

# Synthesis of cardanol Schiff base ligands, characterization and biological activity of Copper (II), Nickel (II) Cobalt (II) complexes

C.M Sofia<sup>1</sup> and C. Isac Sobana Raj<sup>2</sup>

<sup>1</sup> Department of Chemistry and Research, N.M.Christian College Marthandam, Affiliated to Manonmaniam Sundaranaar University, Abisekapaty Tirunelveli, Tamil Nadu, India

<sup>2</sup> Department of Chemistry and Research, N.M.Christian College Marthandam, Tirunelveli, Tamil Nadu, India

## Abstract

Cardanol 'C15' are chained aliphatic unsaturated compound, which naturally contains substituted phenol derived from CNSL (Cashew Nut Shell Liquid) was utilized for the development of Schiff base polymeric ligand by using P-toluidine. The complexes 'Schiff base' of Copper (II), Nickel (II) Cobalt (II) from di- $\alpha$ -formyl methoxybis (3-pentadecenylphenyl) methane (DFMPM) and P-toluidine were extracted in three steps. The Spectroscopy IR characterized the complexes and ligand, UV-visible and SEM analysis were studied. The conductance assessments specify that each of the complexes of nonelectrolytes. Spectral report from UV-visible provides structural information of these complexes. The IR spectra specify the location of nitro imino group and M-N bonds. The SEM analysis revealed the nanocrystalline nature of the complexes. The result shows that the Copper, Nickel and Cobalt complexes, are hexa-coordinated and consist moderate antimicrobial activity. The intake of ion metal signifies the ligand could be utilized for the separation of these ions metals from water molecules.

**Keywords:** Antimicrobial activity; Cardanol; DFMPM; Schiff base; Ions Metal

## 1. Introduction

From the natural renewable source, the synthesis of fine chemicals has exploited great importance in the synthetic community and is becoming a challenging and significant theme for investigators of both the industrial and academic areas. Amongst the renewable materials, cardanol has attracted considerable attention because of its unique nature [1]. Concern with the sources that are limited, rising demand, and toxicity inherent of such polymer-based

phenol substances, efforts have stood forward to subsidize them with bio-phenolic origins. In the midst of several possible substitutes, cardanol has been measured to be an attractive process due to its agro-waste supplies, basic extraction methodology, potentiality, and nontoxicity to reduce because of bio-phenolic origins. The most abundant production of cardanol was from India, Brazil, Vietnam, Thailand, and Indonesia. India specifically accounted for a production approaching 15,000 MT in 2015 [2]. A low-cost resource considered as 'Cardanol,' that is occurring naturally through a renewable phenolic mixture of non-isoprenoid lipid from CNSL. Through industrial thermal treatment, CNSL is generally composed by cardanol (almost 70%), a long chained phenolic-carbon which contributes this compound high hydrophobicity and for organic synthesis with several reactive sites [3]. Also, it does not depict mutagenic or cytotoxic effects [4]. Presently several investigators have analyzed its extraction [5], chemistry and composition [6]. As per literature, CNSL is already in use in the manufacture of peculiar phenolic resins [7]. CNSL may displace phenol in several processes with better or equivalent results. CNSL comprises four main molecular compounds namely cardol based resorcinol 5-pentadecenyl, anacardic based salicylic acid 6-pentadecenyl, cardol based resorcinol 2-methyl and cardanol based phenol 3-pentadecenyl.

This functionality based reactive make them applicable for a number of polymerization responses with relent as well as reduction mechanisms. Cardanol, one of the vital components of CNSL elements that has been stated as a prospective raw material for oligomer/polymer synthesis relating several chemistries such as alkyd, phenolic, polyurethane, polyol, epoxy, etc and has shown to be

an exceptional material for coating when formulated for diverse coatings types like altered coatings of epoxy, liquid-borne coatings, coatings that are UV based curable, phenolic coatings, altered polyurethane coatings and coatings based on alkyd (Balgude and Sabnis, 2014; Xiong et al., 2014) [8,9]. Schiff bases are significant ligand based classes because of their flexibility synthetic, their sensitivity and selectivity amongst the central atom metal, similarities in structural with substantial natural biological activity and also due to existence of (N=CH-) imine group which acts in exposing the mechanism of rasemination and transformation reaction in natural system [10-12].

The present of study aims to achieve synthesis, characterization and biological studies of the Schiff base complexes of Copper, Nickel and Cobalt derivatives from DFMPM and P-toluidine.

## 2. Literature Review

Polymerization oxidative of cardanol had been carried out using oxidative enzymes for the development of coating anti-biofilm materials [13,14]. A study conducted by Jisha and Raj, [15] on 'Cardanol' unsaturated aliphatic substituted phenolic-chain derived from CNSL. The preparative way involved (a) the conversion of cardanol into bis (3-pentadecenyl phenol) methane (BPPM) with formaldehyde. (b) BPPM into di- $\alpha$ -formylmethoxy bis (3-pentadecenyl phenylmethane). DFMPM on reacting with epichlorohydrin alongside with sodium periodate. (c) DFMPM to ligand base Schiff with aniline and lastly (d) complexes of Schiff base Nickel, Cobalt and Copper salt metals. The complexes and ligand were categorized by UV-Spectra, <sup>1</sup>H NMR, IR and analysis of element, point of melting, ion metal intake, conductivity, and the antimicrobial activity was studied. The outcome of the study indicates that the Copper, Nickel and Cobalt complexes are hexacoordinated and contains moderate antimicrobial activity.

An earlier study by Raj et al [16] on 'Cardanol' based phenol substitution resulting from CNSL for the Schiff base polymeric ligand preparation using ethylenediamine. The measurements of conductance specified that all its complexes are nonelectrolytes. The IR spectra indicated the M-N bonds and nitro imino coordinates. UV-metric spectra provided information about complexes structure. <sup>1</sup>H NMR records are also reliable with IR spectral records. The outcome indicated that the complexes of Nickel, Cobalt and Copper are hexacoordinated and have reasonable antimicrobial activity.

An analysis by Lalitha et al [17] reported the extraction based on cardanol of complex metals contained a unit of hydrophobic that is unsaturated and acrylated epoxidized linseed oil (AELO)

developed via double bonds epoxidation trailed by acrylation. The investigation to limit the biofilm formation by using potential synthesized metal compounds (A and B) also the persistent state for the bacteria. Both the compounds had significant biofilm inhibiting activity. The minimum concentration that was necessary to inhibit biofilm formation by A was 100  $\mu\text{g mL}^{-1}$  for SA, PA, and UPEC, whereas the STM biofilm was subdued at nearly 90% at 500  $\mu\text{g mL}^{-1}$  as compared to the control.

Dawson and Wasserman, [18] reported that nitro hydrogenated cardanol could be used as a constituent in rubber compounds. Amino-3-pentadecylphenol can be used as a transitional for the preparation of a variety of useful dyes in the compounding of rubber. Ghatge and the Iron, Cobalt and Chromium complexes obtained from (ONNO) tetradentate ligands base Schiff, 1, 4-bis[3-(2-hydroxy-1-naphthaldimine) propyl]piperazine and 1, 8-bis [3-(2-hydroxy-1-naphthaldimine)-pmenthane, show moderate antimicrobial activity [19] compared to standard antibiotics [20]. The antimicrobial activity of tridentate based Schiff, obtained by reduction of 2-amino-3-carboxyethyl-4-5-dimethylthiophene with salicylaldehyde, was predicted to increase on chelation with metal transition ions [21]. Nickel, Copper, Zinc and Cobalt base Schiff complexes resultant from  $\alpha$ -aminobutyric acid and vanillin were also found to exhibit higher antibacterial activity compared to the free Schiff bases [22]. Several binuclear and mono transition and di benzoylmethane, are more effective fungicidal and bactericidal than the ligand. Sharma and Piwnicka-Worms, [23] reported complexes of Schiff base that target hemozoin accumulation like the chloroquine drug for antimalarial activity.

## 3. Materials and Methods

### 3.1 Chemicals

All ingredients used are of the standard grade (SR) and maximum pureness obtained. They included cobalt(II)chloride (AlfaAesar), nickel(II)chloride (Sigma), Copper(II) chloride(Sigma), formaldehyde (Aldrich), Phenylhydrazine (Aldrich), L-valine (Aldrich),  $\alpha$ -naphthyl ammine (Aldrich), 3,3'-diaminobenzidine (Aldrich), L-phenylalanine (Aldrich) and Cardanol (Aldrich). The micrological records (C, H, N) were gathered by Perkin Elmer 2400 device. Infra-Red bands were acquired by PE-IR spectrum, Device brand: System 2000. <sup>1</sup>H NMR readings were achieved by AMX-300MHz, FT NMR device. Automated bands of the ligands and its complexes are acquired by Elmer Perkins Lamda-25 UV-Visible device model in the range of 190-700 nm. A morphological surface study was attained using JW-5610 electron scanning microscope.

### 3.2 Synthesis of DFMPM

Production of bis (3-pentadecenylphényl) methane (BPPM), diglycidylether of bis (3-pentadecenylphényl) methane (DEBPPM), di-a-formylmethoxybis (3pentadecenylphényl) methane (DFMPM) was obtained as per the previous method [24].

### 3.3 Synthesis of Schiff base ligand with DFMPM

The ligand based Schiff were prepared by the stated procedures [25, 26]. An ethanolic equimolar solution of DFMPM and P-touline were intermixed and refluxed for an hour about. Pour the intermixed product in ice, (1+2) ligand based Schiff was achieved [27]. The precipitate of yellow composite was sieved cleansed with H<sub>2</sub>O and desiccated over unhydrated calcium chloride. The crude extracted sample was recrystallized from 50% absolute alcohol yield=62%. Melting point =223°C.

### 3.4 Synthesis of Copper, Cobalt and Nickel Schiff based complexes metal

Each of the complexes metal were prepared by mixing an ethanolic solution of Schiff base ligands (L1-L5) with the equivalent aqueous metal salt portion of Copper nitrate, Cobalt nitrate and Nickel nitrate in 1:2 molar ratios. The subsequent combination was refluxed for about twelve hours at 70-80°C [28], a colored solution appeared on standing. The obtained product mixture was sieved, ethanol cleansed, acetone, diethyl ether, and boiling water and lastly desiccated under vacuum at 90°C yield=60%.

### 3.5 Estimation of intake metal ion

The filtrate acquired in the overhead approaches was gathered, and the gatherings were utilized for the valuation of the metal transition ions used for complexation by using standard methods [28].

### 3.6 Antibacterial activity

The in vitro growth inhibitory action of the complexes was confirmed against bacteria by a disc diffused method using agar as nutrient [26]. The standard drug Gentamycin dissolved in DMSO which acts as control was also tested at the same concentration under similar conditions to that of complexes. The solution medium comprised with microbial subcultures, then was autoclaved for 20 minutes at 121°C at a pressure of 15 lb before incubation. The microbes were incubated in broth of nutrient medium at 37°C for a day. The microbes were injected into Petri dishes (100 mm x 70 mm) in

the amount of 0.01 cm<sup>3</sup>; 15 ml of potato dextrose agar were homogeneously distributed into the sterilized Petri dishes. Each of the complexes was introduced into vacant pasteurized disc consuming antibiotic with the diameter of 6mm in the volume of 30 ml. The developed mixtures were dissolved in DMSO to a closing stage of 2000 ppm and socked in sieve paper. The cultured plates were kept at 4°C for 2 hours and incubated at 25°C for a day. The thickness of the progressed zone of inhibition around the sieved paper was measured after 24 hours' gestation, and the action of all treatment were made in replicates.

## 4. Results and Discussion

### IR Spectra

Bands of IR spectral for selected ligand and its ion metal complexes are represented in Table 1 and Figure 1-4. The IR spectrum of the free ligand is characterized mainly by the strong peaks at 3355 cm<sup>-1</sup>, 2973 cm<sup>-1</sup>, 1667 cm<sup>-1</sup>, 1387 cm<sup>-1</sup> and 1047 cm<sup>-1</sup> which are attributed the flexible frequencies of C–O, C–H and C = N (azomethine) correspondingly [29]. The free ligand from spectral IR was related with the spectral metal ion complexes. The characteristic absorption bands 3381-3338 cm<sup>-1</sup> ranges were attributed to –OH group of the coordinated or lattice water [30]. The absorption bands in the range 2973 cm<sup>-1</sup>, 2256 cm<sup>-1</sup> –1667 cm<sup>-1</sup> and 1614 cm<sup>-1</sup> –1387 cm<sup>-1</sup> are assigned to C–O, C–H and C = N respectively [31]. The iminal peak within the complexes metal displayed modification in shifts compared to the ligand representing the direction of the nitrogen iminal atom to the ion metal because of coordination. Another absorption bands at 1306 cm<sup>-1</sup> – 1047 cm<sup>-1</sup> are assigned to M-N bond, and 655 cm<sup>-1</sup> – 541 cm<sup>-1</sup> is assigned to M-O bond [32, 33].

**Table 1: Selected FT IR frequencies (cm<sup>-1</sup>) of the ligand and its complexes**

Ligand/Complex	vO-H (H <sub>2</sub> O)	vC-O	vC-H	vC=N	vM-N	vM-O
Ligand	3355	2973	1667	1387	1047	-
Copper Ligand Complex	3338	-	1630	1411	1292	655
Cobalt Ligand Complex	3381	-	2256	1614	1306	541
Nickel Ligand Complex	-	-	-	1483	1289	629

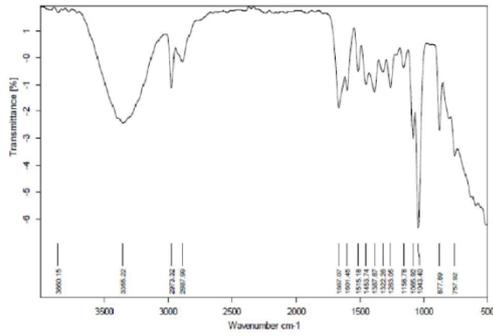


Figure 1 Bands of IR spectral for Ligand

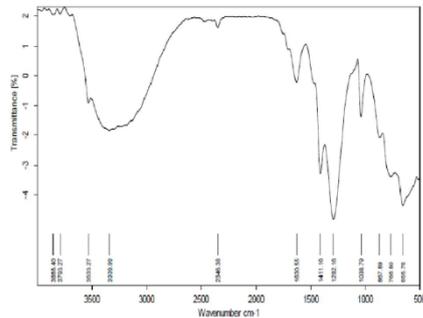


Figure 2 Bands of IR spectral for Copper Ligand Complex

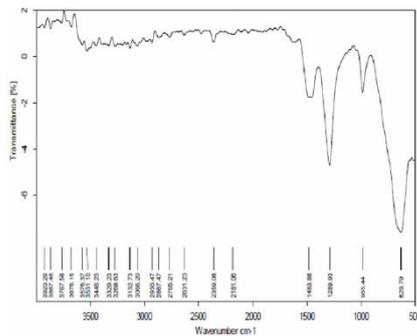


Figure 3 Bands of IR spectral for Cobalt Ligand Complex

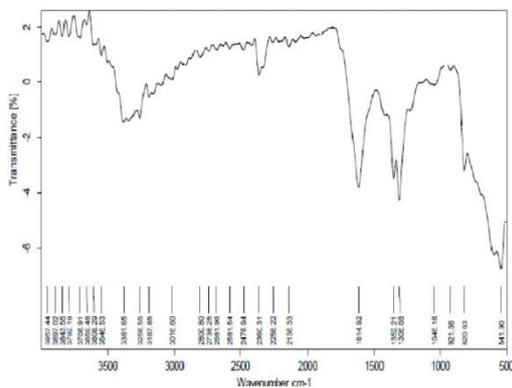


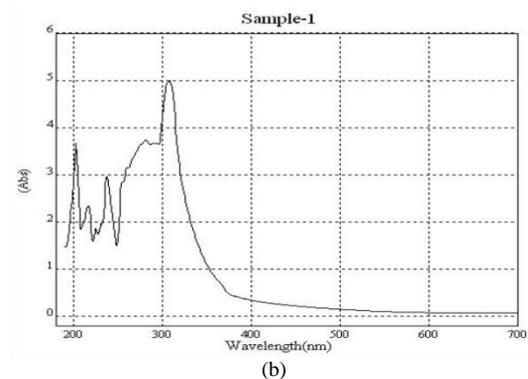
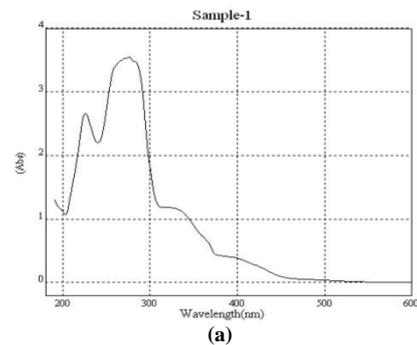
Figure 4 Bands of IR spectral for Nickel Ligand Complex

### UV - Visible Spectra

The region of absorption geometry and assignment of the complexes and ligand are given in Table 2 and Figure 5, and showed a broadband at 276 nm which is assigned to  $\pi$ -  $\pi^*$  transition of the C = N chromophore [34]. This bond complexation was altered to a wavelength of lower with signifying the coordination of iminal nitrogen with central ion metal. The UV and visible spectra of the Cu (II) complex showed three absorption bands at 308 nm, 281 nm, 238 nm and 203 nm giving an octahedral geometry with field transitions. The position and broadness of the band favour biased geometry octahedral for copper complexes due to the effect of Jahn-Teller. The spectral range of Co(II) and Ni(II) complexes showed absorption bands at 261 nm alone and 276 nm, 231 nm, and 192 nm respectively suggesting octahedral geometry for the complexes.

Table 2 UV-Visible Spectral data of the ligand (L) and its complexes

Ligand/ Complex	$\lambda_{max}$ (nm)		
Ligand	283	276	226
Copper Ligand Complex	308	281	203
Cobalt Ligand Complex	276	231	192
Nickel Ligand Complex	261	-	-



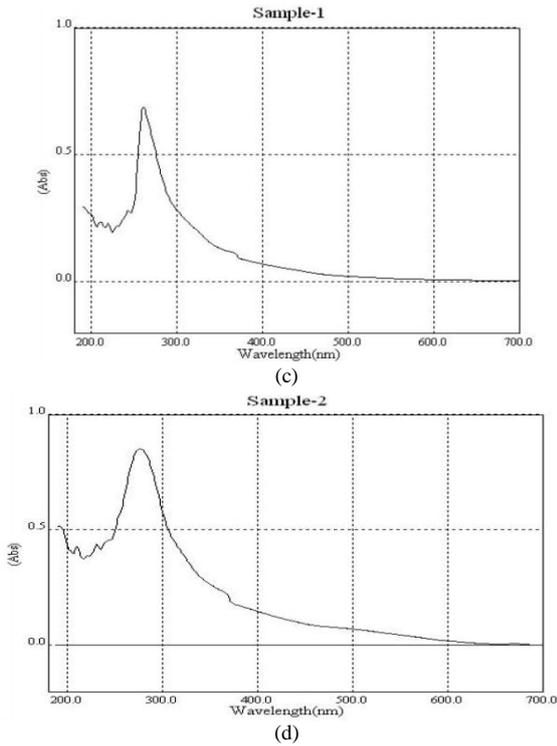


Figure 5. (a) Ligand L (b) Copper complex (c) Cobalt complex and (d) Nickel complex

### SEM Analysis

The surface morphology of Copper, Cobalt and Nickel [35] complexes have been studied using electron based Scanning Microscope, and the relevant images are given in Fig. 6-8.

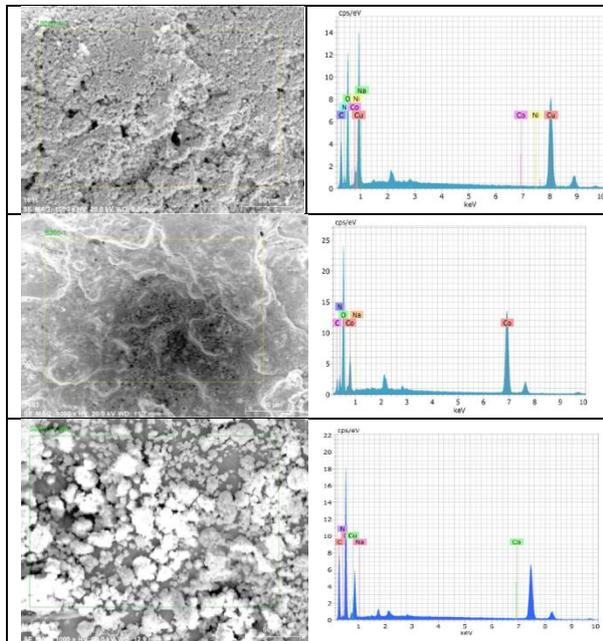


Figure 6 SEM image of Powder Sample Cu and 6a represents Cu elemental analysis (II) 7 SEM image of Powder Sample Co, and 7a represents Co elemental analysis (II) 8 SEM image of Powder Sample Ni (II), and 7a represents Ni elemental analysis

SEM study reveals that all the complexes are crystalline in structure, under low magnification, while a beaded appearance on slightly higher magnification and the surface morphology of the complexes appeared as rough, amorphous deposition and pitted surface.

Microanalysis of carbon and nitrogen contents present in the samples were recorded. By using SEM primary electron beam that is responsible for the release of an electron from the elements that creates up the specimen upper layers. The emitted electrons are gathered, and the amplified motion is exhibited on a tubular cathode ray. By scanning the primary electron beam across the specimen's surface in synchronization with the cathode ray tube, it is possible to convert the emitted electrons into an image of the specimen for display in the Figures (6a, 7a, and 8a). The critical facts recommend that all the multiplexes are mononuclear with the coordinated ligand to the central metal ion and the ligand to metal ratio in all complexes was 1:1, and their empirical formulae have been computed and are given in Table 3.

**Table 3 Elemental Analysis of the ligand and its complexes**

Compound	Yield %	Elemental analysis,	
		C	N
Copper	60	11	4
Cobalt	43	7	8
Nickel	29	27	4

### 5. Conclusion

Schiff base metals complex such as Copper, Nickel and Cobalt were extracted/synthesized from cardanol using different amine and aldehyde were characterized and described by spectral and analytical data. Three bidentate O, N donor type Schiff bases were prepared by using di- $\alpha$ -formyl methoxybis (3-pentadecenylphenyl) methane (DFMPM) and P-toluidine were extracted in three steps. These ligands were further complexed with transition metals to produce their new metal complexes. Spectral data and elemental analysis of the uncomplexed metal (II) and their ligands complexes were found to be in good agreement with their structures, representing high purity of all the compounds. Elemental analysis displays the metal to ligand ratio is 1:2. Conductivity measurements show all complexes are non-electrolytes. From SEM analysis crystalline nature of complexes is confirmed. The anti-bacterial study shows that all complexes are more active than ligand. This higher activity may be due to chelation process which decreases the polarity

of the metal ion by coordinating with ligands, and the metal complexes were more active than the Schiff bases arising from the cell permeability and the structure of a metal complex.

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