CHAPTER 1

Introduction

1.1 History of carbon nanotubes

Faraday (1857) and Gustav Mie (1908) began the history of nanoparticle using roman glassmakers, glasses containing nanosized metal particles Lycurgus cup. In diffused light In focused light Explained by Michael and Photographic films using silver halide photochemistry (silver nanoparticles) by Richard Feynman(1958)..H. Davy, C. Maxwell (1861), G.Eastman (1883) predicted the existence of electron beam lithography, scanning tunneling microscope and building circuits on the scale of nanometer for powerful computers In diffused. Through the decade nanoscale Materials Inc., neutralizer for chemical hazards of dry powder formulation, (Reactive Nanoparticle (RNP™)) to bind with a variety of chemical warfare agents and toxic chemicals, and chemically convert them to safer by-products, NanoScale Materials Inc.(2004).

There are three main methods to produce CNTs. These are arc discharge, laser vaporization and chemical vapor deposition (CVD) methods *(Baddour and Briens, 2005).* Among these, CVD method is suitable for mass production where we used in this method. Catalyst material is very important for CNT growth. Transition metals are appropriate for CNT growth but especially Fe, Co, Ni are the best ones according to many researchers studied previously. These metal particles are generally supported by an inorganic porous material to provide high surface area for active component. There are many methods to produce catalyst. Impregnation, precipitation, combustion and sol-gel are most common ones. Gel-combustion method has some advantages; it gives high specific surface area and good dispersion of the active sites for catalyst *(Rashidi, et al. 2007).*

Carbon nanotubes have caught scientists attention with its special properties since their detailed observation TEM by *Iijima in 1991* Carbon nanotubes divide into two groups; single wall carbon nanotubes (SWNTs) and multi wall carbon nanotubes (MWNTs) depending on number of concentric graphene cylinder that tube contains *(Dresselhaus, et al. 2001).* Some of carbon nanotube's excellent physical properties are high aspect ratio, high Young modulus, high tensile strength , high thermal and electrical conductivity *(Salvetat, et al. 1999).* Because of their high young modulus and low weight they are useful as reinforcing agents in composite materials and in a variety of applications such as sensors, field emission devices, flat panel displays, energy storage, electrochemical devices and electronic devices *(Paradise, et al. 2006).*Catalyst material is very important for CNT growth. Transition metals are appropriate for CNT growth but especially Fe, Co, Ni are the best ones according to many researchers. *(Rashidi, et al. 2007).*

1.2 Research problem

The aim of this work is to synthesize carbon nanotubes (CNTs) of iron Fe and cobalt Co catalyst particles by Low pressure chemical vapor deposition (LPCVD) method and then grown high quality and large scale CNTs by optimizing CNT growth parameters of acetylene chemical vapor deposition gas. Besides that, pretreatment and growth atmosphere gas composition, hydrogen, argon, and acetylene flow rates, growth temperature and growth time should be the main problem to be studied. Therefore, there is a need to study quality, structure and physical properties of the nanocomposite dependent strongly on the growth conditions e.g. temperature, catalyst and environment condition (gas rates).

1.3 Research Methodology

The methodology of the research is based on using low pressure chemical vapor deposition device to synthesize carbon nanotubes (CNTs) of iron and cobalt catalysis. Finally; implications of the scanning electron microscopic SEM nanotubes images results were used fractal methods to explain the diameter of the carbon nanotube.

1.4 Research Objectives

The objectives of this study are:

1. To study the effect of acetylene rates and temperature variations of iron

and cobalt nanoparticles in carbon nanotubes CNTs

2. Modeling of Surface morphologies of Carbon Nanotubes (CNTs) catalytic results of Iron and Cobalt (Fe and Co) nanocatalysis using mixed model (R-Program, Image-J, Harfa Program).
3. To use fractal program to estimate carbon nanotubes diameters with their sizing effects in CNTs growth and yield.

1.5 Literature Review

By rolling a graphene sheet into a cylinder and capping both end of the cylinder with a half of fullurene molecule a carbon nanotube is formed. Harry Kroto discovered C_{60} molecule in 1985 *(Kroto, et al. 1985)* while experimenting a laser ablation system for the vaporization of graphite by laser beams and depositing them on a copper collector and it was the beginning of a new area in carbon material science. At 1990s arc discharge method was reported in order to make large quantities of the C60 molecule. In 1991, Iijima experimented this technique in order to observe fullerene and by passing large current between two graphite rods, he vaporized them and condensed them on Cu tip. When he looked at the result through an electron microscope, he noticed something unexpected; he discovered carbon nanotubes *(Iijima, et al. 1991)* at the negative electrode of an arc discharge. They were tiny tubes of pure carbon with a large amount of other forms of carbon. These first carbon nanotubes were like Russian dolls, several concentric layers with caps at the end, so they were called multi-wall carbon nanotubes. Only two years later, in 1993, single-wall carbon nanotubes could be grown using Co metal catalysts by arc discharge method *(Iijima, et al. 1993).*

Former researchers studied a novel carbon nanotube (CNT) synthesis method by chemical vapor deposition (CVD) using catalysts obtained from carbon rich fly ash. In this approach two fly ash samples (S1 and S2) were treated by heating at 750 0C, and then analyzed using scanning electron microscope (SEM) and energy dispersive spectroscopy (EDS) *(Numan Salah, et al. 2012).* In the CVD method, a hydrocarbon gas is allowed to flow over a heated layer of catalyst particles *(J. Kong, et al, 1998).* Carbon nanotubes (CNTs) were synthesized on cobalt catalyzed film using low pressure chemical vapour deposition system. These studies CNTs grown by the catalytic deposition of acetylene gas C_2H_2 at 750ºC, at a chamber pressure of 100 Torr and the growth time was kept fixed at 5 minutes. Initially, nitrogen gas was passed for 10 minutes *(Zishan H. Khan. et al. (December 2011).* Other studied the Fe-catalyzed chemical vapor deposition of carbon nanotubes by complementary in situ grazing-incidence X-ray diffraction, in situ X-ray reflectivity, and environmental transmission electron microscopy *(Christoph T. Wirth, et al, 2012).*

A large variety of growth mechanisms have been suggested for Fe catalyzed CNT growth *(Yoshida, H.; et al. 2008).* Chemical vapour deposition (CVD) has been widely used to grow CNTs in recent years *(M Meyyappan; et al. 2003).* In this approach, a feedstock also at: Eloret Corporation. such as CO or a hydrocarbon is heated from 800–1000˚C with a transition metal catalyst to promote nanotube growth *(Kong J, Soh H T, et al. 1998, Li J, et al.1999).*

1.6 Thesis Outline

Chapter 1 presents the introduction, history and objectives of the study and literature review. Chapter 2 gives a brief literature background about the properties of Carbon Nanotubes (CNTs), and syenthesization of iron and cobalt Catalytic Nanoparticles on substrate. The techniques used to characterize Fe, Co nanotubes production were also explained. The experimental work is presented in Chapter 3. Chapter 4 contains the results, discussion and conclusion.