

XRD Analysis of Aniline based Schiff base ligand (4 - chloro-salicylidene Aniline)

Ankit Singh Chouhan ¹, Suprajnya Thakur ²

Department of Physics, SVIS, Shri Vaishnav Vidyapeeth Vishwavidhlaya, Indore, India^{1,2}

Abstract: Schiff's base and their metal complexes hold significant properties as catalysts in various biological systems, antimicrobial activities, polymers, dyes, antifungal activities, antitumor and antiviral activities insecticides. In the past scientists have been identified properties of Schiff bases and their metal complexes and also lots of work is going on to studied the unique properties of the Schiff bases. In this paper we have reviews the method of Synthesis of one of Schiff base ligand 4 - chloro-salicylidene Aniline (SB1). The synthesized ligand was characterized by XRD (X-Ray diffraction).

Keywords: Schiff base, Ligand, Aniline, XRD.

I. INTRODUCTION

Schiff bases are multipurpose ligands synthesized from the condensation of an amino compound with carbonyl compounds. German Scientist, Hugo Schiff were first synthesized Schiff bases (R-N=C-R') and so named. [1-3] Schiff's base and their metal complexes hold significant properties as catalysts in various biological systems, antimicrobial activities, polymers, dyes, antifungal activities, antitumor and antiviral activities insecticides. [4-5] Aniline is the simplest of the primary aromatic amines. Aromatic amines can be formed by reduction of the equivalent nitro compound, the ammonolysis of a phenol, and by direct amination of the aromatic ring. [6-7]

Looking into this, in this paper we have attempted to prepare and characterize one of Schiff base ligand using 4- chloro Aniline with salicylaldehyde.

X-ray diffraction (XRD) is a unique technique used for determination of crystallinity of a compound. XRD is largely used for Identification of crystalline material and polymorphic forms, Distinguishing between amorphous and crystalline material, and Quantification of the percent crystallinity of a sample. It is based on bragg's law that $2d \sin \theta = n\lambda$ (λ =wavelength, d =distance). For typical powder patterns, data is collected at 2θ from $\sim 5^\circ$ to 70° . [8]

II. EXPERIMENTAL

A. Synthesis of Schiff base

This ligand we take 1ml salicylaldehyde by micro pipette in 100ml flask. This was mixed with 10 ml of ethanol and then we added 1.281 gram 4- chloro aniline to this solution of salicylaldehyde. After this, we mix this solution with a glass rod for 4-5 minutes and magnetically stirred with the help of a stirrer for 2 hour at 350–380 rpm. After the whole process we get a dark yellow solution. We left this solution overnight at room temperature. Next day, the solid colored product formed was filtered and washed with ethanol. [9]

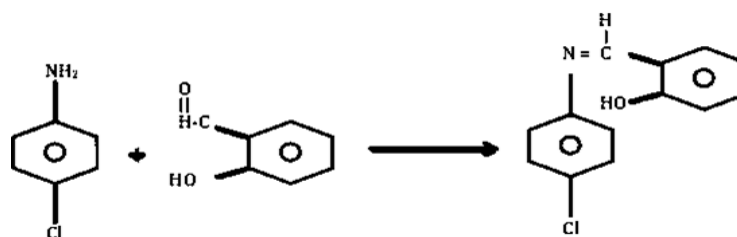


Fig 1: chemical equation for Synthesis of Schiff base

B. Characterizations

Prepared Schiff base (SB1) was characterized by X-Ray Diffraction (XRD). The Bruker D8 Advance X-ray diffractometer was used for XRD measurement. The range of 2θ is taken 10-70° with a step size 0.02. Wavelength of X-ray was taken for this measurement is 1.54 Å (Cu Kα radiation).

III. RESULT AND DISCUSSION

Figure-2 shows the XRD pattern of 4 - chloro-salicylidene Aniline (SB1). The major peaks of in the XRD spectrum for SB1 arising from various diffraction positions were assigned to different hkl planes assigned as (111), (200), (201), (210), (211), (202), (220), (301) and (311) appearing at 2θ values ~ 13.67°, 17.17°, 20.34°, 23.74°, 25.06°, 29.49°, 33.86°, 40.22°, and 46.60° respectively.[10-11]

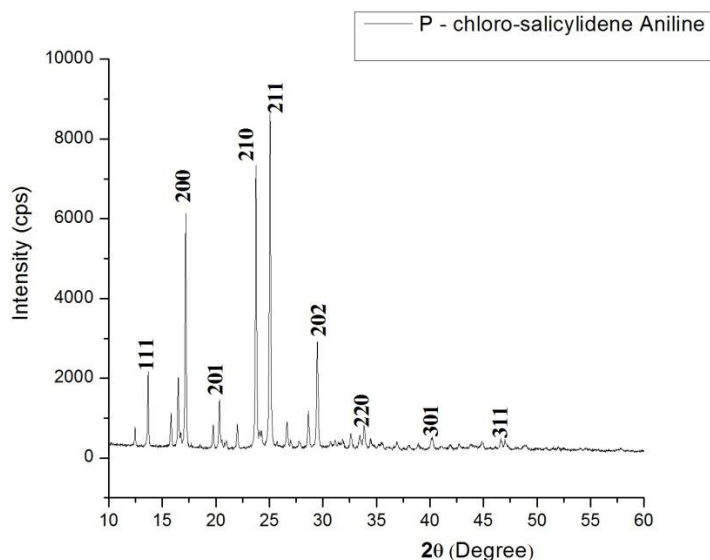


Fig. 2: XRD pattern of 4 - chloro-salicylidene Aniline Aniline

The average crystallite size of SB1 was estimated using the Scherer’s formula:

$$L = \frac{0.9\lambda}{B \cos(\theta)} \dots\dots\dots (1)$$

Where, B is half width (in radians), θ is the Bragg angle and λ is the wavelength.

The average crystallite size was found to be ~59.2927 nm.

The value of lattice parameter for the SB1 was determined by:

$$a^2 = \frac{\lambda^2 (h^2 + k^2 + l^2)}{4 \sin^2(\theta)} \dots\dots\dots (2)$$

The calculated lattice parameter value was 9.154 Å.

IV. CONCLUSION

In this paper we described a Chemical root method to synthesize the schiff base ligand. XRD result of SB1 shows that the nature of the SB1 is crystalline with CUBIC structure and the crystalline size and lattice parameter were found to be 59.2927 nm and 9.154 Å respectively.

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