

Research Article

Common Wet Chemical Agents for Purifying Multiwalled Carbon Nanotubes

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Purification and functionalization of multiwalled carbon nanotubes (MWCNTs) are challenging but vital for their effective applications in various fields including water purification technologies, optoelectronics, biosensors, fuel cells, and electrode arrays. The currently available purification techniques, often complicated and time consuming, yielded shortened and curled MWCNTs that are not suitable for applications in certain fields such as membrane technologies, hybrid catalysis, optoelectronics, and sensor developments. Here we described the H_2O_2 synergy on the actions of HCl and KOH in purifying and functionalizing pristine MWCNTs. The method (HCl/H₂O₂) showed 100% purification yield as compared to HCl and KOH/H₂O₂ with purification yields 93.46 and 3.92%, respectively. We probed the findings using transmission electron microscope, energy dispersive X-ray spectroscope, attenuated total reflectance infrared spectroscope, Raman spectroscope, thermal gravimetric analysis, and X-ray powder diffraction. The study is a new avenue for simple, rapid, low cost, and scalable purification of pristine MWCNTs for application in versatile fields.

1. Introduction

Carbon nanotubes (CNTs) are one of the most fascinating nanomaterials having many attractive and useful physicochemical properties such as mechanical [1, 2], thermal [3], electrical [2, 4], and optoelectronic properties [5]. Since CNTs were defined by Iijima in 1991 [6], they have played significant shoot in many domains including water purification technologies [7–9], polymer, and composites [10–12], conductive cable fibers [13], hydrogen storage media [14], biomedical sciences [15], field emission displays [16], electrochemistry, and nanosensors [17], nanoelectrodes and microarrays [18], and many other versatile fields [16].

CNTs are often received or synthesized with other generic impurities such as nonnanotube or amorphous carbons, ash, and metal catalysts with extreme hydrophobicity [19]. The existing CNT synthesis methods added the impurities such as metal catalysts to increase the yield and reduce cost [20].

The level of these unwanted matters depends on the specific method used for CNT synthesis. Whatever might be the method of choice, impurities often hinder CNTs performances and confuse the understanding of their original functionalities, limiting their applications in many important fields. For instance, this triggered CNT aggregation in various polymers [21], which destabilizes polymer's mechanical strength and electrical conductivity [21].

In order to purify pristine CNTs, three classes of CNT purification methods such as chemical, physical, and a combination of both were developed [19]. Chemical purification method was effective because of its selectivity, sensitivity, faster rate kinetics of the oxidation of carbonaceous impurities, and metal catalysts dissolution from the pristine CNTs [22]. In addition, chemical agents are widely available and cost-effective and need simple laboratory settings. In contrast, physical method was involved in removing graphitic sheets and carbon nanospheres from CNT [19]. The method was not effective to remove carbon impurities, often complicated and time consuming [19]. Therefore, based on pristine CNT impurities, one can select chemical, physical, or a combination of both in order to get pure CNT with desired properties.

HCl, H₂O₂, and KOH are easily available simple wet chemicals that are commonly found in most of the ordinary laboratories. Here we treated MWCNTs with HCl [23, 24] as a reference. But HCl is often incapable of completing removal of nonnanotube impurities [25]. Pristine few walled carbon nanotubes (FWCNTs) were oxidized and purified by H_2O_2 [26], but the method thus far is not extensively studied for multiwalled carbon nanotubes (MWCNTs) purification except few [22, 27]. Although single-walled carbon nanotubes (SWCNTs) were purified by a mixture of HCl and H₂O₂ [25, 28-31], no study has yet been adopted for MWCNT purification by using that mixture. Therefore, we studied here the synergistic effects of HCl/H₂O₂ mixture for improving the carbon yield and getting purified well graphitic layer of MWCNTs. A basic treatment involving NH₄OH/H₂O₂ was effective in purifying MWCNTs [32]. We developed and applied another basic wet oxidizing technique involving a mixture of KOH/H₂O₂ in purifying MWCNTs. The methods (HCl, HCl/H₂O₂, and KOH/H₂O₂) were effective to get oxidized MWCNTs, but high purification yield was observed for HCl/H₂O₂ compared with HCl and KOH/H₂O₂. We focused on MWCNTs because of their low preparation costs and availability in large quantities.

Transmission electron microscope (TEM) was used to study the morphological changes of MWCNTs. Energy dispersive X-ray spectroscope (EDX) was used for the analysis of elemental composition and state of impurities. Attenuated total reflectance infrared (ATR-IR) spectroscope was performed to estimate the degree and type of functionalization. Raman spectroscope was conducted to measure the defects and character of graphite bands, and thermal gravimetric analysis (TGA) was used to record the mass profile of pristine and treated MWCNTs. Finally, X-ray powder diffraction (XRD) was used to define crystallinity, in-plane regularity, and lattice profiles.

2. Experimental Section

2.1. Materials. Pristine MWCNTs of 12 ± 5 and 4 nm in outer and inner diameters and >1 μ m in length were bought from Bayer MaterialScience AG (Germany). The tubes were prepared by catalytic chemical vapor deposition (CCVD) and contained >95% carbon by weight and were used as received. Hydrochloric acid and hydrogen peroxide were purchased from Merck Sdn Bhd. (Malaysia). Potassium hydroxide, ethanol, and acetone were purchased from Sigma-Aldrich Sdn Bhd. (Malaysia). The purity of all reagents was ≥99% except hydrochloric acid (37%), hydrogen peroxide (30%), and ethanol (70%) in water.

2.2. Instrumentation. TEM (Hitachi-HT7700, Japan) was used for the morphological characterizations of the MWC-NTs. It was performed at 120 kV. An EDX coupled with a FE-SEM (QUANTA FEG 450, FEI, USA) was used for

elemental analysis. An X-Max Silicon Drift detector (Oxford, UK) of 80 mm² was used to identify the elements and energy and relative intensity of emitted X-rays were analyzed at 10 Kev. ATR-IR spectra were recorded on a KBr using an IR spectrometer (IFS 66 v/S, Bruker, Germany). Raman spectra were acquired for 10 min at a laser power of 100 on Ar⁺ laser (514 nm) focused (50X objective) on a spot size of about 1.5–2.0 μ m (Renishaw inVia, UK). TGA (TGA/SDTA 851, Mettler Toledo, USA) was performed under air flow (50 mL) between 25 and 1000°C at 10°C/min. XRD diffracted patterns were collected at Ni filtered Cu K α radiation (40 kV, 40 mA, λ = 1.5401 Å) (XRD/D8, Bruker, Germany).

2.3. Wet Chemical Treatments of MWCNTs. Three wet chemical treatments were performed to purify and oxidize the asobtained pristine MWCNTs.

(*i*) *HCl Treatment*. Pristine MWCNTs (0.5 g) were treated with 100 mL of hydrochloric acid (36 wt%) and sonicated at 50°C for 3 h in an ultrasonication bath (Series 400; POWERSONIC, 40 KHz; Korea) [24]. The method was as a reference.

(*ii*) HCl/H_2O_2 Treatment. Pristine MWCNTs (0.5 g) were dispersed into 25 mL mixture (70:30) of hydrochloric acid (36 wt%) and hydrogen peroxide (30 wt%), and the final mixture was sonicated at 50°C for 5 h at 40 KHz.

(*iii*) KOH/H_2O_2 Treatment. Pristine MWCNTs (0.5 g) were dispersed into a 20 mL mixture (50:50) of potassium hydroxide (25 wt%) and hydrogen peroxide (30 wt%), and the mixture was sonicated at 50°C for 5 h at 40 KHz.

2.4. Removal of the Residual Impurities. All treated MWCNTs were extracted from the residual acids, bases, metallic byproducts, and carbonaceous impurities by repeated cycle of dilution and centrifugation (dissolved in 1L of deionized water and centrifuged (Eppendorf-5430R; Germany) at 7000 rpm for 30 min). The supernatant was carefully collected when the MWCNTs were precipitated at the bottom of the polyethylene centrifuge tube. The procedure was repeated 5-6 times until the resistivity of the supernatant was greater than 0.5 MΩ·cm and pH was ~7.0. The treated MWCNTs were then rinsed with ethanol (70 wt%) and dried overnight in a vacuum oven at 100°C.

3. Results and Discussion

3.1. Predicted Chemical Reactions of HCl, H_2O_2 , and KOH with MWCNTs. To get pure MWCNTs, agents such as HCl, HCl/H₂O₂, and KOH/H₂O₂ were found to be promising (Figure 1). The method (HCl/H₂O₂) can purify MWCNTs through different routes. The metals that are usually present in pristine CNTs act as catalysts to produce hydroxyl radical (OH^o) through Fenton's chemistry [33] (Figure 1(a)), which is stronger oxidizing agent than H₂O₂:

 $H_2O_2 + metal (reduced) \longrightarrow OH^{\circ} + OH^{-} + metal (oxidized)$ (1)



FIGURE 1: Schematic of the localized catalytic reactions of HCl/H₂O₂ (a), (b), and (c) and KOH/H₂O₂ (d), (e), and (f) with pristine MWCNTs.

The radical (OH^o) is then reacted with amorphous carbon impurities of pristine CNTs [22] and converted them into CO_2 [25] (Figure 1(b)):

$$4OH^{o} + C \longrightarrow CO_{2} + 2H_{2}O$$
⁽²⁾

The oxidized metals and other impurities are then dissolved into HCl (Figure 1(c)), which are subsequently removed through filtering and washings.

However, a mixture of KOH and H_2O_2 was unable to complete amorphous carbon oxidation and removal of metal impurities from MWCNT. This is because of the chemical reactions between KOH and H_2O_2 (Figures 1(d), 1(e), and 1(f)). KOH decreases the availability of H_2O_2 in the system, so there is the least chance to produce free radicals and other etching agents. The ultimate products of the reaction were KOH and O_2 . Oxygen was evaporated while KOH may have some chemical interactions with amorphous carbons, which might be negligible to remove MWCNT core impurities.

3.2. TEM Analysis. TEM microscope was used to closely examine the contents of amorphous carbon and location of metal catalysts trapped into the tubular interstitial spaces of pristine and treated MWCNTs (Figure 2). Pristine MWCNTs reflected clumped, cloudy, and amorphous carbon containing MWCNTs (Figure 2(a)). The impure carbonaceous particles were found to be wrapped around the nanotube structures, and metal catalysts were trapped into the MWCNTs. Although the overall amorphous carbons and metals were removed from the nanotube surface after HCl treatment, some MWCNTs were thick suggesting small percentages of nonnanotube carbonaceous agents and impurities may present on MWCNT surfaces (Figure 2(b)). The HCl/H₂O₂ treatment produced much cleaner, fresh, and complete amorphous carbon and metals-free MWCNTs and the nanotubes appeared in thin and loosely connected bundles (Figure 2(c)). Although the oxidizing strength of H₂O₂ is high (Pka 11.6), it did not produce vigorous CNT fragmentations upon the purification process in presence of HCl. In the KOH/H₂O₂ treatment, some of the amorphous carbons from MWCNT surfaces were removed. However, the effects were local since the presence of some nonnanotube carbon impurities was globally obvious (Figure 2(d)) and the nanotubes appeared more flattened and thick than those of HCl (Figure 2(b)) and HCl/H₂O₂ (Figure 2(c)) treated MWCNTs.

3.3. EDX Analysis. EDX is a significant characterization tool for measuring the extent of CNT oxidation and elemental composition [22]. EDX findings of the pristine and treated MWCNTs are shown in Figure 3 and representative analysis is listed in Table 1. Purification yield of pristine and treated MWCNTs was calculated based on the following:

Purification yield (%) :
$$\frac{W_0 - W_t}{W_0} \times 100,$$
 (3)

where W_0 is the metal content of the pristine MWCNT (%) and W_t is the metal content of purified MWCNT (%).

As we observed in Table 1, by far, the largest element in the as-received pristine MWCNT is carbon (either graphitic

FIGURE 2: TEM images of pristine (a), HCl (b), HCl/H₂O₂ (c), and KOH/H₂O₂ treated MWCNTs (d).

TABLE 1: Elemental analysis of before and after MWCNT treatments in this study.

Specimen	Treatment time (h)	Elemental composition (Wt%)							
		С	Ο	Со	Mn	Al	Mg	Sum	Purification yield (%)
Pristine MWCNT	0	97.35	1.12	0.48	0.46	0.3	0.29	100	_
MWCNT-HCl	5	98.77	1.13	0	0	0.1	0	100	93.46
MWCNT-HCl/H ₂ O ₂	5	98.78	1.22	0	0	0	0	100	100
MWCNT-KOH/H ₂ O ₂	5	95.19	3.34	0.45	0.42	0.3	0.3	100	3.92

or amorphous), with some extent of oxygen (Figure 3(a)). However, pristine MWCNTs were highly contaminated with metal impurities such as Co, Mn, Al, and Mg (Figure 3(a)). After wet chemical agent treatments, it was observed that the amount of graphitic carbons was slightly increased, since most of the metal impurities diminish significantly, given the established role of HCl and HCl/H₂O₂ as good purification yields of 93.46 and 100%, respectively (Figures 3(b) and 3(c)). In contrast, KOH/H₂O₂ was incapable of completing removal of metal impurities and showed lowest purification yield 3.92% (Figure 3(d)). Herein we hypothesized that the HCl/H₂O₂ mixture can be a judicial choice for the complete purification of pristine MWCNTs compared with HCl and KOH/H₂O₂ treatments.

3.4. ATR-IR Analysis. ATR-IR spectroscope was performed for characterizing the functionalities produced following wet chemical treatments (HCl, HCl/H₂O₂, and KOH/H₂O₂) resulting in MWCNT purifications. The IR spectra of the pristine and treated MWCNTs are depicted in Figure 4. The dominant IR spectrum at 3409 cm⁻¹ was assigned to the stretching vibration of intermolecularly hydrogen bonded OH : OH groups (Figures 4(a), 4(b), 4(c), and 4(d)) [34, 35]. The intensity of this band was low in pristine MWCNTs

(Figure 4(a)), but it was significantly increased and broadened following wet chemical treatments and purifications especially at KOH/H_2O_2 (Figure 4(d)) indicating the formation of huge -OH groups upon chemical treatments [36]. The IR transmittance peak at 2907 cm⁻¹ which was dominant in HCl/H₂O₂ (Figure 4(c)) and KOH/H₂O₂ (Figure 4(d)) treated MWCNTs was assigned to sp² and sp³ C-H stretching vibrations [37]. The transmittance bands at 2422 and 2279 cm^{-1} were observed for pristine (Figure 4(a)), HCl/ H₂O₂ (Figure 4(c)), and KOH/H₂O₂ (Figure 4(d)), but was absent in the HCl (Figure 4(b)) treated MWCNTs, respectively, may pointing out the grafting of some CO and COO⁻ groups, respectively [38]. The peak at 1630 cm⁻¹ (Figures 4(a), 4(b), 4(c), and 4(d)) was due to the stretching vibration of C=C [36] and C=O of quinone [38] that was created on MWCNT surfaces following wet chemical treatments. The highest intensity of this peak was found following KOH/H_2O_2 treatment (Figure 4(d)), suggesting the presence of more -CO groups. The prominent peak at 1044 cm⁻¹ (Figures 4(a), 4(b), 4(c), and 4(d)) was due to -OH group generated because of the atmospheric oxidation or oxidation from wet chemical treatments [38]. In addition, a peak that appeared at 804 cm⁻¹ was due to epoxy and oxirane rings evolved from C-O- groups and ring deformation of



FIGURE 3: EDX profiles of pristine (a), HCl (b), HCl/H₂O₂ (c), and KOH/H₂O₂ (d) treated MWCNTs.

substituted aromatic structures [36]. Some weak peaks that appeared in $2500-3500 \text{ cm}^{-1}$ region in pristine MWCNTs (shown by asterisks) (Figure 4(a)) disappeared following chemical treatments (Figures 4(b), 4(c), and 4(d)). It clarifies the presence of some minor functional groups of the pristine MWCNTs anchored by amorphous carbons and other carbonaceous fragments which were successfully removed by wet chemicals processing.

3.5. Raman Spectroscopy. Typically, MWCNT represents two significant high frequency bands called D- and G-bands at

1330 and 1585 cm⁻¹ for CNT structural defects and graphite in-plane vibration, respectively [39, 40]. In this study, D- and G-bands appeared at ~1349 and ~1588 cm⁻¹ both in treated and pristine MWCNTs, respectively (Figure 5).

The intensity of the D-band which is induced by nonzero center phonon mode usually depends on the presence of disordered carbon atomic networks [31, 41]. However, Figure 5 shows the D-band intensities were practically constant in both pristine and treated MWCNTs. This indicates that MWCNTs were purified with less defects density. This might be due to the milder reaction conditions among graphitic



FIGURE 4: ATR-IR spectra of pristine (a), HCl (b), HCl/ H_2O_2 (c), and KOH/ H_2O_2 (d) treated MWCNTs.



FIGURE 5: Normalized Raman spectra of pristine and treated MWCNTs.

carbons of MWCNTs and HCl, HCl/H₂O₂ and KOH/H₂O₂. The etching properties of OH^o, which was generated by Fenton's chemistry [33], may have direct affinity to oxidize amorphous carbons due to the presence of many active sites on it [32] (Figure 1(b)) rather than oxidizing graphitic layer's carbon atoms. On the other hand, KOH/H₂O₂ was unable to directly react with graphitic skeleton, since most of the amorphous carbons were wrapped around the pristine MWCNTs (Figures 1(d), 1(e), and 1(f)). However, the G-band intensities were significantly increased in treated MWCNTs especially for HCl/H₂O₂ treated MWCNTs. This clearly indicates that the HCl/H₂O₂ removed nonnanotube carbon



FIGURE 6: TGA curves of pristine and treated MWCNTs (down) and their derivative spectra (up).

impurities and metal catalysts and generated well graphitic MWCNT structure [41] compared to HCl and KOH/ H_2O_2 while maintaining intact MWCNT integrity (Figure 5).

Finally, the purity states of the pristine and treated MWCNTs were compared from the intensity ratio of the G ($I_{\rm G}$) and D-bands ($I_{\rm D}$) [32]. The highest ratio (0.91) of $I_{\rm G}/I_{\rm D}$ was found for HCl/H₂O₂ treated MWCNTs, suggesting the better efficiency of HCl/H₂O₂ in removing amorphous and carbonaceous materials from MWCNTs [26]. The $I_{\rm G}/I_{\rm D}$ ratios for HCl (0.81) and KOH/H₂O₂ (0.73) treated MWCNTs were less effective in complete removal of nonnanotube carbon impurities and metal catalysts from pristine MWCNT surfaces (Figure 5).

3.6. TGA Analysis. TGA was performed to measure the amorphous carbons, oxidation defects, and overall quality of purified MWCNTs. TGA of pristine and treated MWCNTs (down) with their derivative spectra (up) are presented in Figure 6. By oxidation temperature, herein, we mean the temperature where MWCNTs lose their weight and thus show the highest derivative weight curve. This can define the stability of MWCNTs at a given temperature. At first pristine and KOH/H2O2 treated MWCNTs showed lowest decomposition temperatures at around 100°C and lost their weights of about 1 and 70%, respectively, which correspond to the pyrolytic evolution of hydroxyl and/or water [32]. Typically, amorphous carbons oxidized at around 500°C [42] due to their lower activation energy and the presence of many heat sensitive active sites [32]. TGA of pristine and KOH/H₂O₂ treated MWCNTs showed highest decomposition temperatures at 550°C and loss of their weights of about 5 and 75%, respectively (Figure 6). However, pure and well graphitic carbon skeletons are commonly reacted at relatively



FIGURE 7: XRD patterns of pristine (a), HCl (b), HCl/H₂O₂ (c), and KOH/H₂O₂ (d) treated MWCNTs.

higher temperature ranges between 600 and 700°C [43]. TGA of HCl and HCl/H₂O₂ (Figure 6) treated MWCNTs started to weight loss of about 5 and 10%, respectively, at 600°C, suggesting the efficacy of these chemicals in purifying MWCNTs. Remaining disordered carbons present in all MWCNTs showed complete weight loss between 650 and 800°C [32].

3.7. XRD Analysis. The two characteristic XRD peaks of MWCNTs for two important phases such as 002 and 100 in the range of $2\theta = (10-60^{\circ})$ (Figure 7) were followed in this study [44]. The peak characterizing the interlayer spacing (002) of CNT tubular walls was observed at $2\theta = 26.08$ and 26.03° (mean) for pristine (Figure 7(a)) and treated MWCNTs (Figures 7(b), 7(c), and 7(d)), respectively. This indicates that the graphitic structure of MWCNTs was not destroyed during the purification process [45]. The peak defining in-plane regularity (100) appeared at $2\theta = 43.34$

and 43.26° (mean) for pristine (Figure 7(a)) and treated MWCNTs (Figures 7(b), 7(c), and 7(d)), respectively. When all parts of MWCNTs are absolutely parallel to the 002 plane, the intensity of the peak 100 often decreases or vanishes [27]. Presence of nonnanotube impurities and debris can change carbon ordering and increase roughness of the CNT surface lattice structure. The intensity at plane 100 was 8.01 < 10.44 < 12.25 < 19.37 for HCl/H₂O₂ (Figure 7(c)), HCl (Figure 7(b)), KOH/H₂O₂ (Figure 7(d)), and pristine MWCNTs (Figure 7(a)), respectively. This suggested that the HCl/H₂O₂ treated MWCNT had well parallel MWCNT lattice structure because of complete elimination of nonnanotube impurities as compared to HCl and KOH/H₂O₂.

4. Conclusions

The purification of MWCNTs using three common wet chemical agents (HCl, HCl/ H_2O_2 , and KOH/ H_2O_2) is presented. The HCl/H_2O_2 mixture produced more cleanly and well orderly MWCNT skeleton compared with that of ordinary HCl. Although considerable removal of amorphous carbon is possible with KOH/H₂O₂ treatment, complete MWCNT purification is out of place. Thus the MWCNT purities depend on the nature of wet chemical agents and impurities present in the overall MWCNT structure.

Conflict of Interests

The authors declare no conflict of interests.

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