2.1 INTRODUCTION

Synthesis of materials at nanometer scale has already become routine practice for supported metal catalysts after decades of research on the subject. However, there is much room for the development of design and control. Nanotechnology has gained substantial popularity recently due to the rapidly developing techniques both to synthesize and characterize materials at the nanoscale, as well as the promises that such technology offers to substantially expand the achievable limits in many different fields including medicine, chemistry, and engineering. In the literature, there are many reports of new researches about interesting phenomena of nanomaterials and their applications. Nano size metal particles had occupied a central place in heterogeneous catalysis for many years long before their recognition in nanotechnology. Thus, it is fitting to critically evaluate the impact of such development on heterogeneous catalysis.\textsuperscript{1,2}

Schiff base ligands are condensation products of primary amines and carbonyl compounds and they were discovered by a German chemist, Nobel Prize winner, Hugo Schiff in 1864.\textsuperscript{3} Schiff bases are the compounds carrying imine or azomethine (C=N) functional group.\textsuperscript{4} The chemistry of Schiff base ligands has been gaining considerable interest primarily because of their fascinating structural diversities.\textsuperscript{5–9} Schiff base ligands are able to coordinate with various metals, and to stabilize them in various oxidation states, enabling the use of Schiff base metal complexes for a large variety of useful catalytic transformations. The preparative ease and variable geometries of the metal complexes makes them important stereochemical models in transition metal coordination chemistry.

Metal complexes, in which a single central metal atom or ion is surrounded by Schiff base ligands, play an important role in catalysis, especially for elements of the $d$–block. The atom in the Lewis base ligand that forms the bond to the central atom is called the donor atom because it donates the electrons used in bond formation. The metal atom or ion, the Lewis acid in the complex, is the acceptor atom. Schiff bases
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composed of \( \text{N}_2\text{O}_2 \) donor atoms are important ligands for designing medicinal and catalytically useful metal complexes.\textsuperscript{10,11} In recent years, there have been numerous reports of their use in homogeneous and heterogeneous catalysis.\textsuperscript{12–15}

Since last few years, the synthesis, characterization and catalytic activities of the metal complexes entrapped in the cavities of zeolites have been extensively investigated due to their industrial significance.\textsuperscript{16–19} These organic–inorganic hybrid materials promise to combine the advantages of both homogeneous and heterogeneous catalysts as the metal complex can be synthesized within the framework and is constrained therein, not by intra–nor intermolecular forces that might perturb the metal complex but because it is too large to escape through the pores. Thus, the metal complex, once formed inside the cavity, does not diffuse out and pass in the liquid phase during the catalytic reaction. Additionally, entrapment of the metal complex can, in principle, provide a stabilizing effect by prohibiting molecular deactivation pathways.\textsuperscript{20–22}

2.2 REAGENTS

All solvents were of AR grade, purchased from Merck (India) and used as received without further purification. For the synthesis of Schiff base ligands, 2–aminobenzoic acid, 3–nitrobenzaldehyde, 4–hydroxybenzaldehyde, 2–hydroxy–benzaldehyde, \( \alpha \)–phenylenediamine, and 4,4’–diaminodiphenylmethane were purchased from Aldrich (Germany). Transition metal salts viz., VO\( \text{SO}_4 \cdot 5\text{H}_2\text{O} \), MnSO\( \text{4} \cdot \text{H}_2\text{O} \), FeSO\( \text{4} \cdot 7\text{H}_2\text{O} \), NiSO\( \text{4} \cdot 6\text{H}_2\text{O} \), and CuSO\( \text{4} \cdot 5\text{H}_2\text{O} \) were purchased from Merck (India). While, the sodium form of zeolite–Y (Si/Al = 2.60) was purchased from Hi–media (India) for the synthesis of metal exchanged zeolite–Y, and entrapped complexes. However, zeolite–Y usually tend to absorb moisture, which must be eliminated upon careful drying of the zeolite material before being used as host material for entrapment of complexes. 30% hydrogen peroxide (H\( \text{2O}_2 \)), cyclopentanone, and cyclohexanone were purchased from Aldrich (Germany) for the purpose of catalytic activities of the synthesized materials.
2.3 SYNTHESIS OF SCHIFF BASE LIGANDS

2.3.1 Synthesis of Schiff base ligands 'nbab' and 'hbab'

Schiff base ligands $(Z)-2-((3\text{-nitrobenzyllidene)amino})\text{benzoic acid}^{23}$ (nbab) and $(Z)-2-((4\text{-hydroxybenzyllidene)amino})\text{benzoic acid}^{24}$ (hbab) were synthesized by heating a mixture of 2-aminobenzoic acid (1.37 g, 10 mmol) with 3-nitrobenzaldehyde (1.51 g, 10 mmol)/ 4-hydroxy benzaldehyde (1.22 g, 10 mmol) in 50 mL of ethanolic medium with constant stirring under reflux condition. The solid product formed was filtered off and dried in an oven (Scheme 2.1).

![Scheme 2.1 Synthesis of Schiff base ligands 'nbab' and 'hbab'.](image)

2.3.2 Synthesis of Schiff base ligands 'pamp' and 'mpamp'

For the synthesis of Schiff base ligands $2,2'-(1E,1'E)-(1,2-\text{phenylenebis(azanylylidene)})\text{bis(methanylylidene)})\text{diphenol}^{25}$ (pamp) and $2,2'-(1E,1'E)-(4,1-\text{phenylene})\text{bis(azanylylidene)})\text{bis(methanylylidene)})\text{diphenol}^{26}$ (mpamp), a mixture of 2-hydroxybenzaldehyde (2.10 mL, 20 mmol) with o-phenylenediamine (1.08 g, 10 mmol)/ 4,4'-diaminodiphenylmethane (1.98 g, 10 mmol) in 25 mL of ethanol were added to a round bottom flask. Then the resulting mixture was heated at 80 °C for 2 h on an oil bath with continuous stirring. The solid product formed was filtered off and dried in an oven (Scheme 2.2).
2.4 SYNTHESIS OF NEAT COMPLEXES

2.4.1 Synthesis of neat complexes [M(nbab)$_2$] and [M(hbab)$_2$]

An aqueous solution of 10 mmol transition metal salt M(II) (Where, M = MnSO$_4$·H$_2$O, FeSO$_4$·7H$_2$O, NiSO$_4$·6H$_2$O or CuSO$_4$·5H$_2$O) was added to an methanolic solution of 20 mmol ligand nbab/ hbab with constant stirring. The resulting solution was refluxed at 80 °C for 4–5 h. Sodium acetate was added to adjust the pH of the solution around 5–6. Then the mixture was cooled and filtered under reduced pressure. The collected solid was washed with diethylether and dried in air to give coloured [M(nbab)$_2$] and [M(hbab)$_2$] complexes (Scheme 2.3 and 2.4).

2.4.2 Synthesis of neat complexes [M(pamp)] and [M(mpamp)]

An aqueous solution of 10 mmol transition metal salt M(II/IV) (where, M = VOSO$_4$·5H$_2$O, MnSO$_4$·H$_2$O, FeSO$_4$·7H$_2$O, or CuSO$_4$·5H$_2$O) was added to 10 mmol ligand pamp/ mpamp solution in 1,4–dioxane with constant stirring. The resulting solution was refluxed at 80 °C for 4–5 h. Sodium acetate was added to adjust the pH of the solution around 5–6. Then the mixture was cooled and filtered under reduced pressure. The collected solid was washed with diethylether and dried in air to give coloured [M(pamp)] and [M(mpamp)] complexes (Scheme 2.5 and 2.6).
Scheme 2.3 Synthesis of neat complexes derived from ‘nbab’ Schiff base ligand.

Scheme 2.4 Synthesis of neat complexes derived from ‘hbab’ Schiff base ligand.
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Scheme 2.5 Synthesis of neat complexes derived from ‘pamp’ Schiff base ligand.

Scheme 2.6 Synthesis of neat complexes derived from ‘mpamp’ Schiff base ligand.
2.5 SYNTHESIS OF METAL EXCHANGED ZEOLITE–Y

Transition metal ion M(II/IV) was exchanged into zeolite–Y framework by an ion exchange method as reported earlier. A 4.0 g of zeolite–Y was first ion–exchanged with 12 mmol transition metal salt M(II/IV) (VOSO$_4$·5H$_2$O, MnSO$_4$·H$_2$O, FeSO$_4$·7H$_2$O, NiSO$_4$·6H$_2$O or CuSO$_4$·5H$_2$O) in deionized water with continuous stirring under refluxed condition for 24 h. The resulting solid material was washed with deionised water till the filtrate was free from any metal ion content, and dried at 150 °C for 4 h. The resulting metal exchanged zeolite–Y were denoted as VO(IV)–Y, Mn(II)–Y, Fe(II)–Y, Ni(II)–Y, and Cu(II)–Y (Scheme 2.7).
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Scheme 2.7 Synthesis of metal exchanged zeolite–Y $[M({\text{II/IV}})–Y]$. 

Where,  

$\text{A} = \text{Zeolite–Y}$  

$\text{B, C, D, E, F} = \text{Metal exchanged zeolite–Y}$
Zeolite–Y entrapped complexes have been synthesized by the formation of the metal complexes inside the cavities of the zeolite–Y via FL method (*Scheme 2.8 – 2.11*). A 1.0 g activated metal exchanged zeolite–Y [M(II/IV)–Y] was added to a solution of an excessive amount of ligand *nbab, hbab, pamp* or *mpamp* in 1,4-dioxane (50 mL). The resulting mixture was heated at 120 °C with continuous stirring for 12 hours, which gives VO, Mn, Fe, Ni, and Cu entrapped complexes. Uncomplexed ligand and the complex molecules adsorbed on the external surface of zeolite material were removed through Soxhlet extraction with 1,4-dioxane and acetonitrile until the filtrate became colorless. The resulting material was dried and further washed with an aqueous solution of 10 mmol NaCl to discard undesired free metal ions. Subsequently, it was washed with deionized water to remove any chloride ions present, and dried in an oven at 120 °C overnight under reduced pressure (*Scheme 2.12*). The resulting catalysts were designated as [M(*hbab*)₂]–Y, [M(*nbab*)₂]–Y, [M(*pamp*)]–Y, and [M(*mpamp*)]–Y.
Scheme 2.8 Synthesis of zeolite–Y entrapped [M(nbab)_2]–Y transition metal complexes.

Where, M = Mn(II), Fe(II), Ni(II), or Cu(II)

nbab = Schiff base ligand

A = Metal exchanged zeolite–Y

B, C, D, E = Zeolite–Y entrapped transition metal complexes
Scheme 2.9 Synthesis of zeolite–Y entrapped [M(hbab)$_2$]–Y transition metal complexes.

Where, $M = \text{Mn(II), Fe(II), Ni(II), or Cu(II)}$

$hbab = \text{Schiff base ligand}$

$A = \text{Metal exchanged zeolite–Y}$

$B, C, D, E = \text{Zeolite–Y entrapped transition metal complexes}$
Scheme 2.10 Synthesis of zeolite–Y entrapped [M(pamp)]–Y transition metal complexes.

Where, $M$ = VO(IV), Mn(II), Fe(II), or Cu(II)

$pamp$ = Schiff base ligand

$A$ = Metal exchanged zeolite–Y

$B$, $C$, $D$, $E$ = Zeolite–Y entrapped transition metal complexes
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Scheme 2.11 Synthesis of zeolite–Y entrapped [M(mpamp)]–Y transition metal complexes.

Where,
- \( M \) = VO(IV), Mn(II), Fe(II), or Cu(II)
- mpamp = Schiff base ligand
- A = Metal exchanged zeolite–Y
- B, C, D, E = Zeolite–Y entrapped transition metal complexes
In summary, entrapment of metal complexes in the cavities of zeolite–Y involve the exchange of transition metal ions $M(II/IV)$ with Na–Y in deionised water to form $M(II/IV)$–Y species, followed by the reaction of the $M(II/IV)$–Y with ligand in 1,4–dioxane to give $[M(L)]$–Y. Soxhlet extraction was carried out with acetonitrile, methanol, chloroform, and 1,4–dioxane solvents to remove the uncoordinated ligand. The uncomplexed metal ions in zeolite were removed by ion exchange with aqueous solution of 10 mmol NaCl. The resulting materials were designated as zeolite–Y entrapped transition metal complexes (Scheme 2.12).

Scheme 2.12 The graphical representation for the synthesis of zeolite–Y entrapped transition metal complexes.
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2.7 PHYSICO–CHEMICAL TECHNIQUES

A variety of physico–chemical techniques have been used for the characterization of the zeolite–Y entrapped complexes to get a better understanding of entrapment, the physico–chemical properties of the complex, distribution of the complex in the host, the influence of the host–guest interaction on the geometry of the complex, the stability of the entrapped complex compared to that of the neat complex, retaining of the zeolite–Y framework even after entrapment, and so on. The following techniques were used for the characterization of synthesized materials.

2.7.1 Nuclear Magnetic Resonance (NMR) spectroscopy

NMR spectroscopy is a powerful analytical technique that can reveal structural information about many organic and inorganic molecules. In NMR, magnetic nuclei of specific isotopes are aligned by a strong external magnet and then perturbed by a weaker radio wave. This external energy applied to the molecule is absorbed, and the perturbed nucleus is said to be "in resonance". The resonance frequency is observed as re–emitted energy, and is related to the identity, quantity, position, and intra–molecular relationships occurring within the analyzed substance. Nuclei that have a Spin Quantum Number of ½ typically give the best spectra. Some of these nuclei are $^1$H, $^{13}$C, $^{19}$F, and $^{31}$P.

$^1$H and $^{13}$C NMR spectra of all the Schiff base ligands were recorded using acetone–d₆, methanol–d₄, and chloroform–d as a solvent and TMS as an internal standard on a model Advance 500 Bruker NMR Spectrometer.

2.7.2 Molar conductivity

Transition metal complexes can behave as an electrolyte in an appropriate solvent if it possesses ionizable counter ions outside the coordination entity of the complexes. In order to find out the electrolytic nature of neat complexes, the molar conductivity was measured on Metrohm 712 Conductometer by using DMF solvent.
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2.7.3 Elemental analysis

The technique used for the determination of CHN is based on the quantitative “dynamic flash combustion” method. The samples are held in a tin container, placed inside the autosampler drum where they are purged with a continuous flow of helium and then dropped at present intervals into a vertical quartz tube maintained at 900 °C (combustion reactor). When the samples are dropped inside the furnace, the helium stream is temporarily enriched with pure oxygen, and the sample and its container melt and the tin promotes a violent reaction in a temporary enriched atmosphere of oxygen. Under these favourable conditions even thermally resistant substances are completely oxidized.

Quantitative combustion is then achieved by passing the mixture of gases over a catalyst layer. The mixture plug of combustion gases is then passed over copper to remove the excess of oxygen and to reduce the nitrogen oxides to elemental nitrogen.

The resulting mixture is directed to the chromatographic column where the individual components are separated and eluted as nitrogen (N₂), carbon dioxide (CO₂), and water (H₂O) with the help of a Thermal Conductivity Detector whose signal feeds the automatic workstation known as Eager 200. The instrument is calibrated with the analysis of standard compounds. All results for elemental analysis are calculated based on a known value of a standard by using the K value factors calculation. This K value is determined by analyzing an organic standard of a known elemental composition. The elemental analysis of the synthesized materials was carried out on Perkin Elmer, USA 2400–II CHN analyzer.

2.7.4 Inductively Coupled Plasma–Optical Emission Spectrometry (ICP–OES)

The ICP–OES separates the light emitted from the plasma into its discrete component wavelengths using a diffraction grating. Each element in the periodic table has its own distinct set of emission wavelengths. ICP–OES can quantitatively measure the elemental content of a material from the parts per trillion range to the weight percent range. The only elements which cannot be measured by ICP methods are C, H, O, N, halogens and the noble gases. In order to analyze a solid sample, it is typically immersed in a highly acidic solution. After the sample is immersed, it will dissolve or
"digest," which allows for analysis of the sample solution. The content of Na(I), Al(III), Si(IV), V(IV), Mn(II), Fe(II), Ni(II), and Cu(II) ions in zeolite–Y modified materials was determined by ICP–OES using Perkin Elmer optima 2000 DV model.

2.7.5 Brunauer–Emmett–Teller (BET) and Barrett–Joyner–Halenda (BJH) Analysis

BET analysis provides precise specific surface area evaluation of materials by nitrogen multilayer adsorption measured as a function of relative pressure using a fully automated analyzer. The technique encompasses external area and pore area evaluations to determine the total specific surface area in m² g⁻¹ yielding important information in studying the effects of surface porosity and particle size in many applications. BJH analysis can also be employed to determine pore area and specific pore volume using adsorption and desorption techniques.

Prior to the BET measurements, samples were de–gasified at 125 °C for 2 h to remove any adsorbed gases. The specific surface area and pore volume were obtained from nitrogen adsorption–desorption isotherms measured by a multipoint BET method using ASAP 2010, micrometrics surface area analyzer. The data are treated according to the Brunauer, Emmett and Teller (BET) adsorption isotherm equation:

\[
\frac{1}{V_a \left( \frac{p_o}{p} - 1 \right)} = \frac{C - 1}{V_m C} \times \frac{p}{p_o} + \frac{1}{V_m C}
\]

Where,

- \( P \) = partial vapour pressure of adsorbate gas in equilibrium with the surface at 77.4 K (b.p. of liquid nitrogen), in pascals,
- \( P_o \) = saturated pressure of adsorbate gas, in pascals,
- \( V_a \) = volume of gas adsorbed at standard temperature and pressure (STP) [273.15 K and atmospheric pressure (1.013 × 10⁵ Pa)], in mL,
- \( V_m \) = volume of gas adsorbed at STP to produce an apparent monolayer on the sample surface, in mL,
- \( C \) = dimensionless constant that is related to the enthalpy of adsorption of the adsorbate gas on the powder sample.
2.7.6 Scanning Electron Microscopy (SEM)

The SEM instrument uses a focused beam of high-energy electrons to generate a variety of signals at the surface of solid specimens. The signals that derive from electron-sample interactions reveal information about the sample including external morphology, chemical composition, crystalline structure and orientation of materials making up the sample. In most applications, data are collected over a selected area of the surface of the sample, and a 2-dimensional image is generated that displays spatial variations in these properties. Area ranging from approximately 1 cm to 5 microns in width can be imaged in a scanning mode using conventional SEM techniques (magnification ranging from 20X to approximately 30,000X, spatial resolution of 50 to 100 nm).

SEM analysis is considered to be "nondestructive"; that is, x-rays generated by electron interactions do not lead to volume loss of the sample, so it is possible to analyze the same materials repeatedly. The morphology of zeolite-Y entrapped complexes was observed before and after Soxhlet extraction by a SEM instrument LEO 1430 VP.

2.7.7 Powder X-ray diffraction (XRD) patterns

XRD is now a common technique for the study of crystal structures and atomic spacing. The principle of powder XRD patterns is based on the interaction of X-rays with polycrystalline material, which acts as a diffraction grating. Incident radiation of fixed wavelength was chosen for the purpose and the diffraction pattern was obtained by observing the intensity of scattered radiation as a function of scattering angle 2θ. The wavelength of incident X-ray beam, the angle of diffraction θ and the interplanar distance d are related by Bragg’s equation:

\[ n\lambda = 2d \sin \theta \]

The characteristic x-ray diffraction pattern generated in a typical XRD analysis provides a unique “fingerprint” of the crystals present in the sample. When properly interpreted, by comparison with standard reference patterns and measurements, this fingerprint allows identification of the crystalline form.
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X-ray diffraction patterns of zeolite-Y modified materials were measured on a Bruker AXS D8 Advance X-ray powder diffractometer with a Cu Kα target and movable detector, which scans the intensity of diffracted radiation within the range of 5° to 70° as a function of the angle 2θ between the incident and diffracted beams. This technique is beneficial to check if the crystallinity of zeolitic matrix remain intact or not upon entrapment of metal complexes.

2.7.8 Fourier Transform Infrared (FT-IR) spectroscopy

FT-IR technique is mainly useful for obtaining information on the basic characteristics of a molecule such as identification of various functional groups, the nature of atoms and their chemical linkages. The frequencies and intensities of IR bands can thus provide information about their structure.

Since every type of bond has a different frequency of vibration, and since two of the same type of bond in two different compounds are in two slightly different environments, no two molecules of different structures have exactly the same infrared absorption pattern, or infrared spectrum. Although some of the frequencies absorbed in the two cases might be the same, in no case of two different molecules will their infrared spectra (the patterns of absorption) be identical. Thus, IR spectrum can be used for molecules such as fingerprint can be used for humans.

FT-IR spectra (4000–400 cm⁻¹) of synthesized materials were recorded on a Thermo Nicolet IR200 FT-IR spectrometer in KBr.

2.7.9 Ultraviolet–Visible (UV–Vis) spectroscopy

The UV–Vis spectra of transition metal complexes arise as a result of electronic transitions just as they do in organic compounds. There can be more than one type of electronic transitions, or excitation, taking place depending upon the nature of the chromophore(s) involved.

The spectrum that we see is the combination of the different types of transitions as they occur within the compound. The electronic transitions that principally give rise to absorption in the visible region and are therefore, responsible for the color of transition metal complexes. These are known as d–d transitions and...
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relate to excitation of the metal ion itself. Transition metals are often defined as forming one or more stable ions with incompletely filled d–orbitals. It is believed that these are involved in generating color.32

When ligands bound to a transition metal ion to form a complex, electrons in the ligands and electrons in the five d–orbitals of the metal ion repel each other. The net effect is that the energies of the d–orbitals are raised; however, they are split into two groups of differing energy. The size of the energy gap between the two groups of d–orbitals will vary with the transition metal ion, its oxidation state and the nature of the ligands. The further apart the groups are split, the greater the energy required to promote an electron (and the shorter the wavelength). Ammonia ligands, for instance, will cause greater splitting than water ligands. Electronic spectra were recorded in the range of 200–1000 nm on UV–1800 spectrophotometer, SHIMADZU using a quartz cell of 1 cm³ optical path. However, the diffuse reflectance (DR) UV–Vis spectra of complexes were recorded on UV reflectance spectrometer (Model: LAMDA 19 UV/VIS/NIR) in the solid phase at room temperature.

2.7.10 Thermogravimetric analysis (TGA)

TGA monitors the mass change of a material as a function of temperature and time in a controlled atmosphere. It is ideally used to assess the volatile, organic, and inorganic content of a material. In addition, the thermal stability, degradation characteristics, lifetime breakdown, and reaction kinetics may also be assessed.

In TGA, a temperature program is applied to a sample located in a sensitive microbalance (capable of detecting weigh changes as small as 0.1 μg). The temperature may then be programmed to increase and the weight loss is monitored. The weight loss of the sample on heating is being recorded as a function of temperature. Typically, the experiments were performed in N₂ atmosphere at a heating rate 10 °C/min in the temperature range 30–700 °C by using Perkin Elmer equipment.
2.7.11 Gas Chromatography Mass Spectrometry (GC–MS)

GC–MS is an instrumental technique, comprising a gas chromatograph (GC) coupled to a mass spectrometer (MS), by which complex mixtures of chemicals may be separated, identified and quantified. The sample solution is injected into the GC inlet where it is vaporized and swept onto a capillary pack column by the carrier gas (usually helium). The sample flows through the column and compounds comprising the mixture of interest are separated by virtue of their relative interaction with the coating of the column (stationary phase) and the carrier gas (mobile phase). The latter part of the column passes through a heated transfer line and ends at the entrance to the ion source where compounds eluting from the column are converted into ions.

Two potential methods exist for ion production. The most frequently used method is electron ionization (EI) and the alternative is chemical ionization (CI). Mass spectra are routinely obtained at an electron beam energy of 70 eV. The simplest event that occurs is the removal of a single electron from the molecule in the gas phase by an electron of the electron beam to form the molecular ion, which is a radical cation. Many of these molecular ions disintegrate in $10^{-10}$ – $10^{-3}$ s to give, in the simplest case, a positively charged fragment and a radical. A number of fragment ions are thus formed, and each of these can cleave to yield smaller fragments. If some of the molecular ions remain intact long enough to reach the detector, we see a molecular ion peak. It is important to recognize the molecular ion peak because this gives the molar mass of the compound. With unit resolution, this mass is the molecular weight to the nearest whole number.

A mass spectrum is a presentation of the masses of the positively charged fragments (including the molecular ion) versus their relative abundance. The most intense peak in the spectrum, called the base peak, is assigned a value of 100%, and the intensities (height $\times$ sensitivity factor) of the other peaks, including the molecular ion peak, are reported as percentages of the base peak. Of course, the molecular ion peak may sometimes be the base peak. Each compound has a unique or near unique mass spectrum that can be compared with a mass spectral database and thus may be identified. Through use of standards, quantitation is also possible. The GC–MS were recorded on a SHIMADZU, QP–2010 equipped with a RTX–5 column.
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2.7.12 Atomic Absorption Spectroscopy (AAS)

In essence, flame AAS involves generating a gaseous population of free atoms by heating a sample in a flame and then passing narrow bandwidth light at a certain wavelength through the atoms in the flame. These conditions result in absorption of radiation that is selective for a particular element. Absorbance is measured and Beer’s Law, which defines the simple linear relationship between absorbance and concentration, is applied to enable quantitative analysis of the sample for the particular element under analysis.

Spectroscopy concerns the interaction of light with matter. When light is absorbed by matter the outcome is an increase in the energy of the molecules or atoms that comprise the sample. In particular, absorption of radiation by the atoms of an element demonstrates itself as a spectrum that is characteristic of the element.

2.7.13 Thin layer chromatography (TLC)

Reaction mixture can be examined by TLC to assess whether the reaction is complete or not. If any impurity is there, then it shows extra spots and this can be detect easily. TLC for Schiff base ligands was performed using mixed solvent system (methanol + chloroform 7:3; ethyl acetate + hexane 2:8).

2.7.14 Soxhlet extractor

Soxhlet extraction of zeolite–Y entrapped transition metal complexes was performed to remove uncoordinated ligands using various solvents such as acetonitrile, chloroform, methanol, and 1,4–dioxane.

2.7.15 Oil bath for catalytic study

The catalytic activity of synthesized materials was performed over oxidation of cyclic ketones using DBK Digital Multi Magnetic Stirrer oil bath.

2.4.16 Melting point apparatus

The melting point of Schiff base ligands and neat metal complexes were measured with Griffin Melting Point apparatus.
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REFERENCES