

Enhanced Dispersion and Functionalization of Multiwalled Carbon Nanotubes via Mild Mixed Acid Sonication Treatment

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ABSTRACT

Multi-walled carbon nanotubes (MWCNTs) have excellent characteristics, including mechanical strength and durability, as well as electrical and thermal conductivity, which make them desirable for materials science, sensing, and nanotechnology applications. MWCNTs are hydrophobic and chemical inert materials, therefore causing considerable agglomeration and poor dispersion when they are to be dispersed in a polar liquid. This study investigated the effectiveness of the MWCNT surface modification via functionalization (adding functional groups to the surface of MWCNTs) and dispersion properties of MWCNTs after mild-acid sonication with either mixed-acid (6 M Nitric Acid and Sulfuric Acid) or single acid (6 M Nitric Acid). The functionalization and dispersion properties were characterized by using FTIR, Raman, XRD, and SEM. FTIR analysis confirmed the formation of oxygen-containing functional groups, with a characteristic C=O peak at $\sim 1720\text{ cm}^{-1}$. Raman analysis showed that functional groups formed covalent bonds and that there was no significant structural degradation. SEM analyses indicated that there is a large difference between pristine MWCNTs, as agglomerated (pristine) and functionalized and dispersed (uniformly dispersed). It was determined that although the mixed-acid sonication approach produced more stable dispersions than single-acid sonication did, there was no difference in MWCNT structures (functional group formation relative to preservation of MWCNT structural integrity). The optimal sonication duration time to achieve the best balance of MWCNT functional group incorporation and MWCNT structural integrity was 2-4 hours. Based on the results of this study, it can be concluded that functionalizing and dispersing MWCNTs using a mild mixed-acid sonication treatment is an effective and simple way to achieve those goals.

Keywords: Multiwalled Carbon Nanotubes (MWCNTs), Mild acid functionalization, Dispersion stability, Mixed acid oxidation, Surface modification, Nanomaterials characterization, Acid functionalization

1. INTRODUCTION

Across a wide range of industries, including nanotechnology, materials science, and biomedical engineering, the need for new materials with improved mechanical, electrical, and thermal properties continues to grow [1]. Multi-walled carbon nanotubes (MWCNTs) are particularly good candidates due to their superior properties, including high tensile strength, high electrical and thermal conductivity, high aspect ratios, and large specific surface areas [2]. These hollow nanostructures composed of layer-upon-layer of graphene, discovered by Iijima in 1991, have been studied for application in sensors, composites, and hybrid nanomaterials since the discovery of carbon nanotubes.

The main barrier to the practical use of MWCNTs, however, is the inert, hydrophobic nature of their surfaces, which means they do not dissolve well in most polar solutions and physiological fluids [3]. The inability to solubilize MWCNTs effectively hampers their compatibility with matrices and devices, making them much less valuable for applications. Many researchers have sought to overcome this issue by

employing various surface functionalization techniques. When surface functionalization is done non-covalently via methods such as van der Waals attraction, π - π stacking, and/or hydrogen bonding, the ability to improve MWCNT dispersal can be achieved while still maintaining the MWCNTs' original sp^2 -carbon structure. Unfortunately, the weak and reversible nature of van der Waals attractions, π - π stacking, and/or hydrogen bonding results in limited stability over time [4], [5].

Parallel with this, covalently functionalizing by means of controlled oxidation, generally through an acid such as nitric acid (HNO_3), either alone or in conjunction with another acid such as sulfuric acid (H_2SO_4), results in the addition of oxygen-containing compounds on the surface of a multiwall carbon nanotube (MWCNT), including -COOH, C=O, and -OH [6]. The traditional method of acid functionalizing MWCNTs is by reflux oxidation (at very high temperature and under extreme acidic conditions) for long periods of time (which can result in a high build-up of structural defects and high energy consumption). However, this study proposes a milder mixed-acid sonication method (ultrasonic treatment) that produces more oxygen-

containing functional groups while causing less damage to the nanotubes, using a lower process intensity and being more efficient at dispersing nanotubes.

Taken together, these functional groups enhance MWCNTs' hydrophilic and active properties, significantly improving their dispersion stability in polar solvents and opening possibilities for further chemical modifications. It must be noted that care must be taken when oxidizing MWCNTs because too many functional groups can result in an imbalance in structural integrity and the introduction of functional groups. Over-treatment of MWCNTs with acids may cause "oxidative cutting," leading to defects and fragmentation, thereby decreasing the aspect ratio of the MWCNTs and adversely affecting their mechanical and electrical properties [7], [8]. To the best of our knowledge, systematic optimization of sonication duration under mild mixed-acid conditions for balancing dispersibility and structural preservation remains insufficiently reported.

The objective of this study is to provide an effective and simple way to functionalize MWCNTs using an easy to perform method for the application of sonication (a process whereby sound or ultrasonic vibrations are used to assist in the addition of functional groups to the surface of a MWCNT) under low levels of acidity, which introduces -COOH groups into the MWCNT and substantially increases MWCNT's dispersion characteristics (without significant degradation). In order to assess how effective each of the two (2) acid treatment approaches to functionalize MWCNT and improve dispersion characteristics was, the mixed acid and single acid approaches (6 M HNO₃) were utilized to compare their effects. Structural and morphological differences were confirmed through characterization methods such as Fourier Transform Infrared Spectroscopy (FTIR), Raman Spectroscopy (RS), X-ray Diffraction (XRD) and Scanning Electron Microscopy (SEM) to ensure that the desired effects were achieved. An optimized process to produce functionalized MWCNTs found in this study is displayed in Figure 1.

2. THEORETICAL BACKGROUND

Multi-walled carbon nanotubes (MWCNTs) are hollow tubes at the nanoscale, composed of multiple concentric layers of a form of carbon called graphene. Each layer consists of sp²-hybridised carbon atoms linked to form a continuous sheet of graphene and the properties of MWCNTs are determined largely due to the fact that MWCNTs are formed from graphitic carbon material, which means that MWCNTs have superior tensile strength, electrical and thermal conductivity, high aspect ratios, and very large specific surface areas [9]. These characteristics make them very valuable to sensor applications, composites, and hybrid nanomaterials.

However, due to the chemical inertness and hydrophobic nature of unmodified MWCNTs, the practical uses of MWCNTs are limited. Also, the strong van der Waals forces and π - π stacking interactions between neighbouring MWCNTs contribute to nanotube aggregation, thereby

limiting their solubility and stability in polar solvents and biological environments. As a result, the effective surface area of the individual nanotubes is significantly reduced (due to aggregation), and the interfacial compatibility between the components in use can be compromised; thus, the performance of MWCNT-based materials can be adversely affected by nanotube aggregation.

Among other means, a widely used method to modify the interfacial characteristics of MWCNTs, and improve their dispersion and stability in solutions, is through surface functionalization. A less common method for functionalizing MWCNTs is non-covalent functionalization, which can improve the dispersion of MWCNTs while maintaining their sp² carbon backbone; however, due to the weak and reversible nature of the interactions between the functionalized groups and the MWCNTs, long-term stability is typically lacking. A more stable and effective means of chemically altering the surface of MWCNTs is through the process of covalent functionalization, which utilizes controlled oxidizing agents to oxidize MWCNTs [10]. Nitric acid has been successfully used to oxidize MWCNTs by means of acid-assisted oxidation and create covalent functional groups on the surface of MWCNTs in order to enhance the dispersion of MWCNTs in an aqueous suspension.

3. METHODOLOGY

3.1. Materials

The Pristine multiwalled carbon nanotubes (MWCNTs) were purchased from Fibermax Composites (Greece), with diameters ranging from 10–40 nm and lengths of 1–25 μ m. Sulfuric acid (H₂SO₄, 98%) and nitric acid (HNO₃, 65%) used for the functionalization processes were purchased from Merck Chemicals (Germany), while other analytical-grade reagents were supplied by Sigma-Aldrich (Malaysia). Deionized water (DIW) was consistently used in all preparation and washing steps.

3.2. Functionalization of MWCNTs

Functionalized multiwalled carbon nanotubes (MWCNTs) through oxidation of pristine MWCNTs was accomplished using two different oxidizing acid treatments: the Mixed Acid Treatment (M1) and the Nitric Acid Treatment (M2). For M1, 0.5 grams of MWCNTs were placed into a 100 mL quick-mixing mixture of sulfuric acid (H₂SO₄) and nitric acid (HNO₃) at a 3:1 (v/v) ratio. The sulfuric acid and nitric acid were diluted to 6M before adding to the MWCNTs. For M2, 0.5 grams of MWCNTs were dispersed in 100 mL of 6 M nitric acid. In both treatments, the MWCNTs were initially vortex-mixed and then subjected to ultrasonic treatment (40 kHz, 200W) for up to 6 hours, while ensuring the temperature did not exceed 40°C. The temperature of the sample was closely monitored during sonication, and ice was added as needed to prevent overheating. In addition to these standard treatments, reaction times of 0.5, 2, 4, and 6 hours were evaluated to assess the time-dependent oxidation of MWCNTs.

After sonication, the samples were cooled to room temperature, filtered through 0.22- μm PTFE membranes, and were then washed many times with deionized water to achieve a neutral pH (~ 7) for the filtrate. The solids were placed into a vacuum oven overnight at 80°C to dry. The

final product of this process will be termed functionalized MWCNTs (f-MWCNTs). These products were preserved for further testing and characterization in sealed containers. The process of functionalizing MWCNTs through these acid treatments can be seen in Figure 1 below.

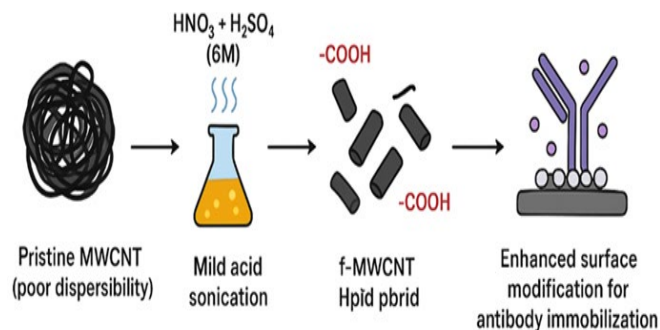


Figure 1. Flowchart of the functionalization process.

3.3. Dispersion Stability Test

In order to evaluate the quality of dispersion of f-MWCNTs, f-MWCNTs at a concentration of 1 mg/mL were dispersed in IPA and submitted to an ultrasound treatment for 30 minutes. The resulting dispersion was allowed to rest for 5 hours and again at 24 hours to determine the stability and settling patterns of the material in the M1 solvent.

3.4. Characterization Techniques

A series of analytical techniques was employed to characterize the structural and chemical properties of f-MWCNTs. FTIR (PerkinElmer Spectrum 100, 4000–400 cm^{-1} , 4 cm^{-1} resolution) was conducted on KBr pellets to identify surface functional groups. XRD (PANalytical X'Pert PRO, Cu $\text{K}\alpha$, $\lambda = 1.5406 \text{ \AA}$) was used to determine crystalline structure, while Raman spectroscopy (Horiba Lab RAM HR Evolution, 532 nm laser) evaluated structural disorder through the I_D/I_G ratio. Morphological features were examined by SEM (JEOL JSM-7001F, 15 kV) after dispersing samples in IPA, drop-casting on SiO_2 wafers, and drying at room temperature.

4. RESULTS AND DISCUSSION

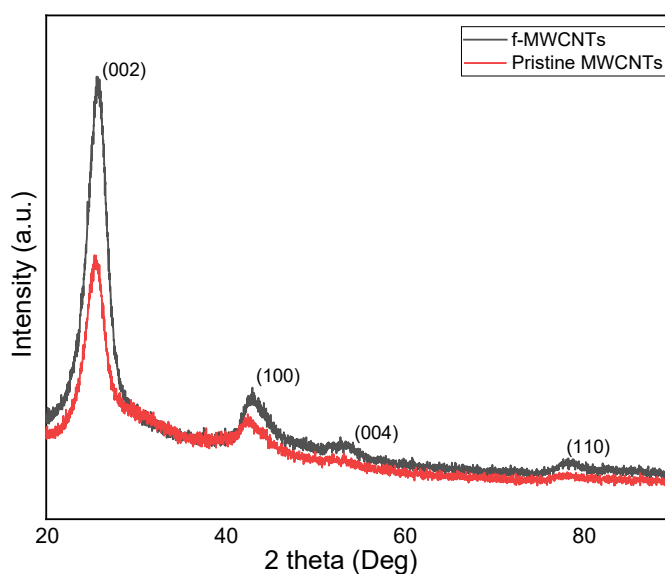


Figure 2. XRD pattern of pristine MWCNTs and f-MWCNTs.

The intensity and sharpness of the f-MWCNTs sample's primary (002) peak were compared to that of the pristine CNT. The f-MWCNT's (002) peak was noticeably sharper and more intense, indicating greater graphitic order and higher crystalline purity than the pristine CNT. The increase in crystalline purity and graphitic order for the f-MWCNTs was due to the removal of amorphous carbon and excess metal catalyst particles from the surface of the CNTs by an acid treatment process [11]. The removal of these materials

reduced the tendency of CNTs to aggregate or bundle due to van der Waals forces and allowed for the introduction of hydrophilic functional groups (i.e., COOH) on the surfaces of the CNTs, thereby giving rise to enhanced dispersibility and colloidal stability for the f-MWCNTs in polar solvents [12]. These properties are critical for the successful integration of CNTs into a variety of applications, such as nanocomposite materials, chemical and biological sensors, and biomedical devices.

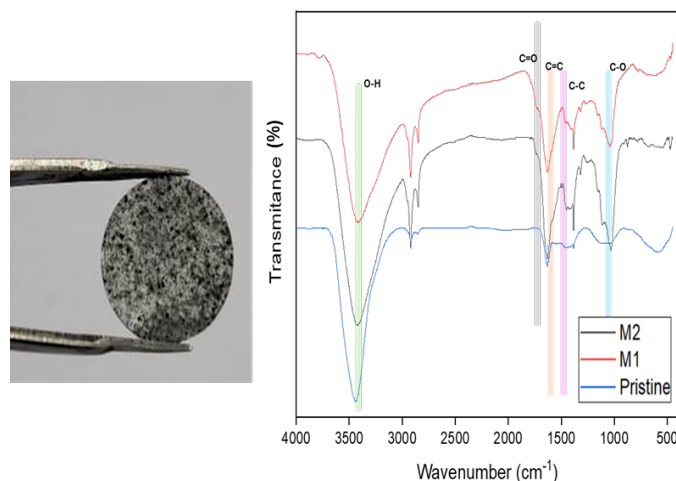


Figure 3. (a) Photograph of the prepared KBr pallet containing f-MWCNTs via mild acid treatment (raw/M1/M2) for FTIR analysis and (b) FTIR spectra of raw, M1, and M2 samples.

Successful covalent functionalization of MWCNTs by mild acid treatment was confirmed by FTIR spectroscopy, which detected the presence of new oxygen-based functional groups. The spectrum of pristine (raw) MWCNTs is used as the zero baseline and reveals only a strong peak at approximately 1580 cm⁻¹ representing the C=C stretching of the graphitic backbone.

Following the application of acid treatment on both M1 and M2, distinct new absorption bands indicative of oxidation were noted. The strongest evidence of oxidation was a sharp, intense absorption band at around 1720 cm⁻¹, corresponding to the C=O stretching vibration of the -COOH functional group on the surface of the nanotubes.

Additionally, noticeable increases in the intensity and width of the O-H stretching (~3400 cm⁻¹) and -C-O stretching (~1170 cm⁻¹) bands also support the presence of hydroxyl and carboxyl functionalization [13].

A comparative analysis of the two modifications (M1 and M2) reveals that the C=O and O-H reverse intensities in M1 are substantially higher than those in M2. Thus, it can be concluded that M1 was more oxidized than M2, indicating a greater amount of functionalization on the surface of M1. It can therefore be surmised that the conditions employed to produce the M1 sample were optimal for enhancing the degree of oxidation of the MWCNT.

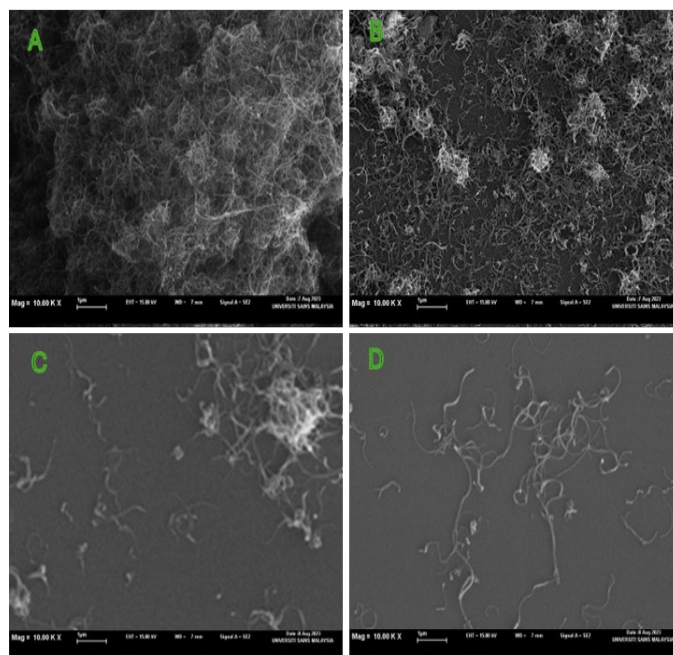


Figure 4. SEM images of (a) pristine MWCNTs, (b) f-MWCNTs, (c) f-MWCNTs with excessive structural damage, and (d) f-MWCNTs obtained at optimal functionalization time.

Analyzing SEM images of MWCNTs treated with mild acid shows that they underwent considerable morphological change and exemplifies the balance between better dispersibility and lesser structural integrity of MWCNTs after treatment with mild acids. MWCNTs in their pristine form, as depicted in Figure 4(A), form very large, tightly packed, branched networks due to extremely large van der Waals forces and π - π stacking between the nanotubes, resulting in agglomeration. The MWCNTs also exhibited significant increases in dispersion following their functionalization. With the optimal treatment duration (4 hours), as shown in Figure 4(D), it was possible to obtain MWCNTs that were highly dispersed and essentially the only nanotube present in each. The presence of oxygenated functional groups ($-\text{COOH}$, $-\text{OH}$) on the MWCNTs then

produces both electrostatic and steric repulsive forces that operate between adjacent tubes, which means that they can overcome the attractive forces of agglomerates, leading to a stable de-agglomerated suspension[14]. Conversely, as with any other type of treatment, extended times in contact with strong oxidizing agents will cause structural degradation on the MWCNTs (shown in Figure 4(C)). Also, extended times increase the likelihood of oxidative cutting. The appearance of noticeably shorter and fragmented MWCNTs is likely a result of the strong acids preferentially attacking defect sites and strained bonds on the MWCNTs. This will cause the scission of the tubes [14], [15]. Therefore, structural degradation may result in the formation of a more poorly wetted, rougher surface on MWCNTs. This is an undesirable effect.

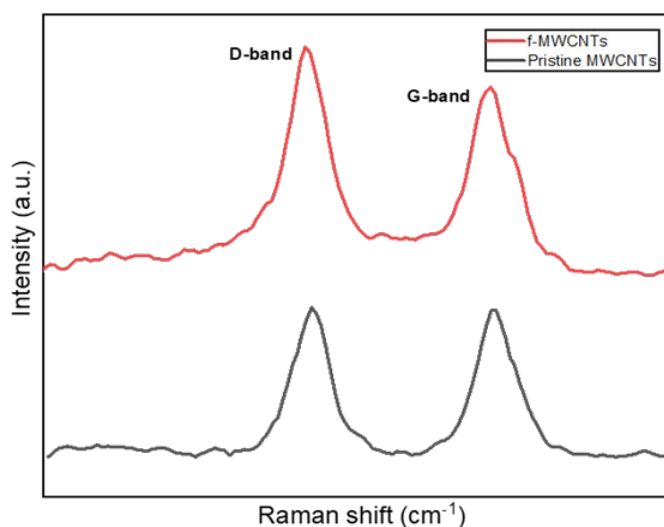


Figure 5. Raman shows structural change after using mild acid treatment.

The Raman spectroscopy analysis of MWCNT structural modifications produced by acid functionalization show that there is a direct correlation between MWCNT performance in biosensing and the functionalization of MWCNT. As illustrated in Figure 5, both pristine MWCNTs and f-MWCNTs exhibit two main spectral features: the D-band ($\sim 1350\text{ cm}^{-1}$), which corresponds to the presence of disorder or defect sites on the material; and the G-band ($\sim 1580\text{ cm}^{-1}$), which is due to the E_{2g} phonon mode of sp^2 hybridized carbon atoms within a graphitic lattice. Furthermore, the intensity ratio of the D-band to G-band (I_D/I_G) is a commonly accepted measure of MWCNT structural disorder and defect density.

The pristine Multi-Walled Carbon Nanotubes (MWCNTs) show a relatively low value of the ratio of I_D/I_G , which means that they have a more ordered graphitic (graphitic is a synonym of graphite, so there would be only one word) structure. The f-MWCNTs have a high degree of the ratio of I_D/I_G indicating that during the acid treatment process there was an introduction of sp^3 hybridization (this is a way of indicating which type of hybridization was produced) that

produced the observed defect with a significant amount of oxygenated functional groups (i.e. $-\text{COOH}$ and $-\text{OH}$) being added to the outer surface and breaking bonds along the sides of the MWCNT [16], [17]. Although excessive damage could negatively affect the material by reducing conductivity, the controlled increase in this case will provide advantages for biosensor applications. The newly formed functional groups provide a chemical point of attachment for the immobilization of biomolecules such as antibodies, enzymes, or DNA probes thereby increasing the specificity and stability of the sensors. In addition to improving wettability and increasing electron-transfer rate at the electrode interface, the addition of defects will promote uniform film formation and enhance electrochemical performance. Thus, it is evident from the Raman Spectroscopy results of the f-MWCNTs that tailoring the surface chemistry with multiple defects will not degrade the structure but it will enhance the usability for integration into electrochemical biosensors.

Table 1 Dispersibility test in solution as a function of mild acid treatment

Time/Type	Pristine MWCNTs	0.5 hour functionalized	2 hour functionalized	4 hour functionalized	6 hour functionalized
After 5 minutes					
After 5 hours					
After 24 hours					

The data presented in Table 1 provide evidence that the amount of time that MWCNTs are treated with acid can affect how well they will be dispersed within an aqueous solution and, therefore, how well they are able to be used successfully to fabricate biosensors through optimal functionalization (the addition of chemical groups to the surface of the MWCNTs). Pristine MWCNTs were the least dispersed, quickly settling after 5 minutes and showing a large amount of sedimentation, or "clumping," after 5 hours and 24 hours, which is an indication of how strong the van der Waals attraction between the MWCNTs is, as well as their hydrophobic properties [18], [19]. The initial treatment time of 0.5 hours shows some improvement in the MWCNTs' suspension. However, substantial sedimentation was still observed at both 5 and 24 hours

post-treatment, suggesting that the degree of grafting of hydrophilic functional groups (i.e., groups that would help MWCNTs disperse in an aqueous solution and form stable suspensions) was insufficient to counteract the forces between the MWCNTs. After undergoing 2 hours of acid treatment. However, the MWCNTs showed significantly improved dispersibility and remained stable with only a small amount of sedimentation after 24 hours of acid treatment (i.e., increased ability to remain suspended) [20]. This means that as the amount of time the MWCNTs have been subjected to the acid treatment increased, a more favorable balance was achieved between the increased incorporation of hydrophilic groups (such as $-\text{COOH}$ and $-\text{OH}$) onto the surfaces of the MWCNTs along with maintaining the structural integrity of the nanotubes

themselves. The functional group modifications increase electrostatic repulsion and steric hindrance between different nanotubes and therefore result in more stable aqueous dispersion(s) of MWCNTs.

With longer treatments (4 or 6 hours), the material will have improved long-term dispersibility due to the uniformly dispersed form obtained after sonication. Additionally, the greater long-term stability of this form may be attributed to oxidative degradation occurring at too severe a level. Oxidative degradation of MWCNTs occurs during acid treatment, resulting in fragmentation as they become shorter and more compressed as the outer graphitic layers are removed. Ultimately, the removal of the outer layer leaves only smaller MWCNT fragments with a more compact tubular morphology, as previously reported [21]. Although this process initially increases dispersibility, it simultaneously reduces the MWCNT's effective aspect ratio and alters its surface charge. Over time, these effects lead to the tendency of the smaller, damaged pieces of MWCNT to reaggregate or precipitate out of solution. As a result, the optimal treatment of MWCNTs achieved after 4 hours of functionalization will provide a highly reliable long-term dispersibility needed to form uniform, stable MWCNT films on biosensor electrodes, enabling accurate, continuous electrode performance when detecting analytes and limiting degradation of MWCNT structural integrity.

5. CONCLUSION

The findings of this study demonstrate that acid-enhanced functionalization can be utilized to increase both the dispersion and surface reactivity of MWCNTs, while not compromising their structural integrity. FTIR and Raman spectroscopy analyses demonstrated the introduction of carboxyl (-COOH) and hydroxyl (-OH) functional groups onto the MWCNT surface. The X-ray diffraction results verified that amorphous carbon had been removed, and that the crystalline purity of the sample was improved. Scanning electron microscopy revealed a clear transformation from highly agglomerated pristine MWCNTs to more uniformly dispersed and well-separated nanotube structures following functionalization.

Furthermore, the results of the compatibility study indicated that mixed acid-treated MWCNTs ($\text{H}_2\text{SO}_4/\text{HNO}_3$) exhibit superior dispersion stability when compared to MWCNTs treated with a single acid. The analysis showed that 2-4 hour sonication durations for mixed-acid treatment resulted in optimal functional-group attachment while maintaining the integrity of the MWCNT structure. Overall, the functionalized MWCNTs produced using the optimized method demonstrated improved surface reactivity and enhanced dispersion capabilities; therefore, they are prime candidates for incorporation into the next generation of advanced nanomaterials. Future work will involve testing the ability of functionalized MWCNTs combined with titanium dioxide (TiO_2) in sensor-related applications.

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