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Preparation, Identification, and Antioxidant Activity of Some New Transition Metal Complexes of Curcumin with Mono-and Bidentate Ligands

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Abstract: Eight ternary copper (II) and nickel (II) complexes of curcumin (Cur) [1,7-bis(4-hydroxy-3-methoxyphenyl)-1,6-heptadiene-3,5-dione] as a primary ligand, o-phenylenediamine (OPD), dimethylglyoxime (H_2dmg), tryptophan (Tryp), bromoaniline (Br-Ani) or thymine (Thy) as a secondary ligand have been prepared and characterized by melting point, conductivity, infrared and electronic spectra. The complexes obtained are stable powders at room temperature, soluble in DMSO and are non-electrolyte. The IR and UV-vis spectroscopic analysis revealed that the complexes adopt either octahedral, tetrahedral or square planar geometry. All the ligands are bound to the cations Cu(II) and Ni(II) via the nitrogen and oxygen atoms. The synthesized compounds were screened for their in-vitro antioxidant activities using 2,2-diphenyl-1-picrylhydrazyl radical (DPPH$^{\cdot}$) free radical scavenging. The results obtained show that these complexes have a good antioxidant activity in comparison with ascorbic acid as positive control. These observations suggest the possibility of using the complexes as antioxidant agents to solve the problems linked to the presence of free radicals in living organisms.

Keywords: Complex, Curcumin, Ligand, Spectroscopic analysis, Antioxidant activity

Introduction

Metal bound organic compounds are known to possess potential activities in the areas of biological, clinical, analytical, catalytic, microbial, insecticidal, antibiotic, growth factors, food additive, tumor inhibitor, cell division etc. This is due to either the unused coordination sites present on the metal and ligand systems, or due to the selective oxidation state of the complexed metal ions in the coordination sphere (Cotton &Wilkinson, 1996). Scientific researches spanning over more than four decades have confirmed the diverse pharmacological effects of curcumin and established its ability to act as a therapeutic agent for several diseases (Gubendran, 2016).

The chemistry for oxime metal complexes has been investigated actively since the first synthesis of nickel (II) dimethylglyoximate and recognition of the chelate (Erdem-Tuncmen & Karipcin, 2012). Copper (II) complexes with dimethylglyoxime are known for their high stability, but their biological activity has been very scarcely studied (Bougherra et al., 2018). Metal-based antioxidants have captured outlook recently for their receptivity to concerve organisms and cells from harm induced by oxidative stress or scavenge free radicals (Mahadi- Hasan et al., 2021).

This paper describes the synthesis and the charcterization of eight new metal transition complexes of curcumin as a primary ligand and some mono-and bidentate organic compounds as secondary ligands. The antioxidant

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activity is evaluated by DPPH radical scavenging activity using ascorbic acid as positive control. The aim of this studies is to examine in vitro the capacity of these complexes to inhibit free radicals present in living organism.

Method

Curcumin (Cur) and all the secondary ligands (o-phenylenediamine (OPD), dimethylglyoxime (H_2dmg), tryptophan (Tryp), bromoaniline (Br-Ani) and thymine (Thy)) were purchased from commercial sources and used as received. Other chemical reagents of reagent grade were used without purification. Melting points were measured using a melting point SMP30. The conductivity measurements are determined using a CONSORT C3030 standard at room temperature in 10^{-3} M solutions of the complexes in DMSO. IR spectra were recorded in the pure, undispersed solid state in a SHIMADZU IRAffinity-1S spectrometer at room temperature holder in the range 4000-400 cm^{-1}. The electronic absorption spectra in DMSO solution were recorded on a thermo-scientific, Evolution 220 UV-visible spectrophotometer in the 900-200 nm range.

Synthesis of Complexes

The solvent used in all reactions is ethanol. The experimental equipment consists of a heating tank topped with a column with cold water circulation to prevent the loss of matter. The reagents are kept under constant stirring at a temperature of 60°C until a solid precipitate was obtained, collected by filtration through a sintered glass filter and washed. Successively with water and ethanol to remove all traces of metal or ligand, then with ethyl ether and left to dry. Three complexes were synthesized by substitution method, whereas five have been prepared by addition method.

Copper(II) Complexes

$[Cu(Cur)_2Br_2] + H_2dmg \rightarrow [Cu(Cur)_2(H_2dmg)]$ **Complex 1**
$[Cu(Trp)_2] + Cur \rightarrow [Cu(Cur)(Trp)]$ **Complex 2**
$Cu(NO_3)_2, 3H_2O + Br\text{-}Ani + Cur \rightarrow [Cu(Cur)(Br\text{-}Ani)_2]$ **Complex 3**
$Cu(NO_3)_2, 3H_2O + OPD + Cur \rightarrow [Cu(Cur)(OPD)]$ **Complex 4**

Nickel(II) Complexes

$[Ni(Hdmg)_2] + Cur \rightarrow [Ni(Cur)(Hdmg)_2]$ **Complex 5**
$Ni(NO_3)_2, 6H_2O + Trp + Cur \rightarrow [Ni(Cur)_2(Trp)]$ **Complex 6**
$Ni(NO_3)_2, 6H_2O + Br\text{-}Ani + Cur \rightarrow [Ni(Cur)(Br\text{-}Ani)_2]$ **Complex 7**
$Ni(NO_3)_2, 6H_2O + Thy + Cur \rightarrow [Ni(Cur)(Thy)]$ **Complex 8**

Antioxidant Activity

Antioxidant activity of the synthesised compounds was evaluated by DPPH (Figure 1) radical scavenging effect. The 2,2-diphenyl-1-picrylhydrazyl radical (DPPH) is a stable free radical, which has been widely accepted as a tool for estimating the free radical scavenging activities of potential antioxidants (Brand-Williams & Cuvelier, 1995). The antioxidant capacity of the complexes was evaluated by the method of Wang et al. (Wang & Rangarajan, 1998). The diluted working solutions of the test compounds were prepared in DMSO. Ascorbic acid was used as positive control in 0.5-100 mmol/l solution. 0.002% of DPPH was prepared in ethanol and 2 ml of this solution was mixed with 40 ㎖ of test compounds at different concentrations. The reaction mixture was incubated in the dark at room temperature for 30 min. The DPPH radical scavenging activity was determined by measuring the absorbance at 517 nm using the UV-vis spectrophotometer. The DPPH radical scavenging activity of ascorbic acid was also assayed for comparison. The absorbance was recorded and radical scavenging activity was expressed as percentage inhibition of DPPH radical and was calculated by the following equation (Embarek, 2005):

% Inhibition = $[A_0 - A/A_0] \times 100$, where A_0 is the absorbance of the negative control; and A is the absorbance of the test compounds.

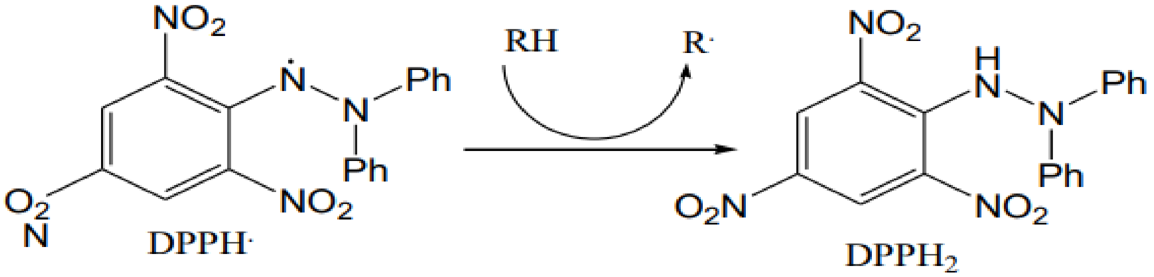

Figure 1. Reduction of the DPPH radical• (DPPH• + RH→ DPPH-H + R)

Results and Discussion

Physical Properties and Analytical Data

The physical properties and analytical data of the synthesized complexes are summarized in Table 1. The compounds are obtained in solid state; they are stable in air and at room temperature. Their melting points are above 165°C. All the complexes are insoluble in water, ethanol, and methanol, slightly soluble in the mixture water-ethanol, and soluble in dimethylsulfoxide. The nonelectrolyte nature of the complexes was confirmed by the low molar conductance values measured at 25°C (10^{-3} M) in DMSO solution.

Table 1. Characteristic and analytical data of complexes

Complex	Mol. Wt (g/mol)	Color	Yield (%)	M.P. (°C)	Λm (Ω^{-1}.cm ^{2}.mol^{-1})
[Cu(Cur)$_2$ (H$_2$dmg)]	916,42	Black	46,67	>350	14,50
[Cu(Cur)(Trp]	636,15	Black	66,7	>350	20,3
[Cu(Cur)(Br-Ani)$_2$]	663,95	Dark gray	14	>350	6,60
[Cu (Cur)(OPD)]	540,06	Light brown	62	254	3,31
[Ni(Cur)(Hdmg)$_2$]	659,31	Dark pink	42	318	2,50
[Ni (Cur)$_2$(Trp)]	999,68	Brown	94	>350	40,2
[Ni(Cur)(Br-Ani)$_2$]	771,13	Grayish white	52	>350	3,24
[Ni(Cur)(Thy)]	553,18	Orange	47	165	5,90

Spectroscopic Studies

FT-IR Spectra

The spectral data of the free ligands are given in Table 2. In the IR spectrum of free Curcumin, the absorption bands at 3505, 1629, 1504 and 1425 cm^{-1} are assigned respectively to ν(O-H)$_{enol}$, ν_{as}(C=O), ν(C=C) and δ(C-O)$_{enol}$ (Refat, 2013). The IR spectrum of free H$_2$dmg shows a broad band at 3196 cm^{-1} assigned to OH group of oxime, a medium intense band at 1436 cm^{-1} characteristic of C=N streching vibration, and a deformation band at 1135 cm^{-1} of N-O (Adkhis et al., 2017; Bougherra et al., 2018).

The infrared spectrum of tryptophan exhibits a band at 3404 cm^{-1} which is attributed to the ν (N-H) stretching vibration frequency of the indole heterocycle. The symmetric and asymmetric ν(NH$_3^+$) vibration bands appeared, respectively, at 3037 and 3081 cm^{-1} (Rabindra & Mohan, 2000). The IR spectrum of Br-Ani shows two absorption bands at 3473 and 3381 cm^{-1} assigned to the asymmetric and symmetric vibrations of the NH$_2$ group, respectively (Adkhis et al., 2017). Two other peaks are also observed at 1274 and 1607 cm^{-1} which correspond, respectively, to the ν(C-NH$_2$) frequency and the ν(C=C) frequency (Afraa, 2017).

In the OPD spectrum, the bands observed at 3185-3373 cm^{-1} are due to NH$_2$ and N-H vibrations, respectively. On the other hand, the band corresponding to the frequency of ν(C=C) is located at 1626 cm^{-1} (Bougherra et al., 2018). The spectrum of thymine shows two bands at 1672 cm^{-1} and 1752 cm^{-1} which are assigned to streching vibration of C=O group (Adkhis et al., 2017).

Table 2. IR spectra data of ligands (cm^{-1})

Ligand	Cur	H_2dmg	Tryp	Br-An	OPD	Thy
ν(O-H)	3505	3196	-	-	-	-
ν(N-H)	-	-	3404	-	3373	-
ν(NH_2)	-	-	-	3473, 3381	3185	-
ν(NH_3^+)	-	-	3037, 3081	-	-	-
ν(C=O)	1629	-	-	-	-	1725, 1672
ν(C=N)	-	1463	-	-	-	-
ν(C=C)	1504	-	-	1607	1626	-
δ(CH)	1271	-	-	-	-	1384
δ(C-O)$_{phe}$	1425	-	-	-	-	-
δ(C-NH_2)	-	-	-	1274	1262	-
δ(NO)	-	1135	-	-	-	-

The IR spectra of the complexes show a band in the region 3502-3520 cm^{-1}. This band exists at almost the same frequency in free curcumin, indicating that the OH group is not involved in the complexation. All the other characteristic bands of free curcumin ν(C=O), δ(C-O)$_{enol}$, ν(C=C), are displaced in the IR spectra of their complexes. The band at 1436 cm^{-1} of dimethylglyoxime assigned to (C=N) of the oxime is shifted to a higher frequency at 1504 cm^{-1} in complex 1 and at 1567 cm^{-1} in complex 5. Whereas the band observed at 1135 cm^{-1} which due to δ(N-O) of the oxime group is shifted in complex 1 to 1152 cm^{-1} and to 1225 cm^{-1} in complex 5. New bands appear in the region 518-522 cm^{-1} and 432-454 cm^{-1} which can be attributed to ν(M-O) and ν(M-N) respectively. This means that the coordination of curcumin with Cu(II) and (Ni) is carried out with the oxygen of the carbonyl groups in the enol form and by the nitrogen atom of the oxime function for H_2dmg (Table 3).

The vibrational frequency band ν(NH) of free tryptophan observed at 3404 cm^{-1} was shifted to lower frequencies at 3366 and 3373 cm^{-1} in complexes 2 and 6. On the other hand, that of ν(NH_3^+) was shifted to higher wavenumbers in these complexes, 1149 and 1139 cm^{-1}. Two new bands appear at 564-565 cm^{-1} and 458-482 cm^{-1} which correspond, respectively, to the M-O and M-N bonds attests that the Cu(II) and Ni(II) are coordinated to the ligands via the oxygen and nitrogen atoms (Refat, 2013; Ismail et al., 2014).

The bands at 3464 and 3373 cm^{-1} assigned to the asymmetric and symmetric stretching vibration ν(NH_2) in complex 3 and the band at 3409 cm-1 corresponding to the frequency ν(NH_2) in complex 7 (Figure 2) indicate the presence of bromoaniline in these two complexes (Adkhis et al., 2017). Thus the appearance of new bands at 530-566 cm^{-1} and in the region 456-489 cm^{-1} corresponding respectively to the frequencies ν(M-O) and ν(M-N).

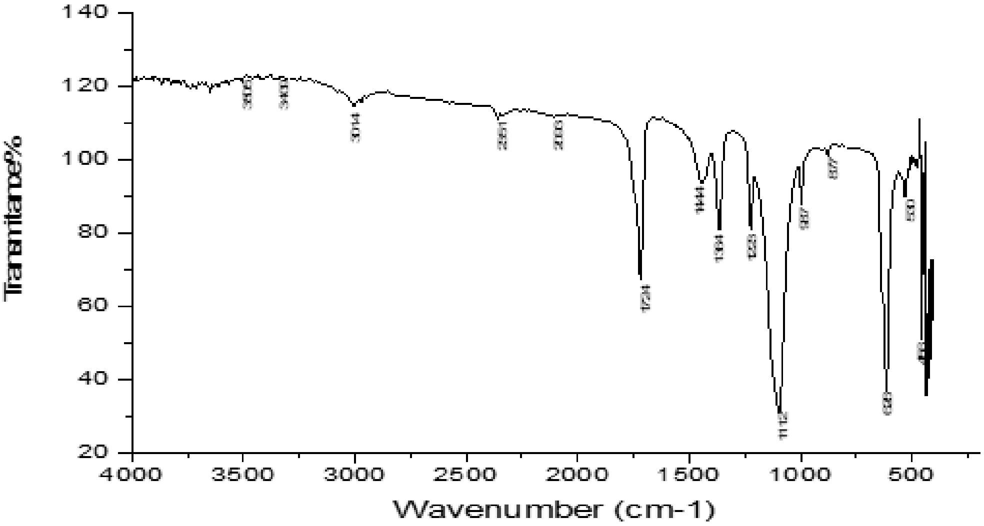

Figure 2. IR spectrum of [Ni(Cur)(Br-Ani)$_2$]

The IR spectrum of the complex 4 with OPD exhibits a characteristic band due to the N-H stretching vibration of the OPD ligand at 3352 cm^{-1}. On the other hand, the band corresponding to the ν(C=C) vibrational mode for complex 4 is clearly shifted to a lower frequency compared to that of free OPD which is located at 1494 cm-1. The bands in the region 1620-1578 cm^{-1} can be attributed to the C=O stretching vibration of curcumin and thymine coordinated in complex 8 (Adkhis et al., 2017). Two new bands appear at 545 and 462 cm^{-1} can be attributed to M-O and M-N vibrations, respectively.

Table 3. IR spectra data of Cu(II) and Ni(II) complexes (cm^{-1})

Complex	1	2	3	4	5	6	7	8
ν(O-H)	3514	3519	3514	3515	3519	3502	3505	3520
ν(N-H)	-	3366	-	3352	-	3452	-	-
ν(NH$_2$)	-	-	3464, 3373	-	-	-	3409	-
ν(NH$_3^+$)	-	3149	-	-	-	3139	-	-
ν(C=O)	1584	1613	-	1600	1567	1572	1724	1620, 1578
ν(C=N)	1453	-	-	-	1436	-	-	-
ν(C=C)	1504	1494	1578	1494	-	1488	-	1516
δ(CH)	1260	1285	-	-	-	-	1364	1362
δ(C-O)$_{phe}$	1419	1397	1383	1411	1368	1417	1444	1411
δ(C-NH$_2$)	-	-	1278	1264	-	-	-	-
δ(NO)	1152	-	-	-	1225	-	-	-
ν(Cu-O)	522	567	566	573	-	-	-	-
ν(Cu-N)	454	482	489	482	-	-	-	-
ν(Ni-O)	-	-	-	-	518	567	530	545
ν(Ni-N)	-	-	-	-	432	458	456	462

UV-Visible Spectra

The spectrum of curcumin shows two bands, one in the ultraviolet region at 267 nm, attributed to the π → π * transition, and another in the visible region at 436 nm which is attributed to the n→π * transition. The spectrum of dimethylglyoxime contains one band in the ultraviolet at 269 nm corresponding to the π → π * transition (Bensiradj &Tidjani-Rahmouni, 2014). Electronic absorption data of all the ligands are given in the Table 4.

Table 4. Electronic spectra data of ligands

Ligand	Cur	H$_2$dmg	Tryp	Br-An	OPD	Thy
λ (nm)	267 436	269	291	283 307	269 305	273
Transition	π → π * n → π *	π → π *	π → π *	π → π * π → π *	π → π * π → π *	π → π *

All complexes display a band in the 437-377 nm region (Table 5), this band is attributed to a charge transfer transition from the ligand to the metal (Amira et al., 2008). Copper (II) complexes 1, 2, and 3 exhibit two bands between 564 and 771 nm corresponding to the d-d transition; these transitions characterize a square-planar geometry. The electronic absorption spectrum of complex 4 exhibited two bands in the visible spectrum, the first at 556 nm and the second at 734 nm, attributed to the d-d transition.

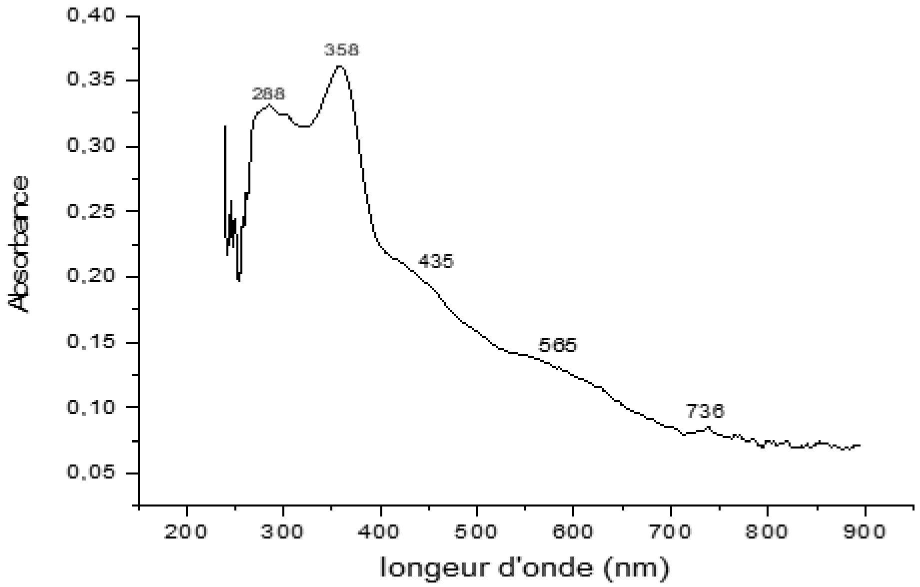

Figure 3. Electronic absorption spectrum of [Ni(Cur)(Br-Ani)$_2$]

Complexes 5 and 6 showed three bands in the visible range around 500 to 800 nm corresponding to the d-d transition. These transitions are characteristic of nickel complexes in an octahedral field (Refat, 2013). The complex 7 exhibits two bands, one at 565 nm and another at 736 nm (Figure 3) which correspond to the d-d transition, and conforms to a square-planar geometry. The electronic spectrum of complex 8 shows a broad band at 436 nm corresponding to the d-d transition which can be masked by the charge transfer transition, because they appear in the same area and whose geometry is tetrahedral.

Table 5. Electronic spectra data of complexes

Complex	1	2	3	4	5	6	7	8
λ (nm)	272 320 387 564 641	269 361 437 585 730	274 363 431 505 771	267 310 426 556 734	266 320 377 582 681 796	274 364 427 569 740 819	288 358 435 565 736	269 303 436
Transition	$\pi \rightarrow \pi^*$ $\pi \rightarrow \pi^*$ Tc (L-M) d – d d – d	$\pi \rightarrow \pi^*$ $\pi \rightarrow \pi^*$ Tc (L-M) d – d d – d	$\pi \rightarrow \pi^*$ $\pi \rightarrow \pi^*$ Tc (L-M) d – d d – d	$\pi \rightarrow \pi^*$ $\pi \rightarrow \pi^*$ Tc (L-M) d – d d – d	$\pi \rightarrow \pi^*$ $\pi \rightarrow \pi^*$ Tc (L-M) d – d d – d d – d	$\pi \rightarrow \pi^*$ $\pi \rightarrow \pi^*$ Tc (L-M) d – d d – d d – d	$\pi \rightarrow \pi^*$ $\pi \rightarrow \pi^*$ Tc (L-M) d – d d – d	$\pi \rightarrow \pi^*$ $\pi \rightarrow \pi^*$ Tc (L-M) d-d

Antioxidant Assay

The mixed metal complexes were estimated in various concentration of the compounds *in vitro* by 2,2-diphenyl-1-picrylhydrazyl radical (DPPH). In qualitative antioxidant assay, the matal complexes revealed free radical scavenging properties through ensuring the presence of yellow color on purple background on the TLC plate. Complexes also confirmed their antioxidant properties in quantitive DPPH free radical scavenging assay (Mubarak et al., 2017). The inhibition concentration of antioxidant was determined by mesearing the IC_{50} value. The antioxidant power of a compound is higher when its IC_{50} is small.

Five complexes showed antioxidant activity compared to the positive control ascorbic acid (AA) with IC_{50} values ranging from 0.05–0.5 mol/l (Figure 4), while ascorbic acid (positive control) showed a value of 0.0007 mol/l. The activity of all these complexes is mainly due to the presence of OH groups of non-deprotonated curcumin during complexation and which can easily release a hydrogen to reduce DPPH. The higher activity of complexes 1 and 5 which are complexes of dimethylgloxime and curcumin is due to the number of OH groups present in these two ligands.

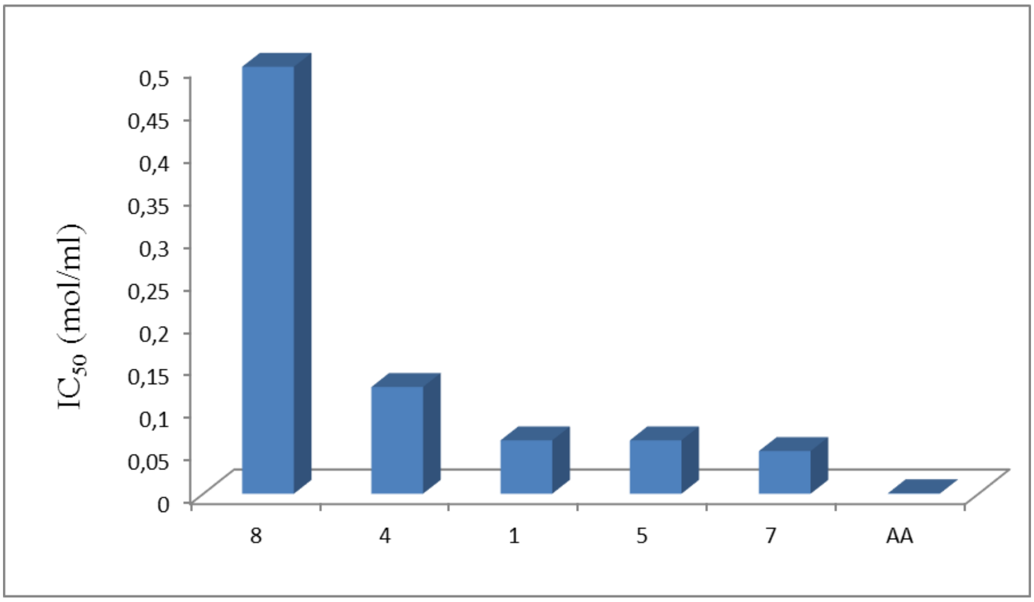

Figure 4. Antioxidant activity of complexes using DPPH assay

Conclusion

In this work, we report the preparation and characterization by different physicochemical and spectroscopic techniques eight ternary copper (II) and nickel (II) complexes of curcumin as a primary ligand, o-phenylenediamine, dimethylglyoxime, tryptophan, bromoaniline or thymine as a secondary ligand. Infrared spectroscopy studies comparing the spectra of the complexes with those of the corresponding ligands revealed the coordination of the ligands with the metals. Based on the UV-visible spectroscopy results we proposed tetrahedral and square planar geometries for Cu(II) complexes and octahedral, tetrahedral and square planar geometry for Ni(II) complexes.

Antioxidant assay revealed that complexes 1, 4, 5, 7 and 8 show an antioxidant effect compared to that of ascorbic acid (AA), due to the presence of protons from the phenols of curcumin. Complexes 1 and 5 show a strong activity due to the presence of OH groups of dimethylglyoxime, in addition to those of curcumin. These observations suggest the possibility of using complexes to solve the problem caused by the presence of free radicals in living organisms.

Scientific Ethics Declaration

* The authors declare that the scientific ethical and legal responsibility of this article published in EPSTEM journal belongs to the authors.

Conflict of Interest

* The authors declare that they have no conflicts of interest

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