The title of this thesis “SYNTHESIS OF POROUS CARBON FROM PLANT BASED PRECURSORS AND THEIR APPLICATIONS” and the experimental investigations embedded in it gives a glimpse of a blend of Chemistry and Engineering in the synthesis, characterization of Porous Carbon (PC) and their diversified application mainly in hazardous management. This thesis covers a detailed method of synthesis of porous carbon from three abundantly available natural sources of carbon precursors namely Tea and Tea Waste (TW), Indian Oleander Seed Hull (IOSH) and Areca Nut Husk (ANH) fiber. Moreover, Magnetic Carbon Composite (MCC) is synthesized by cobalt ferrite magnetic nanoparticle (MNP) impregnated onto TW using combined sonochemical and precipitation method. The as prepared PCs are characterized by various sophisticated analytical techniques and investigated their potentiality as adsorbent for removal of different toxic dyes [e.g.; Methylene Blue (MB) and Eosin Yellow (EY)] and metals [e.g.; Chromium (Cr), Cadmium (Cd) and Nickel (Ni)] from aqueous solution.

The Thesis is organized in the following chapters:

**Chapter 1:** Introduction and Literature Review

**Chapter 2:** Synthesis and optimization of porous carbon

**Chapter 3:** Adsorptive removal of methylene blue dye by thermochemically activated porous carbon from TW, IOSH and ANH

**Chapter 4:** Adsorptive removal of eosin yellow dye by thermochemically activated porous carbon from TW, IOSH and ANH

**Chapter 5:** Adsorption of Cr(VI) on thermochemically activated porous carbon from TW, IOSH and ANH

**Chapter 6:** Adsorption of Cd(II) on thermochemically activated porous carbon from TW, IOSH and ANH

**Chapter 7:** Adsorption of Ni(II) on thermochemically activated porous carbon from TW and magnetic carbon composite
ABSTRACT

This present work has been divided into following seven chapters as mentioned above and is briefly discussed below.

CHAPTER 1

This chapter includes an introductory discussion of PC starting with its historical background and brief discussion about water pollution being rapid industrializations in present day context. The existing preparation method of PC and their advantages/disadvantages are discussed in detail. The characteristics of a PC are tunable varying the source of raw material, chemical activating agents and the activation method. In this chapter, various earlier observations for choosing different raw materials, chemical activating agents are also covered along with the diversified application of PC.

Here, the sources of dyes and metal pollutants and their adverse effects to human as well as aquatic life were described in detail. All the existing different treatment technique for both the pollutants and their pros and cons are discussed for removing dye and metal from aqueous solutions. The details literature review on the important scientific findings till date for PCs, dyes and metals are also included here. Based on the outcomes of the most significant literatures as reported, the objectives and the importance of this research work have been made.

CHAPTER 2

This chapter presents the preparation method of PC by thermochemical activation method mainly from three carbon precursors viz.; Tea and TW, IOSH and ANH using H$_3$PO$_4$ (PA), NaOH (SH) and KOH (PH) as chemical activating agents. Different preparation parameters are investigated and the synthesized PC was characterized by various characterization methods to find out the best possible PC from each carbon precursor.

$N_2$ adsorption/desorption isotherm was used to determine BET surface area and the textural properties of all PC. Surface texture, pore morphology and fundamental physical properties of adsorbent surfaces of PC were characterized by SEM. The surface organic
functional groups of PC were carried out by FT-IR spectrophotometer using Diffuse Reflectance Attachment (DRS) assembly. The surface inorganic components of the prepared PC’s were measured by the powder XRD. The thermal stability of these PC’s was measured by the TGA. The magnetic property of magnetic carbon composite (MCC) prepared by the cobalt ferrite magnetic nanoparticle (MNP) impregnated onto TW and further carbonized to make PC at 500°C (MCC) was investigated by VSM.

The synthesized PC from each precursor was optimized based on BET surface area. Further adsorption studies were carried out using the optimized PC from each class of carbon precursor.

**CHAPTER 3**

This full chapter present the adsorptive removal of MB dye by thermochemically activated PC from TW, IOSH and ANH. Various experimental parameters viz.; solution pH, effect of contact time and adsorbent dose for MB dye adsorption from aqueous solutions on PC were studied and compared their adsorption behavior using batch technique. The adsorption process was found to be strongly dependent on pH and maximum removal of dye was found to be pH 7 for the entire studied PC.

The equilibrium data have been analyzed using Langmuir and Freundlich adsorption isotherm model and the characteristics parameters and related correlation coefficients reveals the best fitness of Langmuir adsorption isotherm model. The maximum adsorption capacity \((Q_0)\) as calculated from the Langmuir adsorption isotherm was found to be 402.25 mg/g, 492.13 mg/g and 421.23 mg/g for TW, IOSH and ANH based PC respectively at 303 K. The suitability of pseudo-first-order and pseudo-second-order kinetic model for the MB dye adsorption was investigated. The pseudo-second-order kinetic model is well fitted to the experimental data for the adsorption of MB dye onto all studied PC for different initial dye concentrations over the entire range. Moreover, the thermodynamic feasibility along with the spontaneity of the adsorption process in terms of the thermodynamic parameters viz., \(\Delta H^0\), \(\Delta S^0\) and \(\Delta G^0\) were discussed and
indicates the endothermic and spontaneous nature of the MB-PC adsorption process.

CHAPTER 4

This chapter is yet another attempt to formulate the adsorption process to remove a hazardous and highly water soluble halogen containing fluorescent dye, EY. In this study, efforts have been made to use optimized PC from three carbon precursors (TW, IOSH and ANH) as adsorbent for EY removal from aqueous solution.

The experimental data were investigated for its adsorption capacity by considering different operating variables and shows the important influences on the adsorption of EY. The maximum removal was obtained at solution pH 7. The equilibrium adsorption data have been analyzed using Langmuir and Freundlich adsorption isotherm model. It was found to follow suitably by Langmuir isotherm from the well fit straight lines at all temperatures suggesting monolayer adsorption of EY dye. The maximum \( Q_0 \) value was obtained as 400 mg/g, 476.19 and 588.23 mg/g for TW, IOSH and ANH based PC at 303 K respectively. Suitability of pseudo-first–order and pseudo-second-order kinetic model for the adsorption of the dye onto the studied PC was investigated and thermodynamic parameters for the dye-PC interaction are presented in detail depicts each of the adsorption processes was spontaneous, feasible and endothermic in nature.

CHAPTER 5

This Chapter includes detailed adsorption studies of Cr(VI) by three best optimized PCs from aqueous solutions in a batch process. The results of this investigation of Cr(VI) removal by three potential PC are found to be highly encouraging. In general, the pH of adsorbate solution plays a pivotal role in the adsorption process and maximum adsorption capacity for entire PC was observed at pH 2. The adsorption was found to be strongly dependent on solution pH and was affected by varying the other parameters such as initial concentration and contact time, adsorbent dose etc. The mathematical description of adsorption equilibrium data were studied by Langmuir adsorption isotherm and Freundlich adsorption isotherm but the first one has an edge over the second one because of their
ABSTRACT

higher regression coefficients. The maximum monolayer adsorption capacity ($Q_o$) of the studied PC from TW, IOSH and ANH was calculated as 47.62 mg/g, 22.99 mg/g and 35.71 mg/g respectively at 303K and pH 2. The observed increasing trend of adsorption capacity with increase in temperature indicates the process to be endothermic in nature. The Kinetic study of the equilibrium data indicates that the adsorption of Cr(VI) onto the studied PC is well fitted to the pseudo-second-order kinetic mechanism. The thermodynamic parameters viz.; $\Delta H^0$, $\Delta S^0$ and $\Delta G^0$ mostly indicates the endothermic and spontaneous nature of the Cr(VI)-PC adsorption process.

CHAPTER 6

The detailed adsorption studies of Cd(II) on thermochemically activated PC from inexpensive agricultural wastes TW, IOSH and ANH are presented in this chapter. Equilibrium adsorption, kinetics and thermodynamics of the reported results were discussed elaborately covering all the aspects for its possible application in removal of Cd(II). Various batch adsorption parameters were studied and investigated the best removal of Cd(II) from aqueous solutions. The adsorption equilibrium experimental data of Cd(II) were analyzed using Langmuir and Freundlich isotherm model and both the model effectively explained as is evident from their straight lines and higher regression coefficients approaching unity. By using the Langmuir isotherm, the monolayer adsorption capacity ($Q_o$) of the studied PC from TW, IOSH and ANH was calculated as 18.45 mg/g, 41.15 mg/g and 42.37 mg/g respectively at 303K and pH 5. The adsorption kinetics for various concentrations was analyzed with the help of pseudo-first-order and pseudo-second-order kinetic models. A pseudo-second-order model sufficiently describes the adsorption kinetics indicating the adsorption process to be controlled by chemisorptions. The adsorption capacity of Cd(II) onto the studied PC was studied as a function of three different temperatures. It has been observed that adsorption capacity increases with increase in temperature indicating the adsorption is of endothermic in nature as well as temperature dependence process.
CHAPTER 7

In this chapter we are investigating the adsorption properties of Ni(II) from aqueous solution by PC from TW and a composite material made by impregnating cobalt ferrite MNP onto PC prepared from TW (MCC). All the parameters that decide the adsorption efficiency of both the PC for separation of Ni(II) from aqueous solution were discussed in detail and optimized the pH, initial concentration and contact time and adsorbent dosage. The removal efficiency was found to be dependent on solution pH and maximum in both cases at pH 5. Adsorption equilibrium data was investigated by two adsorption isotherm models. Using the Langmuir isotherm, the maximum monolayer adsorption capacity \( Q_0 \) of the studied PC from TW and MCC was calculated as 107.53 mg/g and 121.95 mg/g respectively at 303 K. A pseudo-second-order model sufficiently describes the adsorption kinetics indicating the adsorption process to be controlled by chemisorptions. Thermodynamic studies were also carried out to estimate the standard free energy, enthalpy change and entropy change for both the adsorbent. The determination of thermodynamic parameters \( \Delta H^0 \) mostly indicates the exothermic and endothermic nature of the Ni(II) - TW based PC and Ni(II) – MCC adsorption process respectively. The negative \( \Delta G^0 \) value for both the PC system indicates spontaneous nature of Ni(II) adsorption. The entire results of Ni(II) adsorption by PC obtained from TW and its magnetic carbon composite (MCC) were investigated and compared its results for better understanding. Hence the newly developed carbon magnetic composite can be well recommended for removal of Ni(II) from wastewater.