Designing, Synthesis and Structural Characterization of Bidentate Schiff Base Derived from 1-Amino-2-Chlorobenzene

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Abstract

In the present study, a bidentate Schiff base Salicylidene-2-Aminochlorobenzene was designed and synthesized by reaction between salicylic aldehyde and 1- Amino- 2-chlorobenzene. The formed crystals of the synthesized Schiff base Salicylidene-2-Aminochlorobenzene were purified by recrystallization with hot ethanol. The manipulation is carried out under a nitrogen atmosphere and the mixture is continuously stirred magnetically for 2 hours at a temperature of 80°C.The structure of this compound has been characterized through different spectroscopic methods such as, FTIR spectroscopy, UV-Vis spectroscopy, ^{1}H - NMR and ^{13}C -NMR spectroscopy. Physical parameters, molar mass and percent yield of synthesized Schiff base were detemined.

Keywords: Schiff base, spectroscopic characterization, NMR spectroscopy, FTIR spectroscopy, 1-Amino-2-chlorobenzene.

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1. Introduction

Schiff bases, a versatile class of organic compounds, have found widespread applications across various scientific and industrial fields. In recent years, researchers have explored the potential biological and medicinal properties of Schiff bases and their metal complexes. Some Schiff bases exhibit antimicrobial, antiviral, anticancer, and antioxidant activities, making them promising candidates for drug development and therapeutic applications [1-7]. Also one of the most significant applications of Schiff bases is the field of metal ion extraction, they can be biodegradable and less harmful to the environment compared to some traditional extraction agents [8-13] .

Many researchers have reported on the synthesis of Schiff base compounds [14-16]. However, very little information has been offered about the synthesis of Schiff bases using 1-Amino-2-chlorobenzene. In this work, we have been interested in the design and synthesis of of bidentate

Schiff base derived from 1-Amino-2-chlorobenzene. This compound has been characterized by elemental analysis, FTIR spectroscopy, UV-Vis spectroscopy, ^{1}H - NMR and ^{13}C -NMR spectroscopy to confirm the structure of the synthesized Schiff base.

2. Experimental

2.1. Reagents and solutions

All chemical used were of analytical reagent grade from Aldrich-Sigma and were used without further purification. Salicylic aldehyde, ethanol, 1-Amino-2-chlorobenzene and chloroform which was employed as the organic solvent.

2.2. Synthesis of Salicylidene-2-Aminochlorobenzene (HSOA)

0.5 moles of salicylic aldehyde are dissolved in 5 ml of absolute ethanol. Then, this mixture was transferred to a flask and heated and stirred until the complete dissolution is achieved. Next, 0.5 moles of 1-Amino-2-chlorobenzene, dissolved in 5 ml of ethanol, are added to the solution. The manipulation is carried out under a nitrogen atmosphere and the mixture is continuously stirred magnetically for 2 hours at a temperature of 80°C [17]. After cooling the solution, a yellow precipitate becomes evident. This compound is separated from the solution through filtration and subsequently dried under vacuum at ambient temperature. Figure 1 show the Reflux assembly used in the synthesis of bidentate Schiff basis.

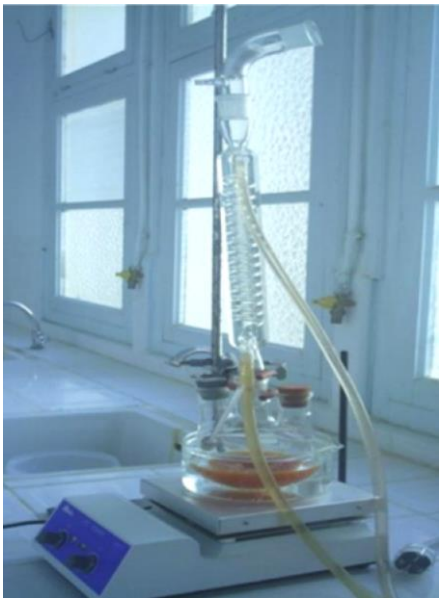

Fig 1. Reflux assembly used in synthesis of salicylidene-2-aminochlorobenzene

3. Results and discussion

3.1. Synthesis of Salicylidene-2-Aminochlorobenzene

The synthetic experiments of salicylidene-2-aminochlorobenzene are shown in Fig 2. The structure of Schiff base was characterized by a combination of elemental, IR, ^{1}H-NMR, ^{13}C-NMR spectral analysis data.

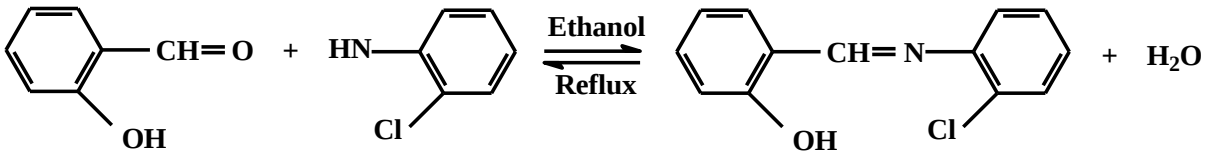

Fig.2. Synthesis of Salicylidene-2-Aminochlorobenzene (HSOA)

3.2. Physical characterization of Salicylidene-2-Aminochlorobenzene

The following table (Table1) summarizes the physical characterizations of the prepared Schiff base of Salicylidene-2-Aminochlorobenzene (HSOA).

Table 1: Physical characteristics of HSOA

Shiff base	Yield $^{0}/_{0}$	Appearance and color	Molar Mass g/mol
HSOA	98.73	Yellow crystals (See Fig 3)	231.45

As shown in figure 3, Yellow crystals of synthesized Schiff base Salicylidene-2-Aminochlorobenzene was obtained with yield more than 98 %.

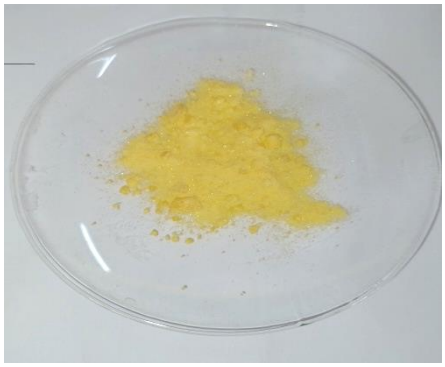

Fig 3. Crystals of synthesized Schiff base Salicylidene-2-Aminochlorobenzene.

3.3. Spectral characterization of Salicylidene-2-Aminochlorobenzene

3.3.1. UV-Visible spectroscopic

The UV-visible spectrum of the synthesis Schiff base salicylidene-2-aminochlorobenzene was performed in the region (300-700 nm) in chloroform solution. The electronic spectrum (II) showed a bond at 396 nm, as seen in Fig.4, this band can be attributed to the transition n π, which correspond to C=N imine group and indicates that the schiff base presents a single enolic form [18].

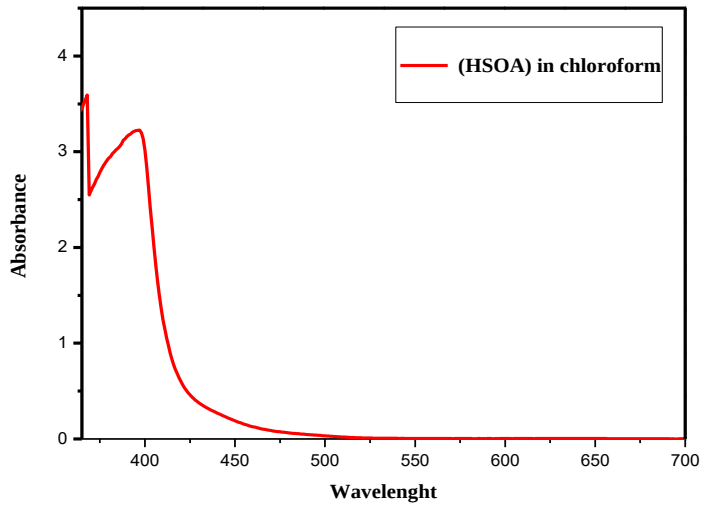

Fig.4. UV-Visible spectrum of salicylidene-2-aminochlorobenzene in chloroform

3.3.2. IR Spectrum analysis

The infrared spectrum of the prepared Schiff base which was derived from 1-Amino-2-chlorobenzene was recorded in the region (4000-400) cm^{-1} on KBr pellets, as shown in Fig.5

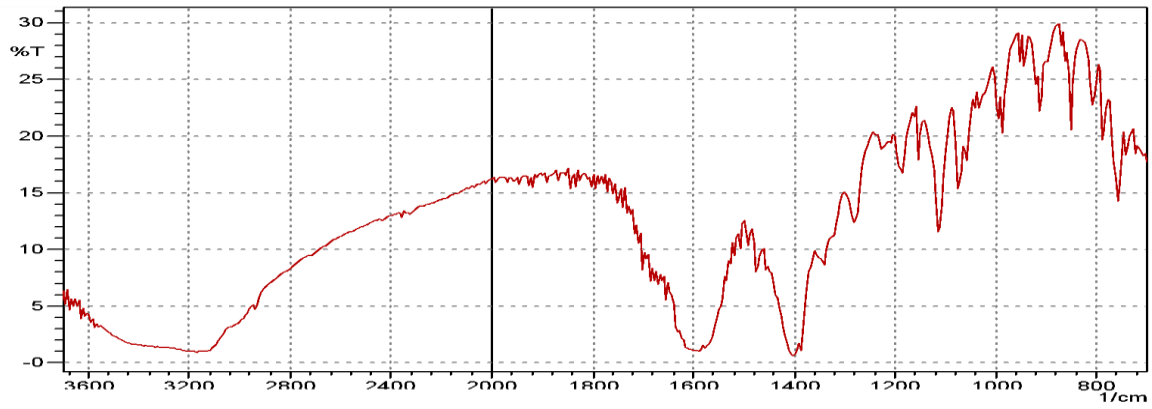

Fig.5. Infrared spectrum of salicylidene-2-aminochlorobenzene in chloroform

The most important absorption bands and their assignments are listed in Table 2. The vibrations of υ(C=N) and υ(C=C) groups have been observed at 1651 and 1589 cm^{-1} respectively. A broad band at 3300 cm^{-1} is due to the phenolic OH group and two medium bands at 1404 cm^{-1} and 1109 cm^{-1} are assigned to υ(C-N) and υ(C-O) vibrations respectively. The band at 2930 cm^{-1} characterize the vibrations of aliphatic groups υ(CH) which present in the synthesized Schiff base [19, 20].

Table 2 Characteristic IR bands (cm^{-1}) of salicylidene-2-aminochlorobenzene

Functional Groups	υ(CH) aliphatic	υ(C=N)	υ(C=C)	υ(OH)	υ(C-N)	υ(C-O)
Absorption band (cm^{-1})	2930	1651	1589	3300	1404	1109
Intensity	Weak	Weak	Medium	Medium	Medium	Medium

3.3.3. ^{1}H-NMR spectrum analysis

^{1}H-NMR spectrum of salicylidene-2-aminochlorobenzene is given by Fig .6. The signals in the region 6.94–7.49 ppm were assigned to the aromatic protons. The signal at 8.63 ppm was assigned to the protons of imine -CH=N group. The Schiff base exhibits resonance due to –CH_2– protons around 1.61 ppm. The peak at 13.24 ppm is attributed to phenolic OH group present in the salicylaldehyde [21, 22].

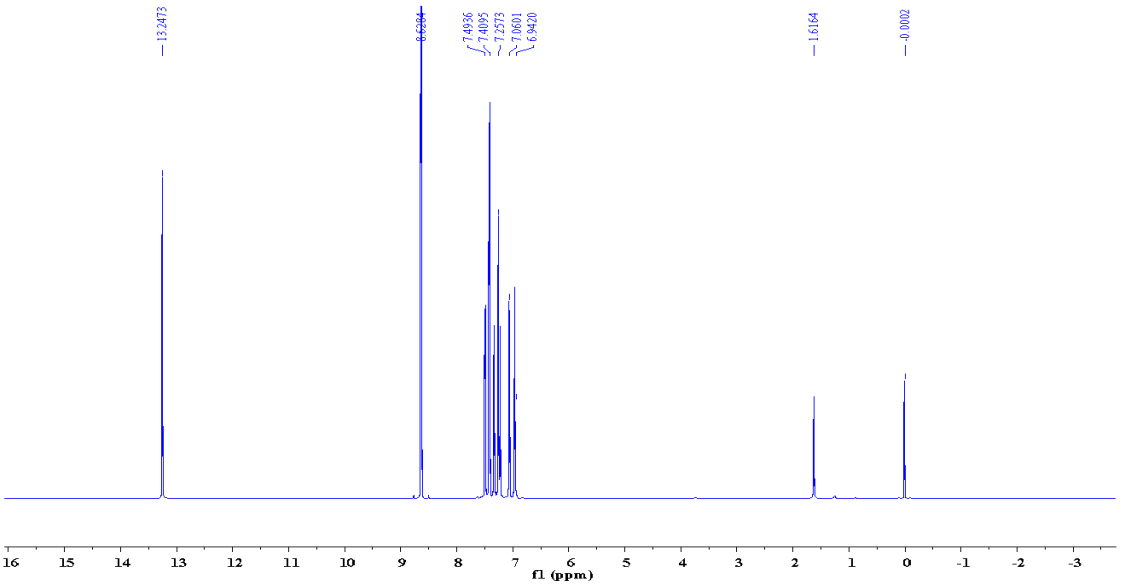

Fig. 6. H-NMR spectrum of salicylidene-2-aminochlorobenzene

3.3.4. ^{13}C-NMR Spectrum analysis

The ^{13}C-NMR spectrum of salicylidene-2-aminochlorobenzene, recorded in $CDCl_3$ is presented in Fig.7 .The signals which appeared in the range of 76.01 and 117.22–133.48 ppm are assigned to aliphatic and aromatic carbon, respectively. The signals 161.23ppm and 162.80 ppm can be attributed to CH=N carbon.

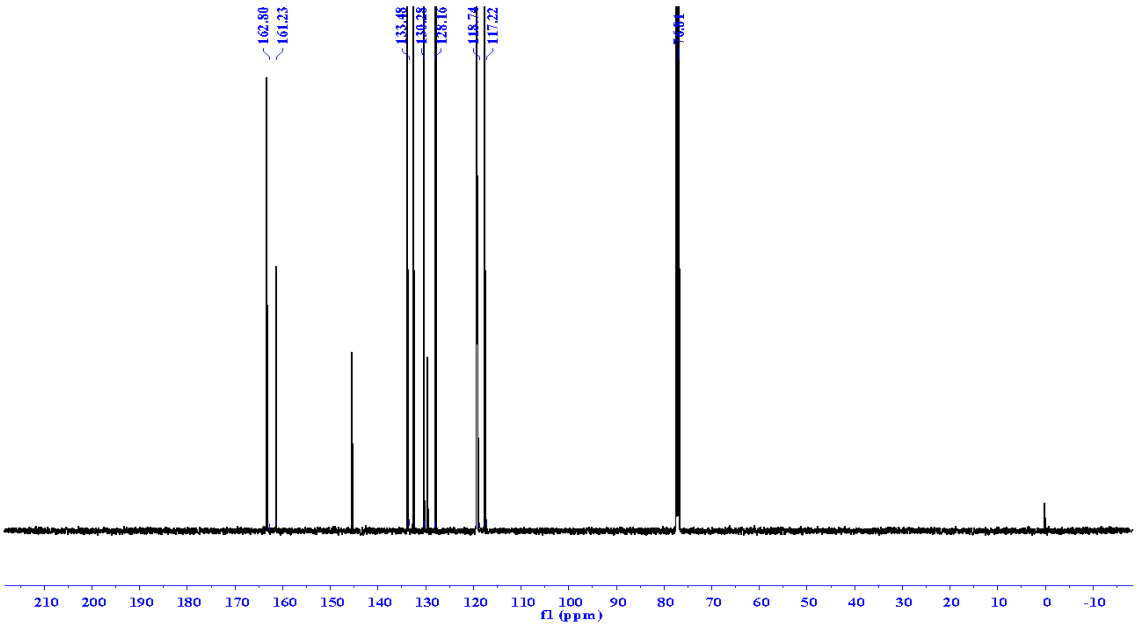

Fig. 7. ^{13}C-NMR spectrum of salicylidene-2-aminochlorobenzene

4. Conclusions

During this work, we have designed and synthesized a Schiff base of the type NO. The structure of the obtained compound was characterized using different spectroscopic techniques of FT-IR, UV-Visible,^{1}H-NMR and ^{13}C-NMR spectroscopy. The characterization methods confirm the presence of functional group imine (C=N) in this compound which act as bidentate Schiff base. Yellow crystals of the synthesized Schiff base was obtained with yield more than 98.73%, the Molar mass was calculated to be 231.45g/mole.

Acknowledgements

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Competing interests The authors declare no competing interests.

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