

CHAPTER 2

EXPERIMENTAL

2.1 MATERIALS

The chemicals used for the synthesis of metal-containing mesoporous MCM-41 molecular sieves were sodium metasilicate nonahydrate (Qualigens), zinc acetate dihydrate (Merck), ferric nitrate nonahydrate (Merck), cobalt nitrate hexahydrate (Merck), chromium nitrate hexahydrate (Merck), manganese acetate tetrahydrate (Merck), nickel nitrate hexahydrate (Merck), titanium trichloride (Merck), ruthenium trichloride trihydrate (Merck), palladium chloride (Merck) and antimony trichloride (Merck) as the source of silicon, zinc, iron, cobalt, chromium, manganese, nickel, titanium, ruthenium, palladium and antimony, respectively. Cetyltrimethylammonium bromide (CTAB) (Qualigens) was used as the structure-directing agent. Sulphuric acid (Merck) was used to adjust the pH of the medium.

Pluronic P123 triblock co-polymer [poly (ethylene glycol)-block-poly (propylene glycol)-block-poly (ethylene glycol)] with molecular weight of 5800 ($\text{EO}_{20}\text{PO}_{70}\text{EO}_{20}$) and n-butanol (n-BuOH) purchased from Aldrich were used as the structure directing agent and co-surfactant respectively. Tetraethylorthosilicate (TEOS) was used as a source of silicon for the synthesis of KIT-6. Hydrochloric acid (wt. % = 35) was purchased from Merck and used as a co-solvent for the synthesis of metal-containing mesoporous 3D cubic KIT-6 molecular sieves. MgO (Merck) was used as a supporting material for the synthesis of MWCNTs. The gases namely

acetylene (99.9 %), nitrogen (99%) and hydrogen (99%) were used as carbon source, carrier gas and reducing agent respectively. Commercial grade of acids such HF, HCl, HNO₃ and H₂SO₄ were purchased from Merck and were used for the purification and functionalization of CNTs. Solvents such as acetone and ethanol were purchased from Merck and double distilled water was used throughout this study.

Diglycidylether of Bisphenol-A (Araldite LY 556) and Trietha tetramine (Aradur HY 951) purchased from Javanthee Enterprises, Chennai, India, were used as the epoxy resin and hardener, respectively. These were used for the fabrication of nanocomposites. All the above said chemicals were of AR (Analar/Analytical) grade and used without any further purification.

2.2 SYNTHESIS OF MESOPOROUS MOLECULAR SIEVES

2.2.1 Synthesis of Si-MCM-41

Si-MCM-41 molecular sieves were synthesized using the gel-composition of SiO₂ : 0.2CTAB : 0.89H₂SO₄ : 120H₂O by the direct hydrothermal method (Beck et al 1992). In a typical synthesis, 28.42 g of sodium metasilicate was dissolved in distilled water and was stirred for 1 h using a mechanical stirrer. The pH of the solution was adjusted to 10.5 by adding 4N H₂SO₄ with constant stirring to form a gel. After 1 h, an aqueous solution of CTAB (6.73 g in 22 mL of distilled water) was added drop by drop and then the mixture was stirred for 1 h at room temperature. The suspension was transferred into a 300 mL stainless steel autoclave which was then sealed and heated in a hot air oven at 145 °C for 48 h. After cooling to room temperature, the product was filtered and washed with distilled water which was dried at 100 °C for 6 h. The dried material was calcined at 550 °C in air for 5 h in a muffle furnace to expel the template.

2.2.2 Synthesis of Fe-MCM-41, Zn-MCM-41 and Zn-Fe-MCM-41

The mesoporous Fe-MCM-41 with various Si/Fe ratios (50, 75, 100 and 125), Zn-Fe-MCM-41 (100) with different Fe:Zn ratios (1:4, 1:3, 1:2, 1:1, 2:1, 3:1 and 4:1) and Zn-MCM-41 with various Si/Zn ratios (50, 75, 100 and 125) were synthesized hydrothermally using the gel composition of $\text{SiO}_2 : x\text{M} : 0.2\text{CTAB} : 0.89\text{H}_2\text{SO}_4 : 120\text{H}_2\text{O}$ (where $\text{M} = \text{Fe}, \text{Zn}$ and $\text{Fe}+\text{Zn}$ and $x =$ various ratios of Fe, Zn and Fe+Zn) according to Savidha and Pandurangan (2004). In a typical synthesis procedure, sodium metasilicate in water was combined with an appropriate amount of metal salts in distilled water and the pH of the solution was adjusted to 10.5 by adding 4N H_2SO_4 with constant stirring to form a gel. After 1 h, an aqueous solution of CTAB was added to it and the mixture was stirred for 1 h at room temperature. The suspension was then transferred into a 300 mL stainless steel autoclave which was further sealed and heated in a hot air oven at 145 °C for 48 h. After crystallization, Fe-MCM-41, Zn-MCM-41 and Zn-Fe-MCM-41 materials were recovered by filtration, washed with distilled water and dried at 100 °C for 5 h. Finally the catalysts were obtained after removing the occluded surfactant by calcining the sample at 550 °C in atmospheric air for 5 h.

2.2.3 Synthesis of Cr-MCM-41 and different Metal loaded Cr-MCM-41

Cr-MCM-41 molecular sieves with different Si/Cr ratios of 50, 75, 100 and 125 were synthesized hydrothermally by adopting the above procedure (Sec. 2.2.2) using a gel composition of $\text{SiO}_2 : x\text{Cr} : 0.2\text{CTAB} : 0.89\text{H}_2\text{SO}_4 : 120\text{H}_2\text{O}$ (where x is various Si/Cr ratios). Transition metals such as Fe, Co, Mn, Ni, Ti, Ru and Pd of 0.2 wt. % was loaded individually over Cr-MCM-41 (100) molecular sieves by wet impregnation method. In a typical procedure, the appropriate amount of iron nitrate was dissolved in distilled water and sonicated for 15 min; the sonicated solution was added drop by

drop to the calcined Cr-MCM-41(100) under constant stirring. The solution was dried under reduced pressure and calcined in atmospheric air at 550 °C for 4 h (Vetrivel and Pandurangan 2005). Similar procedure was adopted for Co, Mn, Ni, Ti, Ru and Pd to obtain respective metal loaded Cr-MCM-41.

2.2.4 Synthesis of Sb loaded Si-MCM-41

Various wt. % of antimony trichloride (wt. % of Sb = 1.0, 2.0, 3.0, 5.0, 10.0, 15.0 and 20.0) were dissolved in distilled water at 20 °C and loaded individually over already synthesized Si-MCM-41 using the wet impregnation method by adopting the above procedure which was followed for loading Fe on Cr-MCM-41 (Sec. 2.2.3).

2.2.5 Synthesis of Fe-KIT-6 and Zn loaded Fe-KIT-6

The mesoporous 3D cubic Fe-KIT-6 molecular sieves with Si/Fe ratios of 50, 75 and 100 samples were synthesized hydrothermally using the gel composition of TEOS : 0.017P123 : 1.83HCl (wt. % = 35) : 1.31n-BuOH : 195H₂O (Kleitz et al 2003). The typical procedure for the synthesis of Fe-KIT-6 is as follows: 6 g of Pluronic P123, 217 g of distilled water and 11.8 g of HCl were taken in a polypropylene bottle. The mixture was stirred for 3 h at 35 °C to make the template dissolved in distilled water and HCl. To this, 6 g of n-BuOH was added under constant stirring at 35 °C. After 1 h stirring, 12.9 g of TEOS and appropriate amount of ferric nitrate were added simultaneously to the homogeneous solution at 35 °C. The resulting mixture was stirred for 24 h at 35 °C and subsequently heated for 24 h at 100 °C in hot air oven under static condition in a closed polypropylene bottle. The solid products obtained after hydrothermal treatment were filtered without washing and dried for 5 h at 100 °C in atmospheric air. The dried material was ground

well and then calcined at 550 °C for 12 h in atmospheric air (Heating rate: 5 °C/min) to expel the template.

The different wt. % (0.25, 0.5 and 0.75) of Zn was loaded individually over Fe-KIT-6 using the wet impregnation method. In a typical procedure, an appropriate amount of zinc acetate was dissolved in distilled water and sonicated for 15 min. The sonicated solution was added drop by drop with mesoporous 3D cubic Fe-KIT-6 under constant stirring at room temperature. The solution was dried under reduced pressure and then calcined at 550 °C for 4 h in atmospheric air.

2.2.6 Synthesis of Fe and Zn loaded MgO

In order to load different wt. % (1.0, 2.0 and 3.0) of Fe and Zn (Fe and Zn ratio = 3:1) on MgO, metal salts were dissolved in distilled water and loaded on MgO by adopting the above said wet impregnation method.

2.3 SYNTHESIS, PURIFICATION AND FUNCTIONALIZATION OF CNTs

2.3.1 Description of CVD Apparatus

The SWCNTs and MWCNTs were synthesized using metal containing mesoporous materials by CVD method. The CVD apparatus basically consists of a horizontal reaction tube, a heating element, a controller and a flow meter and valves connected to respective gas sources. A schematic diagram of CVD apparatus for CNTs growth is shown in Figure 2.1. The horizontal reaction tube was made of quartz. The resistance heater was kept in the straightly grooved area in ceramic container, so that the heating coil can go around the quartz tube. The horizontal tube goes through the heating coils is fixed on the either support. The Process Integral Derivative (PID)

programming and current control were manually operated. Temperature was controlled by PID programmable controller with K-type thermocouple placed in the middle of the horizontal furnace. Set and current temperatures were displayed on the controller unit and the current is automatically adjusted by the controller until temperature gets stabilized. Even after the set value is stabilized, heat loss caused by gas flow occurs. This loss is corrected by the controller. An electronic digital flow meter is connected to the gas cylinders with gas line by manual operation.

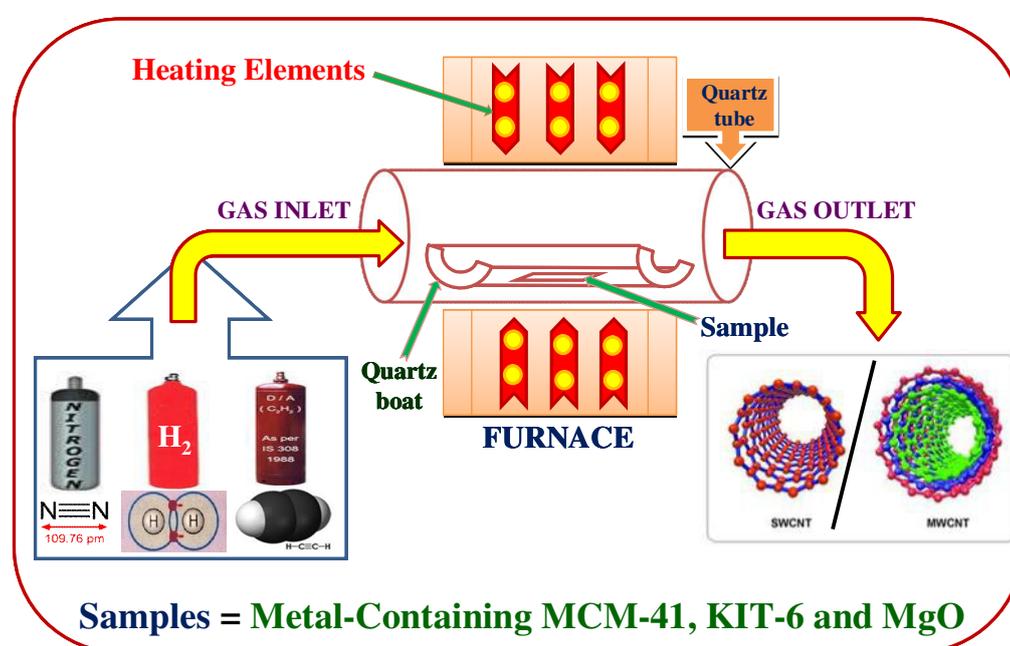


Figure 2.1 Schematic diagram of CVD reaction setup

2.3.2 Synthesis of CNTs using Mesoporous Materials

The catalytic reaction for the synthesis of CNTs were carried out using different metal-containing mesoporous MCM-41 and KIT-6 molecular sieves by CVD method (Kong et al 1998, Somanathan and Pandurangan 2009 and Somanathan et al 2011). The catalytic reactions for the synthesis of SWCNTs and/or MWCNTs were carried out using Fe-MCM-41

(Si/Fe ratios = 50, 75, 100 and 125), Zn-MCM-41 (Si/Zn ratios =50, 75, 100 and 125), Zn-Fe-MCM-41 (Si/(Fe + Zn) = 100 with Fe:Zn ratios: 1:4, 1:3, 1:2, 1:1, 2:1, 3:1 and 4:1), Cr-MCM-41 (Si/Cr ratios = 50, 75, 100 and 125), M/Cr-MCM-41 (M = Fe, Co, Mn, Ni, Ti, Ru and Pd), Sb/MCM-41 (wt. % of Sb = 1.0, 2.0, 3.0, 5.0, 10.0, 15.0 and 20.0), Fe/Zn/MgO, Fe-KIT-6 (Si/Fe ratios = 25, 50, 75, 100 and 125) and Zn loaded Fe-KIT-6 (wt. % of Zn = 0.25, 0.5 and 0.75) molecular sieves by CVD method. In a typical growth experiment, 200 mg catalyst was placed in a quartz boat inside a quartz tube. Before starting the experiment, the quartz tube reactor was flushed with nitrogen to ensure the removal of oxygen. The furnace was heated till the required reaction temperature at the rate of heating was 5 °C/min. The catalyst was purged with nitrogen gas at a flow rate of 100 mL/min for 30 min to remove water as well as to pre-treat the catalyst and then with hydrogen gas at a flow rate of 100 mL/min for 30 min to reduce the metal particles. The reaction was carried out using acetylene as the carbon precursor with various temperatures (700, 750, 800, 850 and 900 °C) at the flow rate of 100 mL/min for 30 min. The furnace was then cooled to room temperature under nitrogen atmosphere and the final product formed was a black material after the completion of the reaction. It usually takes about 3–4 h to cool the reaction tube to room temperature.

2.3.3 Activity of Mesoporous Materials for the Growth of CNTs

The activity of the mesoporous catalytic templates were evaluated by measuring the deposition of carbon, reaction yield and CNT selectivity on them and they were calculated using the equations (2.1), (2.2), (2.3) (Fonseca et al 1996 and Ciambelli et al 2005)

$$\text{Carbon deposition yield (\%)} = \frac{m_{\text{tot}} - m_{\text{cat}}}{m_{\text{cat}}} \times 100 \quad (2.1)$$

where m_{cat} = Weight of the catalyst taken before CNT growth
 m_{tot} = Weight of the catalyst and CNT grown on the catalyst

$$\text{Reaction yield (\%)} = \frac{m_{\text{cd}}}{m_{\text{ci}}} \times 100 \quad (2.2)$$

where m_{cd} = Deposited carbon mass
 m_{ci} = Mass of carbon fed

$$\text{CNT selectivity (\%)} = \frac{m_{\text{cnt}}}{m_{\text{tot}}} \times 100 \quad (2.3)$$

where m_{cnt} = Mass of CNTs
 m_{tot} = Total mass of deposited carbon.

2.3.4 Purification of CNTs

The as-synthesized CNTs contain the mesoporous materials, metal particles and a small amount of carbonaceous impurities like amorphous carbon and microcrystalline carbon. A purification scheme was designed to remove all of these undesired impurities. In a typical purification experiment, for the removal of silicate layer, about 200 mg as-synthesized material was mixed with 10 mL of HF (40%) and 30 mL of distilled water (Zheng et al 2002). Then the resultant mixture was stirred for 5 h and then centrifuged to remove the silica and HF. The CNTs obtained still contained many metal nanoparticles, presumably because the nanoparticles are coated by carbon layers. For further purification, air oxidation was carried out to remove the carbonaceous impurities and liquid phase oxidation treatment which includes the usage of 5 mL concentrated HNO_3 was used to dissolve the metal oxide that is formed during the purification process. It is believed that oxidants breach the carbon shell and oxidize the metal catalysts to the corresponding

metal oxide or hydroxide (Chiang et al 2001). When the metal is oxidized, the volume increases and the metal oxides cracks to open the carbon coatings. Furthermore, oxidants tend to initially attack CNTs from the end, which have highly strained five-membered rings. However, amorphous carbon can be attacked from all sides leading to its preferential removal over CNTs. All of these oxidation processes were accompanied by dissolution and leaching out of the metal/metal oxide impurities (often necessitating a separate procedure to remove the metal itself), generally yield relatively pure CNTs. Such oxidation steps makes the carbon coating found on the catalyst particles exposed to acid attack which subsequently burns. This leaves the nanotubes unaffected because of the higher stability of NTs against oxidation compared with amorphous carbon. Thus the purified product consists of mainly CNTs in bundle form. Finally, the purified CNTs were calcined at 450 °C (especially SWCNTs at 400 °C) for 2 h in atmospheric air in order to remove the carbonaceous impurities on the surface of the CNTs.

2.3.5 Functionalization of MWCNTs

About 500 mg of purified MWCNTs were mixed with 160 mL of 1:3 ratios of HNO₃ and H₂SO₄ mixture. The final mixture was refluxed at 80 °C for 48 h under constant stirring. After cooling, the final mixture was filtered and then washed with distilled water. The precipitate was dried at 70 °C for 10 h in hot air oven. The nature of the obtained f-MWCNTs samples in powder form were analyzed by FT-IR and SEM techniques.

2.4 FABRICATION OF NEAT EPOXY, MWCNTs/EPOXY AND f-MWCNTs/EPOXY NANOCOMPOSITES

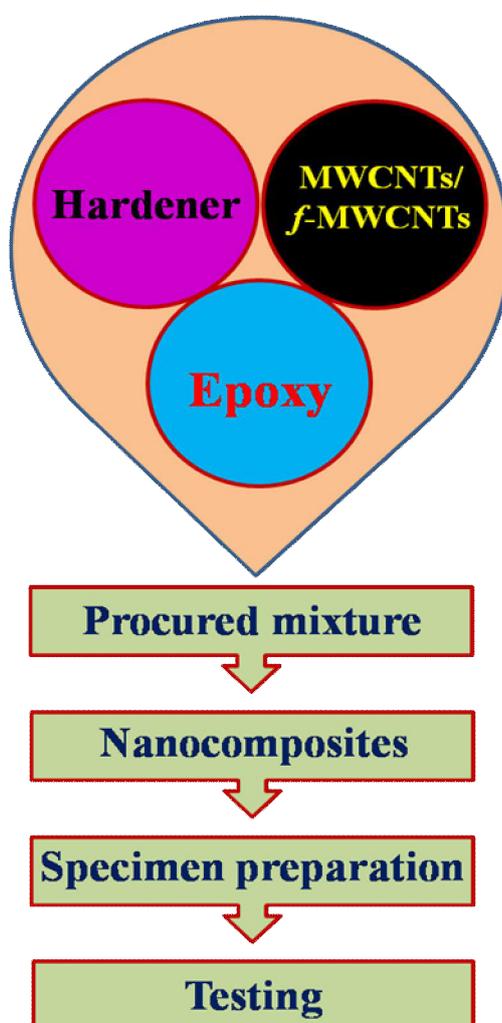
To prepare epoxy nanocomposites, Araldite LY 556 was taken because of its high mechanical properties, very low cure shrinkage and its

dimensional stability. Aradur HY 951 was used as a hardener in order to perform curing at room temperature or temperature between 50 and 120 °C for shorter time. It gives laminates excellent water resistance. MWCNTs and f-MWCNTs were used as filler materials for the fabrication of nanocomposites. Neat epoxy and MWCNTs/Epoxy nanocomposites were prepared by casting method. In a typical synthesis procedure, 10 g of hardener and 100 g of epoxy resin was mixed thoroughly. The mixture was sonicated for 5 min and then poured into a stainless steel mould. Wax was spread on the mould evenly before pouring into the mould. The mould with the sample was cured at 100 °C for 3 h. It was cooled to room temperature and then epoxy resin sheet was removed from steel mould.

In order to achieve better dispersion, MWCNTs were first added to minimum amount of acetone under sonication process and then these treated MWCNTs were dispersed in the epoxy resin under intensive sonication to overcome the clustering nature of MWCNTs. Appropriate amount of hardener was added into the MWCNTs dispersed epoxy resin and mixed thoroughly. This mixture was also sonicated for 10 min. Wax was spread on the mould evenly before pouring into the mould in order to prevent unnecessary binding and shrinkage. The resultant mixture was poured into the stainless steel mould. The mould with the sample was cured at 100 °C for 3 h in hot air oven. After that it was cooled to room temperature and then MWCNTs/Epoxy matrix was removed from stainless steel mould. Similar procedure was adopted for fabrication of f-MWCNTs/Epoxy nanocomposites. The nanocomposites with different wt. % of MWCNTs or f-MWCNTs (wt. % = 0.5, 1.0 and 1.5) were prepared which is listed in Table 2.1. Schematic diagram of the above said procedure was briefly given in Figure 2.2.

Table 2.1 Compositions of fabricated epoxy nanocomposites

Wt. % of filler	Composite
0	Neat Epoxy
0.5	MWCNTs/Epoxy and f-MWCNTs/Epoxy
1.0	MWCNTs/Epoxy and f-MWCNTs/Epoxy
1.5	MWCNTs/Epoxy and f-MWCNTs/Epoxy

**Figure 2.2 Methodology of nanocomposites fabrication**

2.5 SPECIMEN PREPARATION FOR TESTING

Specimens were prepared from the neat epoxy, MWCNTs/Epoxy and f-MWCNTs/Epoxy nanocomposites as per the ASTM standards for thermo-mechanical investigations. Standard tensile test specimens with a dimension of 165 mm X 19 mm X 3 mm were cut from the composite plate and test was conducted in UTM as per ASTM D3039. Flexural test specimen with a dimension of 127 mm X 12.7 mm X 3 mm were cut from the composite plate and was tested in three point loading system applied by a simply supported beam as per ASTM D790. Standard hardness test specimens with a dimension of 173 mm X 56 mm X 42 mm were cut from the composite plate and test was conducted in UTM as per ASTM D2240. Flammability of neat epoxy and MWCNTs/Epoxy nanocomposites were studied by using flame retardant test. The method uses ASTM D635 standard (a modified version of ASTM D3801). Rectangular bars measuring 13 mm wide, 76 mm long and 3 mm thick were used as the test specimen.

2.6 CHARACTERIZATION TECHNIQUES

The above synthesized materials were characterized by various physico-chemical techniques such as ICP-AES, XRD, N₂ sorption isotherms, TGA, FT-IR, DRS-UV, SEM, HR-TEM, Flame retardant test, Tensile strength test, Flexural strength test, Hardness test and Raman spectroscopy to establish the structure, surface area, thermal stability, co-ordination environment and morphology.

2.6.1 ICP-AES Analysis

The amount of metals (Metals = Fe, Zn and Cr) present in the MCM-41 and KIT-6 decides the activity of the catalysts. The amount of metal

loaded in the MCM-41 and KIT-6 were analyzed and determined by Inductive Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES, Perkin Elmer OPTIMA 3000). The samples were dissolved in a mixture of HF and HNO₃ before the measurement.

2.6.2 X-ray Diffraction Analysis

The XRD patterns of all the samples were recorded on a PANalytical X'Pert diffractometer, using nickel-filtered CuK α radiation ($\lambda = 1.54 \text{ \AA}$) and a liquid nitrogen-cooled germanium solid-state detector. The diffractograms were recorded in the 2θ range of $0.5\text{--}10^\circ$ for mesoporous materials and $5\text{--}80^\circ$ for CNTs at the scanning rate of 0.02° with a counting time of 5s at each point. The peaks were identified with reference to compilation of simulated XRD powder patterns. The d-spacing values were calculated by the Bragg's equation (2.4).

$$n\lambda = 2d \sin\theta \quad (2.4)$$

where 'n' represents the order of reflection, ' λ ' represents the wavelength of the incident X-ray beam and 'd' represents the spacing between reflecting crystal planes.

2.6.3 Nitrogen Sorption Isotherms

The surface area, pore volume and pore size distribution were measured by nitrogen sorption at -197°C using an ASAP-2010 porosimeter from Micromeritics Corporation. The samples were degassed at 473 K and 10^{-5} Torr 8–10 h prior to the adsorption experiments. The mesopore volume was estimated from the amount of nitrogen adsorbed at a relative pressure of 0.5 by assuming that all the mesopores were filled with condensed nitrogen in

the normal liquid state. Pore size distribution was estimated using the BJH algorithm (ASAP-2010) available as built-in software from Micromeritics.

The surface area of the samples was calculated by applying the BET equation (2.5).

$$\frac{P/P_0}{V(1-P/P_0)} = \frac{1}{V_m C} + \left(\frac{C-1}{V_m C} \right) \frac{P}{P_0} \quad (2.5)$$

where, V is the volume of nitrogen gas adsorbed at equilibrium pressure P, P₀ is the saturated vapour pressure of nitrogen gas in equilibrium with liquid nitrogen (77K), V_m is the monolayer volume and C is a constant depending on the heat of liquefaction (Q_l) and heat of adsorption (Q_a) of nitrogen gas. From the adsorption isotherm, V_m was obtained and from that specific surface area was calculated.

Pore size distribution was computed from the desorption branch of isotherm using BJH analysis by adopting Kelvin equation (2.6).

$$\ln \frac{P}{P_0} = \frac{-2\sigma V \cos \theta}{r_k R} \quad (2.6)$$

Where,

R is the gas constant

θ is the wetting angle

σ is the surface tension

r_k is Kelvin radius

V is the volume adsorbed.

The pore volume was estimated from the amount of nitrogen adsorbed at a relative pressure of 0.9 by assuming that all the pores were filled with condensed nitrogen in the normal liquid state.

2.6.4 Thermogravimetric Analysis

TGA and DTG of the materials were performed simultaneously on a high-resolution TA Instrument, SDT Q600. TGA measurements were carried out under atmospheric air. About 15 mg of the catalytic sample and nanocomposite samples (cut into small pieces and were machined using the mechanical grinder) were used in each experiment. The sample was heated in air at a heating rate of 20 °C/min in the temperature range from 35 to 1000 °C.

2.6.5 FT-IR Spectroscopy

Mid-infrared spectra of mesoporous molecular sieves were recorded on a PerkinElmer (Spectrum RX1) instrument using a KBr pellet technique. About 4 mg of the sample was ground with 200 mg of spectral grade KBr to form a mixture, which was then made into a pellet using a hydraulic press (under a pressure of 5 ton/cm²) into a self-supported wafer of 13 mm diameter. This pellet was used for recording the infrared spectra in the range 400–4000 cm⁻¹.

2.6.6 DRS-UV Measurements

DR-UV Visible spectroscopy was used to confirm the presence of metal ions in the framework of mesoporous materials. The DRS of all the catalysts were recorded in UV-Visible spectrophotometer (Shimadzu UV 2450 model) in the range of 200–800 nm equipped with an integrating sphere and powdered BaSO₄ used as reference.

2.6.7 Scanning Electron Microscopy

The resolution in the SEM was determined by the size of the incident electron beam and beam-specimen interaction. SEM was performed

on a JEOL with beam energy of 4 kV. To improve contrast, SEM samples were coated with metallic layer using metals Au or Pt. All the samples were suspended in ethanol or acetone and the specimen stub was dipped into the liquid and removed. The powder got evenly deposited onto the surface of the stub when ethanol evaporated. This specimen was coated with gold for two minutes using an ion sputter coater.

2.6.8 Transmission Electron Microscopy

The samples for TEM analysis were initially dispersed in ethanol or acetone by ultrasonication for 30 min which was then allowed to settle. A drop of the supernatant liquid was transferred on to a carbon coated copper grid and mounted onto the transmission electron microscope (JEOL 3010) operated at 300 kV and the TEM images were recorded.

2.6.9 Raman Spectroscopy

The Raman scattering was excited in infrared at 1064 nm with Nd: YAG laser, equipped with liquid nitrogen cooled charge-coupled array detector at 200 mV. The diameter of SWCNTs was estimated from the widely used relationship (2.7) (Jorio et al 2001) and/or (2.8) (Bandow et al 1998).

$$d = 248/\omega \quad (2.7)$$

or

$$d = 224/\lambda \quad (2.8)$$

where,

d is the diameter (nm) of SWCNTs

ω is the RBM Raman shift (cm^{-1})

λ is the RBM Raman shift (cm^{-1})

Raman spectra were recorded with a Micro-Raman system RM 1000 Renishaw using a laser excitation line at 532 nm (Nd-YAG), 0.5 to 1 mW, with 1 μ m focus spot in order to avoid photodecomposition of the samples.

2.6.10 Flame Retardant Test

Flammability of nanocomposites was studied by flame retardant test. The rate of burning for the nanocomposite material was found by burning it in horizontal position as per ASTM D635. The test specimen (3 mm X 13 mm X 76 mm) was kept over the blue flame and the flame diminishing time was determined.

2.6.11 Mechanical Studies

The mechanical properties were investigated for neat epoxy matrix, MWCNTs/Epoxy and f-MWCNTs/Epoxy nanocomposites by UTM as per ASTM standards. The tensile strength of the neat epoxy matrix and the nanocomposites were investigated by using UTM (Model-H50. K-S Hounsfield test equipment Ltd, UK). Typically, the testing involves placing the sample over a fixed cross-section area and then pulling it with a controlled grip by gradual increasing force until the sample changes shape or breaks. The cross head speed was 2 mm/min and the gauge length of the specimen was 150 mm. The tensile strength was evaluated as per ASTM D3039 for nanocomposites.

Flexural strength, also known as modulus of rupture, bend strength, or fracture strength, a mechanical parameter for brittle material, is defined as a material's ability to resist deformation under load. The transverse bending test is most frequently employed, in which a rod specimen having either a circular or rectangular cross-section is bent until fracture using a three point

flexural test technique (Figure 2.3) as per ASTM D790. The flexural strength represents the highest stress experienced within the material at its moment of rupture. It was measured in terms of stress (σ) according to equation (2.9).

$$\sigma = 3FL/2bd^2 \quad (2.9)$$

where,

F is the load (force) at the fracture point

L is the length of the support span

b is width

d is thickness

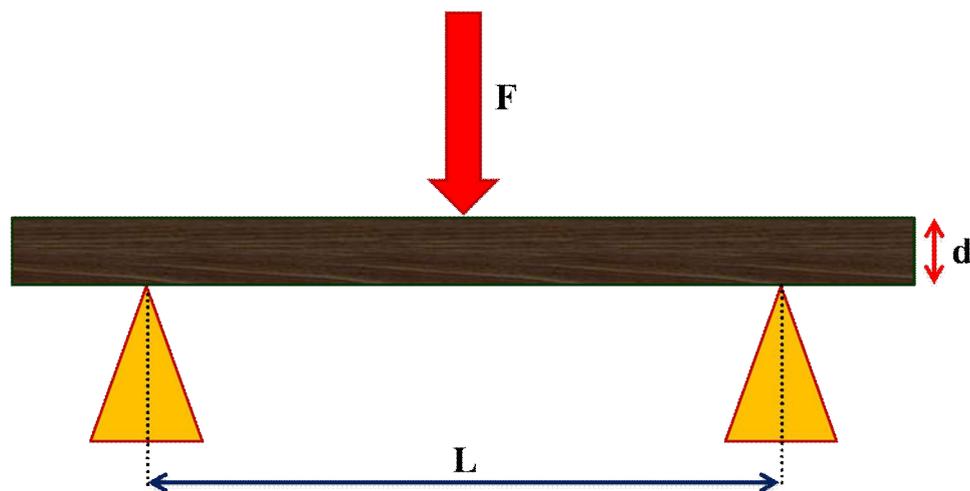


Figure 2.3 Beam under three-point bending setup

Hardness is the property of a material that enables it to resist plastic deformation, usually by penetration. However, the term hardness may also refer to resistance to bending, scratching, abrasion or cutting. Hardness is not an intrinsic material property dictated by precise definitions in terms of fundamental units of mass, length and time. A hardness property value is the result of a defined measurement procedure. Hardness of the nanocomposites was measured by UTM as per ASTM D2240.