Summary

In this study the synthesis of MCM-48 and modified MCM-48 such as Ti-MCM-48, Mn-MCM-48, Ru-MCM-48 and Al-MCM-48 were synthesized by hydrothermal method. The XRD, SEM. TEM, IR, TGA, DRS and BET Surface area of the samples reveal formation of desired framework and the d-values in XRD are in good agreement with those reported for the same framework.

The hydrothermal synthesis and characterization of purely siliceous MCM-48 molecular sieves were done. The characteristic $d_{220}$ peak in the XRD of MCM-48 was obtained at 32.07 Å. Other weak peaks may be indexed at $d = 23.8$ Å (400 plane), 20.6Å (420 plane), 19.02 Å (332 plane), 18.8 Å (422 plane) and a very strong peak at $d = 39.14$Å (211 plane) was observed in the study. The IR spectra shows stretching vibration of silanol -OH at 3446 cm$^{-1}$ and the bending vibration of H$_2$O at 1591cm$^{-1}$, the asymmetric and symmetric vibrations of Si-O at 1084 cm$^{-1}$ and 795 cm$^{-1}$ respectively. The SEM image reveals the nearly spherical shaped of size 660-840 nm and shows non agglomerated uniform and unique crystalline morphology. The TEM image shows a uniform square pore network arrangement. The nitrogen adsorption isotherm shows the type IV isotherm characteristic of mesoporous solid with the hysteresis loop. The average pore diameter of MCM-48 calculated by BJH method from adsorption isotherm was 2.5 nm, BET surface area was 1021 m$^2$ g$^{-1}$ and pore volume was found to be 0.84 cm$^3$/g. $^{29}$Si-MAS-NMR spectroscopy of MCM-48 is shows the three broad overlapping peaks from $Q_4$(-115ppm), $Q_3$(-101.45ppm) and $Q_2$(-88ppm). TGA plot of MCM-48 shows the thermal stability. The outside hydroxyl of the surface of MCM-48 was
encapped by reaction with Hexamethyldisilazane (HDMS) and then the drug ampicillin was loaded which was confirmed by IR and TGA study. The Simulated Body Fluid (SBF) were prepared by using the suitable reagents where the drug release were examined by stirring ampicillin loaded MCM-48 in it. From the study, it clears that 50% of the drug was released by modified MCM-48 within 1.5 hours, while rests of the adsorbed drugs were released slowly over a time period of 9 hours. In unmodified carrier the drug was adsorbed mostly in outside surface physically and 50% of the drug was released rapidly in 1 hour. After that slow release takes place which may be due to chemically adsorbed drugs inside the pores.

Ti-MCM-48 has been synthesized by hydrothermal method using Tetraethylorthosilicate, Titaniumisopropoxide and N-Cetyl-N, N, N-trimethylammoniumbromide as the template. Ti-MCM-48 was characterized by XRD, SEM, TEM, IR, DRS, TG and surface area measurements. The catalytic activities of Ti-MCM-48 were tested for (i) hydroxylation of benzene and (ii) Knoevenagel condensation reactions between Aromatic carbonyl compound and Ethylcyanoacetate. The conversion of benzene were found to be 87% and 76% to Phenol in acetone and methanol solvent respectively. The Knoevenagel reaction gave a product of 78% yield. The study of Photocatalytic decomposition of Rhodamine B by Ti-MCM-48 were also done and found that the decomposition of Rhodamine B is neglible without the catalysts but in presence of Ti-MCM-48 the decomposition is faster and it is even faster than that of TiO2. The decomposition of RB is increased upon prolonging the irradiation time.

The Mn-MCM-48 was synthesized by impregnation of MCM-48 with Mn(acac)3 or KMnO4 solution. The nitrogen adsorption isotherm gave the BET surface area 510 m²
g\textsuperscript{-1}, BJH adsorption average pore diameter 2.75 nm and single point adsorption total pore volume of pores was 0.379 cm\textsuperscript{3}/g. The catalytic application of Mn-MCM-48 was done by employing the epoxidation reaction of styrene and the product 2-phenyloxirane was identified by GC-MS and the percentage of yield was 87%.

Ru-MCM-48 was synthesized by post synthesis impregnation of MCM-48 with Ru-nano sols. The BET surface area found was 422 m\textsuperscript{2} g\textsuperscript{-1} comparatively less because of blocking of some of the pores by Ru-nano particles. The catalytic application of Ru-MCM-48 has been tested by catalytic hydrogenation reactions of acetophenone. The hydrogenation products was identified by GCMS. The reaction was completed in 6 hours and the yield (1-hydroxyphenyl ethane) was found to be 91%.

Al-MCM-48 has been synthesized by hydrothermal method. The IR bands at 523 cm\textsuperscript{-1} and 913 cm\textsuperscript{-1} corresponding to Al-O-Si bending and Al-OH bending respectively and confirms the incorporation of Al into MCM-48. The UV-Visible diffuse reflectance spectroscopy shows broad band at 260 nm which may be attributed to charge transfer from oxygen to aluminum. The average pore diameter of Al-MCM-48 calculated from adsorption isotherm was 2.8 nm and BET surface area was 812 m\textsuperscript{2} g\textsuperscript{-1}. The acidic and basic nature has been studied by adsorption of ammonia and pyridine. The catalytic application of Al-MCM-48 has been tested by carrying out acylation reaction (Friedel-craft’s acylation) of benzene and benzoyl chloride. The product formations were identified by GCMS and the % of product (benzophenone) formation was 68%.

Amino functionalized MCM48 was synthesized by refluxing a mixture of calcined MCM-48 and aminopropyltriethoxysilane (APTES). The adsorption average
pore diameter of NH$_2$-MCM-48 calculated from adsorption isotherm was 2.46 nm and BET surface area was 557 m$^2$ g$^{-1}$. This amino functionalized MCM-48 is an excellent base catalyst and is used in Michael addition reaction between benzaldehyde and 2-hydroxyacetophenone. The product Flavanone formation was identified by GCMS and the product percentage was found to be 67%.