# DEDICATED WITH EXTREME AFFECTION AND GRATITUDE TO MY BELOVED PARENTS & MY SUPERVISOR

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It is certified that the work contained in the thesis titled "Synthesis, Characterization and Application of Nano-Adsorbents for Removal of Metallic Species from Aqueous Solutions" by "Shikha Dubey" has been carried out under my supervision and that this work has not been submitted elsewhere for a degree.

It is further certified that the student has fulfilled all the requirements of Comprehensive, Candidacy and SOTA.

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### DECLARATION BY THE CANDIDATE

I, Shikha Dubey certify that the work embodied in this thesis is my own bonafide work and carried out by me under the supervision of Prof. Yogesh Chandra Sharma from July 2013 to July 2016 at the Department of Chemistry, Indian Institute of Technology, Banaras Hindu University, Varanasi. The matter embodied in this thesis has not been submitted for the award of any other degree/diploma. I declare that I have faithfully acknowledged and given credits to the research workers wherever their works have been cited in my work in this thesis. I further declare that I have not willfully copied any other's work, paragraphs, text, data, results, etc., reported in journals, books, magazines, reports, dissertations, theses, etc., or available at websites and have not included them in this thesis and have not cited as my own work.

**Date:** 13.10.2016 **Place:** Varanasi

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It is certified that the above statement made by the student is correct to the best of my knowledge.

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## PREFACE

The past several decades have witnessed an impetus in exponential population growth and civilization expansion, opulent lifestyles and resources consumption, and proceeding industrial and technological advancements that have led to climbing wages, as well as to a sharp modernization and metropolitan growth. The development of any country reckons on its industrial as well as agricultural activities and the natural resources such as air, water, soil, and plant as well as animal life constitute the natural capital on which man depends on to satisfy his needs to achieve his aspirations for development. However, rates of exploitation of these natural resources as well as extensive industrial activities are far surpassing the past with a negative connotation of accompanying environmental degradation. Therefore, the planet has experienced an assortment of pressing environmental challenges such as climate change, global warming, waste, and water pollution, leading to serious environmental degradation. The contamination of water resources by hazardous pollutants has pulled in much serious attention in the last few decades. This is particularly due to their toxic, acute and chronic health effects that seriously threaten the human health and environment. Providing clean and affordable water is considered as one of the most basic humanitarian goals, and it becomes a grand challenge of the 21<sup>st</sup> century.

Heavy metal contamination due to tremendous increase in industrial applications as well as man's indifferent behaviour towards nature is of a significant concern today. Heavy metals, among various contaminants are of special concern due to their recalcitrant and persistent nature in the environment and are reported to cause life-threatening effects even at trace concentrations.

A wide range of treatment technologies have been developed for treatment of water rich in heavy metals, of which adsorption process is of major interest due to its simplicity, economic viability, and technological feasibility. A number of different conventional low cost adsorbents are being used for treatment of water and wastewater and are being superseded by nano-adsorbents in terms of their large surface area which enables them to adsorb larger amounts of metal ions with enhanced adsorption capacity and rapid sorption rate. Additionally, their ability of regeneration and reuse; low dose requirement make their application economical.

The limitations of classical methods of studying a process by maintaining other factors involved at an unspecified constant level can be overcome by response surface methodology (RSM) where all the affecting parameters can be optimized collectively resulting in improved product yields, reduced process variability, closer confirmation of the output response to nominal and targeted requirements, as well as reduced development time and overall costs. Box-Behnken (BBD) design of RSM is slightly more efficient than the central composite design, but is much efficient than the three-level full factorial designs in terms of cost when the number of factor is higher than 2. Also, it does not contain combinations for which all factors are simultaneously at their highest or lowest levels. Linear regression for the best fitting of isotherm and kinetic data is the most feasible tool for analysing adsorption parameters, but due to its inappropriateness for isotherms with more than two parameters and also inherent biasness associated with it, alternative isotherm parameter sets were determined by non-linear regression. The intention of this thesis is to contribute the scientific findings investigated by me, keeping in view all these facts, a humble attempt has been made to cover the overview of treatment of water laden with heavy metals, synthesis of nano-adsorbents and their characterization for applications for removal of metallic species from aqueous solutions as well as optimization of the process in the present thesis. Best efforts are made to illustrate the scientific findings in the thesis with appropriate justifications, figures, and references.

In the light of above, outcomes of present research investigations are subdivided into eight chapters. First chapter is an introductory chapter; second chapter gives a brief preview of the up-to-date survey of the literature in the area related to the research in the thesis. Third chapter depicts various materials experimental procedures and methods involved all through the research work; and fourth chapter illustrates the synthesis and characterization of nano-adsorbents. The experimental findings of the present thesis are presented in chapters fifth to eighth where each of these chapters begins with a brief review of literature relevant to the work presented in that chapter to put the appropriate outlook, results and discussion followed by conclusion. The chapter wise summary of the work is compiled at the end followed by the list of references.

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