CHAPTER 1

INTRODUCTION

1.1 BACKGROUND

Nanotechnology is the science that creates functional materials and devices on the nanometer ($10^{-9}$ m) scale. This length scale reaches the atomic level providing the opportunity to explore micro and macro phenomena and properties. Nanotubes (CNTs) and other carbon nanostructures are supposed to be the key components of this nanotechnology. Having realized its tremendous application potential in nanotechnology, a huge amount of efforts are being in CNTs projects worldwide. Till date, the art of CNTs synthesis lies in the optimization of parameters for selected group materials in a particular experimental set-up. From the perspective of green chemistry, sustaining the environment implies sustaining the human civilization. The long-term key of a sustainable society lies in a stable economy that uses energy and resources efficiently. Therefore, it is high time to evaluate the feasibility of introducing green techniques in nanotechnology.

Chemical Vapor Deposition (CVD), incorporating catalyst-assisted thermal decomposition of hydrocarbons, is the most popular method of producing CNTs. It is indeed a low-cost and scalable technique for mass production of CNTs (Kong et al. 1998). Mostly petroleum products such as methane, ethylene, acetylene, benzene and xylene are used as precursors for synthesizing CNTs using CVD method. Apart from this volatile petroleum based hydrocarbons, carbon nanotubes have been synthesized from polymers,
metallocenes and domestic fuels such as kerosene and liquefied petroleum gas (Kumar & Ando 2010).

Besides the outstanding physical properties, over the past few years the chemical properties of CNTs have also aroused an increasing interest from scientists. The chemical functionalization is of significant importance since it can tailor the properties of CNTs and therefore aid in the development of applications. Functionalization has been demonstrated at the ends and sidewalls of CNTs.

According to the principles of green chemistry, the feed stock of any industrial process must be a renewable natural resource. Hence, it is the time’s prime demand to explore renewable natural materials for CNTs synthesis. The researchers have established the conditions for growing Multi-walled carbon nanotubes (MWNTs), Single-walled carbon nanotubes (SWNTs) and vertically aligned MWNTs on the suitable catalytic support by a simple and inexpensive CVD technique using volatile natural precursors. Researchers have prepared good Morphology of Multi-walled carbon nanotubes (MWNTs) and vertically aligned ones by thermal decomposition of turpentine oil and camphor (Aswasthi et al. 2010; Suriania et al. 2013). Andrews et al. (2006) have synthesized pure Single-walled carbon nanotubes (SWNTs) by catalytic decomposition of camphor and its anologes. Ghosh et al. (2007; 2008) have prepared single-walled carbon nanotubes from turpentine oil and eucalyptus oil.

In this work, plant derived hydrocarbon oil such as methyl ester of *Madhuca longifolia* oil, methyl ester of *Oryza sativa* oil and methyl ester of *Brassica juncea* oil having vaporizing temperature around 240-320 °C have been used as carbon precursor for synthesis of CNTs. Unlike any fossil or petroleum product, there is no fear of its being depleted as it is a regenerative source and can be obtained easily by cultivating as much quantity as required. As
the chosen precursors evaporate at comparatively lower temperatures, spray pyrolysis method is adopted for synthesis of CNTs. It is to note that spray pyrolysis method not much utilized for CNTs synthesis. The spray pyrolysis method is similar to chemical vapor deposition method with the only difference that the vaporization and pyrolysis of carbon source occur simultaneously in spray pyrolysis whereas in CVD it is a two-step process.

The present work has assessed the yield and morphology of the carbon nanostructures produced using natural carbon precursors viz. methyl ester of *Madhuca longifolia* oil, methyl ester of *Oryza sativa* oil and methyl ester of *Brassica juncea* oil by spray pyrolysis method at different temperatures, catalysts composition and precursor flow rate conditions.

### 1.2 RESEARCH OBJECTIVE AND SCOPE

The main objectives of the present work is to evaluate the suitability of unconventional natural plant based carbon precursor oils with vaporizing temperature around (240-320 °C) for CNT synthesis. This work explores whether pyrolysis of plant derived precursors such as methyl ester of *Madhuca longifolia* oil, methyl ester of *Oryza sativa* oil and methyl ester of *Brassica juncea* oil over Fe, Co and Mo catalysts supported on silica under N\textsubscript{2} atmosphere employed for synthesis of good quality carbon nanotubes at low temperature conditions using spray pyrolysis method. As the properties of carbon nanotubes are expected to be dependent on their morphology, efforts have been made to study growth mechanism of CNTs in order to control the morphology diameter of CNTs.
The present work was undertaken especially to study and illustrate the following

1. Chosing suitable plant-based precursor

2. Chosing catalyst-support material

3. Synthesis of CNTs using chosen plant derived precursor with varying the parameter like Catalyst composition, Temperature and precursor flow rate

4. Characterization using SEM, HRTEM, Raman Spectroscopy and XRD analysis

5. To synthesize well crystalline MWNTs from chosen precursors viz. methyl ester of Madhuca longifolia oil, methyl ester of Oryza sativa oil and methyl ester of Brassica juncea oil over Fe catalyst supported on silica by spray pyrolysis under N₂ atmosphere.

6. To optimize the process parameters for synthesis of CNTs such as temperature, combination of catalyst and precursor flow rate on order to obtain good yield with desirable morphology.

7. To study the characteristics of carbon nanotubes prepared from chosen precursors using SEM, HRTEM, Raman spectroscopy and XRD analysis.

8. To explain the feasibility of synthesis of MWNTs with high yield and good morphology from chosen precursor oils by optimization of process parameters.