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Green synthesis, Characterization and Antimicrobial Assay of Schiff base Ligands derived from Vanillin and Amino Acids (Glycine and phenylalanine) and their Co (II) complexes

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ABSTRACT:
TwoSchiff base ligands were synthesized from condensation of 4-hydroxy-3-methoxybenzaldehyde (vanillin) with amino acids (glycine and phenylalanine) and Co(II) complexes were obtained using mechanochemical (mortar and pestle) method of green synthesis. Both ligands (GlyVan and PheVan) and their Co(II) complexes were characterized using spectroscopic method of FT-IR, Uv-Visvble and various physicochemical and antimicrobial properties were determined. The result obtained revealed that, the ligand GlyVan had milk colour with 96% yield,, while ligand Phevan had yellow colour with 98% yield. The ligands GlyVan, PheVan and Co(II) complex were soluble in water and in all common organic solvents tested except acetone, chloroform and ethyl-ether. The melting point of the ligands range from (210-220)^{o}C while that of Co(II) complexes ranges from 248-285)^{o}C. The FT-IR spectra of ligand GlyVan showed important bands at $_{vO-H}$(33008), $_{vC-H}$(2891), $_{vC=N}$(1675), $_{vC=O}$(1689), $_{vC=C}$(1581),$_{vC-O}$(1282), $_{vC-N}$(1114, 1170). Ligand Phevan showed relevant bands at FT- IR (cm^{-1}) $_{vO-H}$(3482), $_{vC-H}$(2833), $_{vC=N}$(1597), $_{vC=O}$(1679), $_{vC=C}$ (1521) $_{vC-O}$(1264), $_{vC-N}$(1132, 1179). While the Co(II) complexes showed bands for Co[glyVan]$_2$ FT-IR (cm $^{-1}$) $_{vO-H}$(3364), $_{vC-H}$(2833), $_{vC=N}$(1674), $_{vC=O}$(1687), $_{vC=C}$(1579) $_{vC-O}$(1280), $_{vC-N}$(1169), $_{vM-N}$(636), $_{vM-O}$(587) and Co[PheVan]$_2$ FT-IR(cm^{-1})$_{vO-H}$(3370), $_{vC-H}$(2800), $_{vC=N}$(1678), $_{vC=O}$(1675), $_{vC=C}$ (1580) $_{vC-O}$(1281), $_{vC-N}$(1169), $_{vM-N}$(636), $_{vM-O}$(587). The Uv- Vis analysis of the ligands had peaks atUv. nm (cm^{-1}) 262 (38167), 334 (29940) for GlyVan while PheVan had peaks at Uv. nm ((cm^{-1}) 255, (39215), 355 (28169).The antimicrobial screening of the Schiff base ligands and the Co(II) complexes revealed that, the ligands and Co(II) complexes showed susceptibility against all the bacteria isolates of Escherichia Coli, Staphylococcus aureus, Klebsiellapneumoniae and Pseudomonas aeruginosa except Salmonella typhi which showed resistance to the metal complexes and the ligand.

Key words: green synthesis, Schiff base, mechanochemical, glycine, Vanillin.

I. INTRODUCTION:

Green chemistry in synthesis of inorganic compounds particularly Schiff base as a ligands in coordination compounds is quite a developing and a modern approach in designing a more ecofriendly means that minimize environmental harm, uses environmental friendly solvents, energy efficient and minimal waste with very low impacts on environment.

It is a new way of looking at organic and inorganic synthesis and the design of drug precursors and molecules, offeringimportant environmental and economic advantages overtraditional synthetic processes. The recent interest in Schiff base synthesis has posed a new challenge for synthetic chemist thatnew reaction conditions need to be foundother than traditional methods which utilizes high energy, emits volatile organic solventsanduses hazardous toxic chemicals, hence the need for green approaches in other to conserve and preserve the ecosystem. They

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improveselectivity, reduce reaction time, and simplify separationand purification of products more than the conventional methods.

Various green methods exist for the synthesis of Schiff base ligands which include use of natural acids as catalyst, use of water or ethanol as solvents, mechano-chemical methods which involve grinding using mortar and pestle and irradiation methods have been identified to be more efficient with high yield within a short period of time.

Schiff bases are condensation products of primary amines with carbonyl compounds and were first reported by Schiff in 1864 (Cimermanet al., 2000). The common structural feature of these compounds is the azomethine group with a general formula RHC=N-R^1, where R and R^1 are alkyl, aryl, cyclo alkyl, or heterocyclic groups which may be variously substituted. These compounds are also known as anils, imines, or azomethines. Several studies (Singh et al., 1975) showed that the presence of a lone pair of electrons in an sp^2 hybridized orbital of the nitrogen atom of the azomethines group is of considerable chemical and biological importance making it a "wonder" functional group due to it applicability in medicine, catalysis, coordination chemistry, biological systems, material science, sensors and biosensors, pharmaceuticals, dyes and pigments and corrosion inhibition. Recently there has been great focused on the synthesis of Schiff base and its metal complexes due to its vast applicability.

A large number of different Schiff base ligands have been used as cation carriers in potentiometric sensors as they have shown excellent selectivity, sensitivity, and stability for specific metal ions such as Co(II), Cu(II), Gd(III), Hg(II), Ni(II), Pb(II), Y(III), and Zn(II) (Abbaspouret al., 2002). Schiff bases have been studied for their important properties in catalysis (Hernandeset al.,2005). They show catalytic activity in the hydrogenation of olefins (Olieet al., 1984). They find applications in biomimetic catalytic reactions.

An interesting application of Schiff bases is their use as an effective corrosion inhibitor, which is based on their ability to spontaneously form a monolayer on the surface to be protected. Many commercial inhibitors include aldehydes or amines, but presumably due to the C=N bond the Schiff bases function more efficiently in many cases (Olieet al., 1984).

The main aim of this study was to synthesized and characterized Schiff base ligands derived from vanillin (4-hydroxy-3-methoxybenzaldehyde) and amino acids (glycine and phenylalanine) and their metal complexes using mechano-chemical method of green synthesis.

II. MATERIALS AND METHODS

Reagents and chemical were used as supplied from Sigma Aldrich without further purification. The chemicals, reagents and solvents used in the experimentation are Vanillin (4-hydroxy-3-methoxybenzaldehyde), glycine, phenylalanine, ethanol, distil water, mortar and pestle, Zinc Chloride, cobalt(II) chloride hexahydrate, copper(II) chloride dihydrate, DMF, DMSO, acetone.Synthesized Schiff bases and their metal complexes were characterized using Gallenkamp melting point apparatus for melting point determination, solubility test were carried out for main organic solvents and water, Attenuated Total Reflectance of solid state IR- spectra were recorded on perkin Elmer Frontier FT-IR spectrophotometer and Perkin Elmer Lambda 25 Uv/Vis spectrometer for electronic spectra.

Synthesis of Schiff base ligand GlyVan

Using mortar and pestle, the mixture of glycine (0.50 g, 6.6 mmol) and vanillin (1.01 g, 6.6 mmol) were grinded for 30-45 min.,the paste was dried in a hot air oven for 5 min. A loose powdered milk color product was obtained with (1.33 g) 96% yield.M.p(210-212), IR(cm^{-1})$_{\nu O-H}$(33008), $_{\nu C-H}$(2891), $_{\nu C=N}$(1675), $_{\nu C=O}$(1689), $_{\nu C=C}$(1581) $_{\nu C-O}$(1282), $_{\nu C-N}$(1114, 1170). Uv. nm (cm^{-1}), 262 (38167), 334 (29940).

Synthesis of Schiff base ligand PheVan

Using mortar and pestle, the mixture of phenylalanine (0.50 g, 3mmol) and vanillin (0.46 g, 3 mmol) were grinded for 30-45 min., the paste was dried in an hot air oven for 5 min. A loose powdered yellow color product was obtained with (0.89 g) 98 % yield.M.p (218-220)^{o}C, FT-IR (cm^{-1}) $_{\nu O-H}$(3482), $_{\nu C-H}$(2833), $_{\nu C=N}$(1597), $_{\nu C=O}$(1679), $_{\nu C=C}$ (1521) $_{\nu C-O}$(1264), $_{\nu C-N}$(1132, 1179) , Uv. nm ((cm^{-1}) , 255, (39215), 355 (28169).

Synthesis of Cobalt(II) complexes Co[glyVan]$_2$ and Co[PheVan]$_2$

Using mortar and pestle, the mixture of glycine (0.50 g, 6.6 mmol) and vanillin (1.01 g, 6.6 mmol) were grinded for 30-45 min., then equimolar amount of cobalt(II) $CoCl_2.6H_2O$ salt (0.79 g, 3.3 mmol) was added and the mixture was further grinded for 15-20 min., the light purple paste was dried in an hot air oven for 5 min. A loose

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powdered light purple product was obtained with 97 % yield. M. p (248-250)^{o}C, FT-IR(cm $^{-1}$)$_{\nu O-H}$(3364), $_{\nu C-H}$(2833), $_{\nu C=N}$(1674), $_{\nu C=O}$(1687), $_{\nu C=C}$ (1579) $_{\nu C-O}$(1280), $_{\nu C-N}$(1169), $_{\nu M-N}$(636),$_{\nu M-O}$(587). Uv. nm(cm^{-1}), 262(38167), 558(17921), 678 (14749).

Using mortar and pestle, the mixture of phenylalanine (0.50 g, 3 mmol) and vanillin (0.46 g, 3 mmol) were grinded for 30-45 min., then in the ratio of 2:1 metal to ligand, (0.7137 g, 1.5 mmol)amount of cobalt(II) $CoCl_2.6H_2O$ salt were added and the mixture was further grinded for 15-20 min, the purple paste was dried in an hot air oven for 5 min. Loose powdered purple products were obtained with 98% yield. M. p.(283-285)^{o}C, FT-IR(cm^{-1}) $_{\nu O-H}$(3370), $_{\nu C-H}$(2800), $_{\nu C=N}$(1678), $_{\nu C=O}$(1675), $_{\nu C=C}$(1580) $_{\nu C-O}$(1281), $_{\nu C-N}$(1169), $_{\nu M-N}$(636), $_{\nu M-O}$(587).Uvnm(cm^{-1}) 256 (39062), 355 (28169) and 472 (21186)

III. RESULT AND DISCUSSION

The solubility test of the Schiff base ligands and Cobalt(II) complexes was carried out at room temperature. Both the metal complexes and ligands were soluble in DMF, water, n-hexane and DMSO while insoluble in acetone, chloroform and ethyl-ether among all the various common organic solvents tested. The percentage yield of Schiff base ligand and metal complexes range from (96-98)% with Co(II) complexes having varying shades of purple colors while Schiff base ligands GlyVan and PheVan had milk and yellow colors respectively.

Infra-red and Uv-vis Spectra

The Schiff base ligand GlyVan showed an azomethine stretch band at 1675 cm^{-1}, C=O band at 1689 cm^{-1}, C-H of aldimine band at 2891 cm^{-1}. The band observed at 3308 cm^{-1} is attributed to OH^- vibrational frequencies, C-O-C band at 1282 cm^{-1}, while 1581 cm^{-1} and 1169 cm^{-1} are characteristics of C=C and C-N vibrational frequencies respectively (Sani et al 2022).

The infra-red spectra of the Schiff base ligand PheVan showed an azomethine stretch band at 1597 cm^{-1}, C=O band at 1679 cm^{-1}, C-H of aldimine band of 2833 cm^{-1}. The band observed at 3482 cm^{-1} is attributed to OH^- vibrational frequencies, C-O-C band at 1264 cm^{-1}, while 1521 cm^{-1} and 1176 cm^{-1} are characteristics of C=C and C-N vibrational frequencies respectively (Sani et al 2018). The Cobalt(II) complexes showed a shift in azomethine bands in the range of C=N (1597-1679)cm^{-1}, C-O (1264-1282) cm^{-1}, C-N (1169-1176) cm^{-1}, M-N (636-641) cm^{-1}, M-O bond showed at 587 cm^{-1}. These shifts clearly support the facts that there was coordination of metal to the ligand via the azomethine and carboxylate O-H bonds.

The Uv-Vis electronic reflectance spectra of the Schiff base ligands and complexes were obtained between 200-400 nm Uv and 400-900 nm Visible region. The electronic spectrum of the Schiff base GlyVanshowed two important bands at 262 nm(38,168) cm^{-1}, and 334 nm (29940) cm^{-1}, assigned to $\pi\rightarrow\pi^*$ and $n\rightarrow\pi^*$ respectively. These are intra ligands transitions and are due to double bonds in C=N and C=C groups of phenyl rings in the ligand structure and lone pairs of electrons present on heteroatom Nitrogen (N), in the azomethines group respectively (Lever, 1968).

The cobalt(II) complex Co[glyvan]$_2$ showed bands at 262 nm (38,168)cm^{-1}, (intra ligand electron transition), and 558 nm (17,007 cm^{-1}), and 678 nm (14,749 cm^{-1}, which may be assigned to the charge transfer (CT), $^4T_{1g}(F) \rightarrow {}^4A_{2g}(F)$, and $^4T_{1g}\rightarrow {}^4T_{1g}$ (P) transitions respectively. (Virparia 2013).

The UV spectra of Co[PheVan]$_2$ complex showed absorption bands at 255 nm (39216) cm^{-1}and 335 nm (29850) cm^{-1} which may be assigned to $\pi\rightarrow\pi^*$ and $n\rightarrow\pi^*$ transitions originating in the benzenoid and aldimine moieties respectively. But the Co(II) complex shows three absorption bands at 256 nm (39062) cm^{-1}, 355 nm (28169) cm^{-1} and 472 nm (21186) cm^{-1}. The new absorption band at 472 nm (21186) cm^{-1} may be related to metal-ligand charge transfer excitations.

Antimicrobial Assay Schiff Base Ligand and Metal Complexes

The results of the antimicrobial screening of the Schiff base and its metal complexes at a concentration of 30mg/mL (0.03g/mL) in DMSO against five (5) antibacterial strains were analyzed. The inhibition Zone was measured in millimeters. The results indicated that the ligands and Co(II) complexes showed susceptibility against all the bacteria isolates of Escherichia Coli,Staphylococcus aureus, Klebsiellapneumoniaeand Pseudomonas aeruginosa except Salmonella typhiwhich showed resistance to the metal complexes and the ligand. In general, at 30mg/mL concentrations Schiff base ligand and Co(II) complexes showed activity in comparison with the standard drug ciprofloxacin.

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IV. CONCLUSION

Schiff base ligands were synthesized using mechanochemical method by grinding vanillin with amino acids (glycine and phenylalanine) at room temperature.Various physical measurements were taken which include melting point determination, solubility test, and colour. FT-IR and Uv-Vis spectroscopic method were used to characterize the compounds. The FT-IR results showed there was formation of "wonder functional group" C=N around 1675cm^{-1} and 1597cm^{-1} for the ligands Glyvan and PheVan respectively. Antimicrobial screening was also done in other to find the applicability of the synthesized compounds. The result showed that the antimicrobial agents showed more activity than the standard drug ciprofloxin.

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