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FUNCTIONALIZATION AND CHARACTERIZATION OF MULTI-WALLED CARBON NANOTUBES (MWCNTS)

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Abstract
Multi-walled Carbon Nanotubes (MWCNTs) are cylindrical structures composed of rolled-up graphene sheets with diameters of a few nanometers and lengths up to several hundred micrometers. The unique properties of MWCNTs, including their high electrical and thermal conductivity, strong mechanical strength and chemical stability make them attractive in various applications. This study functionalized the MWCNTs via chemical methods using 60 % nitric and sulphuric acid solutions for 3 hours each. The material was washed thoroughly with hot deionized water until neutral pH was obtained and the powdered sample was dried in an oven at 105 ^oC for 3 hours. The powdered sample was characterized by Scanning Electron Microscopy (SEM), Elemental Dispersive X-ray (EDX), Fourier Infrared Spectroscopy (FT-IR), Brunauer Emmett-Teller (BET) and X-ray Diffraction (XRD). SEM confirmed large cavities and sparsely web shape while EDX gave atomic distribution of the material and revealed carbon and oxygen as major elements, and sodium and sulphur as minor elements present in the MWCNTs. The (FT-IR) result of the MWCNTs spectra showed broad band peaks at 3448.84 cm^{-1} and 3414 cm^{-1} which corresponds to O-H stretching mode of hydroxyl groups on the surface of the nanotube. The BET surface area result of the material indicated higher surface area with 865.66 m^2/g. The (XRD) results confirmed prominent diffraction peaks which is common to all carbonaceous material.
Keywords: *Carbon, Characterization, Functionalization, MWCNTs, Nanotube*

Introduction

The discovery of carbon nanotubes (CNTs) in the early 1990's reported by Sumio Iijima and the potential of developing novel carbon-based nanomaterial have attracted substantial interest in the area of analytical chemistry, environmental protection and other scientific researches worldwide. CNTs have great potential as a novel type of adsorbent due to their unique properties such as chemical stability, large diameter and length/diameter ratio, highly porous and hollow structure, light mass density, strong interaction between carbon and hydrogen molecules and mechanical and thermal stability. The uniqueness of these properties has led to various applications including water treatment, protein purification and hydrogen storage. CNTs are very hydrophobic and tend to aggregate in aqueous solutions because of high Van der Waals interaction forces in the tube. As a result, they are not readily dispersible in water. However, their dispersibility in aqueous solutions can be increased by functionalization such as surface oxidation or by addition of surfactants.

The structure of a carbon nanotube is formed by a layer of carbon atoms that are bonded together in a hexagonal (honeycomb) mesh. This one-atom thick layer of carbon is called graphene, and it is wrapped in the shape of a cylinder and bonded together to form a carbon nanotube. Nanotubes can have a single outer wall of carbon, or they can be made of multiple walls (cylinders inside other cylinders of carbon). Carbon nanotubes have a range of electrical, thermal, and structural properties that can change based on the physical design of the nanotube.

Single-walled carbon nanotubes can be formed in three different designs: armchair, chiral and zigzag. The design depends on the way the graphene is wrapped into a cylinder. For example, rolling a sheet of paper from its corner can be considered one design, and a different design can be formed by rolling the paper from its edge. A single-walled nanotube's structure is represented by a pair of indices (n, m) called the chiral vector.

There are two structural models of multi-walled nanotubes. In the Russian Doll model, a carbon nanotube contains another nanotube inside it (the inner nanotube has a smaller diameter than the outer nanotube). In the Parchment model, a single graphene sheet is rolled around itself multiple times, resembling a rolled-up scroll of paper. Multi-walled carbon nanotubes have similar properties to single-walled nanotubes, yet the outer walls on multi-walled nanotubes can protect the inner carbon nanotubes from chemical interactions with outside materials. Multi-walled nanotubes also have a higher tensile strength than single-walled nanotubes.

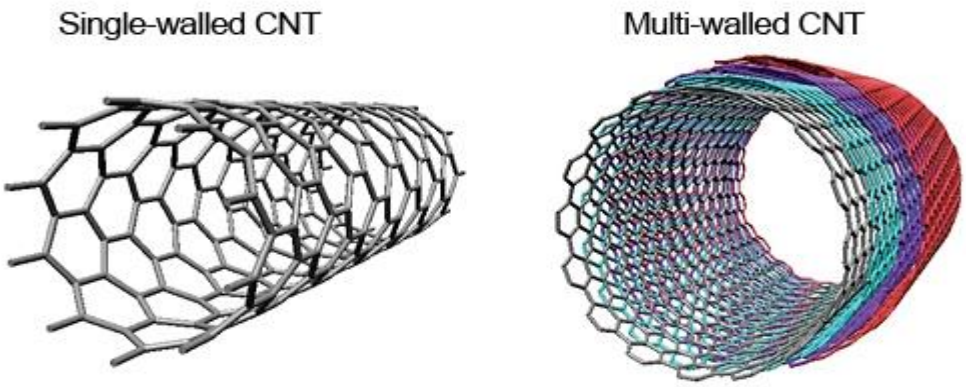

Fig. 1: Single-walled CNT and Multi-walled CNT

Functionalization is the introduction of functional groups to a surface. Carbon nanotube surfaces can be chemically (covalently) or physically (non-covalently) functionalized to achieve highly desirable surface properties. Nanotube functionalization typically starts with oxidative conditions, commonly by refluxing in nitric or sulfuric acid or combination of both to generate carboxylic acid moieties on the defect sites. The end caps of nanotubes have extra strain energy because of their high degree of curvature with pentagons and heptagonal carbon atoms are most vulnerable to the reaction with acid. However, this study will focus on the functionalization and characterization of multi-walled carbon nanotube.

Materials and Methods

Multi-walled carbon nanotubes with average diameter between 50 and 90 nm (95 %), HNO_3 (≥ 65 %), H_2SO_4 ($95.0 - 97.0$ %) were purchased from Sigma-Aldrich which are analytical grade.

All glassware and polyethylene containers were sourced locally and thoroughly washed with hot detergent solution. They were soaked in 20 % HNO_3, rinsed with distilled water and finally with deionized water in order to avoid any contamination during analysis.

Other common laboratory equipment or apparatus used include analytical (digital) weighing balance (PA 313 model), mortar and pestle, stop watch, hammer, magnetic stirrer (78HW-1), oven (model OV-160) and pH meter (PH-2603). Atomic Absorption Spectrophotometer (AAS) (210VGP model), Fourier Transform Infrared Spectrometer (FT-IR) (SHIMADZU 8400S model), Scanning Electron Microscope (SEM) (Tescan MIRA 3), Electron Disperse X-ray Spectrometer (EDX) (FEI model), Brunauer Emmett-Teller (BET) and X-ray Diffraction Spectrometer (XRD) (Rigaku D/Max-lllC) were used for characterization studies.

Functionalization of the MWCNTs

A slightly modified procedures of were adopted. Two different chemicals were used for the functionalization processes (400 mL of 60 % each of HNO_3 and H_2SO_4 acid concentrations) in order to achieve reasonable surface oxidation of the tubes. First, chemical purification was performed with the HNO_3 solution under reflux at 100 ^oC for 3 hrs with 20 g quantity of commercial MWCNTs. The mixture was filtered and the residue was washed several times with deionized water until neutral pH was obtained. The treated MWCNTs was dried in an oven at 105 ^oC for 3 hrs. The dried treated material was used for second chemical purification.

Second chemical functionalization was performed under reflux at 100 ^oC for 3 hrs with the H_2SO_4 solution on the dried sample from the first chemical treatment. The resulting solution was thoroughly washed with deionized water by filtration until the pH of the filtrate was approximately 7. The resulting precipitate was finally dried in an oven at a 105 ^oC for 3 hrs.

Results and Discussion

Important characterization signatures were provided by SEM, EDX, FT-IR, and XRD results which are very imperative to this study.

SEM Result of the MWCNTs

Scanning Electron Microscopy (SEM) is widely used to study the surface morphology, including the pore structure, surface structure and pore arrangement on surface of a material. The SEM micrograph of functionalized MWCNTs is presented in Fig. 2. The SEM image of MWCNTs shows the numerous thick pores of the multi-walled carbon nanotube with dense web shape which resulted in opening of the end-caps, indicating that the carbons are more reactive and this is similar to literature reports. The SEM microgram was in line with findings reported by.

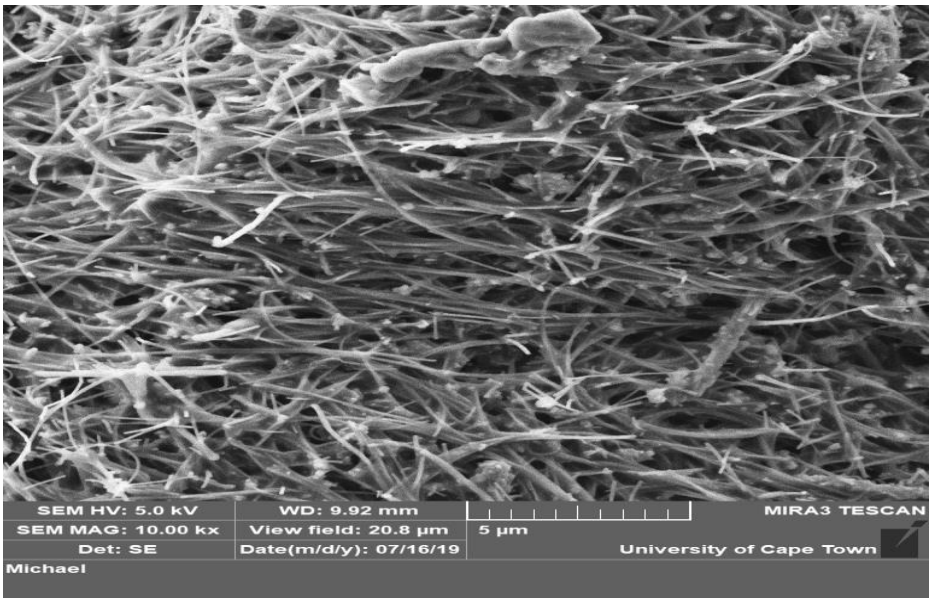

Fig. 2: SEM Micrograph of MWCNTs

EDX Result of the MWCNTs

Energy-dispersive X-ray spectroscopy is an important analytical tool which gives information on the surface for atomic distribution and the chemical elemental composition of the nanoparticles. The spectrum for this result was presented in Fig. 3. The major elements confirmed by the EDX analysis are carbon (90.93 %), oxygen (8.62 %), sulphur (0.45 %) and

sodium (0.02 %). The oxygen atom on the functionalized carbon can have strong effects on the adsorption of contaminants. Many studies showed that ion adsorption onto functionalized carbon nanotubes occurs due to the ion exchange with protons in oxygen functional groups. According to metal ion classification, hard metal ions (Zn^{2+}, Ni^{2+}) are adsorbed to the surface of the functional group (-COOH, -OH). However, activated carbon has π electrons on the surface of micropores, where the soft metal ions (Cd^{2+}, Pb^{2+}) tend to be adsorbed.

Table 1: Summary of Elemental Composition of the MWCNTs

Elements	**Composition (%)**
Carbon (C)	90.93
Oxygen (O)	8.62
Sulphur (S)	0.43
Sodium (Na)	0.02
Total	100

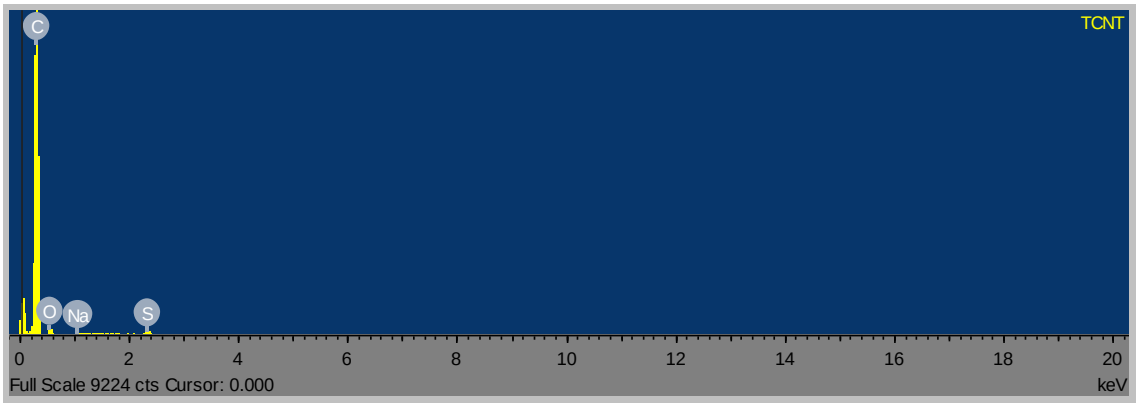

Fig. 3: EDX Spectrum of MWCNTs

FTIR Results of the MWCNTs

Surface chemistry is the main parameter for determining the functional group of a material. The sample spectrum was recorded over the range of 400-4000 cm^{-1} as shown in Fig. 4 and the summary of the major peaks are presented in Table 2. The spectrum of MWCNTs from Fig. (4) shows sharp peak band at 3458.48 cm^{-1} which may be attributed to O-H group resulting from the treatment of multi-walled carbon nanotubes with strong acids containing oxygen group. However, combination treatment of nitric and sulphuric acid significantly contributed in improvement of surface functional groups such as hydroxyl groups (-OH), carbonyl group (C=O) and carboxylic group (-COOH). The presence of peaks related to the main oxygen containing groups such as O-H, C=O and C-O on the surface of the MWCNTs were expected to form strong surface complexes with metal ions. The values obtained are in support with analysis of functionalized MWCNTs by [10]. Noticeable peak at 2910.68 cm^{-1} appears after purification and functionalization corresponding to C-H deformation in alkane which shows the stability of carbon nanotubes suspensions in the aqueous phase. However, peak at 1637.62 cm^{-1} indicates C=O stretching vibrations of carbonyl groups signifying the expansion of carboxylation on the surfaces of the functionalized multi-walled carbon nanotubes. The vibration mode at 1541.18 cm^{-1} may be attributed to C=C in aromatics or C=O moieties of conjugated system. The bands at 1340.57 and 1508.36 cm^{-1} are connected with sulphates and nitrates respectively. The bands below 873.78 cm^{-1} may be related to out of plane bending modes. The various FT-IR spectrum obtained were in agreement with functionalized MWCNTs biosorbent reported by and functionalized MWCNTs with tris (2-aminoethyl) amine of .

Table 2: Assignment of Some Major Peaks on the MWCNTs

Assignments of the major peaks	Wave number (cm^{-1})	References
-OH stretching vibration	3458.48	[2]
C=O	1637.62	[23]
C-H bending vibration	2910.68	[8]
C=C vibration mode	1541.18	[23]

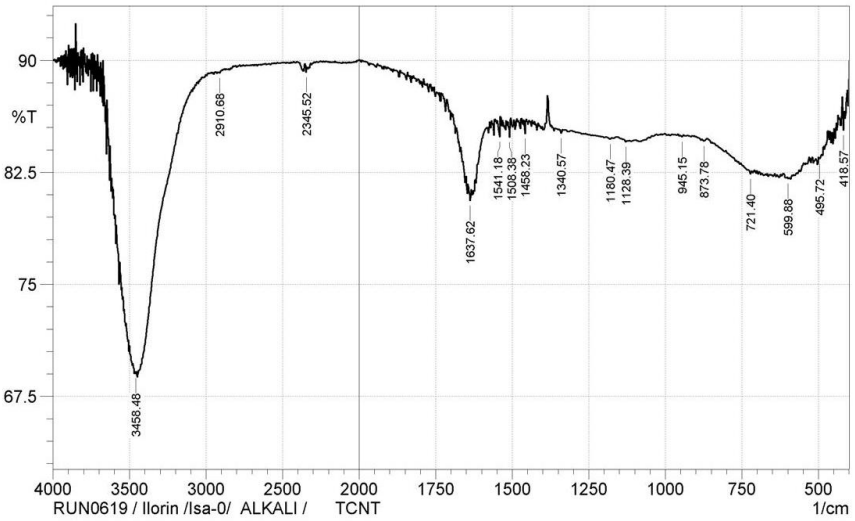

Fig. 4: FT-IR spectrum of MWCNTs

BET Result of the MWCNTs

Brunauer Emmett-Teller (BET) Theory seeks to explain the physical adsorption of gas molecules onto solid surfaces. The N_2physiorption isotherm of the MWCNTs is displayed in Fig. 5 and the summary of the BET surface area results are as presented in Table 3. It can be observed from Table 3 that BET surface area of MWCNTs was 865.66 m^2/g. The surface area results obtained are higher compared with 246 m^2/g value report for Jujube pit adsorbent by. The value obtained from the study is within the minimum range of 500 to 1500 m^2/g needed for industrial application and removal of small molecules from aqueous solution. Similarly, the N_2 adsorption-desorption isotherms results showed that the material belongs to type IV isotherms with values in the range of 2 to 5 nm according to IUPAC classification of adsorption isotherm. The pore volume and pore area distributions of the material determined from the BET as shown in Table 3 indicate mesoporous nanomaterial with capillary condensation.

Table 3: Summary of the BETresults on the MWCNTs

BET parameters	Values
	865.660
Surface Area (m^2/g)	
Single point surface area at P/P_o	985.660
t-Plot micro-pore area	850.440
t-Plot external surface area	0.220
Langmuir Surface Area	750.580
BJH Adsorption cumulative surface area of pores	650.445
BJH Desorption cumulative surface area of pores	650.455

Pore volume (cm^3/g)	
Single point adsorption total pores volume	0.624668
t-Plot micro-pore volume	0.050150
BJH Desorption cumulative volume of pores	0.453300
Pore size (nm)	
Adsorption average pore width (4V/A by BET)	3.35540
Desorption average pore width (4V/A by BET)	3.35544
BJH Adsorption average pore width (4 V/A)	5.5250
BJH Desorption average pore width (4V/A)L	5.0550

BET = Brunauer-Emmett-Teller, Surface Area Analysis BJH = Barrett-Joyner-Halenda, Pore Size and Volume Analysis

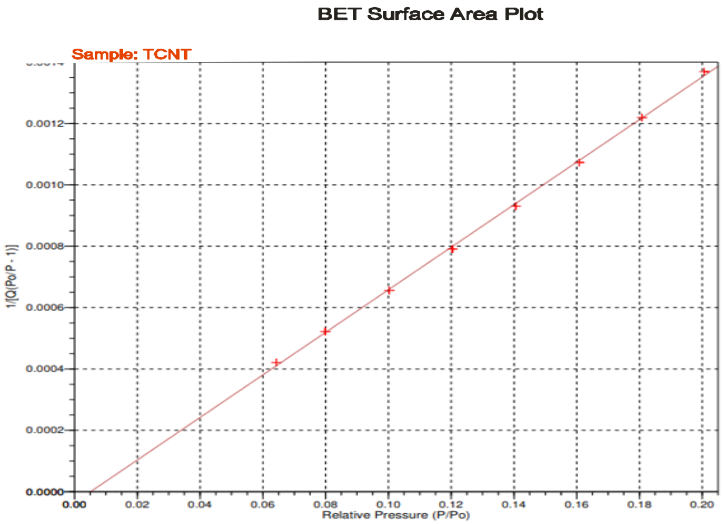

Fig. 5: BET Plot of MWCNTs

XRD Result

The X-ray diffraction pattern of a pure substance is like a fingerprint of the substance. The powder diffraction method is thus ideally suited for characterization and identification of polycrystalline phases. The characterization reaction may lead to changes in molecular and crystalline structure of the MWCNTs, hence XRD will provide plausible explanation on the proportion of each crystalline phase of the material. An overlay of the diffractogram of the MWCNTs is shown in Fig. 6. Similarly, the list of scattering angles of $2\theta^o$ of the material is presented in Table 4.

It can be observed from the information of XRD that the diffraction peaks at $2\theta = 34.85^o$ is related to graphene structure of the MWCNTs and the value obtained is in agreement with literature reported by. Other prominent diffraction peaks obtained are in the range of $20^o < 2\theta < 80^o$ which is common to all carbon containing materials, and corresponds to the sp^3 lattice reflections. The positions of all maxima coincided with the peak characteristics of crystalline structure of cellulose and all the peaks obtained were in agreement with literature reports of [33-35]. Obviously, the peaks obtained are in accordance with the International Centre for Diffraction Database (ICDD) and Joint Committee on Powder Diffraction System (JCPD).

Table 4: List of Scattering Angles of the MWCNTs

Code	Observed Scattering Angles, 2θ^o
MWCNT	10.53^o, 20.44^o, 34.85^o, 40.93^o, 58.18^o, 70.36^o, 80.49^o

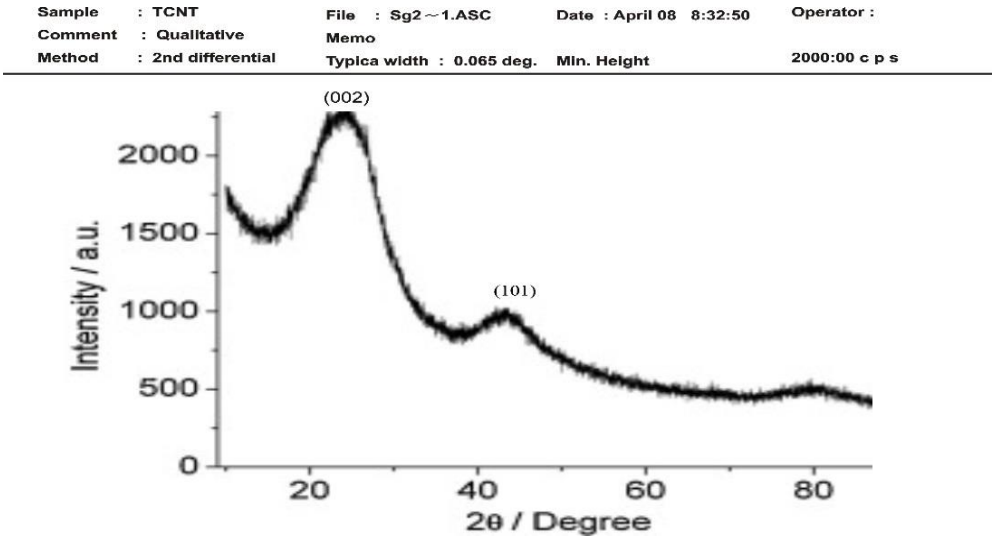

Fig. 6: XRD Spectrum of MWCNT

Conclusion

In this study, the Multi-Walled Carbon Nanotubes were successfully prepared and functionalized using HNO_3 and H_2SO_4 acid solutions as chemical medium under reflux condition. The morphological study was carried out using Scanning Electron Microscopy (SEM) which confirmed large cavities and sparsely web shape while Energy Dispersive X-ray (EDX) gave atomic distribution of the adsorbents and revealed carbon and oxygen as major elements. The (FT-IR) result confirmed major surface functional groups such as hydroxyl groups (-OH), carbonyl group (C=O) and carboxylic group (-COOH) on the nanotube. The BET surface area result indicated that the MWCNTs have higher surface area. The investigation of mineral structure and degree of crystallinity were determined by X-ray Diffraction (XRD) and the results confirmed prominent diffraction peaks which is common to all carbonaceous material. Therefore, the rapidly improving field of nanotechnology, nanomaterials are on the leading front. Their special property most especially the size gives them an edge over other materials. This improves their applications in various human activities.

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