Our Heritage(UGC Care Listed)
ISSN: 0474-9030 Vol-68, Special Issue-12

National Conference on Recent Trends in Physics, Chemistry and Mathematics(RTPCM-2020)
Held on 4th February 2020Organised by: Department of Physics, Chemistr and Mathematics, Sunderrao Solanke Mahavidhyalaya, Majalgaon, MS

Synthesis And Characterization Of 2-(2-Hydroxybenzylideneamino) Acid Derivatives and It's Ni(Ii), Cu(II) Complexes As Microbial Growth Inhibitors

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Abstract:

2-(2-hydroxybenzylideneamino) acid derivative i.e. ligands L_1-L_2 have been obtained by the reaction of amino acids A_1-A_2 with salicylaldehyde. The current article focused on the syntheses, characterization and antimicrobial studies of the Complexes (P_1-P_4),these complexes were synthesized and studied by their spectral data such as FTIR, Mass, UV-Vis., melting point and elemental analysis.From spectral analysis, the probable structure of the complexes was suggested[1][2].

Antibacterial activities of ligands L_1-L_2 and its metal complexes (P_1-P_4) have been tested by screening the compounds against various Gram positive and Gram negative bacterial strains.

Key words: *Amino acid, salicylaldehyde, Antibacterial activities, etc.*

Introduction:

According to WHO andCoordinating group on antimicrobial Studies reported thatlife is at risk today because of growing antimicrobial resistance [3]. Drug-resistant diseases could cause 10 million deaths each year by 2050 and damage to the economy as catastrophic as the 2008-

2009 global financial crisis. By 2030, antimicrobial resistance could force up to 24 million people into extreme poverty. Without investment from countries in all income brackets, future generations will face the disastrous impacts of uncontrolled antimicrobial resistance [4].

Antimicrobial resistance happens when microorganisms (such as bacteria, fungi, viruses, and parasites) change when they are exposed to antimicrobial drugs (such as antibiotics, antifungals, antivirals, antimalarials, and anthelmintics). Microorganisms that develop antimicrobial resistance are sometimes referred to as "superbugs" [5][6]

Antimicrobial resistance is one of the greatest threats that we face as a global community. This reflects the depth and scope of the synthesis and design, cheapest and more effective new anti-microbial drugs is responsibility nurturing scientist, Scientist and chemist researcherneeded to control its rise and protect a century of progress in health [7]. By considering the above importance of novel antimicrobial agent and in continuation of my research work on novel microbial resistant molecules [8][9], here we thought to synthesize biologically active more potent molecule to destroy modern genetically mutated microbe.

Material and Methods

The IR spectra were recorded on a Perkin Elmer FT-IR spectrum 65 at the range of 450-4000 cm^{-1}using ATR samples were kept directly without KBr pallets and Mass Spectra were elemental analysis wereestimated by microanalysis. The melting points of ligand and its metal complexes were determined by open capillary method on digital melting point apparatus (optics technology) and melting points are not corrected. Allchemicals and solvents were commercial grade materials and were used without further purification.Purification of ligands were carried out by column chromatography using commercial column chromatography grade silica gel (60-120 mesh) purchased from s. d. fine-chemicals Ltd. using mixture of ethyl acetate and n-hexane as eluting agent. All known compounds were characterized and compared with the literature reports. IR spectra of metal complexes were recorded in KBr at 4000-400 cm-1 at SAIF Kochi. 1H NMR spectra were obtained on a Perkin-Elmer 300 MHz spectrophotometer using TMS as internal standard in CDCl3 as solvent at SAIF Kochi. Elemental analyses were performed on

elementarvario EL-III at SAIF Kochi.Biological activities at department of Biotechnology Aditya College, Beed.

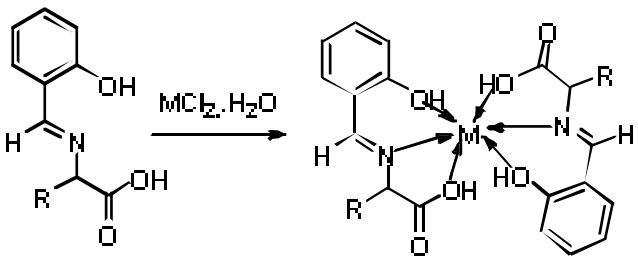
Experimental section

General procedure for the synthesis of Ligands L_1-L_2:

To solution of Amino Acid (10mmole) in 10 ml methanol containing (10mmole) NaOH, add salicylaldehyde (10mmole) in 15 ml methanol as added drop wise with constant stirring and heated under reflux for 3-5 hrs. On a heating stirrer at 60 ^{0}C. Then the reaction was cooled at room temperature, Precipitate was filtered off, 2-3 times washed with ice cold water and dried in hot air oven. Purification of ligands (**L_1-L_2**) was carried out by column chromatography. Yield60-78%.

General Procedure Preparation of the metal Complexes (P_1-P_4):

The ethanolic solution of the metal salt was added dropwise to the hot ethanolic solution of the ligand in the molar ratio of 1:2 with constant stirring. Immediate precipitation resulted in each case was obtained. The precipitate was filtered, washed and dried in hot air oven.



R= H, CH2-Ph M= Ni or Cu

Scheme: 1

Spectral data of compounds:

2-(2-hydroxybenzylideneamino)acetic acid(L_4):

Yield: 78% FTIR (ATR cm^{-}); 3251, 1680, 1612, 1531, 1330 ^{1}H NMR(400MHz $CDCl_3$)δ4.51(S, 2H), δ7.23-7.92(M, 4H),δ8.12(S 1H) ,δ8.51(S, 1H), δ12.0(S, 1H), Mass m/z: 197.058(100%), M+1 peak 180.061(9.8%), Elemental analysis: C(60.33%), H(5.06%), N(7.82%), O(26.79%)

2-(2-hydroxybenzylideneamino)-3-phenylpropanoic acid (L_2):

Yield : 89% FTIR(ATR cm^{-}) : 3137, 1679, 1637, 1521, 1332 ^{1}H NMR δ3.12-3.17(d, 2H), δ4.42 (t 1H), δ7.14-7.66(m, 9H), δ8.14(s, 1H),δ8.52(s, 1H), δ11.71(S, 1H),Mass m/z: M+1 270.01

Elemental analysis:C(71.36%), H(5.61%), N(5.20%), O(17.82%)

P_1: $[Ni(L_1)_2]$

Yield: 86% FTIR (ATR cm^{-}): 1641, 1604, 1518, 1298, 568,418, Elemental analysis: C(51.97%), H(4.12%), N(6.73%), O(23.06), Ni(14.11), Mass: m/z 418.04

P_2: $[Ni(L_2)_2]$

Yield: 73% FTIR(ATR cm^{-}) : 1652, 1598,1517,570,405, Elemental analysis:C(64.35%), H (5.06%), N(4.69%), O(16.07%),Ni(9.83%)Mass: m/z: 598.14

P_3: Yield: 78%, Mass: m/z: (M+1): 422.04, Elemental analysis: C(51.37%), H(4.07%),N(6.66%), O(22.81%), Cu(15.10%)

P_4: Yield: 81%, Elemental analysis: C(63.83%), H(5.02%), N(4.65%) O(15.94%),Cu(10.55%) Mass: m/z: 603.01

Result and discussion:

Antibacterial screening of the L_1-L_2 and P_1-P_4 were carried out using four bacterial strains of Gram-positive (**S.pyogenes**,**S.aureus**spp.) and Gram-negative (***E. coli***,**P.aerug** spp.) bacteria. The zones of inhibition values in Table 1. Ampicillin and ciprofloxacin was used as a reference compound for antibacterial activities. These bacterial strains are used because they are known as common pathogens of human beings. The antimicrobial studies suggested that the Schiff bases

are biologically active and their metal complexes showed significantly enhanced antibacterial activity against microbial strains in comparison to the free ligands.

The spectral data indicate that the complexes have the general empirical formula [ML2], where L is an amino acid Schiff base. The complexes was found to be easily soluble in DMSO and DMF at room temperature, whereas it is insoluble in acetone, water and chloroform.

Table No.1 In vitro antimicrobial activity of 2-(2-hydroxybenzylideneamino) acid derivatives and its Complexes (**concentration ofsolution 10µg/ml)**

Entry	**Antibacterial activity(zone of inhibition in mm±1)**			
	Gram Positive Bacteria		**Gram Negative Bacteria**	
	S.pyogenes	**S.aureus**	**E.coli**	**P.aerug**
L1	17	13	16	19
L2	09	11	11	06
P1	18	**33**	**29**	**32**
P2	22	**43**	**45**	**21**
P3	06	07	**29**	**34**
P4	18	09	**27**	**29**
Ampicillin	**23**	**18**	**21**	**29**
Ciprofloxacin	**27**	**33**	**26**	**28**

Conclusion:

The current article focused on the synthesis, spectral analysis and Microbial activities of 2-(2-hydroxybenzylideneamino) acid derivatives. The antibacterial results shown that most of the synthesized compounds shows promising activity against the Gram-negative bacteria rather than Gram-positive bacteria.

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