvents. For preparation and recrystallization of metal complexes super dry distilled pure ethanol utilized.

Synthesis of β-diketone Ligand[22]

Synthesis of metal complex metal complexes[22]

Characterization

Melting points were determinator by using capillary method. The elemental study performed via FLASH EA 1112 series Thermo Finnigan. FTIR scanned with the help of FTIR instrument made Bruker Optics ALPHA-T. The complex is sited on ATR crystal. The Conductance is confirmed with help of Equip-Tronic conductivity meter. Magnetic susceptibility is reported through Gouy balance. The physical possessions of the metal complexes are studied. In metal complexes presence of coordinated water molecules due to this the probable geometry is octahedral.

Table 1: Metal complexes pH range of precipitation, colour, decomposition temperature and yield

Sr. No.	Name of Complex	pH range of precipitation	Colour	Decomp. Temp./m.p. °C	Yeild (%)
1	Ni-complex	7.0-7.5	Light green	368°C	80
2	Fe-complex	7.0-7.5	Blackish brown	380°C	79
3	Cr-complex	7.0-7.5	Brown	376°C	85

Table 2: Elemental analysis of Metal complexes

Name of Complex	Molecular Formula	% Found (Calculated)			
		C	H	O	Metal
Ni-complex	$C_{38}H_{42}NiO_{10}$	63.91(63.62)	6.49(5.90)	22.87(22.30)	8.74(8.18)
Fe-complex	$C_{38}H_{42}FeO_{10}$	64.35(63.87)	6.24(5.92)	22.74(22.39)	8.13(7.81)
Cr-complex	$C_{38}H_{42}CrO_{10}$	64.58(64.22)	6.47(5.96)	23.11(22.51)	7.79(7.32)

Magnetic susceptibility and Molar conductance of metal complexes

Ni-complex

Ni-complex molar conduction is estimated with the help of solvent DMSO at 10^{-4} M concentration. Ni-complex molar conduction is 49.2 Ω^{-1} cm^2 mol^{-1} that shows Ni-complex nature is covalent and non-electrolyte.

Ni-complex magnetic moment is estimated at room temperature is 2.77 B.M. Ni-ion showing an octahedral geometry.

Table 3: Molar conductances and magnetic property of metal complexes

Sr. No.	Name of complex	Molar conductance Ω^{-1} cm^2 mol^{-1}	μ_{eff} (B.M.)
1	Ni-complex	49.2	2.77
2	Fe-complex	64.5	5.99
3	Cr-complex	26.7	3.78

Fe-complex

Fe-complex molar conduction is estimated with the help of solvent DMSO at 10^{-4} M concentration. Fe-complex molar conduction value is 64.5 Ω^{-1} cm^2 mol^{-1} shows that Fe-complex nature is covalent and non-electrolyte.

Fe-complex magnetic moment is estimated at room temperature is 5.99 B.M are near to the required values for an octahedral geometry.

Cr-complex

Cr-complex molar conduction 26.7 Ω^{-1} cm^2 mol^{-1} shows Cr-complex is covalent and non-electrolyte.

Cr-complex magnetic moment is estimated at room temperature is 3.78 B.M which is near to the required values for an octahedral geometry.

FT-IR of metal Complexes

Ni-complex

In the Ni-complex IR stretching vibration of carbonyl frequency 1606.1 cm^{-1} that is below IR stretching vibration of ligand 1624.3 cm^{-1} [23]. This decreasing stretching vibration of complex display that ligand link with Ni. New-fangled stretching frequency at 633.7cm^{-1} is because of metal to oxygen bond in metal complex. It make sure that metal link with ligand via oxygen. Appearance of peaks at 2966.3 cm^{-1} and 3391.2 cm^{-1} shows presence of coordinated water in the complex[24].

Fe-complex

In the Fe-complex IR stretching vibration of carbonyl frequency 1604.9 cm^{-1} that is below stretching vibration of ligand 1624.3 cm^{-1}. This decreasing stretching vibration of complex display that ligand linked with Fe. New-fangled stretching frequency at 633.2 cm^{-1} is because of metal to oxygen bond in metal complex. It make sure that metal link with ligand via oxygen. Appearance of peaks at 2966.9 cm^{-1} and 3436.4 cm^{-1} shows presence of coordinated water in the complex.

Cr-complex

In the Cr-complex IR stretching vibration of carbonyl frequency 1603.5 cm^{-1} that is below stretching vibration of ligand 1624.3 cm^{-1}. This decreasing stretching vibration of complex display that ligand linked with Cr. New-fangled stretching frequency at 562.6 cm^{-1} is because of metal to oxygen bond in metal complex. It make sure that metal linked with ligand via oxygen. Appearance of peaks at 2966.9 cm^{-1} and 3432.7 cm^{-1} shows presence of coordinated water in the complex.

The IR frequencies of the ligand and Cr-complex are equivalent with the exception of very slight minor changes in absoprtion frequency band created via metal ion.

Thermogravimetric examination of nickel complexes

Ni-Complex

Thermally decomposed of designated metal complex was performed at a hotness quantity of 10°C min^{-1} with the help of N_2 environment between the thermal area 21.85°C to 1000°C. The Ni complex thermogravimetric spectra expressions mass decrease between the temperature area 21.85°C to 200°C that express the appearance of coordinationnated and lattice water molecules in Ni complex. The following mass decrease of obs. 5.02%, cal. 5.96% correspond to the breakdown of the connect (H_4O_2) part of the synthesized ligand.[25] The nickel complex offers breakdown from thermal area 200°C to 425°C, by obs. 77.94%, cal. 77.44% mass decrease because of removal of the ($C_{38}H_{38}O_4$) a portion of synthesized ligand, the exothermal method (T_{DTA}) peak at area for it. Again on continueous heat to 968.97°C, the residual weight correlate to the nickel oxide (NiO). As mentioned in Figure 1.

Fe-Complex

Thermally decomposed of designated metal complex was performed at a hotness quantity of 10°C min^{-1} with the help of N_2 environment between the thermal area 29.16°C to 1000°C. The Fe complex thermogravimetric spectra expressions mass decrease between the temperature area 29.16°C to 200°C that express the appearance of coordinationnated and lattice water molecules in Fe complex. The following mass decrease of obs. 5.04%, cal. 5.25% correspond to the breakdown of the connect (H_4O_2) part of the synthesized ligand. The iron complex offers breakdown from thermal area 200°C to 510°C, by obs. 82.41%, cal. 82.07% mass decrease because of removal of the ($C_{38}H_{38}O_6$) a portion of synthesized ligand. Again on continueous heat to 974.50°C, the residual weight correlate to iron oxide (FeO).[35] As mentioned in Figure 2.

Cr-complex

Thermally decomposed of designated metal complex was performed at a hotness quantity of 10°C min^{-1} with the help of N_2 environment between the thermal area 28°C to 1000°C. The Cr complex thermogravimetric spectra expressions mass decrease between the temperature area 28°C to 200°C that express the appearance of coordinated and lattice water molecules in Cr complex. The following mass decrease of obs. 5.07%, cal. 4.49% correspond to the breakdown of the connect (H_4O_2) portion of the synthesized ligand. The chromium complex offers disintegration from thermal area 200°C to 518°C, by obs. 78.61%, cal. 78.73% mass decrease because of removal of the ($C_{38}H_{38}O_4$) a portion of synthesized ligand. Again on continueous heat to 974.84°C, the residual weight correlate to iron oxide. As mentioned in Figure 3.

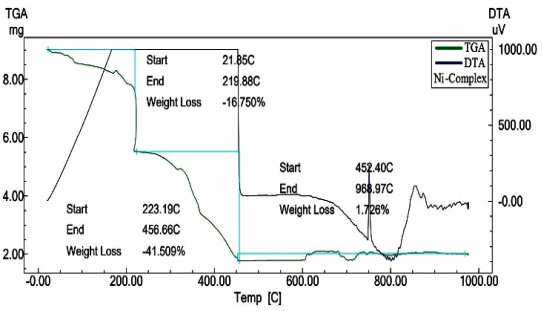

Fig. 1. DTA/TGA Arc of Ni-complex

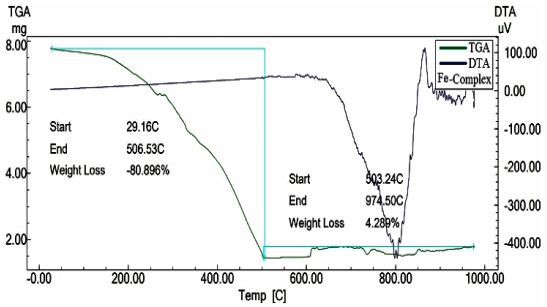

Fig. 2. DTA/TGA Arc of Fe-complex

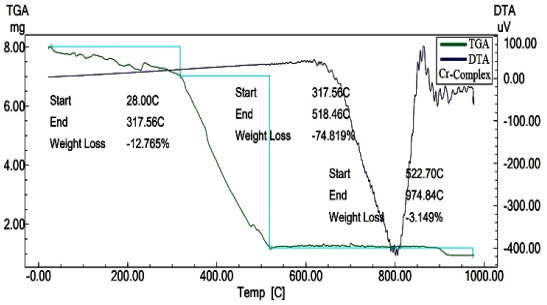

Fig. 3. DTA/TGA Arc of Cr-complex

Antibacterial Property

The optimistic regulator is taken streptomycin drug and express area of standby of 10mm and 16mm in against of *E. coli and B. subtilis* correspondingly. Adverse regulator include DMSO doesn't express any antibacterial action.

Ni-complex and Cr-complex shows antibacterial activity against both bacteria *E. coli* and *B. subtilis*. Fe-complex doesn't express any activity contrary to *E. coli and B. subtilis*.

Antifungal Property

Optimistic regulator for antifungal action is clotrimazole drug and express area of shyness of 6mm against *A. niger*. Adverse control held DMSO doesn't express any antifungal action.

Cr-complex complex shows antifungal activity against *A. niger*. Ni-complex and Fe-complex doesn't express any antifungal action contrary to *A. niger*.

Antioxidant activity

Antioxidant activity of Ni-complex, Fe-complex and Cr-complex tested contrary to the reference as ascorbic acid.

Antioxidant activity in terms of percent inhibition of free radical is 75.98% for Fe-complex at concentration of 1000 µg/mL which is 19.27% less than that of standard Ascorbic Acid 95.25% at same concentration. The IC_{50} value for Fe complex is 448.27 µg/mL and that of reference Ascorbic acid is 126.84 µg/mL. Thus Fe-complex has significant antioxidant activity similar to standard ascorbic acid. Least activity was shown by Cr-complex (32.21%) at 1000 µg/mL. Ni-complex shows intermediate antioxidant activity of 54.82% at concentration 1000 µg/mL.

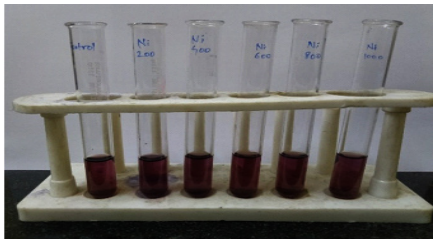

Fig. 4. antioxidant activity of Ni complex at various concentration

Table 4: Antimicrobial action of Ni-complex, Fe-complex and Cr-complex

S.No.	Compounds	Antibacterial action Zone of progress (mm)		Antifungal action Zone of progress (mm)
		E.coli	*B.subtilis*	*A.niger*
1	Ni-complex	7	11	–
2	Fe-complex	–	–	–
3	Cr-complex	9	15	2
4	Streptomycin	10	16	–
5	Clotrimazole	–	–	6

Table 5: %inhibition and IC_{50} values

Sample/Standard	%inhibition at Concentration (µg/mL)					IC_{50}µg/mL
	200	400	600	800	1000	
Ni-complex	14.21	27.55	37.12	45.93	54.82	882.61
Fe-complex	37.91	48.52	56.35	67.14	75.98	448.27
Cr-complex	9.8	15.82	22.41	28.65	32.21	1580.45
Ascorbic acid	52.55	65.41	76.32	86.95	95.25	126.84

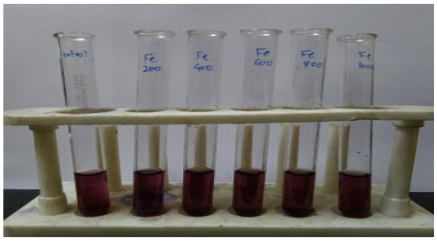

Fig. 5. antioxidant activity of Fe complex at various concentration

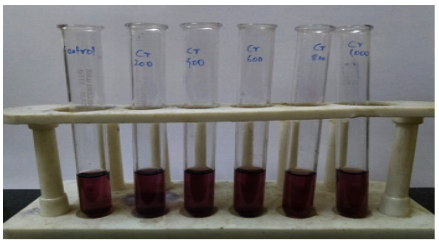

Fig. 6. antioxidant activity of Cr complex at various concentration

Anti-inflammatory action

The anti-inflammatory action of complexes was find out by using HRBC membrane stabilization technique.

Table 6: Absorbance at 560nm

Sr. No.	Concentration (µg/ml)	Absorbance at 560nm			
		Diclofenac	Ni-complex	Fe-complex	Cr-complex
1	0 (Control)	0.925	0.925	0.925	0.925
2	100	0.731	0.899	0.756	0.911
3	200	0.703	0.856	0.725	0.902
4	300	0.678	0.812	0.699	0.889
5	400	0.645	0.765	0.668	0.875

Table 7: % Protection

Sr. No.	Concentration(µg/ml)	% Protection			
		Diclofenac	Ni-complex	Fe-complex	Cr-complex
1	0 (Control)	0.00	0	0	0
2	100	20.97	2.81	18.27	1.51
3	200	24.00	7.46	21.62	2.49
4	300	26.70	12.22	24.43	3.89
5	400	30.27	17.30	27.78	5.41

Observation and Result

The percent protection of Fe-complex is higher than Ni-complex and Cr-complex complex. At highest concentration of 500 µg/mL its anti-inflammatory activity in terms of percent protection is 30.92% which is only 3.02% less than standard diclofenac (33.94%). On the other hand Cr-complex shows negligible anti-inflammatory action of 7.89% at highest concentration of 500 µg/mL. Ni-complex shows intermediate anti-inflammatory action of 20.76% at concentration of 500 µg/mL.

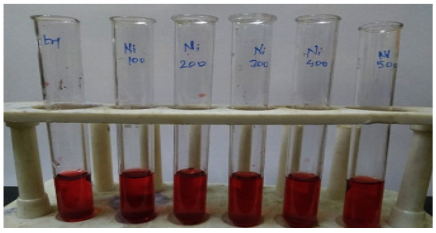

Fig. 7. Dose dependent anti-inflammatory activity of Ni complex

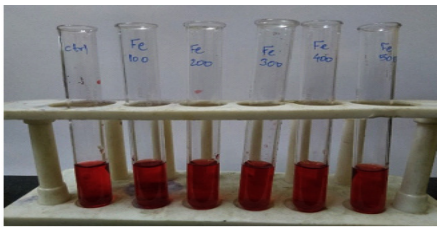

Fig. 8. Dose dependent anti-inflammatory activity of Fe complex

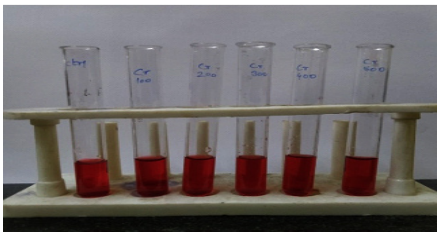

Fig. 9. Dose dependent anti-inflammatory activity of Cr complex

CONCLUSION

Ni-complex, Fe-complex and Cr-complex

are investigated for antioxidant action, anti-inflammatory action, antibacterial action and antifungal action. Ni-complex and Cr-complex shows antibacterial action, Cr-complex shows antifungal action opposite to various fungiform and bacteriological species. Ni-complex, Fe-complex and Cr-complex express antioxidant action and anti-inflammatory property.

ACKNOWLEDGMENT

This research did not receive any specific grant from funding agencies in the public, commercial, or not-for-profit sectors.

Conflicts of interest

No conflict of interest.

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