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Novel functionalization treatment of MWCNTs for unmanned aerial vehicle structure

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Abstract. Present typescript encompasses the influences of functionalization on the carbon nanotubes (CNTs) using acid oxidation which has been widely reported not only as a purification process but also as functionalization process because it is an effect on the outer surface on CNTs. Therefore, using strong acid effect and high power sonication in addition to the nature of the acid mixture (H_2SO_4/HNO_3) with CNTs confirm that adequate volume of carboxyl groups decorate the outer surface, this functionalization process improve the dispersibility of the CNT in the response of the final dimension of the CNTs. Because of using a high temperature for a long time more than 6 hours.

This work interested in the aspect ratio (L/D) and the final shape of the tubes by using a mild temperature process 50°C with concentration acid for not long time 5 hours, in an attempt to make experimental condition which guarantees good functionalization of the surface of MWNTs with minimizing the CNTs damage, The results were confirmed by Thermogravimetry Analysis (TGA), Raman Spectroscopy, Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM) analysis.

In addition to the functionalization process we try to save the loss in the weight during the filtration step, in addition to decrease the time during filtration of the materials after sonication. Where the results show that actually functionalization has been done with an adequate amount at this condition, and the dilution for 2 days before filtration have been approved to be easier and faster than direct filtration after treatment.

1. Introduction

After discovering Carbon nano-tube by Iijima in 1991 [1], it is becoming extremely difficult to ignore amazing new carbon allotrope exist. It has tube structure within nano-scale and high aspect ratio (length/diameter). Carbon sp² hybridization has a special different structure, such as fullerene (0D), carbon nanotubes (CNTs, 1D), graphene (2D), and graphite (3D) [2]. CNTs have 3 different shapes which can be imagined as one sheet of graphene rolled up to consist one tube (single-walled CNTs, SWCNTs) or up to tens and hundreds of graphene cylinders concentrically stacked with an adjacent layer spacing (Multi-walled CNTs, MWCNTs) seamless or only two sheets of graphene to form (double-walled CNTs, DWCNTs). CNTs can be a synthesis in large quantities by three dominant techniques: arc discharge [1], laser ablation [3] and chemical vapor deposition (CVD, including high-pressure carbon monoxide (HiPco) process) [1].

Arc discharge simply about two electrodes (at least one electrode is made of graphite) through which a direct current (DC) is passed in a gaseous atmosphere. MWCNTs can be obtained by arc discharge without any metal catalyst, while mixed metal catalysts inserted into the anode are required when synthesizing SWCNTs by this method. In laser ablation for producing CNTs, an intense laser beam is used to ablate/ vaporize a target consisting of a mixture of graphite and metal catalyst in a flow of inert



gas. This method favours the growth of SWCNTs with a controlled diameter depending on the reaction temperature.

CVD always contain catalyst-assisted decomposition of hydrocarbons growth of CNTs over the catalyst (usually transition metals such as Ni, Fe, Co, etc.) can be achieved by using CVD. Control over diameter, shell number, and growth rate of CNTs are also realized with this method. The chief drawback of CVD is the high defect density of the obtained CNTs owing to low synthesis temperatures, compared with arc discharge and laser ablation.

CNTs have potential mechanical, electrical, and thermo-mechanics properties but to get maximization of the benefits of these proprieties one parameter must be reached an optimum situation, it is the dispersion where to get utilize from the power of the CNTs, it should first achieve good dispersion of CNTs with a polymer. Therefore, scientist directed to improve CNTs dispersibility, as well as improve interfacial bonding in the composite, there was different method to enhance the dispersion of CNTs (mechanically, chemically...etc) in aqueous solutions and to develop new hybrid materials. The chemically method one of the reliable way to improve dispersibility by functionalization, The most effective method for functionalization and purification in the same time is the wet chemical oxidation. Liquid phase oxidation would promote the oxygen content on the CNTs which indicated to the functional group (OH, O=H, COOH) to the MWCNTs, carboxyl group has a great effect in dispersing MWCNTs in an aqueous solution which is useful in the fabrication of CNTs/polymer composite. The applications of polymer/CNT nano-composites are also very broad in industrial and structural applications such as aerospace, radar-absorbing material for plane, sporting goods, fuel cell, and many more.

The most widely used method in liquid oxidation procedure is the acid treatment using any of the following reagent (HNO_3 , H_2O_2 , H_2SO_4 , KMNO_4 ...) or mixture of them [2], although one of the main disadvantages of acid oxidation method is highly damaging in the graphitic network and CNTs fragmentation. For suitable load transfer between polymer and reinforcement (CNTs) large aspect ratio (>200) is required, therefore that high aspect ratio also improve electrical prosperities due to low electrical percolation and as a result get good dispersion.

Rosca and co-workers [3] oxidized MWCNTs using nitric acid for up to 48 h and obtained successful CNT oxidation at the expense of large CNT shortening caused by the prolonged exposure time to concentrated HNO_3 . Su et al. [4] report on purification of MWCNTs employing different concentrations of $\text{HNO}_3/\text{H}_2\text{SO}_4$ for 5–20 h, observing also CNT destruction controlled by the exposure time. Zhou et al. [5] reported that a mixture of HNO_3 and H_2SO_4 applied for more than 6 h to MWCNTs can successfully purify and oxidize the nanotubes, but it also causes CNT fragmentation, and they also mention the influences of temperature on the process.

Relatively fewer researches have been reported on the relationship between the functionalization parameters (acid concentration, treatment time, and sonication power.) and the amount of functional groups “nano-defects” on the tube. It was always the primary objective from this process is the purification and functionalization of CNTs, however. There is a close relationship between the latter parameters and the CNTs damage, for example, higher treatment temperatures easily cause significant weight loss in CNTs and hyperthermal acid mixture leading to serious degradation of sidewalls even for a short treatment time (<1 h). So it seems reasonable to find adequate mild experimental conditions that can drive to successful functionalization without seriously CNT damage. So it obvious that we should have control on the pre-treatment process to preserve CNTs high aspect ratio, Besides the acid concentration and exposition time, the high and localized ultrasonic power. Therefore, the objective of this study was to examine the feasibility of mild acid treatment to functionalize the surface of MWCNTs without causing severe CNT fragmentation.

Many of Researchers have studied CNT to define its suitability in applications in aerospace and aeronautical fields. In aerospace there were different topics under active study to improvement the mechanical and the electrical properties of composite made from epoxy resin and CNTs. The advanced features of CNTs that provide to strengthen resins and polymers has amplified entrance in different scientific fields, at the forefront of the scientific fields is Aerospace engineering. That is due to introduce

of nano-composite materials a larger extent in the currently advanced commercial aircraft construction (such as Airbus A380 and Boeing 787).

CNTs can be achieved main requirements of the applications in aeronautics and unmanned aerial vehicles, such as “lightweight, increased speed, and manoeuvrability”, but there were many Constraints in the use of CNT in aerospace applications such as improve the dispersibility, increase the scale of production, alignment issue, and misunderstanding of their toxicity. So this work are interested in enhance the dispersibility to get the most out of CNTs used with nano-composite materials.

2. Experimental methods

2.1. Raw materials

The pristine MWCNTs were provided by Petroleum Research Institute (EPRI), Egypt by means of the catalytically chemical vapor deposition. The average diameter of the pristine MWCNTs is about 10-40 nm calculated by TEM, and the average length is around 10-15 μm measured by SEM and with purity >90%. The sulphuric acid (H_2SO_4 , 97%) was obtained from scharlau and nitric acid (HNO_3 , 61%) was obtained from alpha chemika.

2.2. Functionalization

Chemical oxidation was carried out using a mixture of nitric acid and sulphuric acid. For the oxidative treatment, 2 g of the agglomerated MWCNTs was mixed with 240 ml of the mixture of acid solutions (HNO_3 : H_2SO_4 = 1:3, v/v) then it was sonicated by the ultrasonic bath for 5 h at 50°C. Then the collected CNTs was washed with distilled water until pH to neutral and collected on the filter paper. The oxidized MWCNTs were dried in oven at 70°C overnight.

So as to control tube damage, low sonication bath power, a comparatively low process temperature and suitable exposure time in acid were used. Therefore, the mixture was sonicated in a conventional ultrasonic bath (150 W, 42 kHz) for only 5h, promoting CNT disentanglement within the acid solution. Then, the slurry was filtered with three methods to get easier and faster one.

- Method (1): After the sonication process, we dilute the mixture in D.W with 1:50 for 3 days to get settling of the CNTs in the bottom, then remove the liquid slowly to get the CNTs from the bottom and direct filter it and called A-MWCNT 1.
- Method (2): In the same manner but dilute the mixture in D.W with 1:5 for 3 days to get settling of the CNTs in the bottom, then repeat the same as the previous method then called A-MWCNTs 2.
- Method (3): Also dilute the mixture in D.W with 1:5 but only for 2 days to get settling of the CNTs in the bottom, then repeat the same as the previous method and called A-MWCNT 3.

2.3. Characterizations

To investigate the quantity of impurity, amount of metal impurities, and the thermo-oxidative stability of MWCNTs, we used thermogravimetric analysis (TGA) which it was conducted on a thermogravimetric analyzer (TA Instruments, TGA Q500, STCE, Egypt) with a heating rate of 10°C/min from 25°C to 800°C and under nitrogen gas flow of 50 ml/min.

The structure defect of MWCNTs was studied by (SENTERRA II, Brucker optic Co. Ltd., Egypt) with 532 nm green lasers and 12.5 μW laser power. The laser beam was focused on the 20x objective.

A Fourier transform spectrometer (FTIR 6300, JASCO, Egypt) was used in detecting the Functional groups in MWCNTs (MWCNTs: KBr = 1:250, weight fraction) were checked using a Fourier in the wave number ranges of 500–4000 cm^{-1} .

The microstructure characterizations of MWCNTs was observed with SEM (ZEISS, JSM 6700F, MTC, Egypt) and with TEM (JOEL, JEM 2100, Epri, Egypt). So as to analyze the chemical structures and morphology of the samples, and In order to get the chemical construction elements EDS are used.

3. Results and discussion

3.1. Thermogravimetric analysis

TGA characterization is effective to indicate the purity and thermal stability of the materials, where there is three important parameter analyses of measured data have an effective role in this characterize (the weight-loss curve, initiation temperature, and the oxidation temperature).

The initiation temperature is the temperature where the material starts to decompose. The oxidation temperature is the point at maximum weight loss, and it appears as a peak in the derivative of the weight loss as a function of temperature. The oxidation temperature is usually attributed to the thermal stability of the material. The oxidation of the materials depends mainly on the crystalline, where highly crystalline MWCNTs should have more resistance to oxidation relative to the other allotrope of carbon[6].

Decorating the nanotubes by function groups or introducing nano-defect to the surface of materials lead to decreasing the oxidation temperature. The size also has an effect decomposition temperature, where compared by Kim et al. [7] MWCNTs with small diameter were have lower decompose temperature than the larger one. The same as length where found that the decomposition temperature was shifted to lower related to decrease the length of MWCNTs [8].

The difference structural of carbon nano-tube lead to a change in oxidation behaviour corresponding to each time for the reactive sites.

For pristine MWCNTs the thermal decomposition of occurring as a multistage process, Start with a weight loss of less than 1% is detected at a temperature of 150°C, then the weight loss between 150°C to 350°C about 3% attributed to Amorphous carbon contaminates which also reflect that MWCNTs purity is so good (above 90%) as mentioned.

The MWCNTs exhibited excellent thermal stability above 600°C without degradation in absence of oxygen due to use nitrogen (inert gas) during oxidation.

For acid-treated as shown in Fig (1), the weight loss for the three sample exhibited an increase in the oxidation rate until temperature about 350°C which attribute to the decarboxylation of the carboxylic groups exist on the MWCNT walls [9]. But the A-MWCNTs 2 showed a sharp decrease in the weight loss which attributes to increase the amount of disorder due to increase the exposition time of MWCNTs with diluted acid with a low amount of dilution 1:5.

At the temperatures above 500°C the observed decomposition corresponds to the thermal oxidation of the remaining amorphous carbon or disordered carbons tend to be oxidized at around 500°C [10]. Also according to the shown curves the A-MWCNTs 1 curve exhibited good thermal stability and the oxidation temperature start above 600°C due to existing oxygen due to acid treatment but the three day with dilute acid lead to more disorder site on the outer surface of the tube.

So as shown in this characterization according to the curves A-MWCNTs 3 is the most recommended method because of increasing oxidation temperature which exhibit high thermal stability and also due to decrease amount of the total weight loss in the sample which attribute to low amount of site defect.

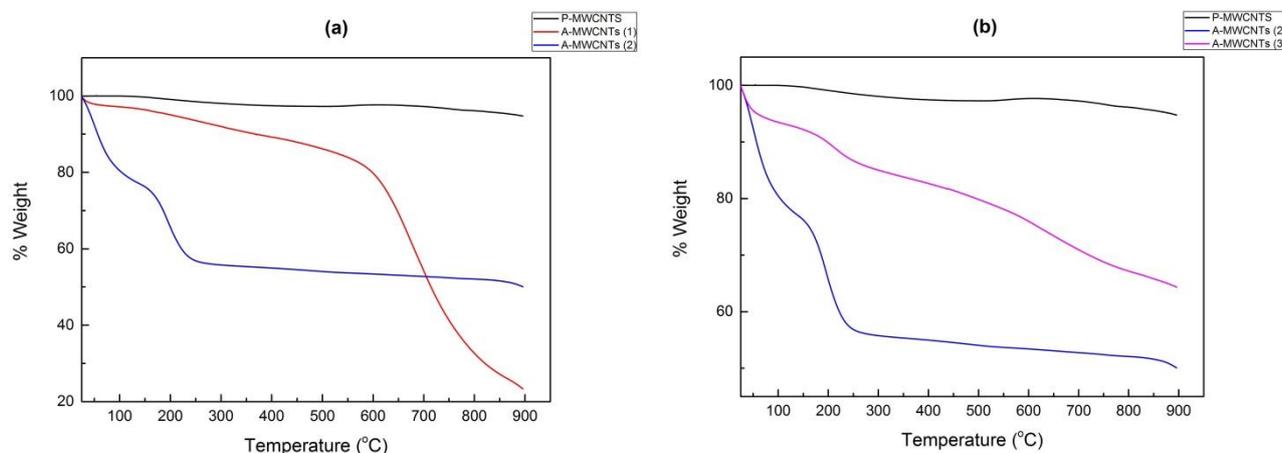


Figure 1. Thermogravimetric curves of as-received and acid-treated MWCNTs. Label (a) change in dilution (b) change in time.

Table 1. TGA analysis data.

Treatments	Total weight (%) loss	Initiation temperature (°C)	Oxidation temperature(°C)
P-MWCNT	5.310%	190.56	620.83
A-MWCNT 1	76.40%	671.57	671.57
A-MWCNT 2	49.93%	52.4	196.09
A-MWCNT 3	35.64%	207.43	652.66

3.2. Raman spectroscopy

Raman spectroscopy of MWCNTs is a great valuable approach on the degree of structural defect, the bonds between carbons in CNTs structure and also Raman can be used to evaluate the surface of CNTs before and after functionalization. The technique used in this research is to analyze a doping of functional group corresponding to the oxidative with the mixture of acids. The pristine MWCNTs and treated with different condition MWCNTs (A-MWCNTs 1, A-MWCNTs 2, A-MWCNTs 3) was comparatively analyzed depends on the destructive level as a result of using acid treatment fig (2).

There were two major peaks, the characteristics and intensity of this band give good impression on the order of layers graphitization of the carbon nano-structured forms, the D-band at 1325 cm^{-1} attribute to the disorder induced to the structure due to structural imperfections or impurities, and peak at 1570 cm^{-1} called G-band which attribute to the tangential c-c stretching vibration due to integrity of hexagonal carbon[11].

The ratio of the intensity of D-band and G-band is a very important parameter to verify if there were functional groups or not by measure the defect in the sample. It was noticed Increase the ratio (I_D/I_G) of functionalized MWCNTs compared with that of pristine MWCNT due to introduce of functional groups to the carbon nanotubes surface. This result gives an indication that quiet defects were introduced on the surface of MWCNTs; therefore, oxygen-containing groups were already linked with MWCNTs side walls.

And also we notice increasing the ratio (R) slightly differ between the three method depending on amount of dilution with D.W and time of settlement which for A-MWCNT1 have dilute with large amount of D.W so the ph of the solution is large compared to the A-MWCNT 2 and as a result the disorder should reduce as shown by the ratio $(I_D/I_G)_1=0.69$, $(I_D/I_G)_2=0.84$.

So the ratio (R) due to the other two methods depends on the time where the samples immersed in more acidity solution which allows attacking the surface so the ratio (R) increases slightly. In case compare between A-MWCNT 2 and A-MWCNT 3 is obviously the influence of time contains acidity solution so A-MWCNT 2 has also increased in the ratio (R) compare to other methods. So the method A-MWCNT 3 is the more preferable in the expanse of interesting the aspect ratio of the tube and easy way to filtration

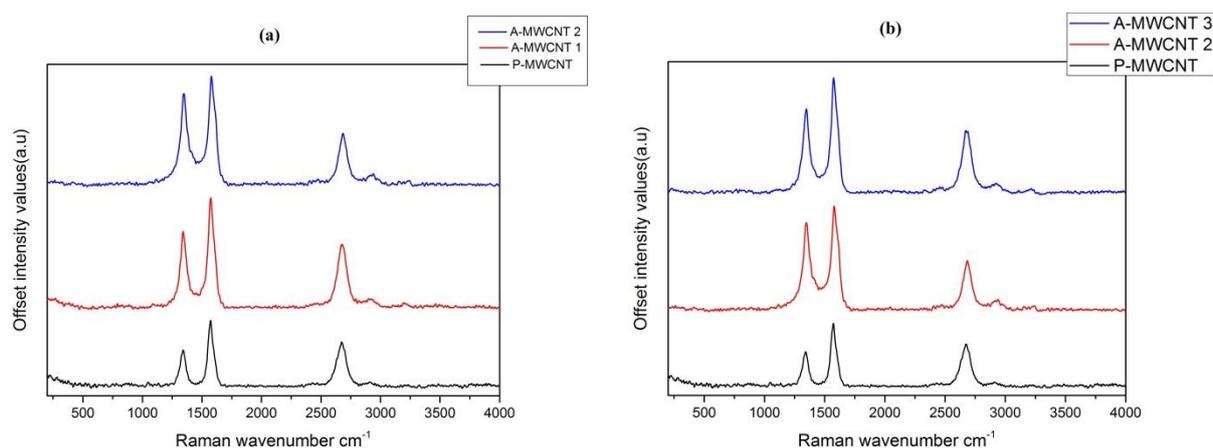


Figure 2. Raman curves of as-received and acid-treated MWCNTs. Label (a) change in dilution ratio (b) change in time.

Moreover, second-order 2D feature at about 2700 cm^{-1} is observed for every sample which also called G' band. Another alternative using the G' band to get an assessment on MWCNT purity, where the typical way use Raman spectroscopy for indicate sample purity have dependant on the intensity ratio of the D-band peak and the G-band peak (I_D/I_G) is not clear as the carbon disorder in the sample effect in these intensities. So the scientists found that the ratio using G' band peak is a more accurate alternative to assess the sample purity and quality [15]. Where the impact of the G' band is because of a two-phonon process, so its capacity is particularly tricky to the virtue, as the perplexity would not take into consideration the coupling sway crucial for the two-photon process. As shown in the table the high value indicate sample purity which proves that the samples get more defect site than pristine as $I_{G'}/I_G$ of the treated decrease compare to the pristine. As proved earlier A-MWCNT 3 is the suitable way according to the previous table (2).

Table2. Raman analysis data.

Treatments	D-position	G-position	I_D/I_G	$I_{G'}/I_G$
P-MWCNT	1342	1572	0.58	0.677
A-MWCNT 1	1341	1573	0.69	0.578
A-MWCNT 2	1346	1578	0.84	0.475
A-MWCNT 3	1344	1574	0.73	0.571

3.3. FTIR spectroscopy

FTIR is mainly used In order to characterize the functional groups of MWNTs, Total Reflection Infrared from 500 to 4000 cm^{-1} spectra has been conducted on pristine MWCNT and acid-treated MWCNT Fig (3). As shown in as-received MWCNT the spectra implies important to peak at 3441 cm^{-1} which assigned to OH stretching, 2928 cm^{-1} which attributed to symmetric and asymmetric CH_2 stretching, notice peak at 1690 cm^{-1} attributed to $-\text{O}-\text{H}$ groups of adsorbed water or covalently bonded functional groups,

however the moisture water in the KBR pellets and the samples has been traced by extensive heating before the measurement (24 h at 70 °C), it could not be surely removed. So we supposed that a majority of the O–H vibrations related to the moisture water in the sample rather than assumed it as functional groups attached to the surface of the CNTs. also, peak at 1616 cm^{-1} band refers to the conjugated C=C stretching, and 1040 cm^{-1} (corresponding to C–O stretch in alcohols).

For acid-treated samples Appearance of peaks at band 1700-1730 cm^{-1} assigns carbonyl (C=O) stretching vibration of carboxyl groups (R-COOH) The high intensity of this band have a great impression that the functionalization process is adequate under these treatments condition, as will be further proved by surface and other spectroscopic analysis.

And also as shown the spectrum of the three methods of filtrations have roughly the same peaks appear around 1713 cm^{-1} , 1620 cm^{-1} and 1395 cm^{-1} which refer to carboxyl groups, carbonyl, and ketone respectively.

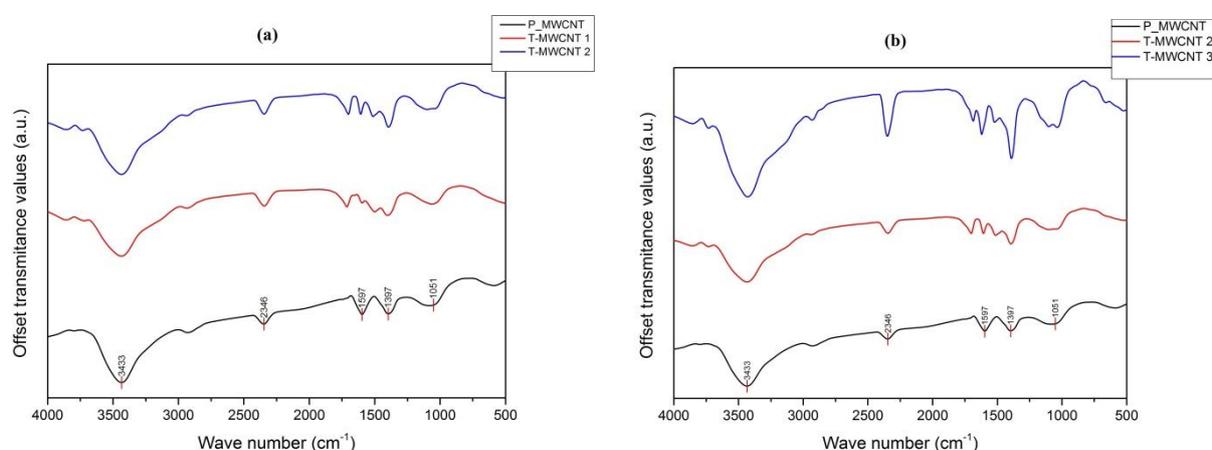


Figure 3. FT-IR spectra of as-received and acid-treated MWCNTs. Label (a) change in dilution (b) change in time.

3.4. Morphology

3.4.1. Scanning electron microscopy (SEM). Scanning electron microscopy was used to provide with a possible indication to MWCNTs fragmentation and change in diameter occurred during treatment. Fig (4) Shows typical SEM images of the pristine CNT (A), (a) as well as those acid-treated 1(B),(b), an acid-treated 2(C),(c), and acid-treated 3(D),(d). In spite of the fact that our various endeavors (by SEM), without a couple of single nanotubes at the edges of the example were hard to get absolutely so a solid measurable length appropriation was impractical to acquire. Despite this restriction, a general but clear assessment can be made depend on image analysis. Several SEM images Analysis for treated MWCNTs give us a reliable length measurement of only a few single tubes, and all of them were around 10 μm , similar to the pristine material. SEM characterization evidence of CNT degradation occur as result to harsh acid treatment has been reported by reducing the micron-length of the pristine nanotubes into small fragments of the order of hundreds of nanometers, or by degrading the length of the long nanotubes to particle material like carbonaceous. But In our study, no severe CNT fragmentation is observed after the acid treatments, see Fig (3). , although a slight statistical reducing in the nanotubes lengths cannot be fully discarded.

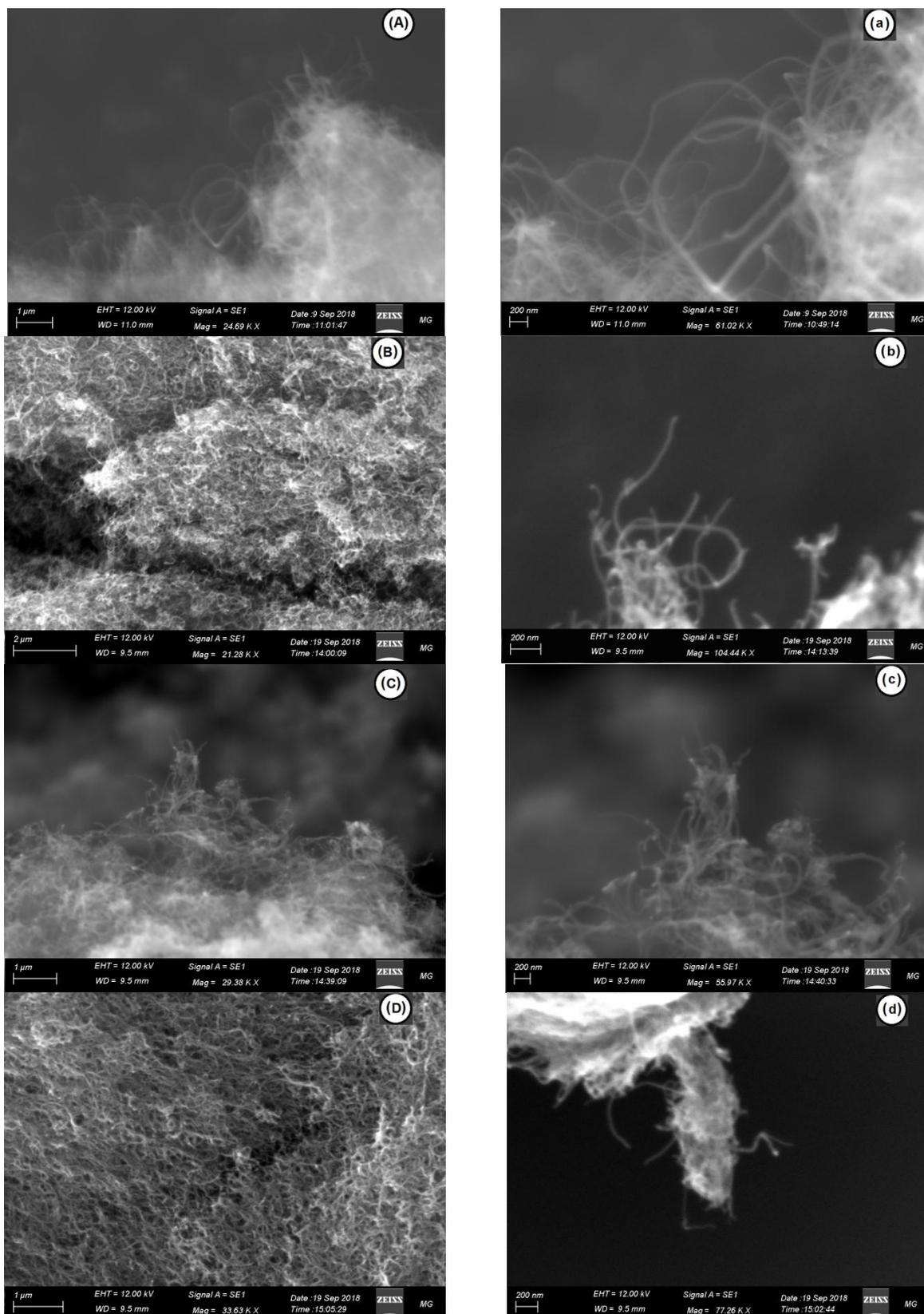


Figure 4. SEM images of as-received and acid-treated MWCNTs. Labels A, B, C and D with Scale bar is 1 μm and Labels a, b, c and d with Scale bar is 200 nm.

EDS analysis was conducted on different areas of the pristine and treated MWCNTs, which give a good indication of the elements and concentration found in the sample as represented in the listed table (3). As apparent in this table, by far, the carbon is the largest element content in the materials (either graphitic or amorphous), with notice oxygen content and some metal impurities such as Mg, Mo, and Co. after the treatment, it is observed that the content of the impurities significantly decrease which give established role of acids as purification agents.

Table 3. EDS analysis data.

Element	P-MWCNT	A-MWCNT 1	A-MWCNT 2	A-MWCNT 3
C	93.37	91.05	89.86	91.65
O	3.84	6.05	8.73	6.93
Co	1.83	1.43	0.09	0.65
Mo	0.96	0.12	0.25	0.77

3.4.2. Transmission electron microscopy (TEM). The tube structure and morphology of the pristine and functionalized MWCNTs were observed with TEM Fig (5). It is obviously that there are change in the outer and the inner surface of the tube.

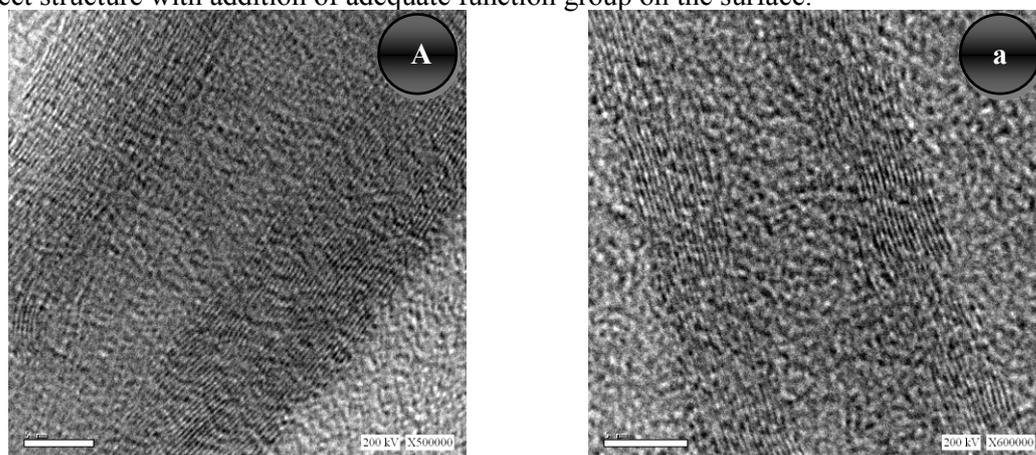
TEM analysis shown for pristine MWCNTs long tube ended with semi spherical end caps. Also black region displaying in the MWCNTs assigned to exist of amorphous carbon. In addition that some dark circle central white spot are noticed in the end of the tubes.

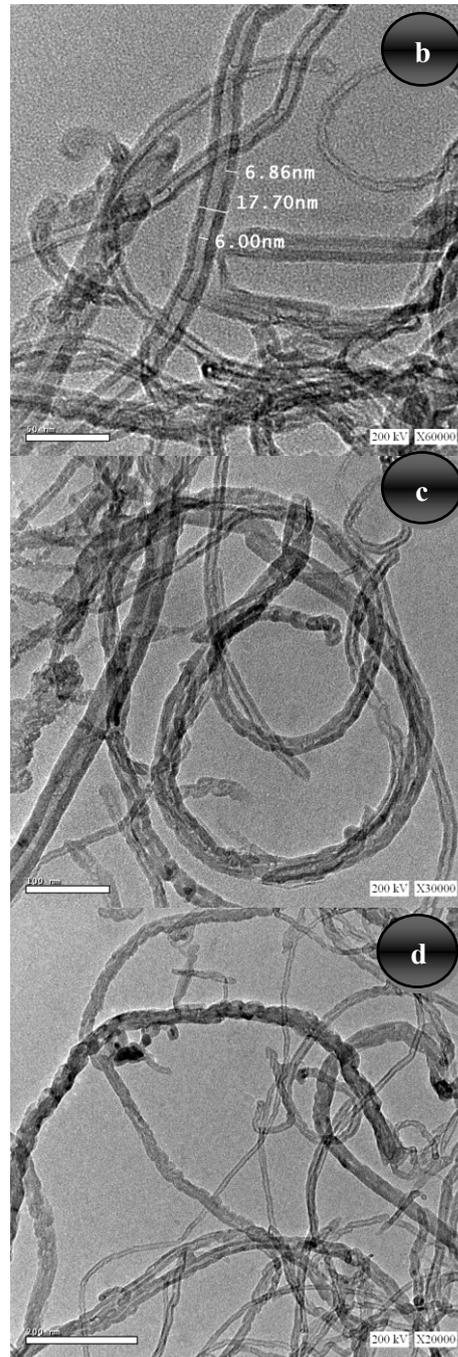
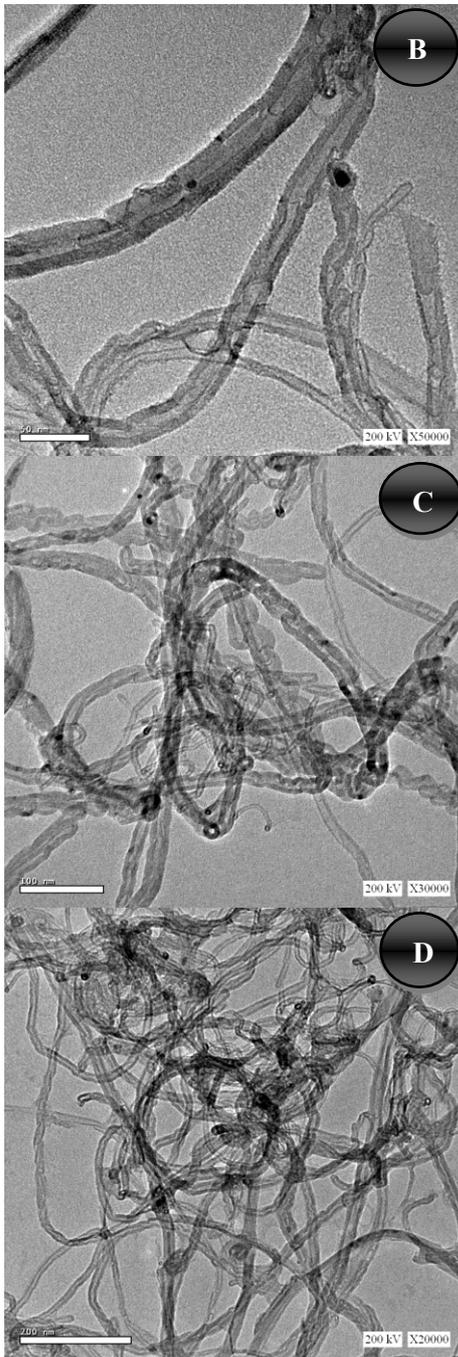
The other inset from Fig (5) (a,b,c,d,e,f) for functionalized MWCNTs display rough surface of the tubes which be an attribute to attachment of carboxylic group on the outer surface. Also shown straight end cut, which mean that the acid success to open it by introducing function group (-COOH). Therefore the acid has to remove most of amorphous carbon, catalyst and other carbonaceous impurities. So it clearly that the functionalization process with that condition preserve the MWCNTs from break and save the perfect structure with addition of adequate function group on the surface.

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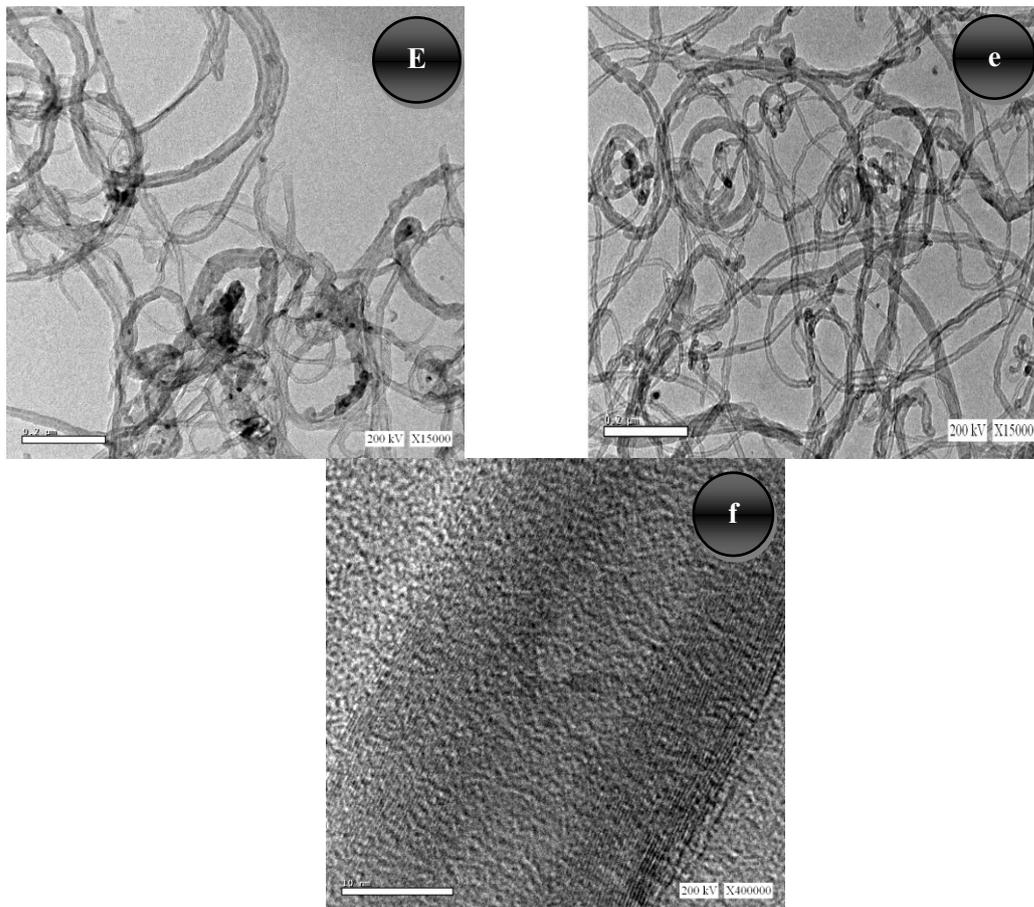


Figure 5. TEM images of as-received and acid-treated MWCNTs. Labels A, B, C, D and E for as-received and Labels a, b, c, d, e and f with Scale bar is (5, 10, 50, 100, 200 nm).

4. Conclusion

In summary, three mild chemical oxidation treatments depend on the mixture of sulphuric and nitric acids have been used to introduce functional groups to the surface as shown in TGA analysis to help in improving the dispersion of carbon nano-tube. Due to decreasing the treatment time (5h), low sonication power, and relative low treatment temperature (50°C) the sample nano-defect can be controlled in order to minimize the sample damage. It was noticed that these conditions can achieve good functionalization of CNTs reducing its fragmentation.

FT-IR analysis promotes an introduction of the OH and C=O groups to the surface of MWCNTs, therefore ID/IG ratio of acid-treated MWCNTs in “Raman analysis” showed an increase as a result to the acid treatments which give an indication to actually introduce functional groups to the MWCNTs surface.

SEM observations revealed that the morphology of the CNTs after acid treatment almost still without aggressive degradation in nanotubes shape, in addition, to preserving the aspect ratio as possible, and the EDS analysis prove an increase in the oxygen content and decrease the metal catalyst impurities.

TEM analysis agree well with the first characterization TGA, FT-IR, Raman, and SEM in good functionalization of MWCNTs with preserve the aspect ratio of the tube from degradation.

MWCNTs subjected to 5 hours of acid treatment were considered to have a homogeneous dispersion in ethanol mainly owing to the formation of carboxyl groups, as confirmed by FTIR spectra. It was promoted that MWCNTs acid-treated for 5 h and diluted with 5:1 distilled water for 2 days “A-MWCNT 3” then filter and dried are qualified for strengthening the composite and create improved matrix

composite due to ensure introduce sufficient carboxylic groups which in turn improve dispersion without any series deterioration of the outer MWCNT layers.

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