2.1 Introduction

The syntheses of various precursor compounds used in this work of the present thesis are described. The different sources of chemicals, solvents and other materials used in the present work are also given. A brief account of various spectroscopic, diffraction and other techniques employed for the identification and structural characterization of the newly synthesized compounds and materials has been given in this Chapter 2.

2.2 Materials

Lignin, NiCl$_2$·6H$_2$O, PdCl$_2$, FeCl$_3$, FeSO$_4$·7H$_2$O were purchased from Sigma Aldrich, USA. Hydrazine hydrate, phenyl boronic acid, organic reagents and substrates for organic transformations were obtained either from Merck Ind. Ltd., or Spectrochem Ind. Pvt. Ltd. Organic solvents were supplied by Spectrochem, SD fine and Loba Ind. Pvt. Ltd., and/or other local supplier. Thin layer chromatography plates used were pre-coated aluminum sheets with silica gel 60 F (254) obtained from Merck Ind. Ltd. Aqueous solutions were made in double-distilled water.

2.3 Experimental

Literature methods were used for the preparation of precursor compounds as described below.

2.3.1 Preparation of standard 1.0 mM Ni(II) solution

NiCl$_2$·6H$_2$O (1.0 mmol) was dissolved in 10-20 mL double distilled water in a 50 mL beaker. This solution was transferred into a 100 mL standard volumetric flask and then the solution was made up to the mark. The resulted Ni (II) standard solution was shaken well for uniform concentration.

2.3.2 Preparation of standard 1.0 mM Pd(II) solution

Anhydrous PdCl$_2$ (1.0 mmol) was dissolved in 10-20 mL double distilled water in a 50 mL beaker. This solution was transferred into a 100 mL standard volumetric flask and then the solution was made up to the mark. The resulted Pd (II) standard solution was shaken well for uniform concentration.

2.3.3 Iron (II, III) Oxide (Fe$_3$O$_4$).

Iron (II,III) oxide is the chemical compound with formula Fe$_3$O$_4$. It occurs in nature as the mineral magnetite. It is one of a mixed of iron oxides, iron(II) oxide
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(FeO), which is rare and the other in iron (III) oxide (Fe₂O₃) also known as haematite. It contains both Fe²⁺ and Fe³⁺ ions and is sometimes formulated as FeO·Fe₂O₃. This iron oxide is encountered in the laboratory as a black powder. It exhibits permanent magnetism and is ferromagnetic, it’s most extensive use is as a black pigment which is synthesized rather than being extracted from the naturally occurring mineral as the particle size and shape can be varied by the method of production.

Fe₃O₄ has a cubic inverse spinel structure which consists of a cubic close packed array of oxide ions where all of the Fe²⁺ ions occupy half of the octahedral sites and the Fe³⁺ are split evenly across the remaining octahedral sites and the tetrahedral sites. The ferrimagnetism of Fe₃O₄ arises because the electron spins of the Fe(II) and Fe(III) ions in the octahedral sites are coupled and the spins of the Fe(III) ions in the tetrahedral sites are coupled but anti-parallel to the former. The net effect is that the magnetic contributions of both sets are not balanced and there is a permanent magnetism.

Fe₃O₄ is used as a black pigment and is known as C.I pigment black 11 (C.I. No.77499) or Mars Black Fe₃O₄ is used as a catalyst in the Haber process and in the water gas shift reaction. The latter uses an HTS (high temperature shift catalyst) of iron oxide stabilized by metal oxide and polymer. Nano particles of Fe₃O₄ are used as contrast agents in MRI scanning.

2.3.4 Synthesis of Fe₃O₄ nanoparticles (Fe₃O₄-NPs)

Fe₃O₄ nanoparticles were prepared [2] by chemical co-precipitation of Fe³⁺ and Fe²⁺ ions with a molar ratio of 2:1. Typically, FeCl₃ (4 g, 24.6 mmol) and FeSO₄·7H₂O (3.419 g, 12.3 mmol) were dissolved in 100 mL deionized (DI) water at 85 °C under N₂ atmosphere with vigorous mechanical stirring (500 rpm). Then, 10 mL of 2 N NaOH was quickly injected into the reaction mixture in one portion. This resulted in the formation of the black precipitate (ppt) of Fe₃O₄ immediately. The stirring was continued for another 25 min and the mixture was cooled to room temperature. Subsequently, this ppt was separated by external magnet and washed several times by DI water and then dried in open air resulting ultrafine powder of Fe₃O₄-NPs.
Lignin was first mentioned in 1813 by the Swiss botanist A. P. de Candolle, who described it as a fibrous, tasteless material, insoluble in water and alcohol but soluble in weak alkaline solutions, and which can be precipitated from solution using acid. He named the substance “lignine”, which is derived from the Latin word lignum, meaning wood. It is one of the most abundant organic biopolymers on Earth, exceeded only by cellulose. Lignin constitutes 30% of non-fossil organic carbon and 20-35% of the dry mass of wood. The Carboniferous Period (geology) is in part defined by the evolution of lignin.

Lignin is a constituent of the cell walls of almost all dry land plant cell walls. It is the second most abundant natural biopolymer in the world, surpassed only by cellulose. Of the polymers found in plant cell walls, lignin is the only one that is not composed of carbohydrate (sugar) monomers.

Lignin is unique in that it is the only large-scale biomass source of an aromatic functionality. It is composed of up to three different phenyl propane monomers, depending on the species. Coniferyl alcohol occurs in all species and is the dominant monomer in conifers (softwoods). Deciduous (hardwood) species contain up to 40% syringyl alcohol units while grasses and agricultural crops may also contain coumaryl alcohol units.

An additional complexity of lignin is that there are many possible bonding patterns between individual units. Thus our knowledge of lignin chemical structure is less precise than our knowledge of other natural and synthetic biopolymers. **Fig. 2.1** shows a representative lignin fragment containing the most important bonding patterns.
The use of biopolymer lignin is particularly advantageous in several aspects. It is stable, second most abundant carbohydrate based natural product only after cellulose thus readily available, environmentally benign. Lignin exhibits high surface area and possess excellent metal-adhesive properties due to the presence of various organic functional groups such as phenolic, alcoholic, alkene, carbonyl, carboxyl and other groups and the large intra- and inter-molecular spacing have also shown to be capable of binding and complexing with metal ions via cation-hydroxyl and cation-π-bonding. It has excellent capability of being structurally modified leading to the adjustment of its properties for specific purposes. Some biopolymers or their modified material has several biological properties for example lignin.

These abilities of lignin facilitate to complex with metal cations, reduce them to atomic metals and then to stabilize metal-NPs in an efficient manner. Bound lignin may also show additional functional properties such as reinforcement fillers, antioxidants, UV adsorbents, antimicrobial agents, carbon precursors and biomaterials for tissue engineering and gene therapy.

Lignin is the most common biopolymer represents about 1.1 million metric tons of the total annual biomass production. Lignin and its derivative are produced on an industrial sacles and used for coating, laminates, optical films as well as for property determine additives in building materials, pharmaceuticals, foodstuffs and cosmetics. However its use as support for catalytic applications is not well explored.
Its application as a reducing agent in materials synthesis has been increasing throughout the years, especially in the field of the nanomaterials synthesis. The application of phenolic compounds in gold and silver nanoparticles synthesis was reported in the literature and involves very quick and easy steps.

**Unique properties of lignin (Substitutes for classical inorganic supports)**

- Lignin is a complex, multifunctional carbohydrate based polymer
- Tumkur is the coconut city of Karnataka
- Cheap and Readily available
- Biodegradable.
- Flexible
- Non-toxic, Naturally occurring -Second most abundant
- Carrying freely available hydroxyl, alkenyl, phenolic, ether and other groups.
- Renewable
- Biocompatible
- Environmental benign

However, its use as a support for catalytic applications is not well explored

### 2.4 Characterization

#### 2.4.1 UV-Vis. Spectroscopy

The UV-Vis. spectrum of the lignin (Fig. 2.2) shows bonds at $\lambda$ max 285 nm (bond I) due to the transition localized within the ring of cinnamoyl system; whereas the one centered at 245 nm (bond II) is for absorbance of ring related to the benzoyl system. They are related to the $\pi\rightarrow\pi^*$ transitions and these absorbent bonds demonstrate the presence of polyphenolics in the category of flavonoids.
2.4.2 FT-IR Spectroscopy

The FT-IR Spectrum of the lignin is shown in Fig. 2.3. The bands obtained in the IR spectrum of lignin (ν, cm\(^{-1}\)) are 3418, 2935, 1595, 1512, 1462, 1426, 1373, 1269, 1221, 1127, 1079, 1030, 966, 855, 818, 620, 566; A broad band around observed at 3418 cm\(^{-1}\) was due to the presence of hydroxyl (-OH) groups. The aliphatic methyl and methylene C-H vibrational band was appeared at 2935 cm\(^{-1}\) and C-H deformation band at 1462 cm\(^{-1}\). Aromatic ring bands were found characteristic obtained at 1595 and 1512 cm\(^{-1}\). Bending vibrations of O-H group was obtained at 1373 cm\(^{-1}\) and C-O stretching and aromatic vibrations were appeared at 1269 and 1221 cm\(^{-1}\) respectively. Dialkyl ether linkages were seen at 1127 cm\(^{-1}\). C-O deformation of methoxy group was observed at 1030 cm\(^{-1}\). These peaks suggested the presence of flavonoid and other phenolics in the lignin.
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Fig. 2.3 FT-IR spectrum of Lignin
2.4.3 Powder X-Ray diffraction (PXRD) of Lignin

The powder X-Ray diffraction spectrum of lignin is given in the Fig. 2.4 shows an obvious diffusion peak at $2\Theta=15$-$25^\circ$ that appeared due to the existence of amorphous carbon.

![Fig 2.4 PXRD spectrum of Lignin](image)

2.4.4 Scanning Electron Microscopy (SEM) of Lignin

![Fig. 2.5 SEM images of Lignin](image)

2.4.5 FT-IR Spectrum of Fe$_3$O$_4$

The FT-IR Spectrum in the range of 4000-400 cm$^{-1}$ are given in Fig. 2.6. For the results of Fe$_3$O$_4$ composite, the peak signals at 580.46 cm$^{-1}$, 1632.45 cm$^{-1}$, 3442.31 cm$^{-1}$ corresponding to the Fe-O vibration of the Fe$_3$O$_4$ particles, the bending vibration of the O-H group, and the stretching vibration of the O-H group, respectively.
Fig. 2.6 FT-IR spectrum of Fe$_3$O$_4$-NPs.
2.4.6 Powder X-Ray diffraction (PXRD) of Fe$_3$O$_4$

The powder X-Ray diffraction spectrum of Fe$_3$O$_4$ is represented in Fig. 2.7. Six characteristic peaks observed at 2θ values 30.1, 35.3, 43.0, 53.3, 57.09 and 62.5° corresponding to the (220),(311),(400),(422),(511) and (440) planes in the PXRD Spectrum of Fe$_3$O$_4$ were appeared sharp and intense indicating their well crystalline structure matched well with the standard JCPDS data (74-748). The size of Fe$_3$O$_4$ nanoparticles was found around 10-15 nm calculated from Debye-Scherrer's equation.

![Fig.2.7 PXRD spectrum of Fe$_3$O$_4$-NPs](image)

2.4.7 Scanning Electron Microscopy (SEM) of Fe$_3$O$_4$

The SEM images of Fe$_3$O$_4$ composites are given in Fig.2.8 these images revealed the composite of Fe$_3$O$_4$ particle morphology.
2.5 Analytical methods used in the present work

A brief account of modern instrumental and analytical techniques used for the characterization compounds and catalytic nano-materials has been discussed. The chemical composition was determined by Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES) and Energy dispersive X-Ray analysis (EDX). The structure, surface morphology, size, optical and photophysical properties were determined by X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM), High Resolution Transmission Electron Microscopy (HR-TEM), UV-Visible Spectroscopy (UV-Vis.), Fourier Transform Infrared Spectroscopy (FT-IR) and Nuclear Magnetic Resonance (NMR) spectroscopy. The details of these techniques are given below.

2.5.1 UV-Vis. Spectroscopy

UV-Vis spectroscopy [5] or absorption spectroscopy is widely used in different fields such as chemistry, physics, biochemistry and nano science. It provides information about the electronic structure of the nanoparticles and materials containing NPs, higher energy radiation in the UV (200-400 nm) and visible (400-700 nm) range of the electromagnetic spectrum causes many organic molecules to undergo electronic transitions. In UV-Vis. spectroscopy the interaction of the UV-Vis. radiation with the sample causes electronic
transition from the ground (low-energy state) to the excited state (high-energy state). The energy difference between the ground and excited state affects the position of the maximum absorption band and the intensity of the molecules. Additionally, the interaction between the beam and electronic system could affect the intensity of the absorption band. A convenient expression which connects the absorbance with path length is Lambert-Beer Law. According to this law the concentration of the sample could be calculated by:

\[
\log \left( \frac{I}{I_0} \right) = \varepsilon c l
\]

Where \( I \) = the intensity of the transmitted radiation, \( I_0 \) = the intensity of the incident radiation, \( \varepsilon \) = the molar absorption coefficient, \( c \) = the concentration of absorbing species, \( l \) = the path length of the absorbing solution [6].

A strong and broad band could be detected in the UV-Vis. spectra for the metallic nanoparticles (> 2 nm). This is called a surface plasmon band (SPB), and it is also known as Mie resonances originated from Gustave Mie who is the most famous in SPB theory. The incoming light interaction with the nanoparticles results in a global scattering. Thus, the surface plasmon band is attributed to the collective electron charge oscillations of the excited metal nanoparticles by light.

2.5.2 Fourier Transform Infrared Spectroscopy (FT-IR)

Fourier transform infrared spectroscopy (FT-IR) technique have been extensively used for the identification present in the sample [7]. The basic principle of this technique implies that a molecule can exist in a variety of vibrational energy levels and can move from one level to another by absorption/release of energy, which is equivalent to the difference in energy of the two involved levels. The absorption/emission of an electromagnetic radiation accomplishes these transitions in the IR region (4000-400 cm\(^{-1}\)) and this forms a basis of vibrational spectroscopy. A particular transition between the two energy states usually ground state (\( E_0 \)) and the first excited state (\( E_1 \)) can be correlated by the following equations. From the fundamentals of IR spectroscopy the equation relating the force constant, the reduced mass and the frequency of absorption is given by:

\[
\tilde{\nu} = \frac{1}{2\pi c} \sqrt{\frac{k}{\mu}}
\]

\[
\Delta E = h\nu = E_1 - E_0 = \frac{hc}{\lambda} = h\tilde{\nu}
\]

\( \Delta E = h\tilde{\nu} \) where \( \Delta E \) is the energy difference between two energy levels, \( h \) is Planck's, \( c \) is the velocity of light and \( \tilde{\nu} \) in the wave number. As chemical bond is assumed to be harmonic
oscillator so from that concept V is the vibrational quantum number may be 0, 1, 2, 3......etc. The most commonly used range of infrared spectroscopy is between 4000 cm\(^{-1}\) at high frequency end and 400 cm\(^{-1}\) at lower frequency end. The range from 4000 to 1500 cm\(^{-1}\) is generally considered as the functional group region and all frequencies below 1500 cm\(^{-1}\) are considered characteristic of the ‘fingerprint’ region.

In the case of nano materials, this technique has been extensively used for identification of the framework structure of the materials, as well as various functional groups of the support and the active catalysts and to measure the surface acidity of the catalysts. [8-10]

2.5.3 Powder X-Ray diffraction

Powder X-Ray diffraction (PXRD) is a non-destructive technique and is one of most preliminary and powerful instrumental technique required for the characterization of nanoparticles and nano-structured materials to know about their crystalline and amorphous nature. The diffraction of X-Ray arises upon interaction with a periodic structure of crystalline material. In this technique, a fixed wave length (\(\lambda\)) is chosen for the incident radiation and Brag’s peaks are measured by observing the intensity of the scattered radiation as a function of scattering angle ‘2\(\theta\)’. By scanning the sample through a range of 2\(\theta\) angles, all possible diffraction patterns of the lattice should be attained due to the random orientation of the powdered material.

Conversion of the diffraction peaks to d-spacing allows identification of the material because each material has a set of unique d-spacing. Typically, this is achieved by comparison of d-spacing with standard reference patterns. The d spacing is calculated from the values of the peaks observed from the Bragg's equation (Equation 2.2). The position of the diffraction peaks gives information about the structure of the material. Successive planes and \(\theta\) is the angle between the incident ray and the scattering planes. Knowing \(\theta\), n and \(\lambda\), the lattice spacing d can be easily calculated.

\[
n\lambda = 2d \sin \theta \quad \text{--------- (2.2)}
\]

Where, \(n\) is the order of reflection and the values are 1, 2, 3... \(\lambda\) is the wave length of the X-Ray radiation, d is the inter planar spacing between two successive planes and \(\theta\) is the angle between the incident ray and the scattering planes.

The position and relative intensity of the lines in the X-Ray diffraction pattern serve as a finger print for a given type of crystalline material. By comparing an X-Ray diffraction
pattern against the patterns collected for known crystalline compounds, the crystallinity and amorphous nature material can be determined [11].

This technique also provides a wealth of useful information about the geometry of the crystal lattice, specific atoms and their arrangement in the unit cell of the crystal structure, degree of crystallinity of the sample, and allows qualitative identification of the crystalline phase. The position and relative intensity of the lines in the X-Ray diffraction pattern serve as a fingerprint for a given type of crystalline material. By comparing an X-ray diffraction pattern against the patterns collected for known crystalline compounds, the crystallinity and amorphous nature material can be determined. X-Ray line broadening of the peak shape of one or more diffraction lines can be used to estimate the crystal size in powder materials. As the particle size decreases, the reflections in the XRD pattern will be broadened. This correlation is used in Scherrer's equation (2.3) to calculate the particle size.

\[
D = \frac{K\lambda}{\beta(2\theta)\cos\theta}
\]

Where 
- \(D\) = mean crystallite diameter
- \(K\) = Scherrer's constant
- \(\lambda\) = X-Ray wave length (1.5418 Å for CuKα radiation)
- \(\beta\) = full width at half maximum
- \(\theta\) = Bragg's angle

In addition to crystallite size of the materials, X-Ray line broadening gives information about dispersion and the degree of metal oxide present at the surface of the support. The minimum detection limit for crystallite size is 4 nm and the two-dimensional metal oxide over layers cannot be detected by XRD. In the present course of work, XRD pattern of the support and catalysts were obtained on a Bruker D8 diffractometer, with nickel filtered Cu Kα radiation (\(\lambda=1.5418\) Å) with an applied voltage and current of 40 kV and 20 mA respectively. Bruker D8 diffractometer has two detectors viz. Scintillation counter detector and lynx eye super speed detector. Scintillation counter detector has been used for the analysis of samples in which 20 value start below 1° known as low angle XRD pattern. On the other hand, lynx eye super speed detector has been used to acquiring the wide angle XRD pattern. [12-21].
2.5.4 Energy dispersive X-Ray analysis (EDX)

EDAX is an X-Ray technique used to identify the elemental composition of materials [25]. From the interactions of the electrons with the sample, X-Ray characteristic emissions are produced and detected by an energy dispersive detector. The detector absorbs the coming X-Rays by ionization, which then transforms into an electrical signal; thus, the characteristic X-Rays coming from the different elements in the sample are separated and identified. The result is an energy spectrum with the characteristic peaks of the elements present in the analyzed specimen. In this way it is possible to obtain an estimation of the chemical composition of the sample.

2.5.5 Scanning Electron Microscopy (SEM)

Scanning electron microscopy technique is one of the most popular and widely used techniques for the characterization of nanomaterials and nanostructure. SEM can be effectively used to characterize specimens down to a resolution of a few nanometres, with images magnifications achievable in the range of ~ 10 to over 300,000. In addition to information on surface topography. SEM can also provide useful information on chemistry, crystal orientation and internal stress distribution. SEM consists of an electron gun emit electron that are focused into a beam, with a very fine spot size of ~ 5nm. Electrons are accelerated to energy values in that ranges of a few 100 eV to 50 KeV . It produces images of the sample by scanning it in a raster pattern on the specimen surface with a focused beam of electrons. The interaction between the electron beam and the specimen surface produces various types of energetic emissions, including back scattered electrons, secondary electrons, auger electrons, continuous X-Rays, and characteristics X-Rays. The electrons interact with atoms in the sample, producing various signals that can be detected and that contain information about the samples surface topography and composition [22,23]. The image displayed on the cathode ray tube comes from the secondary and back scattered electrons. The secondary electrons are the excited electrons emitted from the specimen due to bombardment of the electron beam with the atom. The SEM also produces images of high resolution, which means that closely spaced features can be examined at a high magnification.

Preparation of the samples is relatively easy since most SEM require the sample to be conductive. The combination of higher magnification, larger depth of focus, greater resolution, and ease of sample observation makes the SEM one of the most important tool
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used in research areas today. Scanning electron micrographs were obtained using a JEOL Model JSM - 6390LV with an energy dispersive spectrometer (EDS) attachment. Because the SEM utilizes vacuum conditions and uses electrons to form an image, special preparations must be done to the sample. All water must be removed from the samples because the water would vaporize in the vacuum. All metals are conductive and require no preparation before being used. All non-metals need to be made conductive by covering the sample with a thin layer of conductive material. Samples for SEM were prepared by adding a very minute amount of the finely powered samples onto a carbon tape. Then the samples was coated with a film of gold and then mounted over the probe for scanning.

2.5.6 Transmission Electron Microscopy (TEM)

Transmission electron microscopy can provide microcrystal structure as well as micro-chemical information with high spatial resolution from each of the microscopic phases individually. TEM is therefore a very powerful tool frequently used for the detailed examination of nano-structured materials. It measures the quantitative particle or grain size, size distribution, and morphology (such as shape, geometry, and dimensions) of the nano-structured materials [24]. Further, in the analysis of microporous materials, TEM techniques give a clear indication of ordered structure with long narrow channels and ordered pore openings. The basic principle of the TEM is same as of the light microscope, but it uses electron instead of light. Transmission electron microscopes use electrons as light source and their much reduced wavelength make it possible to achieve resolutions of one thousand times better than with a light microscope.

In this technique, a beam of electrons is transmitted through a sample containing specimen and images are formed from the interaction of the electrons. Then, the image is magnified and focused onto a fluorescent screen with the help of electromagnetic lenses or detected by sensor such as a charge couple device (CCD) camera. In terms of magnification and resolution, TEM has an advantage compared to SEM. TEM has up to a 50 million magnification level while SEM only offers 2 million as a maximum level of magnification. The resolution of TEM is 0.5 angstroms while SEM has 0.4 nanometres. However, SEM images have a better depth of field compared to TEM produced images. Another point of difference is the sample thickness, “staining,” and preparations. The sample in TEM is cut thinner in contrast to a SEM sample. In addition, SEM sample is “stained” by an element that captures the scattered electrons.
2.5.7 Inductively Coupled Plasma - Atomic Emission Spectrometry (ICP-AES)

ICP-AES is an emission spectrophotometric technique; the principle of ICP-AES is that the excited electrons emit energy at a given wavelength as they return to ground state after excitation by high temperature Argon plasma. The fundamental characteristic of this process is that each element emits energy at specific wavelengths peculiar to its atomic character. The energy transfer for electrons when they fall back to ground state is unique to each element as it depends upon the electronic configuration of the orbital. The energy transfer is inversely proportional to the wavelength of electromagnetic radiation, although each element emits energy at multiple wavelengths, in the ICP-AES technique it is most common to select a single wavelength (or a very few) for a given element. The intensity of the energy emitted at the chosen wavelength is proportional to the concentration of that element in the sample being analysed. Thus, by determining which wavelengths are emitted by a sample and by determining their intensities, the analyst can qualitatively and quantitatively find the elements from the given sample relative to a reference standard. The wavelengths used in AES ranges from the upper part of the vacuum ultraviolet (160 nm) to the limit of visible light (800 nm). As borosilicate glass absorbs light below 310 nm and oxygen in air absorbs light below 200 nm, optical lenses and prisms are generally fabricated from quartz glass and optical paths are In present study Inductively Coupled Plasma Atomic Emission Spectroscopic (ICP-AES) analysis (model: PS 3000 uv, (DRE), Leeman Labs, Inc., USA) was carried out for analysing the presence of metals in catalyst.

2.5.8 NMR Spectroscopy

Nuclear Magnetic Resonance is a powerful tool for investigating nuclear structure. Nuclear Magnetic Resonance is a branch of spectroscopy in which radio frequency waves induce transitions between magnetic energy levels of nuclei of the molecule [26]. NMR spectroscopic technique arises with radio frequency induced transitions between magnetic energy levels of nuclei of a molecule, in which magnetic energy levels are created by keeping nuclei in magnetic field. NMR technique offers information regarding the detailed structure and configuration of organic molecules. $^1$H and $^{13}$C ($^1$H) used for the determination of chemically and magnetically non–equivalent proton and carbons respectively [4,27]. Chemical shift values will be given in $\delta$ ppm with TMS as an internal standard.
2.6 References


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