Rapid growth of manufacturing industries dumps huge volume of effluents day by day, which leads contamination of water bodies by toxic pollutants; such as persistent organic compounds, azo dyes, metal ions, etc. Among various water treatment techniques, Advanced Oxidation Process (AOP) using heterogeneous photocatalyst is one of the widely accepted practices due to its economic nature, flexibility, reusability, and effectiveness in remineralization of pollutants. Different groups are working on the utilization of visible light (from sunlight) for AOP. Recently, mixed metal oxides (MMOs) had taken a lead position in visible light photocatalysis. The preparation of MMOs with enhanced surface properties can be done *via* synthesis of layered double hydroxide (LDH) followed by thermal decomposition. Hence, in this work, different types MMOs have been synthesized *via* co-precipitation method, characterized, and studied for their suitability towards visible light photocatalysis of water pollutants.

The thesis work is distributed into six chapters, including a general introduction in Chapter 1. In Chapter 2, n-p type semiconductor has been synthesized by coupling of ntype ZnO with p-type Bi₂O₃, and then the ZnO-Bi₂O₃ was co-assembled with RGO (ZB-RGO). The ZB-RGO has been applied for the photocatalytic removal of 2-chlorophenol (2-CP) and 2,4-dichlorophenol (2,4-DCP). Chapter 3 has two parts. Chapter 3A elaborates the synthesis of heterojunction photocatalysts by coupling of cation (Mg²⁺ and Al^{3+}) doped ZnO with $ZnCo_2O_4$ [Co(X%)/ZMA], and studied their application for the removal of MO dye, 2-CP and 2,4-DCP. Among the catalysts, Co(20%)/ZMA exhibited the highest photocatalytic activity. In Chapter 3B, annealing temperature of Co(20%)/ZMA has been optimised in order obtain heterojunction material with high crystalinity, stable morphology, suitable band gap energy and highest photocatalytic activity under visible light. In Chapter 4, cerium doped CuMgAl-mixed metal oxide (CMA-X% Ce) has been presented, where the copper-cerium interaction through redox equilibrium was established. CMA-1.0% Ce exhibited excellent photodegradation of 2,4-DCP with high rate constant due to redox cycle and high surface area of the catalyst. In chapter 5, Preparation of europium doped CuMgAl- mixed metal oxides and their smart photoredox applications have been described. By our strategic design of catalyst, we could demonstrate simultaneous reduction of Cr(VI) to Cr(III) (in conduction band) and oxidation of toxic 2,4-DCP (in valance band) effectively. Control studies revealed •OH as main reactive species, helped in the photocatalysis. Overall, we synthesized a series of visible light active mixed metal oxides having heterojunctions, and demonstrated their suitability for photocatalytic removal of organic and inorganic pollutants. Based on the control studies and analysis of degradation products, plausible mechanisms of photocatalysis and suitable degradation pathways of each pollutant has been proposed.