Day-by-day increase of environmental pollution in aquatic reservoirs and atmosphere impose seriously concern worldwide. Semiconductor based heterogeneous photocatalysis and chemiresistive gas sensing phenomena have become sustainable and widely practiced techniques for complete removal of toxicants, and precise detection of gases, respectively. This class of materials has good stability, less-toxicity and cost-effective tools, and can be activated either by light or heat energy. However, a majority of such solid materials obtained from traditional synthetic routes fail to deliver expected performance in the above applications owing to their lack of their active-surface structure and unsuitable redox/electronic properties. Thus, in this thesis work, suitable design strategies have been investigated including facile wet-chemical methods and coordination compounds templated routes for syntheses of targeted semiconductors/their hybrids and they were characterized using different instrumental techniques. The performance of the materials was studied on various toxic pollutants and selected gases. The research findings are summarized into five chapters in the thesis (**Chapter 2-6)**. In **Chapter 2**, porous Ag_3PO_4 microspheres (SPB MS) are achieved using a facile and green protein colloid template assisted precipitation routes. The prepared porous microspheres exhibited improved surface area and superior visible light driven photocatalytic activity towards degradation of RhB and 2, 4 DCP than that of other Ag_3PO_4 architectures. In **Chapter 3**, a facile three-step wet-chemical synthetic route was developed for the fabrication of a new three-component hybrid (ZAB: ZnO-Ag-Bi_2S_3), and it has been applied as a potential Ag nanoparticles (NPs) mediated Z-scheme and LSPR sensitized photoredox catalyst for the effective degradation of methyl orange (96% in 1.5 h) under the full spectrum of light. In **Chapter 4**, silver (I) based nano/microstructure coordination polymer (Ag(I)-CP) of 1, 4 benzenedicarboxylate (bdc^{2-}) obtained from precipitation route, were utilized as a new class of UV active semiconductor photocatalyst for the degradation of tartrazine. Later, mixed ligand coordination polymer of Ag(I) (MLCP X, X: 20, 40) with a rational proportion of 2-amino 1, 4 benzenedicarboxylate (NH_2-bdc^{2-}) and their coupled structure (Ag@MLCP 40) with Ag NPs had been synthesized through the same route, and which were utilized as visible light photocatalysts for the degradation of tartrazine. Ag@MLCP 40 achieved the fastest rate of degradation, and more reusable than that of the MLCP 40 counterpart. In **Chapter 5**, a modified Co(II) nanoscale coordination polymer template route has been employed to synthesize (111) faceted, Co^{2+} rich Co_3O_4 octahedra, and it was further modified with SiO_2 to achieve COSI 71. Co_3O_4 octahedra exhibit a ten times higher chemiresistive response towards 100 ppm H_2 gas with respect to Co_3O_4 powder. It is further enhanced by two times in COSI 71 as a result of improved surface area. In **Chapter 6**, Ni(II) based coordination compound templated synthesis of porous 1D (ND 3506) and 2D (NBD 4506) NiO were made with higher surface area and oxygen vacancies in contrast to traditionally prepared NiO (NOH3506). Both NBD 4506 and ND 3506 exhibited higher chemiresistive response and faster recovery time towards 300 ppm ethanol than the traditional NiO sensor (NOH3506). Overall, we synthesized various semiconductors and their hybrids with tuned geometrical and surface functions, demonstrated their suitability for redox reaction by studying their photocatalytic and chemiresistive actions, and their mechanism on particular applications. Moreover, the findings of the studies encourage the strategic development of effective catalytic materials towards different environmental applications.