



Original Research Article

Formation, Characterization and Antioxidant Study of Mixed Ligand Complexes Derived from Succinyl Chloride

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ARTICLE INFO

Article history

Submitted: 2022-05-16

Revised: 2022-07-29

Accepted: 2022-08-10

Manuscript ID: CHEMM-2205-1528

Checked for Plagiarism: Yes

Language Editor:

Dr. Behrouz Jamalvandi

Editor who approved publication:

Professor Dr. Hassan Karimi-Maleh

DOI:10.22034/CHEMM.2022.342599.1528

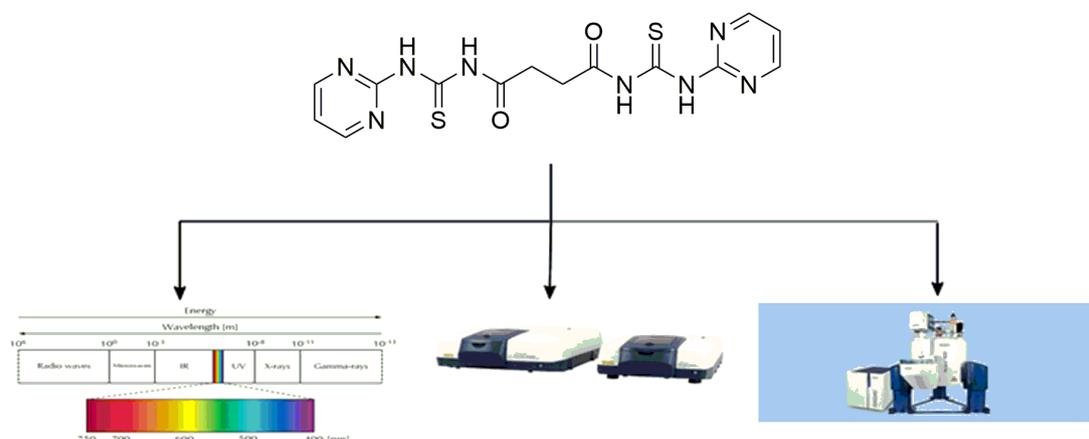
KEYWORDS

Succinyl chloride
2-Aminopyrimidine
Biological activity
Antioxidation

ABSTRACT

In the present study, mixed ligand compounds of Mn(II), Ni(II), Co(II), Cu(II), Cd(II) and Hg(II) were synthesized using new Ligand *N*1,*N*4-bis (pyrimidin-2-ylcarbamoithiyl) succinimide (NPS) derived from [Butanedioyl diisothiocyanate with 2- aminopyridine] as first ligand, and proline (pro) as second ligand and evaluation of their antioxidant activities for ligand, nickel and cobalt complex towards 1.1-Di-phenyl-2picrylhydrazyl (DPPH) were compared with the standard anti-oxidants (i.e. the ascorbic acid). The materials led to the results that came from exhibiting different activities of the radical scavenging for all of the compounds. The compounds observed were then confirmed through the Fourier-transform infrared (FT-IR), proton nuclear magnetic resonance (¹H NMR), ultraviolet-visible (UV-vis), micro-elemental analyses (CHNS), thermal analysis (TGA), carbon13 nuclear magnetic resonance (¹³C NMR), atomic absorption flame (AAF), magnetic susceptibility and conductivity. The proposed geometry for all complexes [M₂(NPS)(pro)₂]Cl₂ was tetrahedral. Furthermore, the antibacterial and antifungal activity was screened for the DMSO solution concerning the ligand (NPS) and its complexes against two kinds of gram; (*Staphylococcus aureas*) positive and (*Esherichia coli*) negative and against (*Candida albican*) fungi.

GRAPHICAL ABSTRACT



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Introduction

Antioxidants of ligand and its complexes are generally donors to the hydrogen or electron donors to the interactive site on the neutralizing free radical types. The scavenging efficacy of a variety of the organic complexes may be assessed with the use of the DPPH free radical, besides the ABTS⁺ tests. Several organic molecules notified already, acting as antioxidants, are quite good, which is why it is highly important to understand the action method besides the efficacy from those antioxidants. There are numerous normal and synthetic antioxidants that are explored; in addition, their antioxidant capability was assessed by using a number of various ways. The antioxidants have been considered as significant nutraceuticals on account of their wide range of the health advantages and are commonly utilized in the area of food industry [1-3]. Heterocyclic chemistry is one of the most complicated parts of the organic chemistry. Industrially produced compounds are useful such as pharmaceuticals and chemical agricultural materials and have an important role in human life. The Pyrimidines can be described as six-member hetero-cyclic ring complexes that are composed of the carbon and nitrogen is one of the most important hetero cyclic. Since the derivatives that have been made based upon 2-amino-pyrimidine have a wide range of the activities, organic synthesis of those compounds have attracted the attention for several decades, hence developing new approaches for synthesizing those compounds has a great deal of importance [4, 5]. Succinyl chloride was used as an important raw material and intermediate in pharmaceuticals, organic synthesis, agrochemicals, dye stuffs and hydrolytic reagent for the determination of water [6]. Prepared mixed ligand compounds differ from the routine metal complexes that have a minimal amount of 2 special ligand types linked to the one metal ion in the complex. As a result of occurrence of a variety of the ligands in same compound, obvious better characteristics for the mixed ligand compounds can be seen, which has resulted in making the mixed ligand complex synthesis interesting with a variety of the properties [7, 8].

This study was concerned with synthesizing and characterizing six complexes of the mixed ligands that have been derived from the new ligand (NPS), obtained by reaction [Butanedioyl diisothiocyanate with 2-amino pyrimidine] as first ligand, proline (pro) as second ligand using transition metal ions such as Co(II), Mn(II), Cu(II), Cd(II), Ni(II), and Hg(II).

Materials and Methods

All of the chemicals and Reagents inside in synthesis of the (NPS) and their complexes were from pure grade and purchased from Fluka, BDH and Merck chemical companies. Infra-red spectroscopy was evaluated with the use of (Shimadzu FT-IR ffinity-1s) device as well as the KBr disc ranging between (400 cm⁻¹ and 4000 cm⁻¹). By using an electrothermal melting point device (in this study it was SMP-10 Stuart), the melting point of compounds that have been prepared in the open tube has been specified. Utilizing (Shimadzu UV1800) visible ultra-violet spectrophotometer with a 3⁻¹⁰ M samples' concentration in the DMSO solvent at temperature of the room as well as a quartz cell that is 1cm long, we evaluated the prepared compounds' electron spectra. Utilizing (Bruker 300MHz NMR spectrometer) device, we recorded chemical displacement values in (NMR spectra ¹³C & ¹H) in (DMSO-d₆ with the TMS). Utilizing A device (Shimadzu (AA680)) was utilized to determine %M in the complexes. We also evaluated the prepared complexes' molar conductivity utilizing a device (Philips pw-Digital conductivity meter) with a (10⁻³ M) concentration in (DMSO) and at temperature of the room. Further, utilizing the (magnetic sensitivity balance (Sherwood Scientific)) device, we assessed (μ_{eff} BM) of compounds at the temperature of the room. Utilizing (Euro EA300) device, we determined (%M, %H, %C, %N, %S) the prepared complexes. To carry out thermal gravity analysis (TGA), An STA PT1000 Linseis at a 0-700 °C temperature range and the argon gas were applied.

Synthesis of ligand NPS [9, 10]

The step A

In dry acetone (20 mL), potassium thiocyanate (0.25 g, 2.57 mmol) was dissolved. Succinyl chloride (0.14 mL, 0.199 g, 1.29 mmol) was added slowly to the first solution while stirring at the temperature of the room for 1 hour. The white precipitate was filtered for potassium chloride.

after which the solution was left at room temperature for one hour. Then, ice powder was added to the bottle and the solution was left until the appearance of the precipitate. Good production was 87% (Scheme 1).

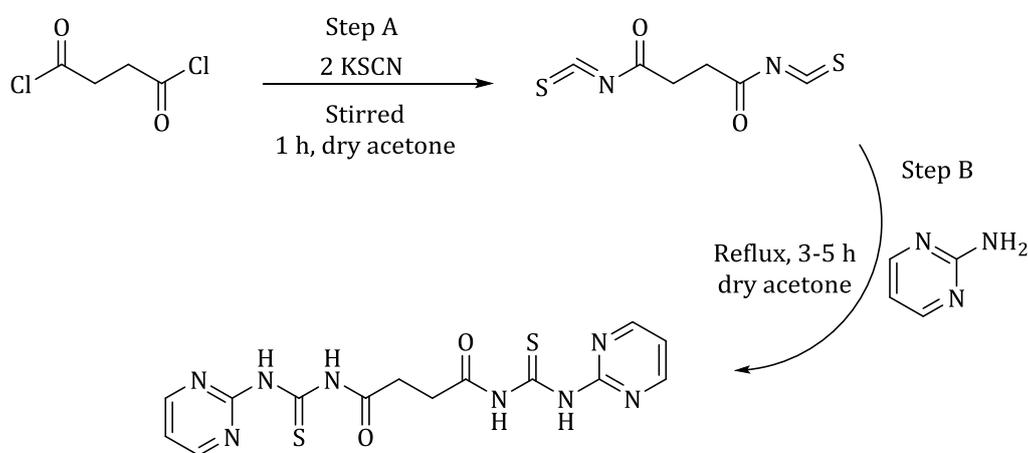
The step B

In dry acetone (15 mL), 2-aminopyrimidine (0.24 g, 2.57 mmol) was dissolved and then added to the solution obtained from the step A with stirring and refluxed at a temperature of 50-55 °C for 3-5 hours

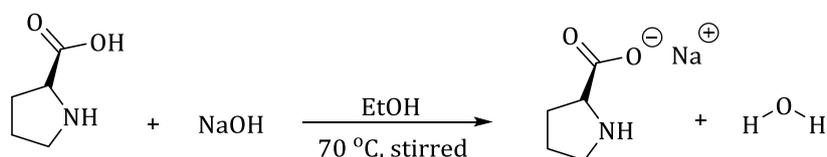
Synthesis of complexes of mixed ligand

I. Synthesis of Sodium Proline

A solution (0.06 g, 0.52 mmol) of L-Proline with (0.02 g, 0.52 mmol) solution of sodium hydroxide in ethanol was deprotonated based on the reaction below, which is represented in Scheme 2.



Scheme 1: NPS preparation course



Scheme 2: The synthesis route of Sodium Proline

II. Synthesis of $[Cu_2(NPS)(Pro)_2]Cl_2$ complex

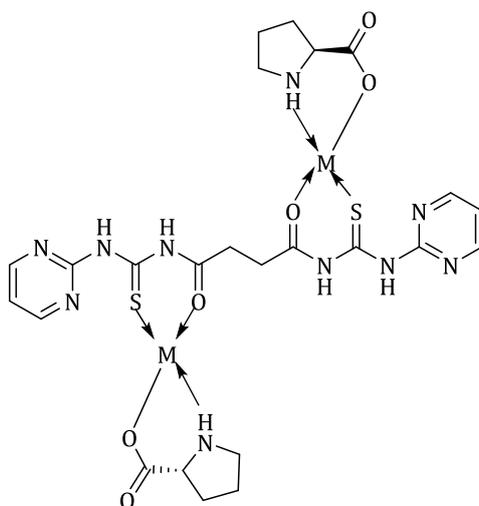
The complexes were prepared in the molar ratio (M: NPS: Pro) (2: 1: 2). The ethanol solution (10 mL) of the metal chloride ($CuCl_2 \cdot 2H_2O$) (0.09 g, 0.52 mmol) was added into the solution of the ethanol (10 mL) of Sodium proline. This mix was left at 70 °C with continuous stirring and reflex for half an hour, then (0.1 g, 0.52 mmol) of the ligand NPS was added after dissolving it in 10 mL ethanol. This mix was returned to the same conditions and for a period of 3-4 hrs thereafter. Precipitate with a color (pale blue) was formed. The precipitate was filtered, washed for a number

of times by using the distilled water and diethyl ether, and recrystallized with absolute ethanol.

III. Synthesis of $[Ni_2(NPS)(Pro)_2]Cl_2$, $[Cd_2(NPS)(Pro)_2]Cl_2$, $[Mn_2(NPS)(Pro)_2]Cl_2$, $[Co_2(NPS)(Pro)_2]Cl_2$, $[Hg_2(NPS)(Pro)_2]Cl_2$, complexes

The approach that has been utilized in order to prepare those complexes has been similar to the approach mentioned in preparation of compound $[Cu_2(NPS)(pro)_2]Cl_2$ in paragraph (II); the obtained solution complex with Ni(II), Co(II), Mn(II), Hg(II) and Cd(II) was washed for several times with distilled water and diethyl ether, and

recrystallized with absolute ethanol as shown in Scheme 3.



Scheme 3: The preparation route of the mixed ligand $[M_2(NPS)(Pro)_2]Cl_2$ complexes

Results and Discussion

The value of the thermal stability as well as the colored solid's nature represent the most significant properties of prepared metal compounds soluble in the DMF and DMSO solvents. The practical as well as the theoretical data of the A.A measurements for all of the complexes that were prepared have been approximated, as listed in Table 1.

The data fragmentation of the mass spectral of the (NPS) [N1, N4-bis (pyrimidin-2-ylcarbamothioyl) succinimide], Figure 1 showed (M^+) at $m/z=311$ as a result of the original molecular ligand NPS weight (390.4), $[C_{14}H_{14}N_8O_2S_2]$ [11]. Additional peaks are displayed in Table 2.

1H -NMR spectra of NPS

The integral intensity values of every one of the signals in the 1H -NMR spectrum of the NPS, as shown in Figure 2, was found to agree with number of various existing proton types. The spectrum showed that singlet signal at $\delta = 13.18$ ppm is assigned to 2H, CSNH group and the singlet

signal at $\delta = 12.98$ ppm is assigned to 2H, CONH group. The chemical shift at $\delta = 8.47$ ppm is assigned to 4H, CH group from pyrimidine. The triplet signal of 2H, CH group from pyrimidine appeared in $\delta = 6.84$ ppm. Ultimately, the duplate signal at the chemical shifting ($\delta = 2.50$ ppm) was determined by group protons (4H, CH₂ group from methylene succinyl) [12, 13]. The results are listed in detail in Table 3.

^{13}C -NMR spectra of NPS

^{13}C -NMR spectrum of the NPS in $(CD_3)_2SO$ solvent showed that chemical shifting at $\delta = 157.56$ ppm was a result of the C₁ for the pyrimidine ring (Figure 3). The C₂ for pyrimidine ring resonated with chemical shifting at 109.32 ppm. C₃ and C₄ for C=S of thioamide and C=O of amide groups resonated with the chemical shifts at $\delta = 177.59$ and 175.00 ppm respectively. Finally, the chemical shift at $\delta = 30.01$ ppm is attributed to C₅ for CH₂-CH₂ aliphatic in succinyl group [14, 15]. The summary of the results is listed in Table 4.

Table 1: Various physical characteristics of prepared complexes

Compounds	M.wt (g.mole ⁻¹)	m.p (°C)	Colour	Metal % Calculated (Actual)	Conducts Ohm ⁻¹ cm ² mol ⁻¹ in solvent (DMSO)

C ₁₄ H ₁₄ N ₈ O ₂ S ₂ (NPS)	390.44	160-162	Yellow	-	3.3
C ₅ H ₉ NO ₂ (proline)	115.13	205-228	White	-	5.45
C ₂₄ H ₃₀ Cl ₂ Mn ₂ N ₁₀ O ₆ S ₂	799.56	220-222	Off-White	13.74 (13.71)	77.19
C ₂₄ H ₃₀ Cl ₂ Co ₂ N ₁₀ O ₆ S ₂	807.55	300 dec.	bluish violet	14.60 (14.58)	76.66
C ₂₄ H ₃₀ Cl ₂ Ni ₂ N ₁₀ O ₆ S ₂	807.07	236-238	bright blue	14.55 (14.58)	70.90
C ₂₄ H ₃₀ Cl ₂ Cu ₂ N ₁₀ O ₆ S ₂	816.78	215-217	pale blue	15.56 (15.50)	72.38
C ₂₄ H ₃₀ Cl ₂ Cd ₂ N ₁₀ O ₆ S ₂	914.52	300 dec.	White	24.59 (24.63)	83.19
C ₂₄ H ₃₀ Cl ₂ Hg ₂ N ₁₀ O ₆ S ₂	1090.87	238-240	White	36.78 (36.72)	72.83

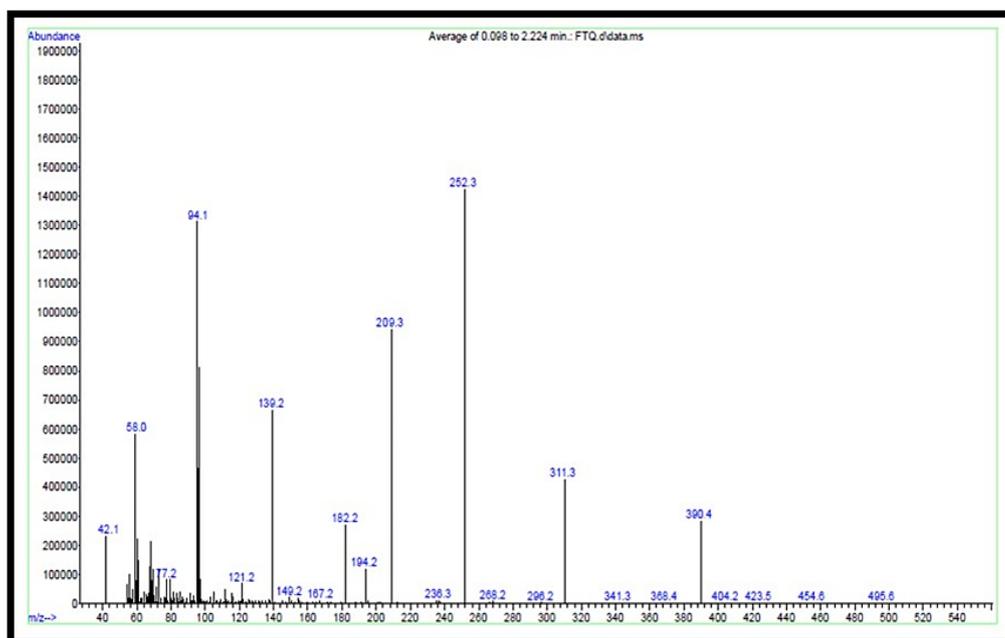


Figure 1: NPS Mass spectrum

Table 2: Fragmentation of the mass spectrum of (NPS)

Fragment ligand (NPS)	Mass/charge (m/z)	Relative Abundance (%)
[C ₁₄ H ₁₄ N ₈ O ₂ S ₂]	390.44	15.79
[C ₁₀ H ₁₁ N ₆ O ₂ S ₂] ⁺	311.36	23.15
[C ₉ H ₁₀ N ₅ O ₂ S] ⁺	252.27	74.94
[C ₈ H ₉ N ₄ OS] ⁺	209.25	50.0
[C ₆ H ₆ N ₄ OS] ⁺	182.20	14.73
[C ₅ H ₄ N ₃ S] ⁺	139.18	35.53
[C ₄ H ₄ N ₃] ⁺	94.10	69.47
[CH ₄ N ₃]	58.0	31.32
[C ₃ H ₆]	42.08	11.58

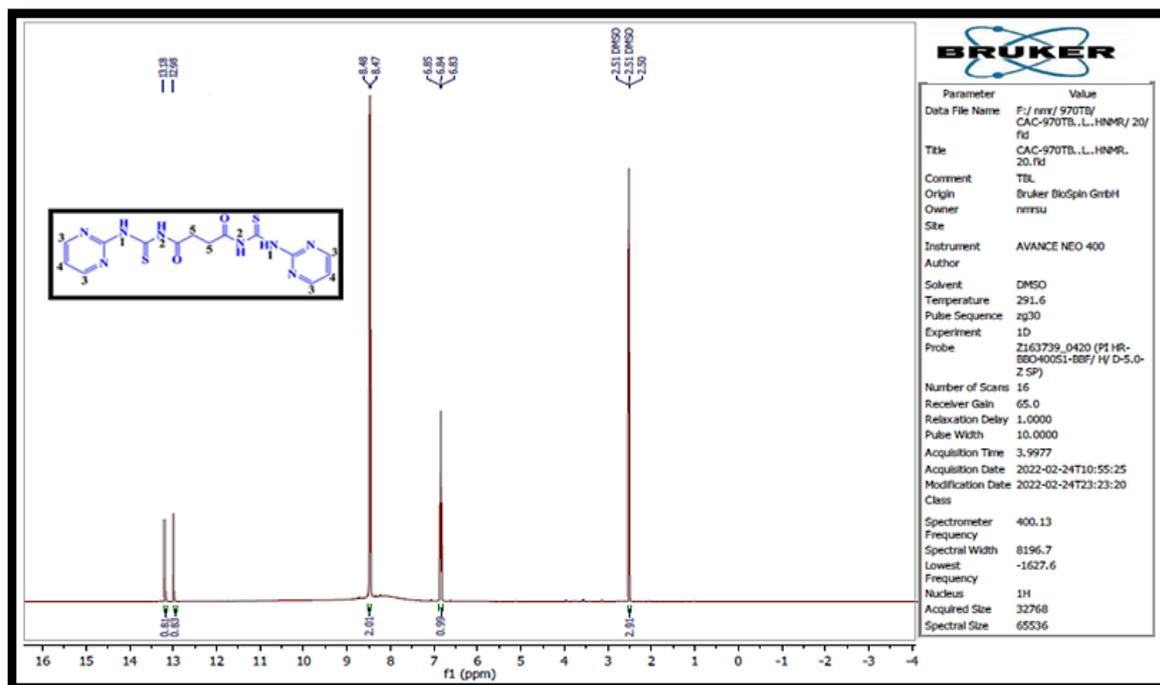


Figure 2: ¹H-NMR spectra of NPS

Table 3: ¹H-NMR data for (NPS) measured in DMSO-*d*₆ and chemical shifting in ppm (δ)

Ligand	Functional group	δ (ppm)
(NPS)	2H (singlate), proton CSNH group	13.18
	2H (singlate), proton CONH group	12.98
	4H (duplate), proton CH group from pyrimidine	8.47
	2H (triplate), proton CH group from pyrimidine	6.84
	4H(triplate), proton CH ₂ group from methylene succinyl	2.50
	DMSO	2.51

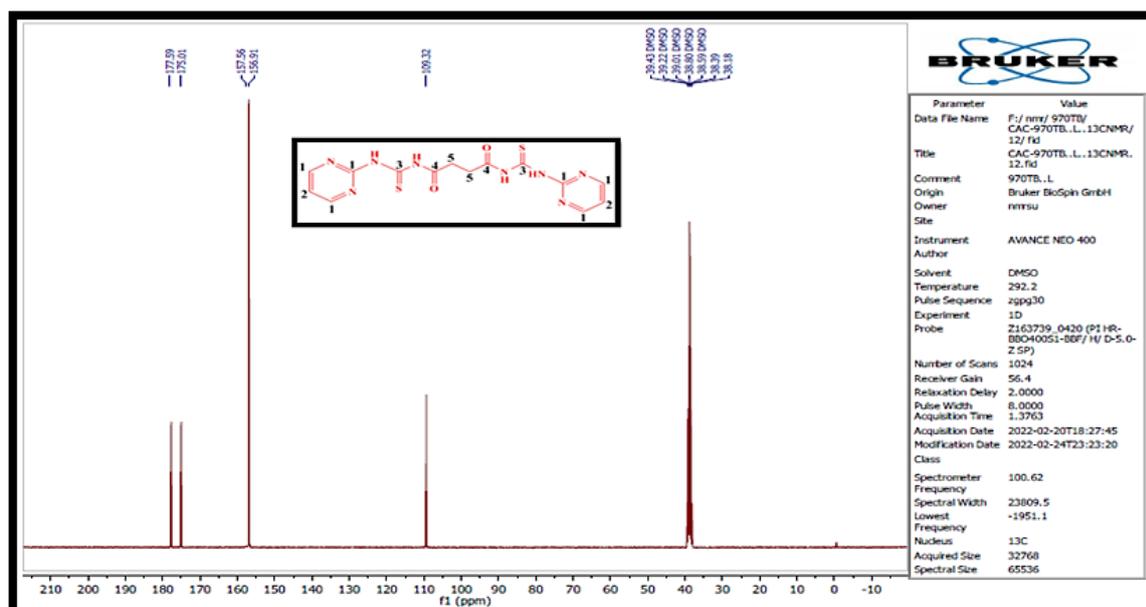


Figure 3: ¹³C-NMR spectrum of NPS

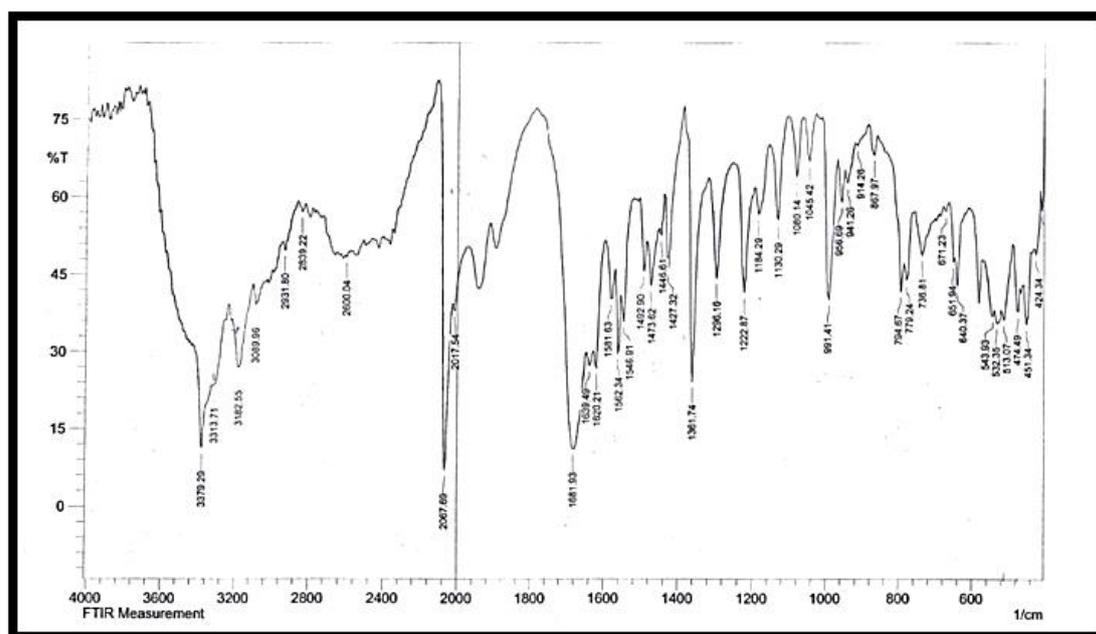
Table 4: ^{13}C -NMR data for (NPS) that has been measured in the DMSO- d_6 and the chemical shifting in ppm (δ)

Ligand	Functional group	δ (ppm)
(NPS)	C ₁ for pyrimidine ring	157.56
	C ₂ for pyrimidine ring	109.32
	C ₃ for C=S, thioamide group	177.59
	C ₄ for C=O, amide	175.00
	C ₅ for (CH ₂ -CH ₂) aliphatic in succinyl group	30.01

Ligand (NPS)

The two bands at 3379 cm^{-1} and 3313 cm^{-1} in NPS spectrum determined by $\nu(\text{NH})$, whereas another band of absorption had shown at 1681 cm^{-1} may be represented as $\nu(\text{C}=\text{O})$ amide, besides the bands of absorption at 1361 and 1130 cm^{-1} determined by $\nu(\text{C}=\text{S})$, and another band of absorption shown at 1296 cm^{-1} may be interpreted as $\nu(\text{C}-\text{N})$ [Figure 4] [16].

As for the other secondary ligand, Proline (pro), it showed bands at 3055 cm^{-1} and 2981 cm^{-1} belonged to stretch frequency band of the $\nu(\text{NH})_{\text{asym}}$ and $\nu(\text{NH})_{\text{sym}}$ group, respectively, and it also showed bands at 1627 cm^{-1} and 1408 cm^{-1} referring to the stretch bandwidth of the $\nu(\text{COO})_{\text{asym}}$ and $\nu(\text{COO})_{\text{sym}}$ group, respectively and finally the appearance of the band at $\nu(948)$ belonged to the stretch bandwidth of the $\nu(\text{C}-\text{N})$ group [17].

**Figure 4:** NPS spectrum's FT-IR

Ligand (NPS) Complexes

Those spectra have shown a noticeable variation between the bands that belong to $\nu(\text{CO}$, amide group) stretching vibration in a range of 1647 cm^{-1} - 1697 cm^{-1} shifted to a variety of the frequency values, suggesting the likelihood of NPS coordination by atom of the oxygen at amide group [18]. The band of the stretching vibration $\nu(\text{C}=\text{S})$ was measured in a range 1363 - 1346 cm^{-1}

and 1138 - 1134 cm^{-1} shifted to a different frequency, indicating the fact that sulfur atom had been part of this coordination [19]. $\nu(\text{N}-\text{H})$ in (NPS) was not related to central ion, confirmed by no change in frequency values of this group, which were fixed at 3456 - 3402 cm^{-1} and 3367 - 3329 cm^{-1} in the complexes, while in proline, the stretching vibration bands $\nu(\text{N}-\text{H})_{\text{asym}}$ and $\nu(\text{N}-\text{H})_{\text{sym}}$ was found in a range 3217 - 3224 cm^{-1} and 3082 - 3044 cm^{-1} shifted to higher frequency values, indicating

the fact that nitrogen atom was involved in coordination. The stretching vibration bands $\nu(\text{COO})_{\text{asym}}$ and $\nu(\text{COO})_{\text{sym}}$ was discovered in a range 1577-1566 cm^{-1} and 1423-1411 cm^{-1} shifted to lower and higher frequency values, meaning that oxygen atom was involved in the coordination. In the ligand complex spectrum, new bands $\nu(\text{M-S, thioamide group})$ and $\nu(\text{M-O, amide group})$ were found in a range of 516-459 cm^{-1} and 609-570 cm^{-1} in ligand (NPS), also $\nu(\text{M-N, amine group})$ and $\nu(\text{M-O, carboxyl group})$

appeared in a range of 538-497 cm^{-1} and 565-552 cm^{-1} in proline. The coordination through sulfur atom in (NH-S=O) group, the atom of oxygen in (NH-C=O) group, the atom of nitrogen in NH_2 group and oxygen atom in COO group with metal ions led to the appearance of new bands that indicated the metal complexes' formation [20, 21]. In Table 5, the FT-IR data are listed. In Figure 5, spectra of NPS as well as its complexes are presented

Table 5: FT-IR data of NPS and its compounds

Compounds	$\nu(\text{N-H})$ (NPS)	$\nu(\text{N-H})_{\text{asym}}$ $\nu(\text{N-H})_{\text{sym}}$ (pro)	$\nu(\text{C=O})$ Amide (NPS)	$(\text{COO})_{\text{asym}}\nu$ $\nu(\text{COO})_{\text{sym}}$ (pro)	$\nu(\text{C-S})$ $\nu(\text{C-S})$ thioamide (NPS)	$\nu(\text{C-N})$ (NPS) $\nu(\text{C-N})$ (pro)	$\nu(\text{M-O})$ (NPS) $\nu(\text{M-O})$ (pro)	$\nu(\text{M-N})$ (pro)	$\nu(\text{M-S})$ (NPS)
$\text{C}_{14}\text{H}_{14}\text{N}_8\text{O}_2\text{S}_2(\text{NPS})$	3379 3313	-	1681	-	1361 1130	1296	-	-	-
$\text{C}_5\text{H}_9\text{NO}_2(\text{proline})$	-	3055 2981	-	1627 1408	-	- 948	-	-	-
$\text{C}_{24}\text{H}_{30}\text{Cl}_2\text{Mn}_2\text{N}_{10}\text{O}_6\text{S}_2$	3432 3367	3124 3048	1662	1566 1415	1354 1134	1288 867	609 563	536	509
$\text{C}_{24}\text{H}_{30}\text{Cl}_2\text{Co}_2\text{N}_{10}\text{O}_6\text{S}_2$	3433 3365	3154 3082	1662	1577 1415	1354 1138	1288 885	570 552	497	459
$\text{C}_{24}\text{H}_{30}\text{Cl}_2\text{Ni}_2\text{N}_{10}\text{O}_6\text{S}_2$	3456 3340	3163 3047	1662	1573 1423	1357 1138	1280 885	582 564	533	516
$\text{C}_{24}\text{H}_{30}\text{Cl}_2\text{Cu}_2\text{N}_{10}\text{O}_6\text{S}_2$	3455 3329	3178 3044	1697	1573 1411	1346 1138	1288 867	574 565	526	486
$\text{C}_{24}\text{H}_{30}\text{Cl}_2\text{Cd}_2\text{N}_{10}\text{O}_6\text{S}_2$	3402 3336	3217 3067	1647	1573 1419	1363 1134	1288 871	576 553	522	513
$\text{C}_{24}\text{H}_{30}\text{Cl}_2\text{Hg}_2\text{N}_{10}\text{O}_6\text{S}_2$	3433 3329	3132 3045	1666	1566 1411	1354 1134	1284 867	578 555	538	462

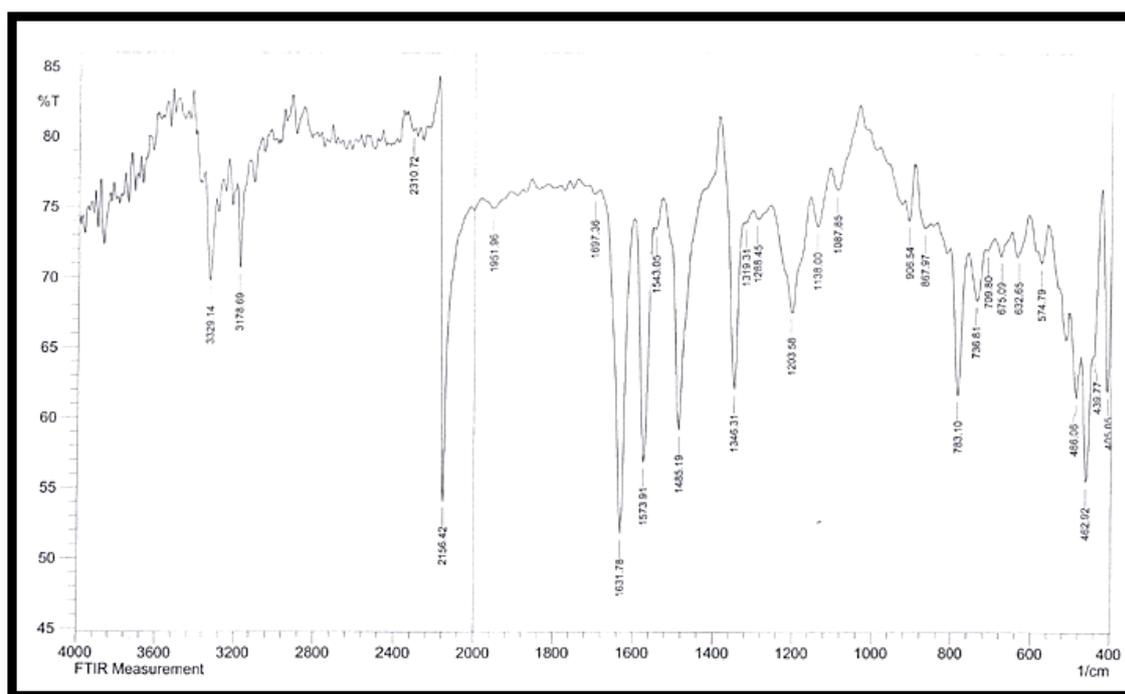


Figure 5: FT-IR spectrum of $[\text{Cu}_2(\text{NPS})(\text{pro})_2]\text{Cl}_2$ complex

Ligand (NPS)

The highest absorption intense was discovered at 33670 cm^{-1} which resulted from transitions ($\pi \rightarrow \pi^*$), in (NPS) electronic spectrum [22]. In Table 6 and Figure 6 data recorded are shown.

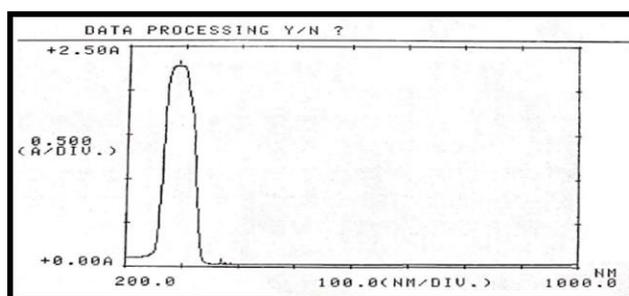
Ligand (NPS) complexes

The Manganese complex electronic spectrum showed bands at 34364, 28735 and 16393 cm^{-1} as a result of I.L, C.T and ${}^6A_1 \rightarrow {}^4T_{1(G)}$ transitions respectively, which suggests that it had tetrahedral geometry [23]. Based on bands in Co complex at 34602, 25000, 16666 and 14925 cm^{-1} , which is back to the I.L, CT, ${}^4A_{2(F)} \rightarrow {}^4T_{1(P)}$ and ${}^4A_{2(F)} \rightarrow {}^4T_{1(F)}$ transitions respectively, tetrahedral geometry of complex has been suggested [24]. Concerning Ni complex, electron spectra in

absorption bands could be assigned to the I.L, CT and ${}^3T_1 \rightarrow {}^3T_{1(P)}$ transition exhibited in 34482, 27777 and 10989 cm^{-1} respectively. Characterizing those bands is an indication of the fact that the compound has tetrahedral geometry [25]. Confirming tetrahedral geometry of Co complex by the appearance of bands at 34843, 28735, 26315 and 11135 cm^{-1} means returns to the I.L, CT, C.T and ${}^2T_2 \rightarrow {}^2E$ transitions [26]. On the basis of bands in Mercury complex at 34013 and 24390 cm^{-1} , which is back to I.L and C.T transitions respectively, the tetrahedral geometry of compound has been proposed [27]. Tetrahedral geometry of Cd complex has been suggested based on the band which appeared at 34482 and 27027 cm^{-1} that is back to the I.L and CT [28]. In Table 6, UV data are shown and in Figure 7, the (NPS) spectra and its complexes are shown.

Table 6: UV-vis data of NPS and its compounds

Compounds	Wave number		ϵ_{max} molar $^{-1}\text{cm}^{-1}$	Assignments	μ_{eff} B.M.	Suggested structure
	nm	cm^{-1}				
$\text{C}_{14}\text{H}_{14}\text{N}_8\text{O}_2\text{S}_2$ (NPS)	297	33670	2355	$\pi \rightarrow \pi^*$	-	-
$\text{C}_{24}\text{H}_{30}\text{Cl}_2\text{Mn}_2\text{N}_{10}\text{O}_6\text{S}_2$	291	34634	2297	Intra ligand	6.027	tetrahedral
	348	28735	119	C.T		
	610	16393	5	${}^6A_1 \rightarrow {}^4T_{1(G)}$		
$\text{C}_{24}\text{H}_{30}\text{Cl}_2\text{Co}_2\text{N}_{10}\text{O}_6\text{S}_2$	289	34602	2231	Intra ligand	4.325	tetrahedral
	400	25000	50	C.T		
	600	16666	100	${}^4A_{2(F)} \rightarrow {}^4T_{1(P)}$		
	670	14925	148	${}^4A_{2(F)} \rightarrow {}^4T_{1(F)}$		
$\text{C}_{24}\text{H}_{30}\text{Cl}_2\text{Ni}_2\text{N}_{10}\text{O}_6\text{S}_2$	290	34482	2253	Intra ligand	3.905	tetrahedral
	360	27777	28	C.T		
	910	10989	15	${}^3T_{1(F)} \rightarrow {}^3T_{1(P)}$		
$\text{C}_{24}\text{H}_{30}\text{Cl}_2\text{Cu}_2\text{N}_{10}\text{O}_6\text{S}_2$	287	34834	2433	Intra ligand	1.925	tetrahedral
	348	28735	1146	C.T		
	380	26315	500	C.T		
	898	11135	45	${}^2T_2 \rightarrow {}^2E$		
$\text{C}_{24}\text{H}_{30}\text{Cl}_2\text{Cd}_2\text{N}_{10}\text{O}_6\text{S}_2$	290	34482	2278	Intra ligand	0	tetrahedral
	370	27027	50	C.T		
$\text{C}_{24}\text{H}_{30}\text{Cl}_2\text{Hg}_2\text{N}_{10}\text{O}_6\text{S}_2$	294	34013	2417	Intra ligand	0	tetrahedral
	410	24390	16	C.T		

**Figure 6:** Electronic spectrum of NPS

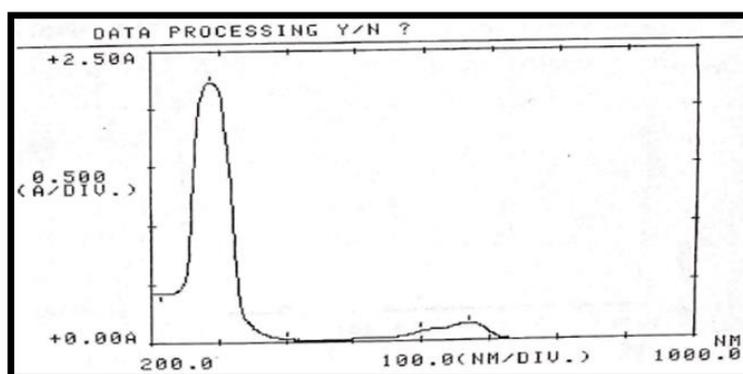


Figure 7: Electronic spectrum of $[Co_2(NPS)(pro)_2]Cl_2$ complexes

Conductivity measurements and Magnetic moments

In Table 6, the measured magnetic susceptibility values and effective magnetic moment (μ_{eff}) for Co(II), Mn(II), Cu(II) and Ni(II) compounds are displayed. Those complexes exhibit μ_{eff} 6.027, 4.325, 3.905 and 1.925 BM, respectively; these normal values have been consistent with the high spin tetrahedral compounds. Nature of the electrolytes (1:2), $M^{+2} = Mn(II), Ni(II), Co(II),$ and Cu(II) compounds of all of the metal compounds were confirmed with the measurements of the

molecular conductivity [29, 30], as can be seen in Table 1.

Thermal analyses

The ligand NPS was prepared and some of its chosen complexes were subjected to the thermal analyses with the use of STAPT1000 Linseis Company 1 Germany. In an argon gas atmosphere, this measurement was carried out with the range of temperature between (0° and 800 °C and 10 °C/min heating rate [31], where the TGA curve in Figure 8 give the results recorded in Table 7.

Table 7: Temperatures for the analyses along with the corresponding values of the weight loss

Ligand	Stage	TGA					Fragmentation
		TGA range (°C)	Mass change		% change		
			found	Calc.	found	Calc.	
(NPS)	1	160-220	1.477	1.48	47.57	47.68	- C ₃ H ₆ , 4N ₂ , O ₂ -11C, 2S
	2	221-800	1.554	1.559	50.07	50.22	

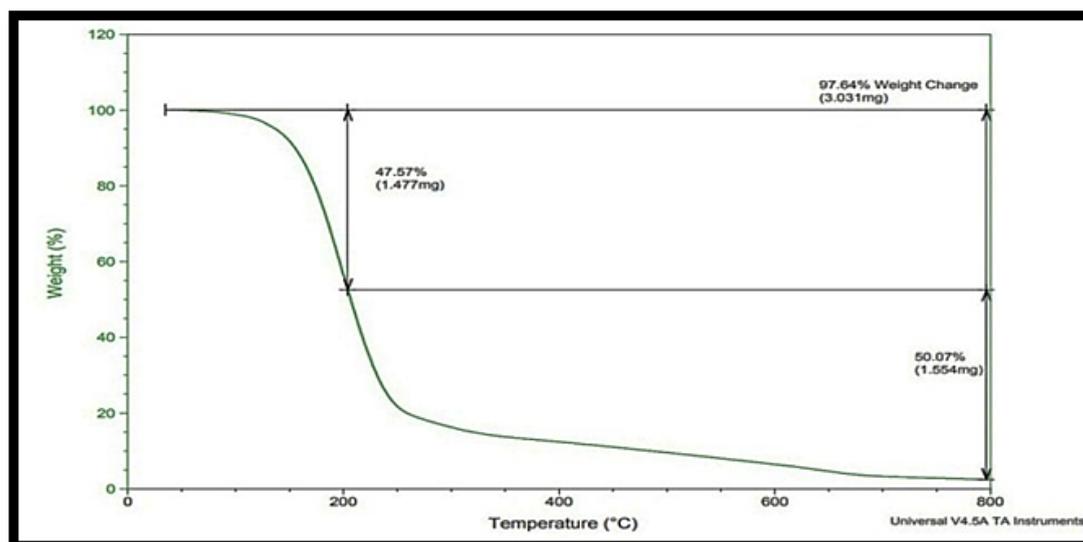


Figure 8: Thermal study of (NPS)

Anti-microbial activity researches

The bacterial, fungi cultures and conditions of the growth, *E. coli* (G⁻), *S. aureus* (G⁺) and *candida albicans* were utilized as testing microorganisms. In organisms that were tested, the surface of the medium was inoculated then covered. Prior to the application disks, the surface of the agar was provided to dry from 3 min to 5 min. By means of sterile forceps, the disks were dipped to beaker of

chemicals and put them within previous medium. At a temperature of 37 °C for 48 hrs, the culture plates of the bacteria and fungi were incubated to grow. In the concentration levels that were prepared, complexes displayed various efficacy for the inhibition of fungi and bacteria spread and fungi compares ligand (NPS) [32, 33]. The data obtained were listed in Table 8, and Figures 9 and 10.

Table 8: The inhibition diameter values of NPS and its compounds

Compound	<i>E. coli</i>	<i>S. aureus</i>	<i>Candida albicans</i>
Controls	-	-	-
Ligand (NPS)	16	16	19
C ₂₄ H ₃₀ Cl ₂ Mn ₂ N ₁₀ O ₆ S ₂	18	15	20
C ₂₄ H ₃₀ Cl ₂ Co ₂ N ₁₀ O ₆ S ₂	15	12	22
C ₂₄ H ₃₀ Cl ₂ Cu ₂ N ₁₀ O ₆ S ₂	15	14	29
C ₂₄ H ₃₀ Cl ₂ Hg ₂ N ₁₀ O ₆ S ₂	31	31	38

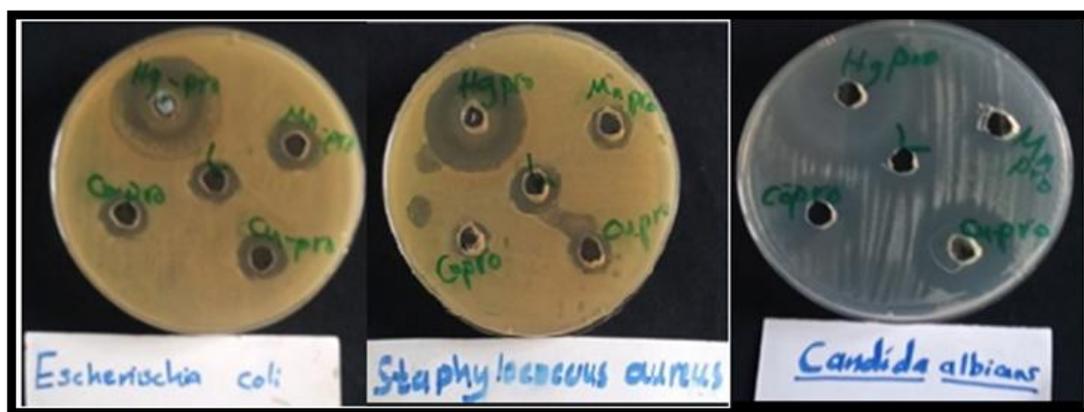


Figure 9: The inhibition diameter values of (NPS) and its complexes against the chosen fungi and bacteria

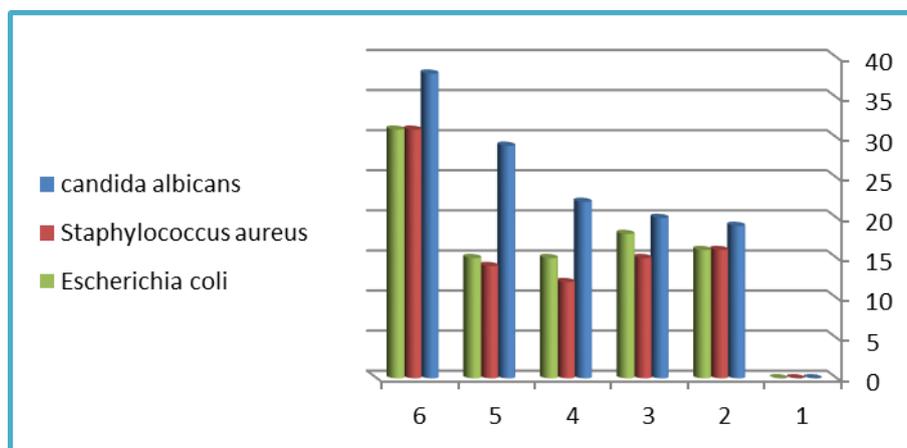


Figure 10: Statistical representation for microbial activity of ligand and its complexes

Table 9: Antioxidant potential activity of compounds

Compounds	DPPH Scavenging activity (IC 50 in μM)				
	200	100	50	25	12.5
Ascorbic acid	84.57 \pm 1.103	76.70 \pm 1.311	66.20 \pm 0.2412	53.28 \pm 1.452	41.17 \pm 1.240
Ligand (NPS)	52.54 \pm 1.910	39.31 \pm 2.270	29.11 \pm 0.4177	16.89 \pm 2.515	14.24 \pm 2.148
C ₂₄ H ₃₀ Cl ₂ Ni ₂ N ₁₀ O ₆ S ₂	79.01 \pm 2.107	66.90 \pm 2.215	50.89 \pm 1.692	39.04 \pm 3.518	26.78 \pm 0.5684
C ₂₄ H ₃₀ Cl ₂ Co ₂ N ₁₀ O ₆ S ₂	53.40 \pm 0.6490	41.74 \pm 2.116	30.17 \pm 1.023	18.17 \pm 0.7529	15.74 \pm 1.838

Antioxidant test

The anti-oxidant potential has been determined by the use of DPPH free radical scavenging assay. Briefly, 30 μl of different concentration levels of the compounds ranging between 12.5-200 μg has been added into 50 μl of the DPPH solution and incubated at temperature of the room for 30 min under dark. The values of the absorbance were recorded after 30 min at 517 nm by considering ascorbic acid as positive reference anti-oxidant activity. After that, the percentage activity computations were performed, when compared with standard reference ascorbic acid, ligand, nickel and cobalt compounds were found to be more active. Similarly, anti-oxidant activity of the nickel and cobalt complex was found to be active anti-oxidant activity than ligand (Table 9) [34, 35].

Conclusion

In this paper, characterization and synthesis of 6 mixed ligand compounds were obtained from reaction [Butanedioyl diisothiocyanate with 2-amino pyrimidine] as first ligand, and proline (pro) as second ligand using transition metal ions such as Mn(II), Hg(II), Cu(II), Co(II), Ni(II), and Cd(II). The ligand (NPS) as bidentate and potent donors was found to be C=O and C=S groups. The NPS and its compounds were observed for the antimicrobial activities towards 1 fungi type and 2 bacteria types. The efficiency of the radical scavenging from the ligand, besides its compounds, was scrutinized with the use of the DPPH screening. The anti-oxidant mensuration from the attended compounds explained that functional groups of -NH-C=S as well as the existence of the electron donating had a significant impact on efficiency of radical scavenging from the complexes.

Acknowledgments

Authors would like to thank the College, as well as its distinguished professors and to lab employees due to the assistance and the support that they have offered for performing all of the research measurements.

Funding

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

Authors' contributions

All authors contributed to data analysis, drafting and revising the paper and agreed to be responsible for all the aspects of this work.

Conflict of Interest

We have no conflicts of interest to disclose.

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HOW TO CITE THIS ARTICLE

Taghreed Q. Abd Alkareem, Enass J. Waheed. Formation, Characterization and Antioxidant Study of Mixed Ligand Complexes Derived from Succinyl Chloride. *Chem. Methodol.*, 2022, 6(12) 914-928

<https://doi.org/10.22034/CHEMM.2022.342599.1528>

URL: http://www.chemmethod.com/article_155318.html