



# Carbon Nano Tube from Natural Source: Banana Peel

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# DECLARATION

This is to certify that the work presented in this thesis is an outcome of the experiment and research carried out by the authors under the supervision of Prof. Dr. A.K.M. Sadrul Islam.

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Although we tried our best to complete this thesis flawlessly, we seek apology if there is any mistake found in this report.

### ABSTRACT

Banana peel has a great potential to be used as a source of renewable biomass energy and for extracting some value-added material like activated carbon and carbon nano tube. Banana is the number one fruit in Bangladesh considering its year round availability, popularity and production. It accounts for 41% of the total fruit production from 21% share in area. The average yield of banana is 15 t/ha, which will result in a great source of banana peel.

Carbon Nano Tubes are very small tubes made solely of carbon molecules. They may be single walled or multi walled having  $Sp^2$  bond on each atom. Due to their unique properties and infinite applications they are considered as the material of the future..

This thesis describes the extraction and derive of carbon nano tube from banana peel activated carbon and 2% mineral oil mixture which was used as a precursor for carbon nano tube and nano carbon. The process of synthesis involves pyrolysis of precursors at 1000°C to 1200°C in a closed stainless-steel tube. The final products were analyzed using SEM, EDX.

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# **INTRODUCTION**

#### 1.1 Overview:

The global energy market is dependent on the fossil fuels viz. coal, oil, and natural gas as sources of thermal energy. Formation of fossil fuels in the earth takes millions

of years. The reserves of fossil fuels are finite and subject to depletion as they are consumed. The sources of biomass are all water- and land-based organisms, vegetation, and trees, or virgin biomass, and all dead and waste biomass such as municipal solid waste(MSW), bio-solids (sewage), animal wastes (manures), forestry and agricultural residues, and certain types of industrial wastes. The biomass is renewable in the sense that it is regenerated within short rotation of time what is used as energy resource unlike the fossil fuel deposits. The size of global carbon sink (standing terrestrial biomass carbon used as an energy resource) is approximately 100 times the global energy consumption annually.

#### **1.1.1** The fundamental concept on biomass energy :

Living plants capture the solar energy by fixing the carbon in its tissue via photosynthesis with the help of chlorophyll. The source of carbon fixed in the plant tissue is the carbon dioxide ( $CO_2$ ) in atmosphere. The photosynthesis process converts atmospheric  $CO_2$  to carbohydrate (glucose, starch, cellulose etc). The product of photosynthesis process is called biomass. The process of photosynthesis in the growth of biomass is expressed by the following equation 1.1

 $CO_2 + H_2O + light + chlorophyll \rightarrow (CH_2O) + O_2$ 

#### 1.1.2 Banana Peel:

Banana peel is one of the major sources of biomass. A banana peel is the outer covering of the banana fruit. As bananas, whether eaten raw or cooked, are a popular fruit

consumed worldwide, with yearly production over 145 million tones , there is a significant amount of banana peel waste being generated as well. Banana peels are used as feedstock as they have some nutritional value. Banana peels are widely used for that purpose on small farms in regions where bananas are grown. The specific nutrition contained in peel depends on the stage of maturity and the cultivar. On average, banana peels contain 6-9% dry matter of protein and 20-30% fiber (measured as NDF). Green plantain peels contain 40% starch that is transformed into sugars after ripening. Green banana peels contain much less starch (about 15%) when green while ripe banana peels contain up to 30% free sugars.

#### **1.2 Value Added Products From Banana Peel:**

Biomass char can be considered as the precursor to a highly valuable product, which is the activated carbon or activated char. Activated carbon has high and fast adsorption capacities due to its well-developed porous structure and very high surface area. One of the most important applications of this material is in water purification in urban areas. Activated carbon is also widely used in chemical industries for gas adsorption and filtration including removal of color and odor compounds in aqueous systems.

Another valuable product which is obtainable is the carbon nano tube. Carbon nano tube is a very small tube which is solely made of carbon molecules having extreme tensile strength and high thermal and electricity conductivity properties. It has a very significant application in Electrical, Biological and Energy storage sector.

It is important to develop an easy to use and easily transportable product that can be accessible to the mass communities. If the banana peel can produce a quality activated carbon and as well as carbon nano tube it is also possible that it could provide a value added commodity for the country from what is at present a waste product.

#### 1.3 Objectives:

The main objective of this study is to investigate the suitability of banana peel forconversion into carbon nano tube and activated carbon. Carbon nano tube will be detected by SEM and EDX testing. Activated carbon will be detected with the particular application to adsorbing color and dyes from aqueous solutions.

The specific objectives are:

- To extract and derive carbon nano tube from natural source.
- To find out the optimum combination of resource for minimizing time & cost.
- To detect the presence of Activated Carbon at various temperature.

## **1.4** Scope of research:

- > Production of carbon nano tube from banana peel of different varieties.
- > Production of activated carbon material from banana peel.
- > To find the techniques for producing carbon nano tube and activated carbon.

## **1.5** Thesis organization:

- Chapter 1 gives an overview of biomass energy generation, importance of banana peel as precursor for carbon nano tube and activated carbon.
- Chapter 2 discusses the studies on banana peel for carbon nano tube and activated carbon from previous studies.
- Chapter 3 discusses the methodology of how the study was carried out.
- Chapter 4 discusses the various sample test and analysis of the results e.g. SEM & EDX analysis of banana peel, adsorption test using methylene blue to detect activated carbon.
- Finally a conclusion and suggestions for improvement/errors is made in Chapter
  9.

# **CHAPTER 2**

# LITERATURE REVIEW

#### 2.1 Value Added Product From Banana Peel :

### 2.1.1 Carbon Nano Tube :

#### 2.1.1.1 Background leading up to Carbon nano tubes:

Until the mid-1980's pure solid carbon was thought to exist in only two physical forms, diamond and graphite. Diamond and graphite have different physical structures and properties however their atoms are both arranged in covalently bonded networks. These two different physical forms of carbon atoms are called allotropes.



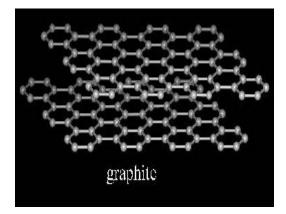


Figure 2.1 : Molecular Structure of Diamond and Graphite

In 1985 a group of researchers led by Richard Smalley and Robert Curl of Rice University in Houston and Harry Kroto of the University of Sussex in England made an interesting discovery. They vaporized a sample of graphite with an intense pulse of laser light and used a stream of helium gas to carry the vaporized carbon into a mass spectrometer. The mass spectrum showed peaks corresponding to clusters of carbon atoms, with a particularly strong peak corresponding to molecules composed of 60 carbon atoms, C60. The fact that C 60 clusters were so easily formed led the group to propose that a new form or allotrope of carbon had been discovered. It was spherical in shape and formed a ball with 32 faces. Of the 32 faces, 12 were pentagons and 20 were hexagons exactly like a soccer ball.

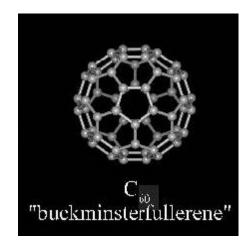


Figure 2.2: Molecular Structure of "Buckminsterfullerene"

After the discovery of "buckminsterfullerene", other related molecules (C36, C70, C76 and C84) composed of only carbon atoms were also discovered and they and the buckyball were recognized as a new allotrope of carbon. This new class of carbon molecules is called the fullerenes. Fullerenes consist of hexagons and pentagons that form a spherical shape.

#### 2.1.1.2 Discovery of Carbon Nano tubes:

The unique geometric properties of this new allotrope of carbon did not end with soccer shaped molecules, it was also discovered that carbon atoms can form long cylindrical tubes. These tubes were originally called "buckytubes" but now are better known as carbon nano tubes or CNT for short. There are two types of CNT:

- Single walled CNT
- Multi walled CNT

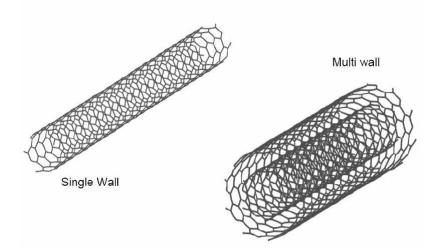


Figure 2.3 : Structure of Single Wall and Multi wall CNT

As Carbon-nanotubes are allotropes of carbon which are actually extremely thin hollow cylinders are considered as the material of the future because of their unique properties and infinite application.

A carbon-nanotube is a one atom thick sheet of Graphite (called graphene) rolled up into a seamless cylinder with diameter of the order of nanometer. Hypothetically one can visualize the formation of single wall carbon nanotube through rolling single graphene sheet into a cylinder. For multiwall nanotubes bi-layer graphene sheet will be the starting material. The diameter of carbon nanotubes typically vary from 0.7-3 nm. Due to such small diameters, nanotubes become quasi one dimensional. They can posses a single shell or multiple shells. Tubes with single shell are called single wall carbon nanotubes (SWNT) while once with more than one shell are multiwall carbon nanotubes (MWNT). The length of nanotubes can be up to centimeters, giving them an astonishing length/diameter ratio of 10<sup>7</sup>.

#### 2.1.1.3 Synthesis of Carbon Nano tubes:

Carbon nano tubes (CNT) were first discovered in the black soot product from a CVD process. Since then, their synthesis techniques have evolved considerably. The last 10 years have seen tremendous research in both nanotube synthesis and their potential use in

electronic circuits, composites, thin films etc. Various ways of synthesis carbonnanotubes were established along with CVD process such as Arc discharge, Laser ablation, Ball Milling and Pyrolysis etc.

#### 2.1.1.4 Pyrolysis:

The production of carbon nanotubes from banana peel by pyrolysis method were examined. Pyrolysis is a thermo chemical decomposition of organic material at elevated temperatures in the absence of oxygen (or any halogen). It involves the simultaneous change of chemical composition and physical phase, and is irreversible.

In general, pyrolysis of organic substances produces gas and liquid products and leaves a solid residue richer in carbon content, char. Extreme pyrolysis, which leaves mostly carbon as the residue, is called carbonization.

The pyrolysis process was carried out according to the following method described below:

The sample was sized in small irregular shape and were fed into the laboratory oven to remove the moisture at 105°C. After removal of moisture banana peel sample was placed in muffle furnace and heated at 400°C to carbonize . Adding Phosphoric Acid in proper ratio the sample was again heated at 600°C. In order to get very fine powder particle 400 mesh screen was used after grinding and crunching the sample. The sample was then pyrolyzed at high temperature by taking the sample in stainless steel tube. The final products after cooling to room temperature were characterized by Scanning Electron Microscopy (SEM) and Energy Dispersive X-Ray Spectroscopy (EDX).

#### 2.1.1.5 Scanning Electron Microscope (SEM):

A scanning electron microscope (SEM) is a type of electron microscope that produces images of a sample by scanning it with a focused beam of electrons. The electrons interact with atoms in the sample, producing various signals that can be detected and that contain information about the sample's surface topographyand composition. The electron beam is generally scanned in a raster scan pattern, and the beam's position is combined with the detected signal to produce an image. SEM can achieve resolution better than 1

nanometer. Specimens can be observed in high vacuum, in low vacuum, and (in environmental SEM) in wet conditions.

The most common mode of detection is by secondary electrons emitted by atoms excited by the electron beam. The number of secondary electrons is a function of the angle between the surface and the beam. On a flat surface, the plume of secondary electrons is mostly contained by the sample, but on a tilted surface, the plume is partially exposed and more electrons are emitted. By scanning the sample and detecting the secondary electrons, an image displaying the tilt of the surface is created.

#### 2.1.1.6 Energy-dispersive X-ray spectroscopy:

Energy-dispersive X-ray spectroscopy (EDX) is an analytical technique used for the elemental analysis or chemical characterization of a sample. It relies on the investigation of an interaction of some source of X-ray excitation and a sample. Its characterization capabilities are due in large part to the fundamental principle that each element has a unique atomic structure allowing unique set of peaks on its X-ray spectrum. To stimulate the emission of characteristic X-rays from a specimen, a highenergy beam of charged particles such as electrons or protons, or a beam of X-rays, is focused into the sample being studied. At rest, an atom within the sample contains ground state (or unexcited) electrons in discrete energy levels or electron shells bound to the nucleus. The incident beam may excite an electron in an inner shell, ejecting it from the shell while creating an electron hole where the electron was. An electron from an outer, higher-energy shell then fills the hole, and the difference in energy between the higherenergy shell and the lower energy shell may be released in the form of an X-ray. The number and energy of the X-rays emitted from a specimen can be measured by an energy-dispersive spectrometer. As the energy of the X-rays are characteristic of the difference in energy between the two shells, and of the atomic structure of the element from which they were emitted, this allows the elemental composition of the specimen to be measured

#### 2.1.2Activated Carbon:

Activated carbon, also calledactivated charcoal, activated coal, orcarbo activatus, is a form of carbon processed to be riddled with small, low-volume pores that increase the surface area available for adsorption orchemical reactions.

Due to its high degree of microporosity, just one gram of activated carbon has a surface area in excess of  $500 \text{ m}^2$ , as determined by adsorptionisotherms of carbon dioxide gas at room or 0.0 °C temperature. An activation level sufficient for useful application may be attained solely from high surface area; however, further chemical treatment often enhances adsorption properties. Activated carbon is usually derived from charcoal.

It is reported that charcoal has been used for water purification for centuries, dating back as far as ancient Egypt and India. Non-polar organic molecules dissolved in water are strongly attracted tocharcoal surfaces and bind due to electrostatic interactions - this gives charcoalmaterials their utility as water filtration media.

Activated carbon sample at three different temperatures (600°C,700°C,800°C) were tested for the presence of activated carbon by adsorption test using methylene blue.

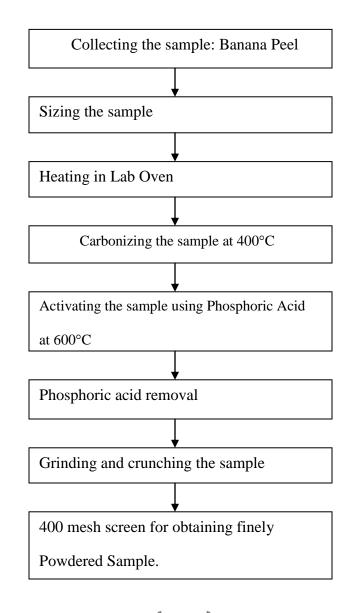
# **CHAPTER 3**

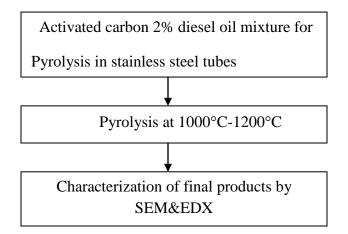
# METHODOLOGY

#### 3.1 GENERAL

The chapter discusses about the procedure that has been followed to conduct the study. It describes about how materials and methods were utilized, by which means carbon nano tube will occur.

#### **3.2 METHODOLOGY**





### **3.2.1 EQUIPMENTS, MATERIALS AND MACHINES**

#### **Muffle Furnace:**

A muffle furnace is a furnace in which the subject material is isolated from the fuel and all of the products of combustion including gases and flying ash.

A muffle furnace is a front-loading box-type oven or kiln for hightemperature applications such as fusing glass, creating coatings, ceramics and soldering and brazing articles.

They are also used in many research facilities, for example by chemists in order to determine what proportion of a sample is non-combustible and non-volatile.



**Fig 3.1: Muffle Furnace** 

#### Laboratory Oven:

Laboratory ovens are ovens for high-forced volume thermal convection applications. These ovens generally provide uniform temperatures throughout.

Process applications for laboratory ovens can be for die-bond curing, drying, Polyimide baking, sterilizing, and other industrial laboratory functions.

Typical sizes are from one cubic foot to 32 cubic feet  $(0.91 \text{ m}^3)$  with temperatures that can be over 650 degrees Fahrenheit (340 degrees Celsius).

Laboratory ovens can be used in numerous different applications and configurations, including clean rooms, forced convection, horizontal airflow, inert atmosphere, natural convection, and pass through.



Fig 3.2 : Lab Oven

#### **Digital Analytical Balance:**

An analytical balance is a class of balance designed to measure small mass in the submilligram range. The measuring pan of an analytical balance is inside a transparent enclosure with doors so that dust does not collect and so any air currents in the room do not affect the balance's operation. This enclosure is often called a draft shield. The use of a mechanically vented balance safety enclosure, which has uniquely designed acrylic airfoils, allows a smooth turbulence-free airflow that prevents balance fluctuation and the measure of mass down to 1  $\mu$ g without fluctuations or loss of product.



Fig 3.3 : Digital Analytical Laboratory Balance

#### **Desicator:**

Desiccators are sealable enclosures containing desiccants used for preserving moisturesensitive items such as cobalt chloride paper for another use. A common use for desiccators is to protect chemicals which are hygroscopic or which react with water from humidity.

The contents of desiccators are exposed to atmospheric moisture whenever the desiccators are opened. It also requires some time to achieve a low humidity. Hence they are not appropriate for storing chemicals which react quickly or violently with atmospheric moisture such as the alkali metals.

Desiccators are sometimes used to remove traces of water from an almost-dry sample. Where a desicator alone is unsatisfactory, the sample may be dried at elevated temperature using Abderhalden's drying pistol.



Fig 3.4 : Desicator

## 3.2.2 Collecting Sample : Banana Peel

Banana is the number one fruit in Bangladesh considering its year round availability, popularity and production. It accounts for 41% of the total fruit production from 21% share in area. The average yield of banana is 15 t/ha, which is lower compared with that of other countries in the world.



Figure 3.5 : Photograph of Banana sample

### **3.2.3** Preparing the sample :

After collecting different types of sample, main ingredient banana peel is obtained from the sample. Fresh and good quality banana peels are generally selected for the sample.



Figure 3.6: Banana peel

## 3.2.4 Sizing the Banana Peel :

Banana peels were cut and sized using scissors & knives to smaller dimensions (1 to 3 square centimeter). In general square shape, banana peel was sized for the next process.



Figure 3.7: Sliced pieces of banana peel sample

#### 3.2.5 Heating in Lab Oven

The banana peel sample was heated in electric oven at 105°C for 24 hours to remove the moisture. The heating was done in 2-hour stages. Around 3-4 batches (dozen bananas in one batch approximately) were heated in the electric oven.



Figure 3.8 : Banana peel at 105°C for 24 hours

#### **3.2.6** Carbonizing the sample at 400°C

The sample from a batch was slightly crushed using mortar and pestle to smaller size. The sample was then placed in a crucible, properly sealed with a cover plate. It was then placed in a larger crucible. Between the two layers of crucible dry charcoal was placed and covered the small crucible completely. This was done to prevent excessive damage and burning of the sample due to the muffle furnace heating. The sample was then placed in muffle furnace and heated for 1 hour at 400 °C .The time and temperature control panel of muffle furnace was closely monitored .The resulting banana peel sample was then carbonized.

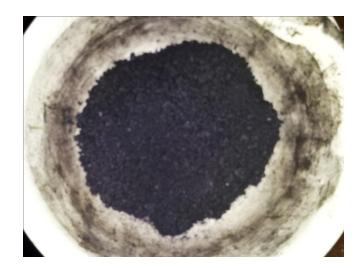


Figure 3.9 : Carbonized sample

## 3.2.7 Activating the sample using Phosphoric Acid at 600°C

The 400°C sample was then activated using phosphoric acid. The phosphoric acidcarbonized banana peel sample was mixed at a ratio of 0.5:1 by volume per weight. The sample was thoroughly stirred for proper mixing of the phosphoric acid and sample.

The activated sample was then heated at 600°C in muffle furnace. In this case the sample was also heated for one hour.



Figure 3.10 : Activated sample

#### 3.2.8 Phosphoric acid removal:

The 600°C activated sample was washed with distilled water three times to remove all traces of phosphoric acid and other impurities.

The sample was then heated in oven at 105°C for 24 hours and placed in desicator for drying the sample.



Figure 3.11: Washing the activated sample with distilled water

#### **3.2.9** Grinding and crunching the sample:

The activated carbon powder was then crunched and grinded for obtaining very small sized sample so that they could be gradually passed through the sieve of 400 mesh screen.



Figure 3.12 : Crushed sample using mortar and pestle

#### **3.2.10** 400 mesh screen for obtaining fine powdered sample:

The very fine powder was sieved through a 400 mesh screen by putting continuous pressure on the sample through a rotating motion as can be seen in the figure.

The resulting sample was collected and weighed in a digital analytical balance.



Figure 3.13 : 400 mesh screen used to sieve sample

### 3.2.11 Activated carbon 2% diesel oil mixture for Pyrolysis in stainless steel tube:

The activated carbon was mixed with 2% diesel oil. The mixture was thoroughly stirred. Then the mixed powder was placed in a stainless steel tube having dimensions (12.5 mm in diameter,1 mm thickness and 200 mm in length).Both ends of the tube were closed by Arc welding which provides proper seal of activated carbon material.



Figure 3.14 : Filling the sample in stainless steel tube

#### 3.2.12 Pyrolysis at 1000°C-1200°C:

The sample 1 in the closed tube was pyrolyzed at 1000°C for 1 hour in furnace. The furnace required around 1 hour for the temperature to rise to 1000°C. After reaching 1000°C the stainless steel tube was put inside the furnace using tongs, and kept for 1 hour. After heating the tube was taken out and cooled to room temperature. The tube was then cut open on one edge and the sample was taken out and isolated in small glass tubes for further testing.

For sample 2 and sample 3 same procedure was followed at 1100°C and 1200°C respectively.



Figure 3.15: 1000°C Sample 1



Figure 3.16 : 1100°C Sample 2

### 3.2.13 Error in pyrolysis:

At 1200 °C a crack in the stainless steel tube was found while carrying out pyrolysis.For this reason the sample 3 could not be analyzed by SEM & EDX test.



Figure 3.17 : 1200°C Sample 3

## 3.2.14 Samples for SEM & EDX testing:

The final products after cooling to room temperature were characterized by Scanning Electron Microscopy (SEM) and Energy Dispersive X-Ray Spectroscopy (EDX).



Figure 3.18 : Final sample

#### 3.2.15 Adsorption test:

The Phosphoric acid- activated carbon sample at 600°C after washing and drying was used as a precursor for adsorption test. Three sample were created (5 gm each) by heating them at three different temperature (600°C,700°C,800°C). A solution of 1000 ml was prepared using 100 ml methylene blue and 900 ml distilled water. Then we separated 100 ml of the solution. Like this 5 more 100 ml solution were separated.

- ▶ 0.05 gm sample at 600°C was placed in 100 ml solution.
- > 0.1 gm sample at 600°C was placed in 100 ml solution
- > 0.05 gm sample at 700°C was placed in 100 ml solution
- > 0.1 gm sample at 700°C was placed in 100 ml solution
- > 0.05 gm sample at 800°C was placed in 100 ml solution
- > 0.1 gm sample at 800°C was placed in 100 ml solution

These samples were jerked, rotated and stirred thoroughly for proper mixing of sample and solution. These mixtures were then observed for two days to see the change of color of solution.

# SAMPLE TEST AND RESULT ANALYSIS

#### **4.1 GENERAL**

This chapter discusses how the sample test was carried out in detail. Analysis of different SEM (Scanning Electron Microscope) and EDX (Energy Dispersive X-Ray Spectroscopy) result.

## 4.2 SEM test images of sample 1 (1000°C)

The sample was tested in various magnification (2000x, 4000x, 10000x etc) which shows the presence of Activated Carbon and non-homogenous distribution of carbon nano tube of narrow tube like structure.

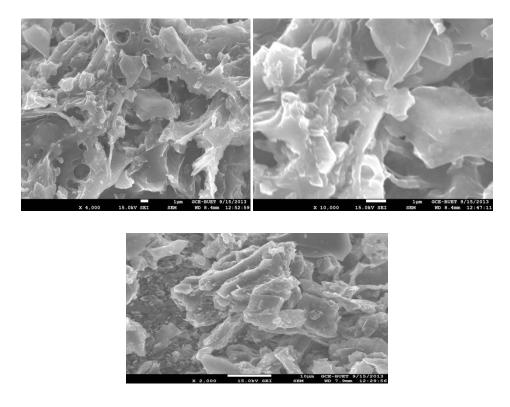


Figure 4.1: SEM micrograph of sample 1

# 4.3 SEM test images of sample 2 (1100°C)

The sample was tested in various magnification (1000x, 3000x etc) which shows the presence of Activated Carbon and non-homogenous distribution of carbon nano tube of narrow tube like structure.

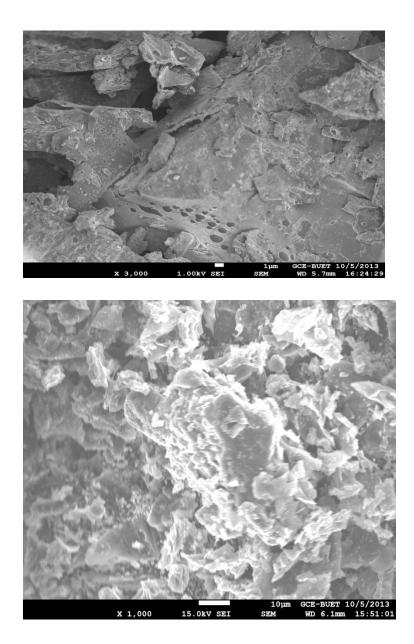
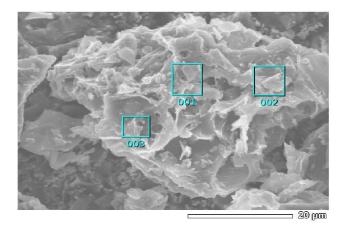
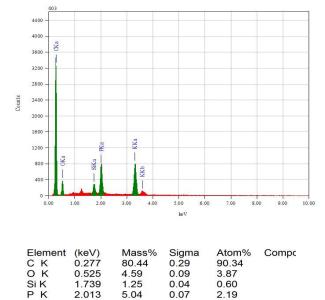


Figure 4.2 : SEM micrograph of sample 2

### 4.4 EDX test images of sample 1 (1000°C)

Three positions were focused as shown in the image. These were tested to find the percentage composition of various elements. The details are shown in the tabulated chart. Mass percentage of Carbon (80.44%) were satisfactory whereas percentage of Potassium (K) (8.68%) was comparatively high.





0.07

0.12

2.19

3.00 100.00

2.013

3.312

KK

Total

5.04

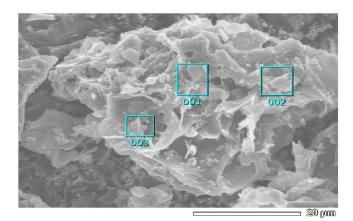
8.68

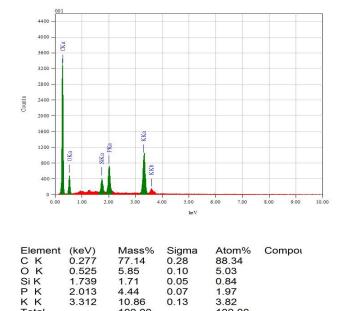
100.00

Figure 4.3 : EDX result of sample 1

### 4.5 EDX test images of sample 2 (1100°C)

For sample 2 similar process analysis was done .Three positions were focused as shown in the image. These were tested to find the percentage composition of various elements. The details are shown in the tabulated chart. Mass percentage of Carbon (77.14%) were satisfactory whereas percentage of Potassium (K) (10.86%) was comparatively high.





0.13

3.82

100.00

10.86

100.00

3.312

Total

Figure 4.4 : EDX result of sample 2

# CONCLUSION AND RECOMMENDATION

#### 5.1 Conclusion

Carbon nanotubes can be obtained from activated banana carbon and 2% mineral oil mixture by pyrolysis in the stainless steel tube at 1000 to 1200°C. The pyrolytic products consisted mainly of straight nanotubes . The nanotubes showed nodular Fe (metal) inside the hollow and the tip interconnections. The pyrolytic products consist mainly of carbon nano tubes and a small amount of carbon amorphous, fullerene, and carbon in chaoite form, Fe3C, Fe2O3, andK2O.

#### 5.2 Recommendation and Discussion

- In case of the mixture used for pyrolysis mineral oil should have been used instead of diesel oil for yielding homogeneous layers of Carbon Nano Tube.
- Potassium mass% was higher than expected. This should be reduced by ammonia treatment.
- Stainless steel tube is expected to act as catalyst, so this could have caused the crack of sample 3 at 1200°C pyrolysis. Another reason could be the poor quality of the material or fault in the welding when the tube was sealed.
- The 400 sieve mesh used had 45µm mesh size which yielded larger size of the particle .In this smaller size 17µm mesh should have been used to get better sample for more accurate result.
- Due to unavailability of magnifying capacity above 300k x sample could not magnified at high resolution which would have yielded better image.

### 5.3 Future Work

- Study may be undertaken using other natural source i.e Sugar bagasse, Bamboo as precursor for producing carbon nano tubes.
- Similar work may be carried out and the results should be compared.
- Study may be undertaken on the application of carbon nano tube from this experiment.

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