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Preparation, physiochemical characterization and biological activity study of new ligands (Azo - Imine)

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Abstract
In this work, Novel ligands and their complexes with Lead ion have been synthesized by the coupling reaction of azo or di azo compounds with imine compounds, the resulting compounds named (azo-imine), which included (azo group linked with imine group at same carbon atom of Schiff base. The structures of tow ligands and complexes with Pb (II) were confirmed by several methods like [FTIR-spectra, H.NMR-spectra, (C.H.N) -analysis (UV-Vis) -spectra, Atomic absorption, molar conductance melting points.

Keywords: cycle ligand, macro ligand, lead ion.

1. Introduction
Imine compounds (Schiff bases) consider starting material to synthesis several organic compounds like hetero cycles macro cycles, reagents in analytical chemistry, it also has biological importance in the metabolism and biosynthesis of amino acids, as a ligands in inorganic chemistry, their complexes have biological properties, like antifungal, antitumor, antibacterial., in other fie1ds [1-6] Azo compounds and their complexes also have a wide range of applications[7, 8] that stretch from their use in analytical and inorganic chemistry with transition metals. In the present work, we have synthesized tow (azo-imine) [8-10] ligands and their complexes with. Lead ion (II) the presence of azo-group (-N=N-) linked with imine (-CH=N) group at same carbon atom of Schiff base makes them poly dentate ligands which gave them important properties in the complexion. As azomethine gaining great importance in all areas and has served a lot of studies to contain one or more of the groups secretary in their structures, [15-19] which attributed her these compounds in the preparation of many compounds of pharmaceutical (8) and use some of them in the industrial field for purification of rubber and as anti-oxidation and corrosion also used some of them as insecticides (13) and some are used as agents of complexity in other studies [20-23] and is characterized by high efficiency and stability of the circles and the basal acid.

2. Experimental: All measurements were carried out by: melting points in electro thermal 9300, LTD, U.K., FTIR in four ever transform infrared Shimadzu 8300, KBr-disc" H, NMR spectra in DMSO-solvent and (C.H.N) -analysis with Atomic absorption in Malaysia, molar conductance in DMSO -solvent, (UV-Vis) -spectrophotometer,

2.1 Synthesis of ligand (BBAP): 2-(S-benzothiadiazole azo)-2- (amine benzene)-phenyl imine. A mixture of benzoic acid (0.0lmole) and thiosemicarbazide (0,01 mole) were reacted in refluxing for (8hrs), the resulting precipitate was amino compound, which dissolved in (2ml) of hydrochloric acid with (0.5gm) solution of sodium nitrite at (0-5)C, ethanolic solution of 2amine benzene phenyl imine added to mixture to give 89% of ligand (BBAP).

2.2 Synthesis of ligand (DBAP):
In a beaker escalation installed condenser reflector and installed on the heater magnetic added 0.2 mole of aldehyde or ketone dissolved in 100 ml of ethyl alcohol absolute to 0.1 ml of the secretary of 3.3 - dual methoxy benzene dissolved in 100 ml of ethyl alcohol absolute then added to the mix output three drops of acetic acid snow escalation process was conducted with constant stirring by a magnetic mixer for two hours after the end of the reaction mixture to

cool to leave a deposit shall be nominated and wash cold ethyl alcohol and then Recrystallization by absolute ethyl several times and took all degrees of melting compound[28] .The use of a measuring function of the type of acidic pH-meter model 820 pH-meter (Kinck-England) and HCl, NaOH for calibration.

2.2 Preparation of Solutions:
Prepare solutions of compounds (1, 2): The preparation of the solution concentration of 10^{3-}as stock solution, from melt 3.1 & 5.43 mg each, respectively, in 10 ml of d. water to: -

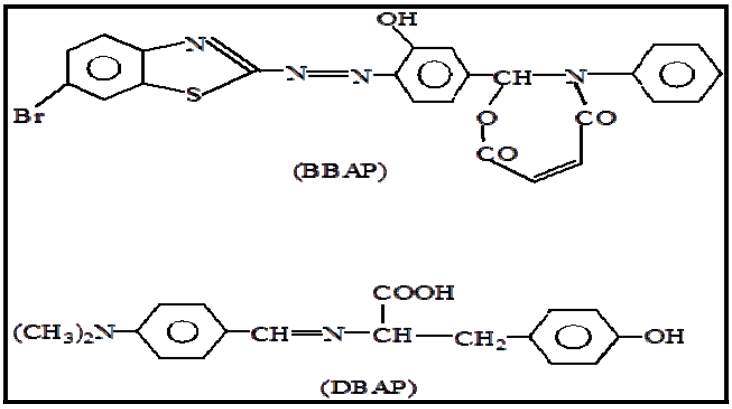
a. Prepare of Calibration Curve for the solution of the compound and in a way the first dilution was obtained on a series of concentrations ranged between (10^{3-}, $8x10^{-3}$, 10^{4-}, $1x10^{4-}$, $2x10^{4-}$, $4 x10^{4-}$, $6x10^{4-}$, $8 x 10^{4-}$) Molar.

b. Choose the optimal focus: were prepared from solutions of different concentration (10^{4-}-10^{5-}) Molar to choose the optimal focus to be affected with Pbcl2 after installing the acidic function at, pH =8 each respectively.

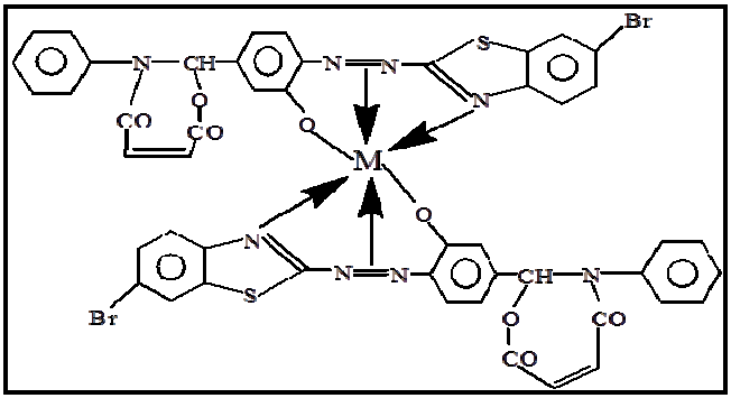
2.3 Preparation of calibration solutions for composite $PbCl_2$: was prepared solution stockpiling standard compound concentration of 10^{3-} Molar irrigation of melt 0.5564 mg in 20 ml distilled water in a bottle volumetric 10 mL and fuller volume to the mark and which has been obtained on a series of concentrations ranging from (10^{4-} - 10^{5-}) Molar way of mitigation and followed the same steps to prepare solution of compounds (1 and 2)at pH =10, 8.

2.4 Synthesis of Complexes with Pb (II):
These complexes were prepared according to procedure (ll), the hot solution of ligand [(1, 2] respectively were added to solution of Lead salt ($PbCl_2$) in mole ratio (metal: ligand) complex for ligand (2) was (metal: ligand) (2:1) "after stirring (lhrs), precipitates formed, dried and re-crystallized to yield (82, 85, 80, 82 and 80) % respectively from complexes of [2].

Scheme (1): preparation of ligands



Scheme (2): Complexes



3. Results and Discussion:
The synthesized ligands and their complexes with $PbCl_2$ have been studied by several methods and techniques:

3.1 FT.IR spectra shown absorption bands in Iigand
The infrared spectrum of the compounds prepared back to the group said the results of the infrared spectrum the most important package absorption frequency 1610 cm^{-1} azomethine (CH= N) and the emergence of a package absorption frequency of 3481 cm^{-1} and 2600 cm^{-1} belonging to the two groups (-OH, carboxyl) package absorption of other frequency 1726 cm^{-1} pack absorption frequency of 1610 cm^{-1} CO-O)) (lactone, carboxyl) belonging to the compound (1) and pack the absorption frequency of 1417 and 1500 cm^{-1} back to azo grub (-N = N-) and other packages belonging to the compound (2) and other packages that reveled in table (2) and forms (1 and 2).

3.2 Results of quantitative analysis

The exact elements carbon, hydrogen, Nitrogen reveal values calculated theoretically noticely near to existing practically as well as when compared to the results of measuring the degree of fusion of the compounds prepared in this research with previous studies, [28] in this area given the substantial convergence in values and also showed degrees of fusion high for these vehicles compared to the previous studies [15, 16] there stability on the direction of the high temperatures and this is further evidence of the compounds prepared as shown in table (1).

Table 1: Physical Properties & Elemental Analysis of Compounds [1, 2].

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Compounds	M.P(°C)	λ_{max}	Product (%)	Calc. / Found		
				C%	H%	N%
(DBAP) $C_{18}H_{20}N_2O_3$	222	310	85 %	69. 230 69. 081	6. 410 6. 278	8. 974 8. 742
(BBAP) $C_{24}H_{15}N_4O_4SBr$	235	496	82 %	53. 841 53. 608	5. 804 5. 657	10. 469 10. 294

Table 2: FT. IR data (cm^{-1}) of Compounds [1, 2].

Compounds	**(CH=N)** imine group	**(-OH)** Phenol , carboxyl	**(-N=N-)** azo group	**(-NH)**	**(-CO-N-)** amide	**(CO-O)** lactone ,carboxyl
(DBAP) $C_{18}H_{20}N_2O_3$	1610	3481 , 2600br	----	----	----	----- 1726
(BBAP) $C_{24}H_{15}N_4O_4SBr$	---	3431 , ----	1417 ,1500	----	1684	1708 -----

Table 3: H.NMR data (ɓ ppm) of Compounds [1, 2].

Compounds	**(CH=N)** imine group	**(-OH)** Phenol , carboxyl	**(CH=CH)**	**(-NH)**	**(O-CH-N-)** oxazine	**(-CH-)** Aliphatic, aromatic
(DBAP) $C_{18}H_{20}N_2O_3$	8.83	11.21 , 12.4	----	----	----	0.97-1.3 , 6.95-7.65
(BBAP) $C_{24}H_{15}N_4O_4SBr$	---	11.14 , ----	5.63	----	9.20	----- 7.1-7.87

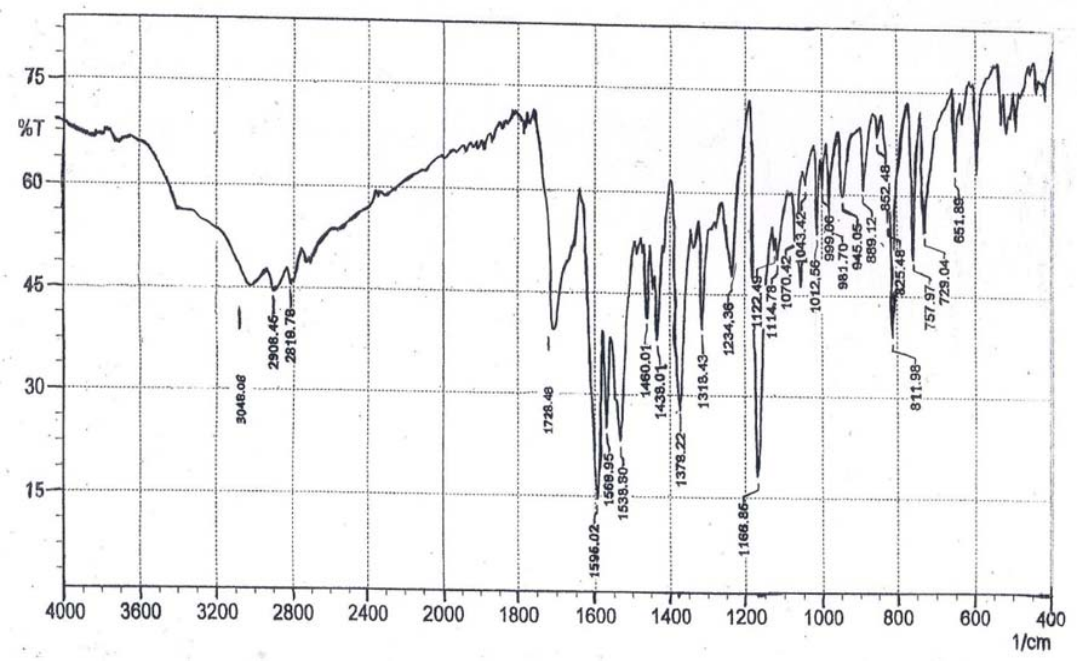

Fig 1: FT.IR spectrum of compound (DBAP)

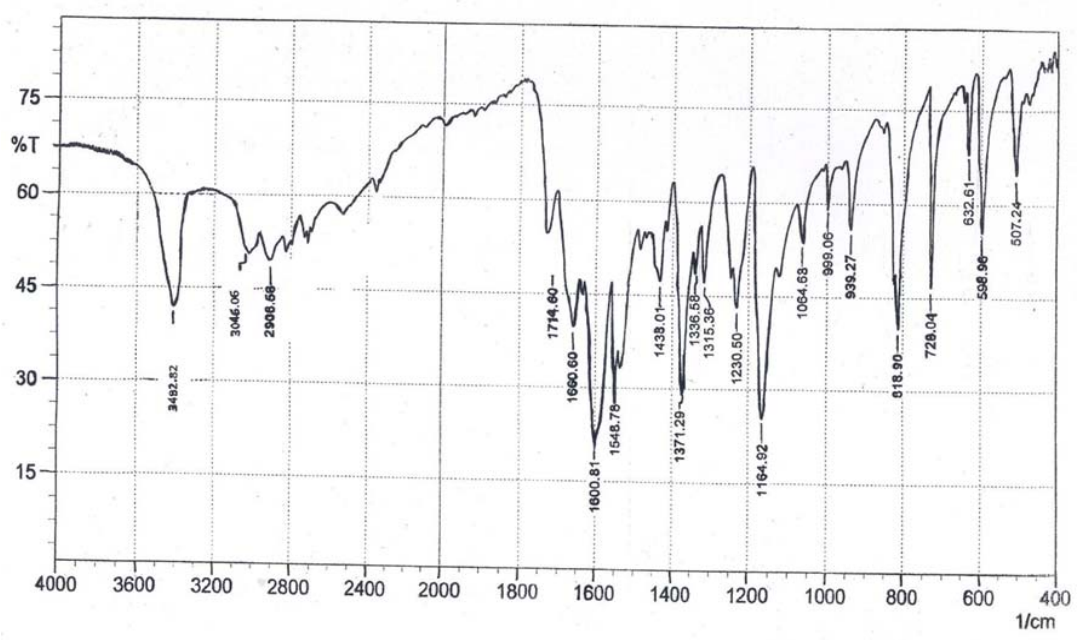

Fig 2: FT.IR spectrum of compound (BBAP)

3.3 Antimicrobial activity assay:

Previous studies have shown [12, 30] Hits biological of such vehicles and the effect of inhibiting the growth of bacterial and fungal for multiple types of bacteria and microorganisms Antibacterial activity was studies using agar diffusion method of all the synthesized compounds (1,2) was dissolved in (ethanol) for the assay, for screening, steriIe, 6mm diameter filter paper disc were impregnated with(0.l) ml of each compound, then the paper discs were placed on to nutrient agar seeded with test organisms (*Pseudomonas .aeruginosa* and *Staphylococcus areas*).the antibacterial activity was evaluated by measuring the inhibition zone diameter(mm).

Table 5: Antibacterial activity of the compounds [1, 2] {diameter of zone (mm)}

Compounds[1, 2] *	diameter of zone (mm)	
	G+: Staphylococcus. aureus	***G-: Pseudomonas. aeruginosa***
(DBAP)	16	10
(BBAP)	20	20
Amoxicillin **	30	26
*Minimum Inhibitory concentration (MIC) of compounds (5mg/ml). **Amoxicillin (0.1mg/ml).		

3.4 Study of spectrum characteristic for compound (1, 2):-

Some spectrum characteristic for compound(2) was Studied in deferent polarity solvents like **Di Ethyl Ethe**r, Ethanol 75%+H_2O 25%, Ethanol, DMSO, Benzene, Dioxan, Chloroform, Methanol, that's result fixed in table (4). that which λmax limited .In figure (3) revealed relationship between Abs. &wave length with Con.0.2*10^{5-}Molar .That indicate to compound(2)have great and broad absorption peak in organic medium (non polar) Di Ethyl Ether at (328-271 nm) with ζ=375000 $L.Mol^{1-}.cm^{1-}$ .This peak du to most grub of transition (**π -π***) and another absorption maxima at (450-496) nm du to **(n - π***) .Whereas (273-331nm) with ζ=395000 in polar medium (**Ethanol)**, but 230-331nm to du to **(π-π***) **in** mixture of Ethanol 75 % + H_2O 25 %. Final that indicate to shift first peak to short waves length direct (Blue – shift) ,and to longer waves length for second peak (Red – shift) were increase polarity of solvents this result interaction lead to change of geometric molecular shape as well as reaction condition and their significantly different physical and electronic properties as show in previous studies [20, 21]

The U.V spectra of new ligand compound (1) shown absorption maxima at (232-351)nm in (non polar SSS) **Di Ethyl Ethe**r with ζ=166500 du to **(π π***) that similar to action of mixture of Ethanol 75 % + H_2O 25 %, and (231-239) nm in(**Ethanol)** polar medium du to**(π π***) whereas another peak appear in (210-317) nm du to **(n π***) Fig.(4).

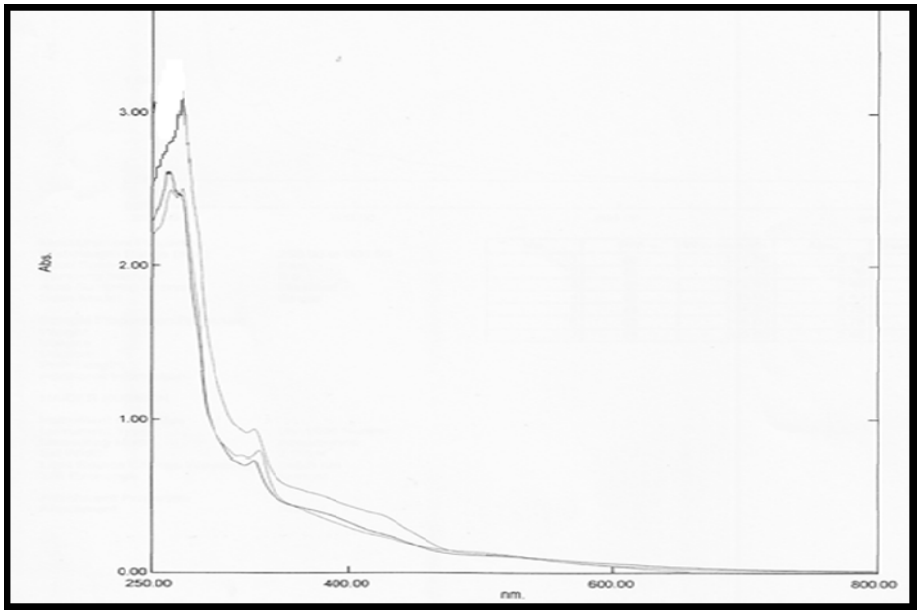

Fig 3: Relationship between Abs.&Wave length for compound (2)

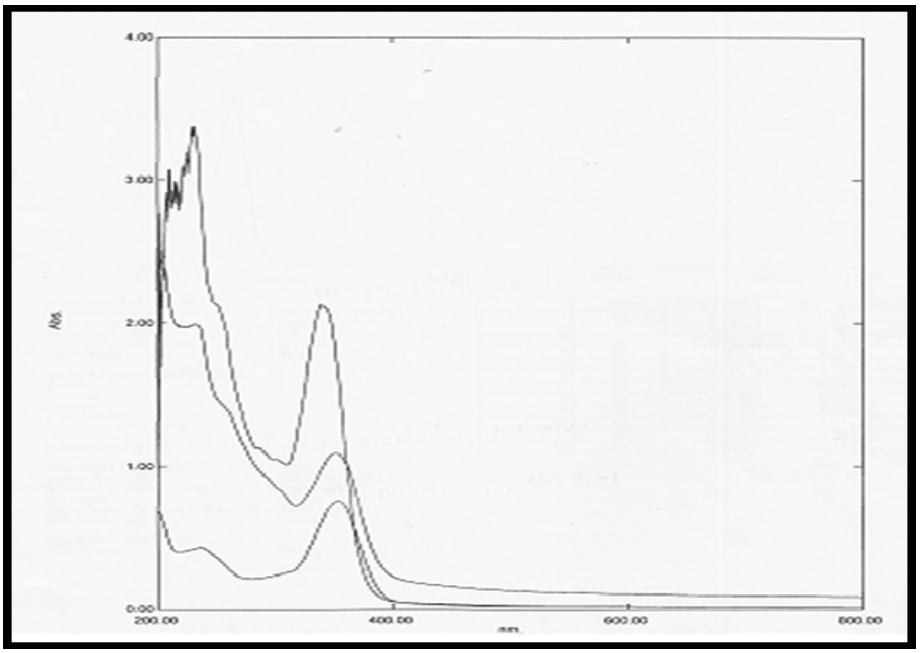

Fig 4: relationship between Abs.&Wave length for compound (1)

Table 4: Solubility of Compounds [1, 2] in Solvents

Solvents	(BBAP)	(DBAP)
Ethanol	+	+
Methanol	+	+
DMSO	+	+
Benzene	_	_
Dioxan	_	_
Chloroform	_	_
Ethanol75%+H_2O25%	+	+
Di Ethyl Ether	+	+

3.5 Study of optimal condition of complexes

This work involved the optimal condition for formation of complexes with pb^{+2} conc.$5X10^{4-}$ at pH=10 for compound 2. mole ratio (M:L) was determination from relationship between the absorption of observed light and mole ratio through series of solutions were prepared having a constant concentration ($IXI0^{-3}$ M) of lead salt ($PbCI_2$), found to be (2:1) as shown in figure(5). But there isn't no interaction between compound no. (1) and solute of lead ion because the operation of composition complexes were very speedily (composed & decomposed), Because of weakness of pentane ring. That was a stable ring.

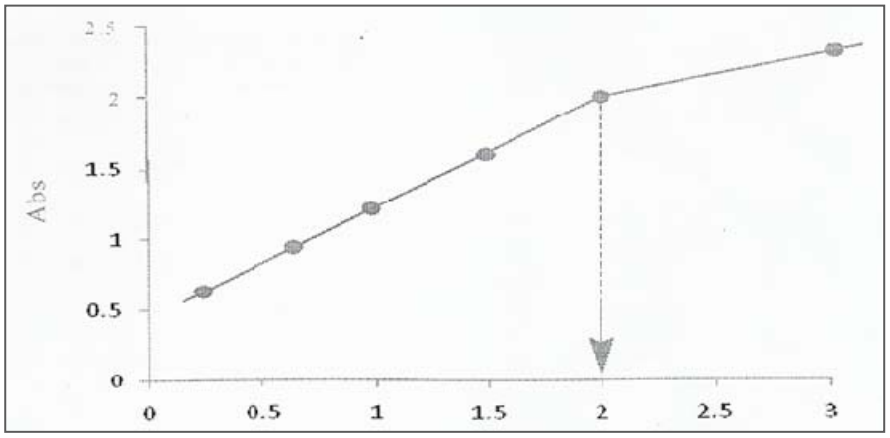

Fig. 5: Mole ratio of Complex

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