Materials Today: Proceedings xxx (xxxx) xxx

Contents lists available at ScienceDirect

Materials Today: Proceedings

journal homepage: www.elsevier.com/locate/matpr

Biological activity and laser efficacy of new Co (II), Ni (II), Cu (II), Mn (II) and Zn (II) complexes with phthalic anhydride

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ARTICLE INFO

Article history:
Received 23 May 2020
Received in revised form 2 July 2020
Accepted 5 July 2020
Available online xxxx

Keywords:
Complexes
Phthalic anhydride
Laser stability
Biological activity
Bridging bidentate

ABSTRACT

This article included preparation and spectroscopic diagnosis of new cobalt(II), copper(II), nickel(II), manganese(II) and zinc(II) complexes with phthalic anhydride (PL) ligand using a mole ratio of 1 Metal: 1 PL. The as-prepared complexes were characterized using ^{1}H NMR, UV–Vis, FTIR, magnetic susceptibility and C.H.N. The measurements indicate that PL ligand was linked to the divalent metal ions as bridging bidentate through oxygen atoms. The microbicide activity for the synthesized complexes against four types of bacteria (*E. coli*, *S. epidermidis*, *K. pneumoniae*, and *S. aureus*) was achieved. Moreover, the stability of the complexes on laser beam was demonstrated and the results prove that the complexes were stable under the laser beams used for 10–30 s.

Selection and peer-review under responsibility of the scientific committee of the International Conference on Advanced Materials Behavior and Characterization.

1. Introduction

The chemical materials including the complex molecules in which metal ions are surrounded by different charged or uncharged molecules are called coordination compounds or usually complex. Coordination chemistry plays a significant role from its commencement up to this day. The complexes have been used a lot in various industrial and medical applications [1–12] and the resulted materials were giving results strong sufficiently to be used on a large scale. Heterocyclic organic compounds, likewise named heterocycle are an important class of organic chemicals portrayed by the way that a number of atoms in their compounds have participate in rings containing one or more atoms (hetero atoms; S, N, O) other than carbon atom [13–17]. The importance of heterocyclic compounds lies in the fact that they constitute many biomedical compounds, such as nucleic acids, vitamins, and many antibiotics [16].

Phthalic anhydride (Fig. 1) which is similarly called as 2-benzofuran-1, 3-dione and isobenzofuran-1, 3-dione, is an oxygen-rich heterocyclic compound with a chemical formula $C_8H_4O_3$. It is prepared from the dehydration reaction of the phthalic acid [18]. The molecular weight of this ligand is 148.12 g/mole and it found as white crystals with a melting point of 131 °C, a boiling point of 285 °C and a density of 1.53 g/cm^3. Furthermore, this ligand is somewhat soluble in water to afford the corresponding acid (phthalic acid) but it dissolves well in organic solvents such as ethanol and diethyl ether [19]. Phthalic anhydride is widely utilized in the chemical industries and one of its most broadly applications is in the preparation of various phthalate esters which extensively used in plasticizers in the plastics industry. Furthermore, phthalic anhydride is used in the preparation of dyes, such as quinizarin pigment as well as in the preparation of phenolphthalein from reacting with phenol [19]. Depending on the chemical composition of this ligand, the expected coordination behavior of phthalic anhydride ligand with the studied metal atoms is bridging bidentate behavior via the exocyclic oxygen atoms to form dimeric or polymeric complexes. Although phthalic anhydride possesses three sites able to coordinate to the metal centre there is rare information about its coordination compounds, therefore, in this article, we tried to provide important information about the coordination chemistry of PL molecule and study the stability of its resulted complexes ($[MCl_2(PL)]_2$; M = Co (II), Ni (II), Cu (II), Mn (II) or Zn (II)) under laser radiation, additionally, we discussed the susceptibility of the prepared complexes as anti-bacteria.

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https://doi.org/10.1016/j.matpr.2020.07.083
2214-7853/
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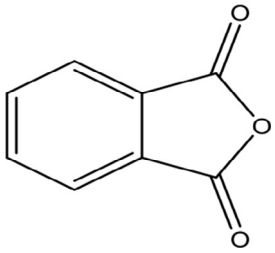

Fig. 1. Chemical structure of phthalic anhydride.

2. Experimental part

2.1. Materials

$CoCl_2{\cdot}6H_2O$, $NiCl_2{\cdot}6H_2O$, $CuCl_2{\cdot}2H_2O$ and ethanol were purchased from Sigma-Aldrich. $ZnCl_2$ and $MnCl_2{\cdot}4H_2O$ were supplied from BDH, while phthalic anhydride was purchased from Alfa Aesar™.

2.2. Instrumentations

Microwave of the type "MAS-II Micro wave synthesis workstation" was used for the synthesis of complexes. Melting points of the complexes were measured by automatic melting point \SMP40. The magnetic measurements of the complexes were performed by using Faraday's method with a type "Brucker BM.6" apparatus. The magnetic correction factor was calculated using Pascal constants for the atoms that forms the prepared complexes. Molar conductivity was measured by a (JENWEAY Molar3 conductivity meter PCM) device using (DMSO) as a solvent with a concentration of (10^{-3}) Molar at a temperature of 25 °C. UV–Vis spectra of the complexes were monitored using Shimadzu UV–Vis of the type UV-1800P spectrophotometry with a 1 cm quartz cells and a range of (200–1100) nm using DMSO as a solvent. IR spectra of the ligand and its related complexes were recorded using FT-IR-800S using KBr in holder and a range of (4000–400) cm^{-1}. Values of quantitative analysis of elements (C.H.N.) using a type 'EuroEA 3000 / Italy device. Nd-YAG laser (wavelengths 1064 nm, energy 0–1000 mJ) was used to study the stability of the prepared complexes

2.3. Method

2.3.1. Synthesis of PL complexes

The PL complexes were synthesized through the addition of PL (1.350 mmol, 0.2 g) in EtOH (25 mL) to a solution of Co, Ni, Cu, Mn or Zn (1.350 mmol) in distilled water (20 mL) under stirring at room temperature. Thereafter the mixture was left to react in a laboratory microwave for further 10 min. The resulted mixture was filtrated, washed with a mixture of ethanol/H_2O and eventually dried for 4 h in vacuum oven.

$[CoCl_2(PL)]_2$, Dark purple solid. Yield: (69%). Anal. calc. for $C_{16}H_8C_{l4}Co_2O_6$: C, 34.57; H, 1.45; N, 0.00, found: C, 34.63; H, 1.48; N, 0.01%. Molar conductance (13 Ω^{-1} cm^{-1} mol^{-1}). FTIR (cm^{-1}): 3080 s, ν(=C–H), 1814vs ν(C = O), 1598 s, 1469 s ν(C = C), 1371 s ν(C-O), 586 m ν(Co-O). Electronic spectrum in DMSO (15400 cm^{-1}) for $^2A_1g \rightarrow {}^2Eg$. μeff (B.M); 2.38, tetrahedral. Melting point: 119–121 °C.

$[NiCl_2(PL)]_2$, Light yellow solid. Yield: (83%). Anal. calc. for $C_{16}H_8C_{l4}Ni_2O_6$: C, 34.60; H, 1.45; N, 0.00, Found: C, 34.71; H, 1.49; N, 0.00%. Molar conductance (21 Ω^{-1} cm^{-1} mol^{-1}). FTIR (KBr, cm^{-1}): 3074 s, ν(=C–H), 1816vs ν(C = O), 1568vs, 1481 s ν(C = C), 1309 s ν(C-O), 574 m ν(Ni-O). Electronic spectrum in DMSO (23699 and 15702 cm^{-1}) for $^1A_1g \rightarrow {}^1B_1g$ and $^1A_1g \rightarrow {}^1A_2g$, respectively. μeff (B.M); 0.01, square planar. Melting point: 99–101 °C. ^{1}H NMR (ppm, 400 MHz, DMSO d_6) δ 7.83 – 7.79 (m, 4H), 7.37 – 7.30 (m, 4H).

$[CuCl_2(PL)]_2$, Light green solid. Yield: (55%). Anal. calc. for $C_{16}H_8C_{l4}Cu_2O_6$: C, 34.01; H, 1.43; 1.45; N, 0.00, Found: C, 34.09; H, 1.53; N, 0.00%. Molar conductance (04 Ω^{-1} cm^{-1} mol^{-1}). FTIR (cm^{-1}): 3066 s, ν(=C–H), 1820vs ν(C = O), 1587, 1479 s ν(C = C), 1341 s ν(C-O), 450 m ν(Cu–O). Electronic spectrum in DMSO (15200 cm^{-1}) for $^2B_1g \rightarrow {}^2A_1g$ and $^2B_1g \rightarrow {}^2Eg$. μeff (B.M); 1.59, square planar. Melting point: 158–160 °C.

$[MnCl_2(PL)_2]_2$, White solid. Yield: (68%). Anal. calc. for $C_{16}H_8C_{l4}Mn_2O_6$: C, 35.07; H, 1.47; 1.45; N, 0.00, Found: C, 35.12; H, 1.55; N, 0.01%. Molar conductance (23 Ω^{-1} cm^{-1} mol^{-1}). FTIR (cm^{-1}): 3089 s, ν(=C–H), 1814vs ν(C = O), 1590vs, 1477 s ν(C = C), 1323 s ν(C-O), 521w ν(Mn-O). Electronic spectrum in DMSO (28001 cm^{-1}) for charge transfer. μeff (B.M); 6.10, tetrahedral. Melting point: 111–113 °C.

$[ZnCl_2(PL)]_2$, White solid. Yield: (78%). Anal. Calc. for $C_{16}H_8C_{l4}Zn_2O_6$: C, 33.79; H, 1.42; N, 0.00, Found: C, 34.00; H, 1.44; N, 0.00%. Molar conductance (02 Ω^{-1} cm^{-1} mol^{-1}). FTIR (cm^{-1}): 3071 m, ν(=C–H), 1807vs ν(C = O), 1594 s, 1492 s ν(C = C), 1336 s ν(C-O), 610 m ν(Zn-O). Electronic spectrum in DMSO (31480 cm^{-1}) for charge transfer. μeff (B.M); 0.01, tetrahedral. Melting point: 174–177 °C. ^{1}H NMR (ppm, 400 MHz, DMSO d_6) δ 7.86 (dd, J^3 = 7.98, J^4 = 4 Hz, 4H), 7.33–7.27 (m, 4H).

2.4. Biological activity study

2.4.1. Collection of bacteria

The used bacterial species were: *K. pneumoniae*, *E. coli*, *S. epidermidis* and *S. aureus*. All bacterial strains have been obtained from Tikrit University, College of Education for Women. The bacteria have been grown up in a nutrient bath at 37 °C, then a sample of 0.5 mL of each bacterial species was carefully put on the surface of a nutrient agar [20].

2.4.2. Antibacterial assay

This assay was done by the diffusion method. In the beginning, the filter paper was cut into 6 mm discs, decontaminated in oven at 140 °C for an hour and then soaked with the germs. All the prepared complexes have been dissolved in dimethylesulfoxide. The control in our experiments was filter paper disc soaked in DMSO. The inoculated plates are incubated at 37 °C for 24 h [21]. The as-prepared complexes showed inhibition zones in centimeter against the growth of the bacterial species. The mean of our experiments was calculated as the antibacterial was measured triplicate.

3. Results and discussion

Treatment of cobalt, nickel, copper, manganese and zinc chlorides with an equivalent molar of PL ligand afforded the dimeric complexes of the type $[MCl_2(PL)]_2$; M = Co (II), Ni (II), Cu (II), Mn (II) or Zn (II).

3.1. Elemental analysis, molar conductivity and magnetic susceptibility

The elemental analysis measurements are in accordance with the proposed formulas for complexes. The molar conductivity values demonstrate that all the complexes have nonionic characters.

The $[CoCl_2(PL)]_2$ complex displayed a magnetic moment equal to 2.38B.M while it was equal to 0.01B.M for the $[NiCl_2(PL)]_2$ complex which are consistent with tetrahedral cobalt(II) and square planar nickel(II) complexes [22,23]. The $[CuCl_2(PL)]_2$ complex showed a magnetic moment equal to 1.59B.M which in agreement with distorted square planar. The magnetic moment value of

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$[MnCl_2(PL)]_2$ were equal to 6.10B.M for the tetrahedral geometry around manganese centre, while it was not measured for the $[ZnCl_2(PL)]_2$ complex, as zinc divalent ion is a diamagnetic.

3.2. Electronic spectra

The electronic spectrum of the $[CoCl_2(PL)]_2$ showed only one absorption as a broad peak at 15400 cm^{-1} which belongs to $2A_1g \rightarrow 2Eg$ for a distorted tetrahedral cobalt ion [24]. In the case of $[NiCl_2(PL)]_2$, the spectrum shows two main peaks at 23,699 and 15702 cm^{-1} which are assigned to $^1A_1g \rightarrow {}^1B_1g$ and $^1A_1g \rightarrow {}^1A_2g$, respectively of square planar nickel complexes [25]. The electronic spectrum of the $[CuCl_2(PL)]_2$ showed only one absorption as a broad peak at 15200 cm^{-1} which fits with $^2B_1g \rightarrow {}^2A_1g$ and $^2B_1g \rightarrow {}^2Eg$ for a distorted tetrahedral cobalt ion [26]. The electronic spectrum of $[MnCl_2(PL)]_2$ complex displays low-intense peak at 28001 cm^{-1}, attributes to charge transfer. The spectrum of $[ZnCl_2(PL)]_2$ gives a broad peak at 31840 cm^{-1}, represents the charge transfer [25].

3.3. Infrared spectra and nuclear magnetic resonance

The FTIR spectrum of PL (Fig. 2) displayed a very strong-intense band at 1833 cm^{-1} belongs to ν(C = O). Furthermore, the spectrum showed a band at 1331 cm^{-1} that attributes to the ν(C-O). For the complexes, the infrared spectra showed a stretching band of ν (M−O) at the range of 450–610 cm^{-1}. Moreover, it was noticed that the frequency of ν(C = O) was shifted towards a lower frequency 1807–1820 cm^{-1} compared to the free ligand; proves the binding of oxygen atom of C = O group to the metal centre [26,27]. The shifting of ν(C-O) towards a higher frequency than in the free PL ligand is a good evidence proves that the oxygen atom of C-O group does not participate in the coordination to the metal atoms. FTIR spectrum of $[CoCl_2(PL)]_2$ is shown in Fig. 3.

^1H NMR spectrum of $[NiCl_2(PL)]_2$ shows two multiplate signals at the ranges 7.83–7.79 ppm and 7.37–7.30 ppm which ascribed to the aromatic protons. Notwithstanding all the estimations mentioned before, the adequacy of the nickel complex in the field of NMR is definitive evidence for the presence of the square planar geometry. ^1H NMR spectrum of $[ZnCl_2(PL)]_2$ shows two signals at the aromatic domain. The first signal was doublet of doublet at 7.86 ppm that assigned to the *para*-situated aromatic protons, while the second signal was found as multiplate at the range 7.337.27 ppm that assigned to the other two aromatic protons.

3.4. Influence of lasers on PL ligand and its complexes

The laser rays in the region 600–700 nm was applied by using a laser device with a capacity of 5 milli-watt. This device has been used to assess the stability of the complexes being prepared. The ligand and complexes were radiated by laser for a period of times 10, 20 and 30 s. The stability of the complexes was confirmed by measuring their melting point and molar conductivity. It was observed that the ligand and its complexes were not affected as PL and its complexes does not show any different in their melting point and molar conductance therefore the complexes show no decompose or polymerize. Therefore, this means that the PL and its complexes have not pretentious by the laser radiation. Furthermore, it was observed that the colors of the complexes did not change within the mentioned range of time, therefore, we could conclude that the prepared complexes are stable, as shown in the Table 1.

3.5. Study of the biocidal efficacy

The biological activity of $[CoCl_2(PL)]_2$, $[NiCl_2(PL)]_2$, $[CuCl_2(PL)]_2$, $[MnCl_2(PL)]_2$, and $[ZnCl_2(PL)]_2$ complexes was assessed against four bacteria species (Gram-positive and Gram negative). The results of antibacterial as inhibition zone (IZ) were illustrated in Figs. 4-7 which indicate that all the prepared complexes show the ability of inhibiting the growth of the studied bacteria. However, the results showed that the inhibition ability increases directly with increasing concentration of complexes. It is noticeable that the $[CoCl_2(PL)]_2$, $[MnCl_2(PL)]_2$ and $[ZnCl_2(PL)]_2$ complexes are the best

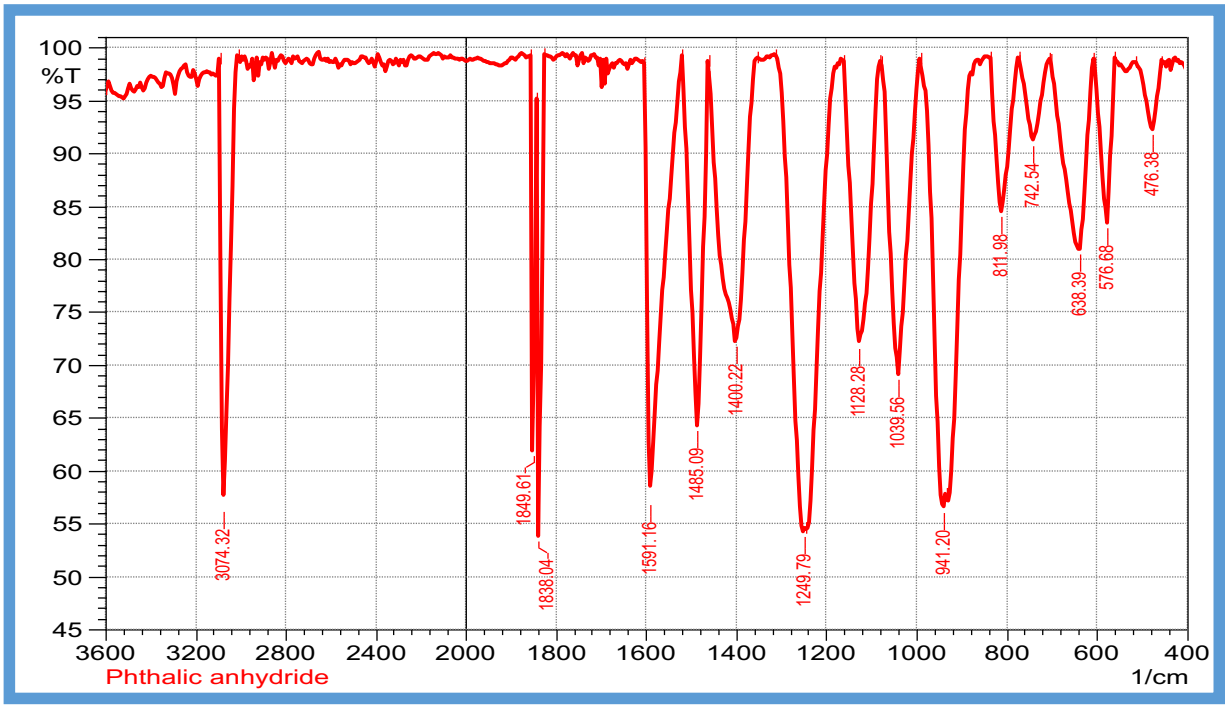

Fig. 2. FTIR of phthalic anhydride.

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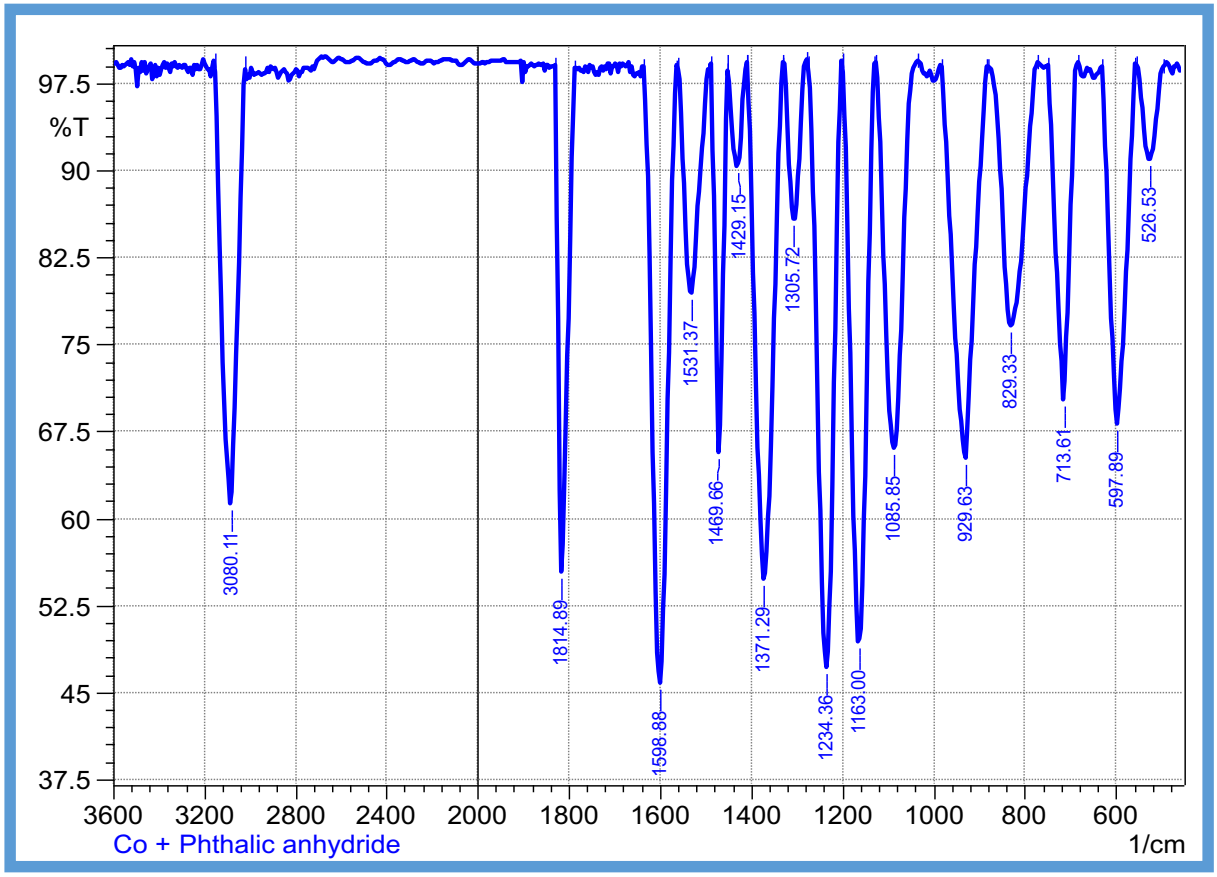

Fig. 3. FTIR of $[CoCl_2(PL)]_2$.

Table 1
The stability results of the complexes under different time of laser irradiations.

Compounds	10 S		20 S		30 S	
	M.P. °C	Molar conduct. (10^{-3}M)	M.P. °C	Molar conduct. (10^{-3}M)	M.P. °C	Molar conduct. (10^{-3}M)
PL	131	–	131	–	131	–
$[CoCl_2(PL)_2]_2$	119–121	13	119–121	13	120–122	13
$[NiCl_2(PL)_2]_2$	99–101	21	99–101	21	99–101	21
$[CuCl_2(PL)_2]_2$	158–160	04	158–160	4.3	158–160	4.3
$[MnCl_2(PL)_2]_2$	111–112	23	111–112	23	111–113	23
$[ZnCl_2(PL)_2]_2$	174–176	02	175–177	02	175–177	02

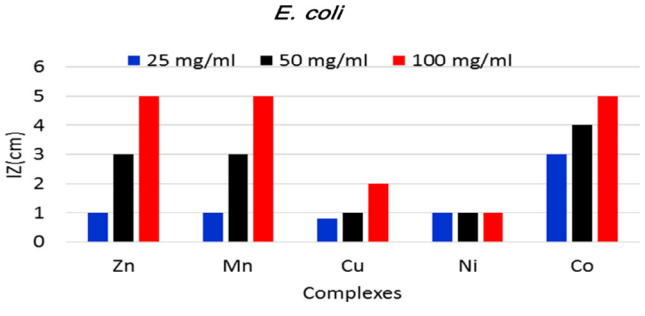

Fig. 4. IZ of the prepared complexes against *E. coli*.

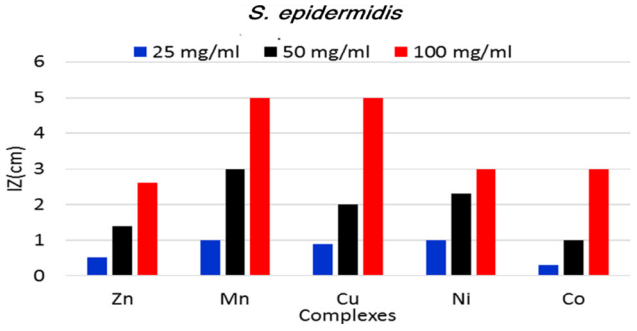

Fig. 5. IZ of the prepared complexes against *S. epidermidis*.

in inhibition of *E. coli* bacteria (Fig. 4), while the $[CuCl_2(PL)]_2$ and $[MnCl_2(PL)]_2$ complexes are the best for inhibiting the *S. epidermidis* (Fig. 5). Finally, the $[NiCl_2(PL)]_2$ complex is the best in inhibiting the bacteria of *K. pneumonia* and *S. aureus* (Figs. 6 and 7) and it must be mentioned here that the highest value of inhibition

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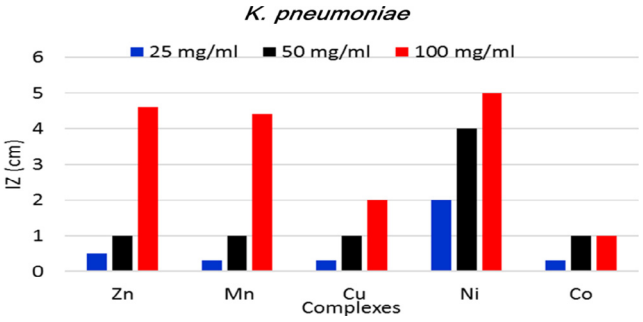

Fig. 6. IZ of the prepared complexes against *K. pneumoniae.*

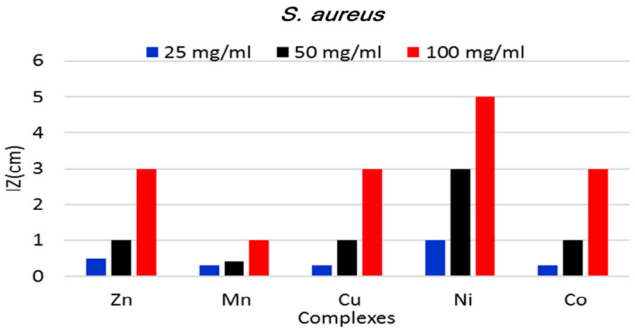

Fig. 7. IZ of the prepared complexes against *S. aureus.*

reached 5 cm and in sum, these complexes can be considered broad-spectrum antibacterial [28–30]. The reason for the inhibition efficacy of the prepared complexes against the studied pathogens can be attributed to the fact that during the decomposition of the complexes, the central metal cations will be released and coordinated to the cell membrane that negatively charged, causing denaturation of proteins and thus killing the bacteria [31–33].

4. Conclusions

This work gave much information about the coordination chemistry of phthalic anhydride ligand which had not been studied before. The results demonstrated that the ligand exhibits bidentate bridging through oxygen atoms of C = O groups to form the homobinuclear complexes. The results of the elemental analysis matched perfectly with the proposed complexes structures. The stability of the resulting complexes was tested under laser irradiations, and the results were good, as no decomposition was shown. Moreover, the complexes were tested for susceptibility to bacterial inhibition. We also concluded that the ability of complexes to inhibit bacteria was as follows: $[CoCl_2(PL)]_2$, $[MnCl_2(PL)_2]_2$, $[ZnCl_2(PL)]_2$ < $[CuCl_2(PL)]_2$ < $[NiCl_2(PL)]_2$ against *E. coli*; $[MnCl_2(PL)]_2$, $[CuCl_2(PL)]_2$ < $[NiCl_2(PL)]_2$, $[CoCl_2(PL)]_2$ < $[ZnCl_2(PL)]_2$ against *S. epidermidis*; $[NiCl_2(PL)]_2$ < $[ZnCl_2(PL)]_2$, $[MnCl_2(PL)]_2$ < $[CuCl_2(PL)]_2$ < $[CoCl_2(PL)]_2$ against *K. pneumoniae*; $[NiCl_2(PL)]_2$, $[ZnCl_2(PL)]_2$, $[CuCl_2(PL)]_2$, $[CoCl_2(PL)]_2$ < $[MnCl_2(PL)]_2$. Thusly, it can be said that all complexes are broad-spectrum antibacterial agents.

CRediT authorship contribution statement

Ban D. Salih: Conceptualization, Methodology, Investigation. **Adil H. Dalaf:** Data curation, Methodology, Software. **Mustafa A. Alheety:** Supervision, Investigation, Writing - review & editing. **Wesam M. Rashed:** Conceptualization, Investigation. **Ibtihal Q. Abdullah:** Conceptualization, Validation.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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