Present thesis deals with investigations on the synthesis, characterization of semiconducting PbO, CdO, PbS, and ZnS nanoparticles by hydrothermal method. The synthesized nanoparticles are further used as catalyst for the degradation of the organic dye, removal of chromium, and in organic transformations. The main goal of the present work is, i) to synthesize the semiconducting PbO, CdO, PbS, and ZnS nanoparticles by hydrothermal method, ii) to encapsulate different metal ion in the synthesized nanoparticles, iii) to characterize the synthesized oxide, sulphide, and their doped products, iv) to study the photocatalytic degradation of dye and removal of chromium using synthesized nanoparticles, and v) to study the catalytic applications of synthesized nanoparticles in organic transformations.

The thesis is divided into four chapters. First chapter deals with the introduction, explaining different methods for the synthesis of nanoparticles, applications of metal oxide, and metal sulphide nanoparticles. Second chapter includes the result obtained and discussion on synthesis of semiconducting PbO, Ni doped PbO, CdO, Cs doped CdO, PbS, Co doped PbS, ZnS, Sm doped ZnS nanoparticles by hydrothermal method, and their characterization. Third chapter contains the investigations on photocatalytic degradation of methyl blue dye, rose bengal dye, methylene blue dye, crystal violet dye, removal of chromium using synthesized metal oxide, and metal sulphide nanoparticles. Fourth chapter covers the use of synthesized nanoparticles as the catalyst in multicomponent organic transformations.

The first chapter contains the introduction, literature survey of metal oxides, metal sulphides, metal doped oxides, and sulphides nanoparticles. It includes the classification, method of synthesis, and applications of nanoparticles. The literature
survey is also given in detail. The work done as well as scope of the present work is also given at the end of this chapter.

The second chapter includes the synthesis, characterization of metal oxide and metal sulphide nanoparticles. This chapter is divided into four sections. **Section-A** deals with the investigations regarding synthesis of PbO and Ni doped PbO nanoparticles using citric acid as a capping agent by hydrothermal method. The synthesized materials were characterized by techniques like FT-IR, XRD, SEM, EDAX, TEM, SAED, UV-DRS, PL spectroscopy, TGA, ESR, and BET surface area analysis. TGA data shows that the decomposition of the lead citrate complex takes place in temperature region 250-500°C and PbO remains stable in the 500-600°C temperature range. The reported PbO nanoparticles show greater uniformity and stability. The incorporation nickel into PbO nanoparticles has been discussed with the help of different analysis techniques. The average crystallite size decreased from 69 to 61 nm with encapsulation of nickel into PbO nanoparticles. From the data of UV-DRS characterization, the calculated band gap of PbO and Ni doped PbO nanoparticles have been found in the range of 3.04-3.13 eV. The optical properties of PbO nanoparticles were determined by using photoluminescence (PL) spectrum, which shows that the nanomaterials exhibit good optical properties with maximum emission peak at about 473 nm. The BET surface area analysis result shows that, the surface area of PbO, and Ni doped PbO nanoparticles have been found in the range of 29.38-34.32 m²/gm.

Similarly **Section-B** deals with synthesis of CdO and Cs doped CdO nanoparticles using Triton X-100 as a capping agent by hydrothermal method. The synthesized materials were characterized by techniques like FT-IR, XRD, SEM, EDAX, TEM, SAED, UV-DRS, TGA, PL spectroscopy, and BET surface area
analysis. This analysis enables us to evaluate the parameters like band gap energy, particle size, and surface area of synthesized CdO and Cs doped CdO nanoparticles. The synthesized CdO and Cs doped CdO nanoparticles have band gap energy is in the range of 3.52-3.64 eV.

**Section-C** includes the synthesis of PbS and Co doped PbS nanoparticles using Triton X-100 as a capping agent and sodium sulphide as a source of sulphide by hydrothermal method. The techniques like FT-IR, XRD, SEM, EDAX, TEM, SAED, UV-DRS, PL spectroscopy, TGA, ESR, and BET surface area analysis were used to characterize the synthesized nanoparticles. The TGA results reveal that the PbS nanoparticles remain stable in the temperature region 425-525°C and the synthesized PbS nanoparticles have greater stability. The incorporation of cobalt ion into PbS nanoparticles has been discussed with the help of different analytical techniques. An energy dispersive analysis by X-ray spectroscopy (EDAX) investigation supports the doping of cobalt into PbS nanoparticles. From the data of UV-DRS characterization, the calculated band gap of PbS and Co doped PbS nanoparticles have been found in the range of 4.07-4.20 eV. The optical properties of PbS nanoparticles were determined by using photoluminescence (PL) spectra, which shows that the nanoparticles exhibit good optical properties with maximum emission peak at about 476 nm. The BET surface area analysis results shows that, the surface area of PbS and Co doped PbS nanoparticles have been found in the range of 15.65-42.80 m²/gm.

**Section-D** reports the hydrothermal synthesis of ZnS and Sm doped ZnS nanoparticles using sodium deodecyl sulphate (SDS) as a capping agent. The synthesized materials were characterized by investigative techniques like FT-IR, XRD, SEM, EDAX, TEM, SAED, UV-DRS, PL spectroscopy, TGA, ESR, and BET surface area analysis. An energy dispersive analysis by X-ray spectroscopy (EDAX)
investigation reveals the stoichiometry in the undoped and doped ZnS nanoparticles. The XRD pattern of synthesized ZnS nanoparticles show single phase system with average particle size of 20 nm and the XRD pattern is in good agreement with hexagonal structure. From the data of UV-DRS characterization, the calculated band gap of ZnS and Sm doped ZnS nanoparticles have been found in the range of 3.59-3.67 eV. The optical properties of ZnS nanoparticles were determined by using photoluminescence (PL) spectrum, which shows that the nanomaterials exhibit good optical properties with maximum emission peak at about 448 nm. The BET surface area analysis results show that, the surface area of ZnS and Sm doped ZnS nanoparticles have been found in the range of 36.30-48.71 m²/gm.

The third chapter includes application of nanoparticles for degradation of organic dyes and removal of chromium from aqueous solution. This chapter is divided into four sections. **Section-A** deals with the photodegradation of methyl blue dye using synthesized PbO and Ni doped PbO nanoparticles. The present investigation reveals that the Ni doped PbO photocatalyst is more efficient than undoped PbO for degradation of methyl blue dye. The variation of pH, concentration of dye, amount of photocatalyst on photodegradation of dye has also been discussed. **Section-B** includes the photodegradation of rose bengal dye using synthesized CdO and Cs doped CdO nanoparticles. The present work report that the Cs doped CdO is more efficient than undoped CdO photocatalyst for photocatalytic degradation of rose bengal dye. The variation of pH, concentration of dye, amount of photocatalyst on photodegradation of dye has also been studied, and discussed in detail. It also includes the removal of heavy metal like chromium using CdO, Cs doped CdO nanoparticles, and results show it is an efficient adsorbent for the removal of chromium. **Section-C** includes the photodegradation of methylene blue dye using synthesized PbS and Co doped PbS
nanoparticles. The present investigation reports that the Co doped PbS is more efficient than undoped PbS photocatalyst for photocatalytic degradation of methylene blue dye. The effect of pH, concentration of dye, and amount of photocatalyst on photodegradation of methylene blue dye has also been studied. **Section-D** deals with the photodegradation of crystal violet dye using synthesized ZnS and Sm doped ZnS nanoparticles. The present investigation also report that the Sm doped ZnS is more efficient than undoped ZnS photocatalyst for photocatalytic degradation of crystal violet dye. The photodegradation of dye was carried out at different pH, concentration of dye, and amount of photocatalyst.

The fourth chapter includes application of synthesized semiconducting nanoparticles as the catalyst in organic transformations. This chapter is divided into four sections. **Section-A** includes the solvent free synthesis of benzylidene malononitriles and tetrahydrobenzopyrans using PbO nanoparticles by grinding at room temperature. All products are known compounds and were characterized by melting point (mp), IR, $^1$H NMR, $^{13}$C NMR, and MS technique. The attractive features of this catalyst is short reaction time, excellent yields, solvent free reaction, easy work up, and recyclable catalyst without significant loss in catalytic activity. **Section-B** deals with the solvent free one pot synthesis of acridinediones using CdO nanoparticles. The effect of solvent on the reaction has been studied, it was observed that solvent free condition is more suitable than reported reaction. All products are known compounds and were characterized by melting point (mp), IR, $^1$H NMR, and MS technique. The reported method has advantages like short reaction time, excellent yields, solvent free reaction condition, green heterogeneous catalyst, easy work up, and recyclable catalyst without significant loss in catalytic activity.
Similarly Section-C includes the solvent free one pot synthesis of amidoalkyl naphthols using PbS nanoparticles. All products were characterized by melting point (mp), IR, $^1$H NMR, and MS technique. The salient features of this catalyst are short reaction time, green procedures, excellent yields, solvent free reaction condition, easy work up, and recyclable catalyst. Section-D deals with the solvent free synthesis of 5-arylidene barbituric acid using ZnS nanoparticles by grinding at room temperature. All products are known compounds and were characterized by melting point (mp), IR, $^1$H NMR, $^{13}$C NMR, and MS technique. The attractive features of this catalyst are environmentally benign, excellent yields, solvent free reaction condition, easy work up, and recyclable catalyst. Section-E deals with comparative study on the catalytic efficiency of synthesized nanoparticles towards the synthesis of pyranopyrazoles. All products were characterized by melting point (mp), IR, $^1$H NMR, $^{13}$C NMR, and MS technique. The synthesized nanoparticles have catalytic efficiency is in the order PbO < CdO < PbS < ZnS nanoparticles. The series of pyranopyrazoles were prepared using ZnS nanoparticles at room temperature by grinding method under solvent free condition.