



Mn^{II} and Pb^{II} complexes of 1,10- phenanthroline and acetylacetonate as mixed ligands and study of their antimicrobial activity

Abdullahi Mustapha*, Shehu Habibu*, Usman Sani[§] Aminu Ahmed[#] and Mohammed Saleh Salga[§]

*Department of Chemistry, Federal University Dutse PMB 7156, Jigawa State Nigeria.

§Department of Chemistry, Umaru Musa Yar'adua University Kastina

#Department of Chemistry, Kano University of Science and Technology, Wudil

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Correspondence author:

e-mail:

abmustapha2004@yahoo.com

ABSTRACT

Complexes of Mn (II), and Pb(II) with 1,10- phenanthroline and acetylacetonate as ligands were prepared and investigated using FT-IR and UV-Visible spectroscopy and other techniques. The Infra-red data revealed the absorption vibrations corresponding to coordination sites of the ligand to the metal ions. The (M-O) and (M-N) bands were found at 850cm⁻¹ and 900cm⁻¹ then 900cm⁻¹ and 1460cm⁻¹ for manganese and lead complexes respectively. The metal complexes were screened for their invitro antibacterial activity against two bacterial species namely: *Escherichia coli*, and *Aspergillus*. The prepared manganese (II) complex was found to have higher antibacterial activity than the corresponding Lead (II) compound.



Introduction

The treatment of infectious diseases still remains a challenging problem due to combination of factors which includes emerging infectious diseases and the increasing number of multi-drug resistant microbial pathogens. In spite of the large number of antibiotics and chemotherapeutics available for medical use, at the same time the emergence of old and new antibiotic resistance created in the last decades revealed a substantial medical need for new classes of

antimicrobial agents (Gupta *et al.*, 2012). There is a real need for the discovery of new compounds endowed with antimicrobial activity, possibly through mechanism of action, which is distinct from those well-known classes of antimicrobial agents to which many clinically relevant pathogens are now resistant. Due to the outbreak of infectious diseases caused by different pathogenic bacteria and the development of antibiotic resistance, researchers are searching for new antibacterial agents.

Therefore, new antimicrobial agents have to be synthesized for the treatment of resistant bacterial diseases (Eleni *et al.*, 2010). The design of metal compounds as antimicrobial agents is currently of interest due to the growing reports of drug resistance reported for known antimicrobial agents. Metal complexes show a wide spectrum of antimicrobial properties (Roth *et al.*, 2007). Antimicrobial activity is a property of both inorganic and organic substances, and the exploitation of such activity is a matter of considerable practical importance in the development of antiseptics, sanitizers, germicides, bactericides, sporocides, virucides and disinfectants (Maurer and Shringapurey, 1977). Schiff bases have played a very important role in the development of coordination chemistry as they readily form stable complexes with most of the transition metals (Taylor *et al.*, 2004). Metal complexes are specifically of interest in bioinorganic chemistry because many provide biological models in understanding the structure of biomolecules and biological processes (Raman and Raja, 2009).

Experimental

Materials

All reagents and solvents used in this work were of analytical grade obtained from BDH and Sigma Aldrich, and used without further purification. The electronic spectra were measured on T60 spectrophotometer. The sample prepared

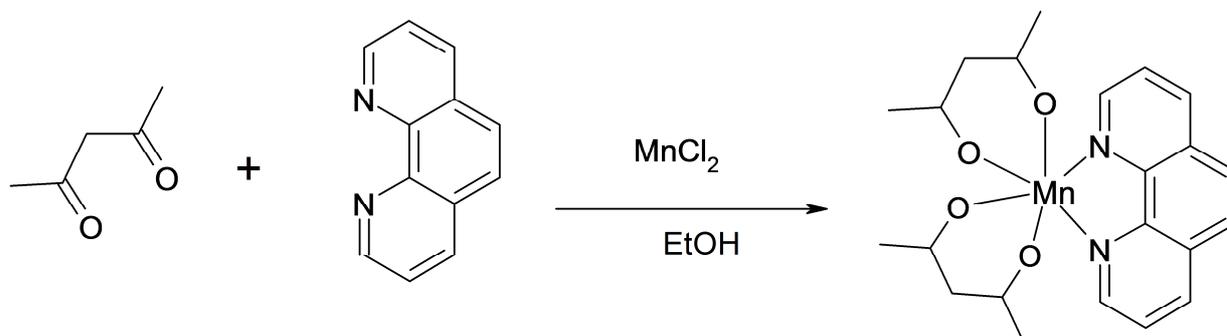
were characterized using KBr disc sampling method. The discs were prepared by grinding the samples (2% by weight) with KBr which was then compressed into a disc and were analyzed with FTIR spectrometer (cary630). The microorganisms used in this study were obtained from the Biology Laboratory of Umaru Musa Yar'adua University (UMYUK) Katsina State-Nigeria and they include: *E. coli*, and *Aspergillus*.

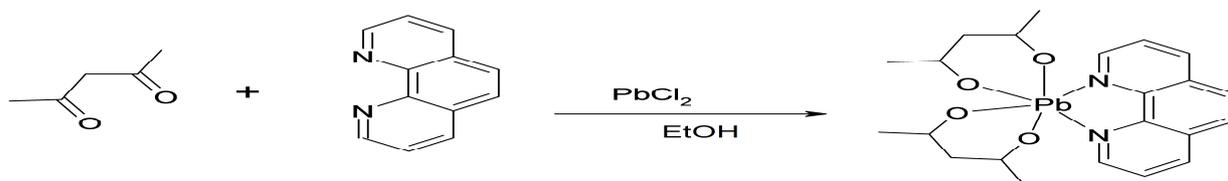
Synthesis of Manganese (II) Complex

To a 50ml absolute ethanol in 250ml round bottom flask was added 1g of acetyl acetone, and stirred. This was followed by the addition of 1.97g of 1-10 phenanthroline, the solution was at room temperature, and 1.5g of manganese (II) chloride salt ($MnCl_2$) was added to the mixture, which immediately gave pale pink color. The coloured mixture was refluxed for 2 hours, and allowed to cool, the pale pink powder was collected by gravity filtration.

Synthesis of Lead (II) Complex

To a 50ml absolute ethanol in 250ml round bottom flask was added 1g of acetyl acetone, and then stirred. This was followed by the addition of 1.97g of 1-10 phenanthroline, the solution was again stirred at room temperature, 2.8g lead(II) salt was added, the mixture immediately turns cloudy. The mixture was refluxed for 2 hours and allowed to cool, the white powder was collected by gravity filtration. The proposed synthetic scheme of the complexes are as follows:





Determination of Solubility

The solubility of each sample was tested in a variety of solvents which include water, ethanol, chloroform, benzene, nitrobenzene, ammonia, ethylacetate, N-hexane and DMSO. 5cm³ of each solvent was added to about 0.1g of the sample in a test tube and shaken vigorously. The solubility was carefully observed and recorded.

Antibacterial studies

16.8g of Nutrient Agar (N.A) was weighed and 500ml distilled water was also measured. The measured distilled water was poured into a 1000ml borosilicate glass beaker and the measured Nutrient Agar was dissolved in it. The mixture was heated to boil over a heating plate with continuous stirring. The heat was turned off as soon as it boiled while the hot agar was then poured into a clean conical flask. The mouth of the conical flask containing the media were plugged with cotton wool and placed in an autoclave. The media was sterilized at 121°C for 15minutes. After sterilization, the conical flask was removed and allowed to cool to 50°C. The medium was then poured in to sterilized petridishes and allowed to solidify before inoculating. The same procedure was carried out using POTATO DEXTROSE

AGAR (PDA) to test the anti fungal isolate (Anacona and Alvarez, 2002).

Results and Discussion

The complexes synthesized are crystalline solids, and have relatively high melting temperature and decomposition temperature. The Manganese complex formed yellow crystalline compound with decomposition within 233° C - 237° C. This high decomposition temperature revealed stability of the complexes. The color of manganese complex is due to the MLCT, the Lead complex gave a White crystalline solid with melting point within 228° - 233° Celsius. The percentage yield for Mn (II) complex was 55.5% while that of Pb(II) complex was 45%. The solubility test carried out on the transition metals complexes synthesized showed that complex of manganese are soluble in ethanol, nitrobenzene, dimethylsulfoxide (DMSO) and water while Lead complex was found to be soluble in chloroform, benzene, nitrobenzene, DMSO and water. Thus their solubility in water shows that they have tendency of forming hydrogen bonding with its two hydrogen atoms.

FT-IR Spectroscopy

The FT-IR spectroscopic information for the synthesized metal complexes is shown in the Table 1:

Table 1: FT-IR spectroscopic information for the synthesized metal complexes

Complexe	V(C-O)	(M-O)	(C-O)	(C-H)	(N-M)
[Mn(acac)(phen)]	1580 cm ⁻¹	850cm ⁻¹	1200 cm ⁻¹	1480 cm ⁻¹	1460 cm ⁻¹
[Pb(acac)(phen)]	1590 cm ⁻¹	900 cm ⁻¹	1100 cm ⁻¹	1450 cm ⁻¹	900.33 cm ⁻¹

From the results obtained, v(C-O) for the two complexes appear at; 1580 cm⁻¹ and 1590 cm⁻¹ for manganese and lead

complexes respectively, while (C-O) bands was at 1100cm⁻¹ - and 1200cm⁻¹ as reported by Ejiah *et al.*, 2012.

However, in the spectra of the two complexes it is observed that the (C-H) bands appears at 1450cm^{-1} and 1480cm^{-1} for the two complexes (manganese and lead respectively) which is in agreement with similar observation made by Brian, 1999. The (M-O) and (M-N) bands was found at 850cm^{-1} and 900cm^{-1} then 900cm^{-1} and 1460cm^{-1} for manganese and lead

respectively, which also conformed with a similar results reported by Ejiah *et al.*, 2012. The UV-Visible result for the complexes shows that manganese complex have peak around 350nm and a shoulder around 450nm; while Lead (II) complex shows peak around 320nm and a shoulder around 430nm and this is in agreement with similar result reported by (Bell *et al.*, 2002).

Antimicrobial Activity



10^1 concentration



10^2 concentration

Bacterial plates showing zone of inhibition



concentration 10^2



10^1 concentration

Fungal plates showing the zone of inhibition:



Fungal growth showing on pouring method at different concentration

The result of the Anti- Bacterial/fungal activity of the complexes is shown in the Table 2.

Table 2: Anti- Bacterial/fungal activity of the complexes

COMPLEXES	ISOLATES	Concentration ($\mu\text{g/ml}$)		
		10^1	10^2	10^3
Sample A	<i>E-coli</i>	0.60	0.30	0.25
	<i>Aspergillus</i>	0.95	0.52	0.45
Sample B	<i>E-coli</i>	0.40	0.58	0.53
	<i>Aspergillus</i>	0.50	0.62	0.75
Sample C	<i>E-coli</i>	0.45	0.41	0.38
	<i>Aspergillus</i>	0.60	0.72	0.65

Zone of inhibition (cm)

KEY: A= Manganese complex
 B= Lead complex
 C= Penicillin (control)

Both the two complexes were successfully screened for their anti-microbial activity, and they show a very interesting behavior. On comparing the two complexes and the penicillin (control) the manganese compound was found to be more active against *E. coli* than the corresponding Lead complex and the penicillin. This may be attributed to the size of inhibition. Similarly on comparing the complex of lead and that of penicillin, it was clearly observed that, the penicillin is more active than lead compound.

Conclusion

This paper report successfully the synthesis of transition metal complexes of manganese and lead using 2, 4-pentanedione and 1-10 phenanthroline. The two complexes crystallized as solid with high decomposition and melting temperature which is an indication of the complexes stability, and finally the complexes were tested for their anti-microbial activity. It was found that the prepared manganese (II) complex possess higher antibacterial activities than the corresponding lead (II) complex.

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