

## **Review** Article

# A Review of Spectral Methods for Dispersion Characterization of Carbon Nanotubes in Aqueous Suspensions

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Characterization is a crucial step in the study of properties of nanomaterials to evaluate their full potential in applications. Carbon nanotube-based materials have properties that are sensitive to size, shape, concentration, and agglomeration state. It is therefore critical to quantitatively characterize these factors in situ, while the processing takes place. Traditional characterization techniques that rely on microscopy are often time consuming and in most cases provide qualitative results. Spectroscopy has been studied as an alternative tool for identifying, characterizing, and studying these materials in situ and in a quantitative way. In this paper, we provide a critical review of the spectroscopy techniques used to explore the surface properties (e.g., dispersion) characteristics of carbon nanotubes in aqueous suspensions during the sonication process.

## **1. Introduction**

The discovery of carbon nanotubes (CNTs) by Iijima [1] in 1991 attracted scientific and technological interest in the use of CNT-based materials. CNTs exhibit remarkable intrinsic properties including high mechanical strength [2], electrical conductivity [3, 4], and thermal conductivity [5]. These properties are of great interest in engineering applications including biomaterials [6], multifunctional composites [7, 8], and electronic components [9]. To utilize carbon nanotubes (CNTs) in various commercial and scientific applications, the graphene sheets that comprise CNT surfaces are often modified to tailor properties, such as dispersion. The buckypaper method, which consists of producing (10-25 microns thick) of free standing carbon nanotubes through multiplestage dispersion and filtration procedures, has been studied as an effective method to disperse CNTs and prepare stable homogenous CNT sheet materials [10]. However, carbon nanotubes have the tendency to aggregate in bundles due to the large Van der Waals forces between surfaces of the tubes [11, 12] and the difficulty to uniformly disperse the

highly entangled bundles in fluids in a repeatable and costeffective way limits the commercial applicability of these materials [13].

The term *dispersion* describes the process during which the smallest dispersible unit of an agglomerate is separated from it and is mixed in the host matrix system but is also used to characterize the achieved state of CNT separation. In this papers context, the smallest dispersible unit is an individual carbon nanotube in a polymer matrix. Figure 1 shows an example of a badly dispersed suspension (a) and a welldispersed suspension (b). Quantitative analysis of dispersion and agglomeration of nanoparticles in a matrix is important to assess the constitutive properties in a more objective way and eliminate the subjective judgments that the microscopybased methods suffer from. Because nanotube dispersion greatly affects suspension quality, the dispersion of CNTs is a vital aspect for the production of quality nanocomposites. CNTs easily agglomerate, bundle together, and entangle, leading to many defect sites in the composites and limiting the efficiency of CNTs. Therefore, the dispersion and incorporation of CNTs as individuals in polymer matrices



FIGURE 1: (a) Poor dispersion, (b) good dispersion.

are important and challenging tasks toward maximizing the translation of CNT properties to the composites [14–18].

A good distribution is achieved when all available CNTs are uniformly arranged in the host matrix. One simple and most convenient method used for dispersion (deagglomeration) of CNTs in liquids, resins, and polymers is the ultrasonication process in which CNTs are first premixed in dispersion media by a standard stirrer or high-shear mixer and then homogenized by ultrasound [19]. During ultrasonication, the shear force generated inside the sonicator bath disperses and distributes CNTs in the liquid. A proper ultrasonication procedure often results in well-dispersed nanotubes and better composite mechanical properties (e.g., [20]). Practically, however, the aim of good dispersion and distribution cannot be achieved in all cases and in several scientific reports it is reported that complete dispersion of CNT agglomerates is difficult. The choice of processing conditions and base materials has a big impact on dispersion [21].

Using the term uniform dispersion or good dispersion to evaluate the CNT dispersion without any distinctive description is often inadequate. To address this limitation, several researchers have attempted to develop indices to quantify dispersion applied to CNTs, nanoparticles, and nanorods [22, 23]. For CNT suspensions, dispersion has two aspects: (i) disentanglement of CNT bundles or agglomerates, which is nanoscopic dispersion, and (ii) uniform distribution of individual CNTs or their agglomerates throughout the nanocomposites, which is more of a micro- and macroscopic dispersion [24]. For dispersion characterization tools based on microscopy and spectroscopy have found widespread application. Four types of microscopy have been used to characterize dispersion, transmission electron microscopy (TEM), scanning electron microscopy (SEM), atomic force microscopy (AFM), and optical microscopy. SEM examination provides an overview of nanostructures, and a more accurate examination by TEM generally reveals many defects [25].

Microscopy methods are able to directly measure the carbon nanotube diameter and lengths. However, they require significant sample preparation time and are largely applied as offline monitoring tools. Spectroscopic methods, including Raman and UV-Vis spectroscopy, can quantify nanotube dispersion properties by applying a light source on the suspension and correlating the amount of light absorbed or scattered with particle size [26]. While they are indirect methods they enable one to conduct measurements in situ and much more rapidly than the direct imaging methods and they can be automated to enable continuous measurements without requiring extensive operator experiences.

UV-Vis is applied to measure absorption in liquid samples. By contrast to bundled CNTs, the completely dispersed CNTs are highly active in the 200 to 1200 nm wavelength region, and the UV-Vis technique is used to detect individual CNTs by relating the intensity of absorption at a specific wavelength to the concentration of CNTs suspended in the solution through the Beer-Lambert law [27]. Spectra obtained can be characterized as a set of superimposed peaks for CNTs of different diameters and chiralities [28]. Further classification of peak wavelengths was achieved for singlewall nanotubes and multiwall nanotubes: multiwalled carbon nanotubes (MWCNTs) show peak absorption at 253 nm (Jiang et al. [29]) while single-walled carbon nanotubes (SWCNTs) showed peak absorption at 972 nm (Wenseleers et al. [30]) and 1710 nm (Meng et al. [31]). However, maximum UV-Vis absorbance has been suggested by many authors as a good indicator of maximum achievable dispersion in sonication-driven dispersion of MWCNTs in aqueous surfactant solution [32]. A spectrum with small change over time is desired to achieve a stable suspension [29]. Maximum absorbance shows an increase at the beginning of the sonication and it reaches a plateau that has a different magnitude depending on the surfactant concentration [32]. Higher MWCNT concentration requires higher total sonication energy while higher surfactant concentration requires less total sonication energy because dispersion rate increases.

The purity of nanotubes was observed to affect the energy required to achieve a maximum absorption. Grossiord et al. [27] proposed a useful classification based on the total energy required for the UV absorbance to reach the plateau for CNTs with varying impurities. It has been observed that CNTs that contain more impurities have less contact areas between nanotubes and can be detected from UV absorbance because they will require less energy to reach the maximum degree of exfoliation and the plateau. SWCNTs absorb light at distinct wavelengths dictated by their specific (n, m) chiral vector. Fagan et al. [33] demonstrated that this enables the isolation of semiconducting and metallic singlewall carbon nanotube (SWCNT) populations at different surfactant concentrations. The method has been suggested as a robust and scalable alternative to current chromatography instruments for SWCNT separation in diverse surfactant environments.

Spectroscopy methods offer the ability of real-time monitoring of CNT dispersion in aqueous media, an important enabler for producing repeatable nanocomposite products. There has been extensive research on UV-Vis based monitoring of CNT dispersion quality in the past two decades. The aim of this paper is to summarize important findings in the literature on the spectral features with respect to CNT dispersion, suspension properties, and sonication settings. We review methods that characterize dispersion based on UV-Vis spectral methods to probe surface CNTs as a consequence of ultrasonication. In Section 2, we discuss spectroscopy theory and the Beer-Lambert law. A discussion of spectral methods to characterize CNT dispersion is presented in Section 3 followed by the review of some recent industrial applications of UV-Vis in Section 4. A discussion of the works reviewed is provided in Section 5 and the paper is concluded in Section 6.

#### 2. Spectral Theory

Spectroscopy is the study of the absorption and emission of light and other radiations by matter, as a function of the wavelength of the radiation. Different types of spectroscopy have been applied to characterize CNTs. Raman spectroscopy is based on inelastic scattering of monochromatic light from a laser source. Monochromatic light, typically from an argongas laser, is passed through a sample, and the light scattered at right angles to the incident beam is analyzed by an optical spectrometer. Raman spectroscopy yields information about the purity, defects, and tube alignment. The technique has been strikingly successful at describing the structural properties of SWCNTs [34] and to characterize CNT dispersion [35– 41]. However, this technique requires that the suspension be first dried into a solid state material and limits the usefulness in real-time monitoring of manufacturing process.

UV-Vis-NIR spectroscopy is based on absorption spectroscopy or reflectance spectroscopy in the ultravioletvisible-near-infrared region [42] collected using a UV-Vis spectrometer. UV-Vis spectroscopy is applicable to characterize CNTs in aqueous suspensions. As the CNTs are dispersed, the solution will become darker and will absorb more light and hence the maximum absorbance can be used as an indicator of dispersion quality. The ability to monitor



FIGURE 2: Parameters of the absorption process affecting the Beer-Lambert law on UV-Vis spectroscopy.

aqueous solution has the potential to detect quality problems at early stages and ultimately to make corrective process adjustments. In order to obtain complete spectra in the UV-Vis range, dual beam dispersive scanning instruments or dispersive multichannel instruments are employed. A UV-Vis scanning spectrometer typically consists of a light source, a monochromator, a chopper (rotating sector mirror or rotating sector disk), a compartment for the sample and the reference, and a detector [43].

The absorption spectrum of a given compound has characteristic peak heights at certain wavelengths which can serve as an aid to identification and will form the basis of a quantitative characterization of the constituent compounds. When radiation travels through the solution of an absorbing compound it is reduced in intensity by each molecule that it encounters according to an exponential law. The amount of radiation absorbed by a solution is thus an exponential function of the concentration of the solution and the distance that the radiation passes through [44, 45]. In practice it is the amount of radiation transmitted by the solution that is measured. This is expressed as the transmittance, which is the ratio of the amount of radiation transmitted to that incident upon the solution.

An important result from physics is the Beer-Lambert law that shows absorption is proportional to the number of absorbing molecules, that is, the concentration of absorbing molecules given as [46]

$$A = \log_{10} \frac{l_0}{l} = \varepsilon LC, \tag{1}$$

where  $l_0$  is the intensity of the incident radiation, l is the intensity of the transmitted radiation,  $\varepsilon$  is the molar absorption coefficient, L is the path length of the absorbing solution in cm, and C is the concentration of the absorbing material. Figure 2 describes the parameters of the equation.  $\varepsilon$  is a useful constant because it is independent of concentration and path length. The other useful information is the wavelength  $\lambda_{\max}$  at which maximum absorption occurs. These coefficients are strongly influenced by the nature of the solvent and for organic compounds by the degree of substitution and conjugation and are used to calculate the concentration of the solution. The Beer-Lambert law can be



FIGURE 3: Schematic showing reduction in particle size with increasing dispersion energy [19].

written for the transmittance *T*, which is also displayed by many instruments, through the expression [47]

$$T = \frac{l}{l_0} \exp\left(-\varepsilon CL\right).$$
 (2)

UV-Vis absorption spectroscopy has evolved to a powerful nondestructive characterization tool of carbon nanotubes and has been employed in various practical applications for measuring nanotube dispersion, purity, and functionalization in the past years [28, 48, 49]. The most important application of the technique is as a means of quantifying dispersion and modem instruments are designed to facilitate rapid and accurate measurements. In Section 3 we will discuss the application of UV-Vis spectroscopy to characterize dispersion properties of CNT suspensions.

#### 3. Dispersion Characterization

The evaluation of the degree of dispersion of CNTs in aqueous media can be achieved by recording the UV-Vis spectra of the dispersions since individualized CNTs show characteristic bands in the UV region [50, 51]. The measured absorbance at a specific wavelength can be related to the degree that the CNTs are dispersed [27, 52]. The treatment time or energy of the ultrasonication process has strong influence on CNT dispersion and within limits, the longer the treatment time (and the higher the ultrasonication energy), the better the CNT dispersion. Figure 3 shows how the CNT bundles are broken apart to smaller particles during sonication process. However, a longer ultrasonication treatment or higher ultrasonication energy may also reduce the aspect ratio and lead to CNT fragmentation and damage [53–55].

We review the literature that employed UV-Vis spectroscopy to characterize CNT dispersion in aqueous suspensions according to the following classification: (i) methods that monitor segments of the curve, (ii) methods that monitor single point on the curve, and (iii) methods that monitor the surfactant efficiency.

3.1. Methods That Monitor Segments of the Curve. In this section, we present works that develop dispersion metrics

based on segments or subsections of the UV-Vis profiles to characterize dispersion of aqueous CNT suspensions. Two parameters have been developed to indicate the sharpness of the spectral features comparative to the background, as illustrated in Figure 4. The first, called resonance ratio, is the ratio of the resonant band area to its nonresonant background [56] (Figure 4(a)). The second parameter, *normalized width*, is the ratio of the width of the band at half height to the peak height on a normalized spectrum (Figure 4(b)). The normalization makes this ratio an intensive property that does not vary with the path length [57]. The two ratios have been used by researchers to quantify the effect of different factors in the dispersion procedure [57]. It was shown that the ratio of half-peak width to peak height decreases with increasing sonication time: the absorption band becomes sharper and more intense as the number of suspended individual nanotubes increases [58]. The resonance ratio increases during the first hour of sonication, and this indicates that, during the first hour of sonication, a large degree of debundling takes place.

Tan and Resasco [56] employed the resonance ratio and normalized width parameters on single-wall carbon nanotubes (SWCNTs) with narrow diameter distributions to verify the effectiveness of the approach. They used these two ratios to compare different dispersion parameters and to obtain an optimal procedure that maximizes the spectral features selected. They have shown that the resonance ratio (area fraction), with a resonant band at 567 nm, stabilizes with respect to sonication time when a stable dispersion is achieved (see Figure 5). In addition, the surfactants with high resonance ratio and low normalized width ratio were found to provide good dispersion.

3.2. Methods That Monitor a Single Point on the Curve. In this section, we present works that quantify dispersion by correlating a single point on the UV-Vis profile, in particular the absorption peak to characterize dispersion of aqueous CNT suspensions. Jiang et al. [29] quantitatively characterized the colloidal stability of MWCNTs using UV-Vis measurements. In their work, they focused on the peak absorbance wavelength. The peak absorption in their aqueous solution of CNTs appeared at 253 nm (see Figure 6). Wenseleers et al. [30] and Meng et al. [31] performed similar work on SWCNT dispersions from the absorbance of peaks at 972 and 1710 nm when comparing a wide range of surfactants, including bile salts. Both works demonstrated that the surfactants containing a single, long, straight-chain hydrophobic segment and a terminal hydrophilic segment proved to be a suitable dispersant for the stable CNT dispersion. The investigation of the stabilization mechanism helped provide a guideline for choosing suitable dispersants to achieve homogeneously dispersed aqueous CNT suspensions.

Kashiwagi et al. [59] introduced a dispersion metric to measure material homogeneity and considered how it correlated with the basic nanocomposite properties. The dispersion metric of SWCNTs was determined by two different methods: one was an absorbance measurement by UV-Vis spectroscopy and the other was a statistical analysis of 100 images taken by confocal microscopy. The analyses



FIGURE 4: Curve parameters to quantify dispersion: (a) resonance ratio, (b) normalized width [56].



FIGURE 5: UV-Vis spectra of NaDDBS (1g/L) surfactant dispersed SWCNT (0.2 mg/mL) at different sonication time (minutes) [56].



FIGURE 6: UV-Vis spectrum of CNTs in aqueous solution [29].

showed that the absorption spectra of the polymer/SWCNT composites vary systematically with the initial loading of the



FIGURE 7: UV-Vis absorption spectra of highly diluted control and suspension samples [61].

SWCNTs. From the experiments, the authors concluded that properly dispersed suspensions produced composites with physical properties that had four orders of magnitudes in variation compared with well-dispersed suspensions.

Pircheraghi et al. [61] studied the characterization of CNT dispersion in melted semicrystalline media using ultrasonication without the aid of solvents. They compared three commercially available multiwalled carbon nanotubes (CNTs) with different aspect ratios to a control using UV-Vis spectroscopy. As shown in Figure 7 the UV-Vis absorption spectra of all samples have an absorption peak around 260 nm

 TABLE 1: Colloidal stability of the dispersed MWCNTs in aqueous solution [32].

Days	0.1 wt %	0.5 wt %	1.4 wt %
0	0.720	0.750	0.730
10	0.700	0.746	0.725
25	0.701	0.760	0.759
30	0.712	0.745	0.733
50	0.711	0.770	0.730
60	0.726	0.751	0.726
90	0.700	0.753	0.734



FIGURE 8: Evolution of the value of the absorbance at different wavelengths for an aqueous HiPCO NT solution [27].

which is consistent with the literature on well-dispersed individual CNTs exhibiting maximum absorption between 200 and 300 nm [29, 32, 62]. Higher aspect ration CNTs resulted in better dispersion with higher absorbance values and this resulted in composites that exhibited better physical properties.

Yu et al. [32] presented an approach to controlling of the dispersion of multiwall carbon nanotubes (MWCNTs) in aqueous surfactant solution. The experimental results concluded that there is minimum energy required to maximally disperse a certain amount of MWCNTs in aqueous solution and this minimum energy can be determined by using UV-Vis spectroscopy combined with plots of sonication energy. They observed that after a certain sonication time was reached the absorbance profiles showed little changes. They also investigated long term stability of MWCNT dispersions using UV-Vis measurements of suspensions and reported stable suspensions can be achieved with maximum absorbance that remains almost unchanged during 90 days (see Table 1). In a similar study, Grossiord et al. [27] presented a timedependent study of the exfoliation of carbon nanotubes in aqueous suspensions using UV-Vis spectroscopy for different types of nanotubes (see Figure 8). The results of experiments

TABLE 2: Efficiency, expressing amount of SWCNT in suspension after sonication and centrifugation [60].

Surfactant	Efficiency (%)	Surfactant	Efficiency (%)
(GT) 10	28	(GT) 15	30
(AC) 10	28	(AC) 10	35
(C) 30	35	(C) 10	27
(T) 30	26	(T) 10	17
(A) 30	4	(A) 10	2
SDBS	45	SDS	52
SCO	42	SDCO	60

with various sizes indicated that absorbance can be maximized at a consistent sonication energy.

Recently, Goak et al. [63] proposed a new metric for evaluating the purity of SWCNTs using UV-Vis spectroscopy by integrating the absorption peaks from the interband electronic transitions of semiconducting and metallic carbon nanotubes. From the constructed sample sets prepared by mixing designed amounts of highly pure SWCNTs and carbonaceous impurities, their methodology demonstrated numerical closeness of the measured purities to the designed ones.

3.3. Methods That Monitor the Surfactant Efficiency. In this section, we review works that investigate dispersion efficiency of surfactants types and the appropriate CNT-to-surfactant ratios using UV-Vis profiles.

The correlation between the resonance ratio and the normalized width was used to distinguish surfactants that provided good dispersion. The most effective surfactants exhibit a low width ratio and high resonance ratio [56]. Haggenmueller et al. [60] contributed to dispersion characterization using similar ratios for SWCNT using various surfactants and biomolecules and proposed UV-Vis-NIR peak clarity measure to compare the ability of the surfactants to suspend SWCNT (see Table 2).

Rastogi et al. [42] contribution to dispersion characterization was a comparison of the dispersion rating of different surfactants. The dispersion of MWCNTs was characterized by UV-Vis spectroscopy and validated by transmission electron microscopy (TEM). The experimental results showed the ranking of the dispersing power of four surfactants. The surfactants they investigated in decreasing order of dispersion efficiency were Triton X-100, Tween 80, Tween 20, and sodium dodecyl sulfate. Triton X-100 was found to achieve the highest dispersion efficiency.

Inam et al. [64] studied the effects of dispersion surfactants on the properties of CNT nanocomposites. Gum Arabic (GA), sodium dodecyl sulfate (SDS) surfactants, and their mixture were used for the dispersion of multiwalled carbon nanotubes in an alumina matrix. Dispersions were evaluated by UV-Vis spectroscopy and agglomerate size analysis. Figure 9 shows the UV-Vis spectra of different CNT suspensions with various surfactant solutions. A major absorbance peak was observed in the range 525–565 nm. It was demonstrated that a mixture of the surfactants disperses CNTs more efficiently compared to a single surfactant.



FIGURE 9: UV-Vis spectra of different aqueous solutions [64].



FIGURE 10: UV-Vis spectrum of aqueous dispersions of the CNTs subject to sonication time [66].

Korayem et al. [65] investigated the adsorption morphologies of dispersant copolymers on the surface of multiwalled carbon nanotubes using UV-Vis spectroscopy. It has been seen that the dispersion of carbon nanotubes in ethanol does not increase continuously with increasing copolymer/CNT ratio. At a ratio of copolymer/CNT below 0.5, the morphology is random, at ratios between 0.5 and 1.0 the morphology is hemimicelle, which is able to prevent the agglomeration of CNTs, and at ratios above 1.0 the morphology is cylindrical, which is observed to provide more stable dispersions at higher concentrations of CNTs.

Sobolkina et al. [66] investigated the effect of surfactant concentration on the dispersion of CNTs mechanical properties using two types of CNTs with different morphologies. In the UV-Vis measurements (see Figure 10), the CNTs display the characteristic peaks in the ultraviolet spectral region. A relationship among the influence factors (i.e., surfactant concentration  $x_1$  and sonication time  $x_2$ ) and the absorbance of aqueous CNT dispersions y was established using a design of experiment. The following regression model was fitted to the experimentally observed UV-Vis spectra:

$$y_{i} = b_{0} + b_{1}x_{1i} + b_{2}x_{2i} + b_{3}x_{1i}^{2} + b_{4}x_{1i}x_{2i} + b_{5}x_{2i}^{2} + b_{6}x_{1i}^{2}x_{2i} + b_{7}x_{1i}x_{2i}^{2},$$
(3)

where  $b_0$  = regression constant;  $b_1, \ldots, b_7$  = regression coefficients;  $i = 1, 2, \ldots, n$ ; n = number of tests. The condition of the CNTs after sonication was studied by examination of dispersion films using SEM and TEM. The best dispersions were found to be produced with a CNT-to-surfactant ratio between 1:1 and 1:1:5 and a sonication time of 120 min. This produced hardened cement paste with a pronounced increase in its compression strength was determined under high strain rate loading.

## 4. Some Industrial Applications of UV Spectroscopy

UV absorbance spectroscopy has found widespread applications in recent years in the industry in areas ranging from electronics and energy-storage to sensing and medicine. In this section we provide an overview of the use of UV spectroscopy in representative applications in these areas.

Carbon nanotubes are promising for drug delivery applications due to their high surface area, allowing for high drug loading, and their unique interaction with cellular membranes. Heister et al. [67] have designed a drug delivery system for the anticancer drugs based on carbon nanotubes. UV-Vis spectroscopy was used to show that samples with CNTs have higher amounts of drug bounded than those with no CNTs, which is an important measure for stable and sustained release of the drug under diverse biological conditions. Gutierrez et al. [68] used the enzyme glucose oxidase to efficiently disperse multiwall carbon nanotubes and achieve biosensing properties to the dispersed nanotubes. UV-Vis spectroscopy was used to study the influence of the sonication time on the dispersion and find the optimum sonicating time and CNT concentration.

Among the electrochemical energy-storage devices, supercapacitors are considered as one of the promising energystorage devices. Qian et al. [69] considered fabricating coreshell structured nanocomposites for supercapacitors and showed that good dispersion is a key characteristic for improved electrical capacitive performance. The authors used UV-Vis spectroscopy to monitor the dynamics of this composite making process and to determine the optimal weight ratio of CNT/polypyrrole. The UV-Vis spectra of dispersions with increasing weight ratios show that a maximum starts to appear between 200 and 300 nm; however, with an excess addition of polypyrrole it starts to disappear, indicating that an optimum amount of polypyrrole can be found.

SWCNTs have excellent potential in electronics and supercapacitors. However, SWCNTs are generally produced as a mixture of semiconducting and metallic tubes, which prevents their usage in large-scale electronic applications. Wang et al. [70] proposed a scalable method for sorting SWCNTs via solution processing to obtain stable solutions of individualized semiconducting SWCNTs with diameters of 1.1–1.8 nm. UV-Vis-NIR absorption spectroscopy was used to show that the polymer-sorted SWCNT solutions showed well-resolved peaks (well dispersed without large bundles) in contrast to the broad peak observed with unsorted SWC-NTs. Ding et al. [71] employed absorption spectroscopy to isolate SWCNTs with high semiconducting purity, which is a requirement for high frequency logic applications. A series of polymer to SWCNT ratios from 0.25 to 8.0 were evaluated and it was found that a ratio of 0.5 gives the deepest valley of the absorption curve around 640 nm, indicating the highest purity.

## 5. Discussion

Commercial production of CNT-based composites in a repeatable and cost-effective way has proved to be a difficult task [72–76]. One of the major concerns has been the dispersion during the aqueous suspension stage of the production process. Researchers have observed a profound effect of CNT dispersion on the mechanical and electrical properties [62, 77–81]. This indicates the need for the development of objective dispersion metrics for correlating how the dispersion of aqueous CNT suspensions affects the properties of final nanocomposite products.

The establishment of a relationship between certain UV-Vis wavelengths to dispersion quantification in aqueous CNT suspensions has proved difficult due to material impurities and environmental and other uncontrollable factors during the sonication process. From our review, we found that the best results were obtained when a wide range of wavelengths were considered simultaneously to quantify dispersion (e.g., resonance ratios and normalized widths) [56, 82, 83]. Another result from our review is that the bands around the peak absorbance wavelengths are critically important in determining the dispersion of the CNT suspension. UV-Vis measurements have been successfully applied to monitor stability of suspensions for long periods (max 90 days). These studies demonstrate that suspensions do not agglomerate quickly and therefore dispersion states do not change drastically for stored suspensions. This is an important result as it allows for suspensions to be made in large amounts and inventoried for extended periods of time in industrial applications.

In addition, our review showed that UV-Vis spectroscopy can be an effective tool for characterizing the dispersion efficiency of surfactants. Various works have advocated that a mixture of surfactants provides a better dispersion than a single surfactant [42]. However, in practice most researchers have adopted using a single surfactant. Moreover, it should be noted that the majority of the works conducted have been applied to SWCNTs. Accordingly, the extension of this strategy to other single and multiwalled nanotubes is required.

## 6. Conclusion

Uniform dispersion is identified as a key bottleneck for scaling up the production of CNT reinforced nanocomposites

in a cost-effective way [54]. The development of reliable in situ dispersion measurement systems is an important enabler for the production of nanotube-reinforced composites with reproducible properties in a cost-effective way. Microscopybased direct imaging techniques have been extensively studied for measuring properties of carbon nanotubes or their bundles suspended in aqueous solutions or in a solid state [19]. While these methods are able to accurately measure the carbon nanotube diameter and lengths they require significant sample preparation time and can be applied as offline monitoring tools. UV-Vis spectroscopy allows one to conduct measurements in situ and much more rapidly than the direct imaging methods to enable continuous measurements without requiring extensive operator experience. Shorter measurement time (comparing to the usually severalhour-long sonication procedure) and the ability to quantify dispersion of suspensions make the UV-Vis methods very attractive for use in online monitoring and quality control of CNT nanocomposites.

This work aims to summarize the spectral characterization techniques that have been used extensively in carbon nanotube research. We classified our review into methods that monitor portions of the spectral curve, methods that monitor a single point on the curve, and methods that monitor the surfactant efficiency. A brief overview of the use of UV absorption in industrial applications is also provided. It has been shown that different wavelengths of the spectral curve are sensitive to dispersion quality depending on the nanotube type and characteristics; however, many authors agree that selection of the feature is application specific, and it is possible that using more features on the curves would be more beneficial to provide more adequate process control. The review is not meant to be exhaustive; however, it is aimed at providing important references to the recent works in literature about spectral methods of CNT dispersion. Some more complete references on CNT-based composites are [84-87] and on spectroscopy include [88–90].

#### **Conflict of Interests**

The authors declare that there is no conflict of interests regarding the publication of this paper.

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